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**PAHs removal from municipal wastewater sludge using  
chemical, electrochemical and biological treatments**

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## RÉSUMÉ

Les HAPs ont été largement étudiés, puisqu'ils sont présents dans presque tous les écosystèmes et sont très toxiques. Du fait de leur forte hydrophobicité, laquelle est liée à la présence de cycles aromatiques, ces molécules s'adsorbent fortement aux matières particulières des sols, des sédiments ou des boues, ce qui les rend peu accessibles à la biodégradation. Dans le cadre de cette recherche, les performances de différents procédés de traitement des boues ont été évaluées pour l'élimination de 11 HAPs ( $5.5 \text{ mg de chaque HAP kg}^{-1} \text{ MS}$ ) préalablement additionnés à des boues d'épuration. Deux procédés biologiques (la digestion aérobie mésophile (MAD) et le procédé simultané de digestion des boues et de lixiviation des métaux (METIX-BS)) ont été testés afin d'évaluer leur capacité de biodégrader les HAPs. En parallèle, deux procédés chimiques (METIX-AC et STABIOX) et un procédé électrochimique (ELECSTAB) ont été testés afin de mesurer l'élimination des HAPs par ces procédés oxydatifs. De plus, la solubilisation des HAPs à partir des boues suite à l'addition d'un surfactant non-ionique (Tween 80) a aussi été explorée. Les meilleurs rendement d'élimination des HAPs ont été obtenus par les procédés biologiques (MAD et METIX-BS) avec plus de 95% d'enlèvement des HAPs à 3 cycles après une période de traitement de 21 jours. L'ajout de Tw80 lors de l'opération du procédé MAD a permis de hausser le taux d'enlèvement des HAPs à 4 cycles. De plus, plus de 45% des HAPs à 3 cycles ont été enlevés à partir des boues d'épuration par METIX-AC et approximativement 62% d'enlèvement des HAPs à 3 cycles par le processus ELECSTAB. Cependant, peu d'enlèvement (< 35%) pour des HAPs à 3 cycles par STABIOX. Aucun des procédés testés ne s'est avéré efficace pour l'élimination des HAPs à haut poids moléculaire ( $\geq 5$  cycles) à partir des boues d'épuration.



## ABSTRACT

Polycyclic aromatic hydrocarbons (PAHs) have been widely studied due to their presence in all the environmental media and toxicity to life. These molecules are strongly adsorbed on the particulate matters of soils, sludges or sediments because of their strong hydrophobicity which makes them resistant to biodegradation. Various treatment processes of sewage sludge were tested to evaluate their performance for PAHs removal from sewage sludge prealably doped with 11 PAHs ( $5.5 \text{ mg each PAH kg}^{-1} \text{ DM}$ ). Two biological processes (mesophilic aerobic digestion (MAD) and simultaneous sewage sludge digestion and metal leaching (METIX-BS)) were tested in order to evaluate PAHs biodegradation in sewage sludge. In parallel, two chemical processes (METIX-AC and STABIOX) and one electrochemical process (ELECSTAB) were tested to measure PAHs removal by these oxidative processes. Moreover, PAHs solubilisation from sludge by addition of a nonionic surfactant (Tween 80) was also tested. Best PAH removal yields have been obtained by biological processes (MAD and METIX-BS) with more than 95% of removal for acenaphptene (ACN), fluorene (FLU) and phenanthrene (PHE) after a 21-day treatment period. Tw80 addition during MAD treatment increased 4-ring PAHs removal rate. In addition, more than 45% of 3-ring PAHs were removed from sludge by METIX-AC and during ELECSTAB process were quiet good with approximately 62% of 3-ring PAHs removal. However, little weaker removal of 3-ring PAHs (< 35%) by STABIOX. None of the tested processes were efficient for the elimination of high molecular weight PAHs ( $\geq 5\text{-ring}$ ) from sewage sludge.



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## LIST OF ABREVIATIONS

ACN	Acenaphtene
BAP	Benzo(a)pyrene
BJK	Benzo(b,j,k)fluoranthene
BPR	Benzo(ghi)perylene
CAM	Ceric-sulfate ammonium molybdate
CMC	Critical micelle concentration
CV	Coefficient of variation
DAC	Doped and autoclaved control
DM	Dry matter
DO	Dissolved oxygen
DOC	Doped control
DSA	Dimensional stabilized anode
EERC	Energy & environmental research center
EU	European union
FID	Flame ionization detection
FLR	Fluoranthene
FLU	Fluorene
FMAE	Focused microwave-assisted solvent extraction
GC-FID	Gas chromatography with flame ionization detection
GC-MS	Gas chromatography with mass spectrometry
HAP	Hydrocarbures aromatiques polycycliques
Hc	Henry's Law constants
HPLC	High performance liquid chromatography
INP	Indeno(1,2,3-cd)pyrene
INRS	Institut national de la recherche scientifique
Kow	Octanol-water coefficient

LD	Limit of detection
MAD	Mesophilic aerobic digestion
MAE	Microwave-assisted solvent extraction
MS	Matière sèche
NDC	No doped control
ORP	Oxidation reduction potential (mV)
PAH	Polycyclic aromatic hydrocarbons
PCB	Polychlorinated biphenyls
PCDD	Polychlorinated dibenzo- <i>p</i> -dioxin
PCDF	Polychlorinated dibenzofuran
PHE	Phenanthrene
PMAE	Pressurized microwave-assisted solvent extraction
POP	Persistent organic pollutants
POR	Potentiel d'oxydo-réduction (mV)
PYR	Pyrene
RPM	Rotations per minute
RRF	Relative response factors
S	Solubility
S/L	Separation solid-liquid
SD	Standard deviation
SDWA	Safe drinking water act
SFE	Supercritical fluid extraction
SIM	Selected ion monitoring
SPE	Solid phase extraction
SPME	Solid-phase micro-extraction
ST	Solides totaux
TLC	Thin layer chromatography
TS	Total solids

Tw80	Tween 80
USEPA	United States environment protection agency
UV	Ultraviolet and visible
Vp	Vapor pressure



## **1. SYNTHÈSE FRANÇAISE**

### **1.1. Introduction**

Les boues d'épuration sont les déchets incontournables du traitement des eaux usées. Elles sont riches en matières organiques, éléments nutritifs comme l'azote, le phosphore et le potassium, composants principaux des engrains agricoles. Au cours des dernières années, le recyclage des boues comme fertilisant a bien été étudié. Aujourd'hui, environ 40% des boues d'épuration produites dans le monde sont utilisées pour la fertilisation des sols (Webber *et al.*, 1996; Recyc-Quebec, 2002).

Dans bien des cas, les boues d'épuration ne peuvent pas être directement appliquées sur les sols puisqu'elles contiennent une très grande variété de pathogène, certains métaux lourds (comme le cadmium, zinc, cuivre, etc.) et des polluants organiques (comme les biphenyles polychlorés (BPCs), les dioxines et furannes (PCDDs/PCDFs), les hydrocarbures aromatiques polycycliques (HAPs) etc.). Ces polluants peuvent être toxiques pour l'humain, les animaux ou les plantes. L'utilisation des boues d'épuration en agriculture est interdite lorsque la concentration d'un ou plusieurs polluants dépasse une valeur limite.

Les HAPs sont des composés organiques neutres, constitués exclusivement de carbone et d'hydrogène et dont la structure moléculaire comprend au moins deux cycles benzéniques. Ces composés sont considérés comme les polluants organiques classiques les plus importants dans l'environnement. De plus, étant hydrophobes, liposolubles et pour la plupart non-volatils, les HAP tendent à s'adsorber à la matière organique ainsi qu'aux particules solides dans les sols, les sédiments ou les boues. Ces HAPs présentent un risque toxicologique important même à de

faibles concentrations pour les microorganismes, les animaux ou les hommes. Plusieurs HAPs sont considérés cancérigènes et mutagènes.

Les HAPs proviennent de sources naturelles et anthropiques. Les sources anthropiques incluent les activités industrielles comme les usines pétrochimiques, les usines de gazéification du charbon etc., mais aussi des sources reliées au domaine du résidentiel et du transport. Les HAPs peuvent également se former naturellement lors de feux de forêts ou d'éruptions volcaniques.

En Europe, la limite de concentration pour total des 11 HAPs<sup>1</sup> dans les boues d'épuration ne devrait pas excéder 6 mg kg<sup>-1</sup> de boues sèches si celles-ci sont destinées à être utilisées pour des épandages agricoles (EU, 2000).

Actuellement, plusieurs groupes travaillent sur différentes méthodes d'extraction (USEPA, 1982; 1990b; Chen et Pawliszyn, 1995; Camel, 2000), de purification (Mangas *et al.*, 1998; Harmsen *et al.*, 2003) et de mesures des HAPs dans les sols (USEPA, 1998; Khim *et al.*, 1999). À l'inverse, le même type d'études sur l'extraction et l'analyse des HAPs dans les boues d'épuration est beaucoup plus rare.

La plupart des HAPs sont peu biodégradables du fait de leur faible solubilité dans l'eau et de leur fort pouvoir adsorbant (Goyer *et al.*, 1995). Diverses approches ont été explorées pour l'élimination des HAPs dans les sols contaminés. Ces technologies comprennent notamment : la biodégradation par les bactéries (Cerniglia, 1992), la photodégradation (Mill *et al.*, 1981; Fukuda *et al.*, 1988; Sigman *et al.*, 1991, 1996, 1998), l'oxydation chimique (Yao et Masten, 1992) et la

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<sup>1</sup> 11 HAP: acénaphthène (ACN), phénanthrène (PHE), fluorène (FLU), fluoranthrène (FLR), pyrène (PYR), benzo(bjk)fluoranthrène (BJK), benzo(a)pyrène (BAP), indeno(1,2,3-cd)pyrène (INP) et benzo(ghi)perylène (BPR).

solubilisation par les surfactants (Bury et Miller, 1993; Volkering *et al.*, 1995; Doong *et al.*, 1996).

Un certain nombre de procédés tels que METIX (Blais *et al.*, 1992, 2004, 2005), STABIOX (Blais *et al.*, 2003) et ELECSTAB (Dogui *et al.*, 2005) ont été testés avec succès pour l'enlèvement des métaux toxiques ou la stabilisation (élimination des pathogènes et des odeurs) des boues d'épuration municipales et industrielles. Toutefois, aucune étude ne s'est portée sur la possible application de ces procédés, ou d'autres technologies de traitement des boues, pour l'élimination des HAPs présents dans les biosolides issus du traitement des eaux usées.

D'un point de vue analytique, le suivi des HAPs dans les boues a été réalisé par la technique d'extraction Soxhlet et saponification, suivi par une purification sur gel de silice et l'analyse des par GC-MS.

En terme de décontamination, divers types de technologies ont été évaluées afin d'identifier les filières de traitement les plus prometteuses. Ces technologies comprennent deux procédés biologiques (METIX-BS et digestion aérobiose mésophile (MAD)), deux procédés chimiques (STABIOX, METIX-AC) et un procédé électrochimique (ELECSTAB). L'étude a également porté sur la possible utilisation de surfactant (Tween-80) pour l'enlèvement des HAPs des boues d'épuration. Enfin, des combinaisons comprenant l'utilisation d'un surfactant avec un procédé biologique (MAD) ou chimique (METIX-AC) ont également été explorées.

## 1.2. Démarche méthodologique

### 1.2.1 Échantillonnage des boues

Les boues de l'usine d'épuration des eaux usées municipales de Haute-Bécancour (Black Lake, QC, Canada) ont été choisies. Ces boues sont générées par des réacteurs biologiques séquentiels (RBS). Les boues ont été conservées à 4 °C dans des contenants de polypropylène jusqu'à leur utilisation après avoir été échantillonnées en mai 2005. La teneur en solides totaux (ST) de ces boues a été ajustée à 18.0 et 30.0 g ST L<sup>-1</sup> pour l'ensemble des expériences. Les teneurs en métaux et en nutriments (mg kg<sup>-1</sup> de matière sèche (MS)) des boues étaient les suivantes: Al (19 000 ± 400), Ca (13 900 ± 200), Cd (3,0 ± 0,1), Cr (139 ± 4), Cu (1 800 ± 40), Fe (37 100 ± 900), K (7 200 ± 200), Mg (17 100 ± 200), Mn (468 ± 7), Na (4 700 ± 100), Ni (134 ± 3), P (19 900 ± 400), Pb (87 ± 1), S (5 000 ± 200), Zn (620 ± 10).

### 1.2.2 Méthode de dopage des boues

Avant les expériences, les boues ont été dopées avec 5 mg kg<sup>-1</sup> MS de chacun des 11 HAPs par ajout de solutions standards Mix 44 (1 000 mg l<sup>-1</sup> dans CH<sub>2</sub>Cl<sub>2</sub>-benzène) et benzo(j)fluoranthène (2 000 mg L<sup>-1</sup> dans CH<sub>2</sub>Cl<sub>2</sub>-benzène). Ces solutions standards ont été obtenues chez Sigma-Aldrich Canada Ltd (Oakville, ON, Canada). Les boues ont été mélangées mécaniquement (Mechanical shaker, Lab-Line Environ-Shaker, modèle 3528) pendant au moins 1 h à la température ambiante (20 ± 2 °C) et conservées à 4 °C pendant 24 h.

### **1.2.3 Enlèvement des HAPs dans les boues d'épuration**

Au cours des traitements et après ceux-ci, des échantillons de boues (300 à 600 ml) ont été prélevés et les phases solide et liquide ont été séparées par centrifugation (2 050 x g pendant 30 min, Beckman Coulter Inc., modèle Allegra<sup>TM</sup> 6 centrifuge) ou filtration sous vide (membrane Whatman No. 4, porosité de 15 à 20 µm). Des étapes d'extraction et de purification des HAPs ont ensuite été effectuées en triplicata sur ces phases solide et liquide.

#### **1.2.3.1 *Essais contrôles***

Dans un premier temps, afin de démontrer l'efficacité de différents traitements pour l'enlèvement des HAPs dans les boues, trois essais contrôles ont été réalisés en utilisant trois volumes d'approximativement 2 L de boues. Le premier contrôle comprenait les boues non-dopées et non-traitées (NDC). Le second contrôle (DOC) a été préparé en utilisant des boues préalablement dopées, mais n'ayant subit aucun traitement. Un troisième contrôle (DAC) a aussi été préparé en stérilisant (autoclavage à 121 °C, sous une pression de 15 psi et pendant 15 min) les boues. Les boues stériles ont ensuite été réparties dans quatre erlenmeyers de 1 L de capacité et ont été dopées selon la procédure décrite précédemment. Par la suite, les boues contenues dans un erlenmeyer ont été séparées par centrifugation. Les phases solide et liquide ont été conservées pour les extractions et les analyses. Les boues présentes dans les trois autres erlenmeyers ont été conservés à température ambiante pendant des périodes respectives de 7, 14 et 21 jours avant d'être centrifugées. Les teneurs en HAPs dans ces boues contrôles sont illustrées au Tableau 1.1.

1. Synthèse française

**Tableau 1.1 Teneurs en HAPs ( $\text{mg kg}^{-1}$  MS) dans les boues non-dopées et dopées**

HAPs	Contrôle non-dopé		Contrôles dopés	
	(NDC)	(DOC)*	(DAC)**	(DAP)***
ACN	0,036 ± 0,001	4,58 ± 0,12	5,56 ± 0,01	5,56 ± 0,01
FLU	0,042 ± 0,002	4,51 ± 0,12	5,56 ± 0,01	5,56 ± 0,01
PHE	0,207 ± 0,010	5,11 ± 0,17	5,73 ± 0,03	5,73 ± 0,03
FLR	0,493 ± 0,012	4,92 ± 0,56	6,03 ± 0,07	6,03 ± 0,07
PYR	0,522 ± 0,028	5,07 ± 0,22	6,06 ± 0,06	6,06 ± 0,06
BJK	0,910 ± 0,084	16,82 ± 0,30	17,72 ± 0,11	17,72 ± 0,11
BAP	0,493 ± 0,012	4,94 ± 0,22	5,93 ± 0,06	5,93 ± 0,06
INP	0,392 ± 0,037	5,65 ± 0,45	6,14 ± 0,05	6,14 ± 0,05
BPR	0,534 ± 0,045	6,02 ± 0,16	6,07 ± 0,04	6,07 ± 0,04
Total (11 HAPs)	3,586 ± 0,214	57,84 ± 2,31	64,79 ± 0,43	64,79 ± 0,43

\* ST = 18,0 g L<sup>-1</sup>,

\*\* ST = 30,0 g L<sup>-1</sup>.

**Tableau 1.2 Conditions expérimentales des différents essais d'enlèvement des HAPs**

Traitements	ST (g L <sup>-1</sup> )	Durée	Tw80 (g L <sup>-1</sup> )	pH final	POR final (mV)
DAC	30,0	21 d	0	7,16	-58
MAD	30,0	21 d	0	7,08	+440
MAD+Tw80	30,0	21 d	0,5	6,43	+446
METIX-BS	30,0	21 d	0	1,42	+461
METIX-AC	18,0	4 h	0	2,08	+326
METIX-AC+Tw80	30,0	4 h	0,5	1,88	+370
STABIOX	18,0	80 min	0	4,00	+290
ELECSTAB	18,0	60 min	0	4,00	+171
Tw80-A	30,0	6 h	0,5	6,60	-70
Tw80-B	30,0	4 h	0,5	6,59	-83
Tw80-C	30,0	2 h	2,0	6,62	-82

#### **1.2.3.2    *Traitemennt par digestion aérobie mésophile (MAD)***

Le procédé conventionnel de digestion aérobie mésophile a été évalué pour la biodegradation des HAPs. Un volume de 3 L de boues dopées a été digéré en mode cuvee dans un réacteur de type cuve agitée (5 L de capacité) à température ambiante. Quelques paramètres opératoires importants sont décrits au Tableau 1.2. Lors de l'opération du procédé, la teneur en oxygène dissout a été maintenue  $> 2 \text{ mg L}^{-1}$ . Des échantillons ont été prélevés aux temps 3, 5, 7, 14 et 21 jours pour les extractions et les analyses de HAPs. Les phases solide et liquide ont été séparées par centrifugation.

#### **1.2.3.3    *Traitemennt MAD+Tw80***

Un ajout de Tween 80 (Tw80), un surfactant non-ionique, a été testé en combinaison au procédé MAD. Une solution de 1,5 g Tw80 L<sup>-1</sup> (750 ml) a été ajoutée à un volume de 1,5 L de boues dopées et le mélange a été digéré dans un réacteur de type cuve agitée (5 L). Des échantillons ont été prélevés aux temps 3, 5, 7, 14 et 21 jours pour les extractions et les analyses de HAPs. Les phases solide et liquide ont été séparées par filtration.

#### **1.2.3.4    *Traitemennt METIX-BS***

METIX-BS est un procédé de biolixiviation des métaux toxiques et de digestion des boues utilisant le soufre élémentaire. Ce procédé exploite la présence d'une microflore indigène composée de thiobacilles capables d'oxyder le soufre élémentaire en acide sulfurique dans les boues d'épuration. Cette production d'acide cause une acidification importante des boues à des valeurs de pH inférieures à 2,0. Un volume de 3 L de boues dopées a été réparti en 15 volumes de 200 ml dans des erlenmeyers de 500 ml de capacité. Une masse de 1,0 g de soufre élémentaire en poudre a été ajoutée à chaque fiole, puis les boues ont été incubées pendant 21 jours, à une

température de  $28 \pm 2$  °C, dans un agitateur gyratoire tournant à 150 rotations par minute (rpm). Des échantillons ont été prélevés aux temps 3, 5, 7, 14 et 21 jours pour les extractions et les analyses de HAPs. Les phases solide et liquide ont été séparées par centrifugation.

#### **1.2.3.5 Traitement METIX-AC**

Le procédé METIX-AC est une technologie efficace et économique pour l'enlèvement des métaux toxiques. Ce procédé est également en mesure d'éliminer les odeurs et les pathogènes par utilisation d'agents oxydants, tels que le chlorure ferrique et le peroxyde d'hydrogène. Un volume de 2 L de boues dopées a été acidifié à pH 2 avec de l'acide sulfurique (5 ml, 5 M) à température ambiante dans un réacteur de type cuve agitée de 5 L de capacité. Par la suite, des solutions de chlorure ferrique (12 ml, teneur en  $\text{Fe}^{3+}$  de 11% p p<sup>-1</sup>) et de peroxyde d'hydrogène (10 ml, 3% p p<sup>-1</sup>) ont été ajoutées afin de maintenir un POR > 400 mV. Les boues ainsi traitées ont été mélangées pendant 4 h. Les phases solide et liquide ont été récupérées par centrifugation.

#### **1.2.3.6 Traitement METIX-AC+Tw80**

L'ajout de Tw80 a également été testé en combinaison avec le procédé METIX-AC. Un volume de 0,6 L de boues dopées a été acidifié à pH 2, dans un bêcher de 2 L de capacité, avec l'acide sulfurique (4 ml, 5 M), suivi par l'addition des solutions de chlorure ferrique (6 ml, 11%  $\text{Fe}^{3+}$  p p<sup>-1</sup>) et de peroxyde d'hydrogène (5 ml, 3% p p<sup>-1</sup>). Les boues ont été mélangées pendant 2 h à température ambiante, puis une solution de 0,5 g Tw80 L<sup>-1</sup> (300 ml) a été ajoutée aux boues, lesquelles ont ensuite été mélangées à nouveau pendant une période de 2 h. Les phases solide et liquide ont été récupérées par filtration.

#### **1.2.3.7 Traitement STABIOX**

STABIOX est un procédé performant de stabilisation (élimination des odeurs et destruction des microorganismes pathogènes) et de conditionnement (amélioration de la déshydratabilité) des boues d'épuration municipales et industrielles. Un volume de 2 L de boues dopées a été acidifié à pH 4 avec de l'acide sulfurique (3,5 ml, 5 M) dans un réacteur de type cuve agitée de 5 L de capacité. Par la suite, des solutions de chlorure ferrique (1,5 ml, 11% Fe<sup>3+</sup> p p<sup>-1</sup>) et de peroxyde d'hydrogène (10 ml, 3% p p<sup>-1</sup>) ont été ajoutées, puis les boues ont été mélangées à température ambiante pendant 80 min. Les phases solide et liquide ont été récupérées par centrifugation.

#### **1.2.3.8 Traitement ELECSTAB**

Le procédé ELECSTAB permet de stabiliser (élimination des odeurs et destruction des microorganismes pathogènes) et d'améliorer la déshydratabilité des boues d'épuration municipales et industrielles. La capacité de ce procédé à détruire les HAP a donc été examinée dans le cadre de cette recherche. Un volume de 5 L de boues dopées a été déposé dans une cellule électrolytique cylindrique, en plexiglas d'une capacité de 12 L (42 cm de haut et 25 cm de diamètre), constituée de deux électrodes concentriques en métaux déployés (DSA : Dimensional Stabilized Anode). L'anode fabriquée de titane (Ti) recouverte d'oxyde de ruthénium (RuO<sub>2</sub>) avait une forme cylindrique de 28 cm de haut, 10 cm de diamètre et 2 mm d'épaisseur. La cathode était une grille en titane déployé et avait une forme cylindrique de 28 cm de haut, un diamètre de 18 cm et une épaisseur de 2 mm. La distance interélectrode était de 4 cm. L'anode était placée au centre de la cellule et la cathode à la périphérie. Les électrodes, fixées mécaniquement, ont été disposées de façon à assurer une bonne répartition de l'eau dans la cellule. Le courant électrique était fourni par une source d'alimentation stabilisée (XFR 40-70, 0-

40 V, 1~70 A). Une addition de 12,5 g de sulfate de sodium (électrolyte) a été effectuée et des solutions d'acide sulfurique (13,5 ml, 5 M) et de peroxyde d'hydrogène (45 ml, 3% p p<sup>-1</sup>) ont également été ajoutées. Les boues ont été traitées par voie électrochimique (8 A et 16 V) pendant 60 min à température ambiante. Les phases solide et liquide ont ensuite été récupérées par centrifugation.

#### **1.2.3.9     *Traitement au surfactant non-ionique***

Le surfactant nonionique (Tween 80) a été choisi pour augmenter la solubilité des HAP dans les boues d'épuration. Le Tween 80 a été utilisé avec succès pour la solubilisation de plusieurs composés organiques toxiques présents dans des sols contaminés. Trois différentes procédures (Tw80-A, -B, -C) ont été testées pour le traitement de boues en utilisant ce surfactant. Ces expériences ont été réalisées avec des volumes de 200 ml de boues dopées déposés dans des erlenmeyers de 500 ml de capacité munis de chicanes et de bouchons en acier inoxydable. Les fioles ont été placées sur un agitateur gyratoire tournant à 150 rpm et gardé à température ambiante. Une solution de 1,5 g Tw80 L<sup>-1</sup> (100 ml) a été ajoutée aux boues lors de l'essai Tw80-A. Après le traitement d'une durée de 6 h, les phases solide et liquide ont été séparées par centrifugation à différentes vitesses (500, 1 000, 2 000 et 3 000 x g pendant 30 min) et par filtration sous vide. Des concentrations de 0,5 et 2,0 g Tw80 L<sup>-1</sup> ont été ajoutées aux boues lors des essais Tw80-B et Tw80-C, respectivement. Après les traitements de 4 h (Tw80-B) et 2 h (Tw80-C), les phases solide et liquide ont été obtenues par filtration.

#### **1.2.4 Extraction, purification et analyse des HAPs**

##### **1.2.4.1 Extraction Soxhlet pour échantillons solides**

Dans cette étude, les HAP dans les fractions solides des boues ont été extraits par une méthode modifiée du Centre d'expertise en analyse environnementale du Québec (Gouvernement du Québec, 2002). Les échantillons solides traités (1 à 10 g) ont été placés dans des bêchers préalablement pesés précisément. La solution combinée d'étalons de recouvrement (250 µl d'une solution à 100 mg L<sup>-1</sup> d'accénaphthène-d<sub>10</sub> et de chrysène-d<sub>12</sub>) a été ajoutée dans les boues solides. Des ajouts de sulfate de magnésium anhydre ont été réalisés afin d'éviter la présence de traces d'eau au sein des extraits. La totalité des échantillons a été transférée dans une cartouche d'extraction en cellulose (43 x 123 mm, Advance MFS Inc.) préalablement pré-décontaminée (traiter dans du dichlorométhane pendant au moins 24 h). Les échantillons solides ont été traités dans un extracteur Soxhlet (Organamation Glassware Heaters, Electrothermal EME 30500/EX1, avec 3 fioles de 500 ml) par 300 ml de CH<sub>2</sub>Cl<sub>2</sub> à un rythme d'environ 4 à 6 cycles h<sup>-1</sup> pendant 24 h (MA. 400-HAP 1.1). Après cette période, l'ensemble a été refroidi et transféré dans un ballon. L'extracteur était rincé à l'aide de CH<sub>2</sub>Cl<sub>2</sub> et le matériel de rinçage transféré dans le ballon. L'ensemble du contenu du ballon était évaporé sous vide à l'aide d'un évaporateur rotatif (marque Büchi, modèle Rotavapor-R) à une température ne dépassant pas 26 °C et ce, jusqu'à un volume de 5 ml.

##### **1.2.4.2 Saponification**

Pour enlever efficacement les composées organiques interférant dans les extraits comme les triacylglycérols, il est important de procéder à une étape de purification sur colonne. Au

préalable à cette étape de purification, il est nécessaire de procéder à une étape de saponification.

Une solution d'hydroxyde de potassium (150 ml d'une solution de méthanol 0.5 M) a été ajoutée dans un ballon (500 ml) qui a été chargé des extraits. Ensuite, un chauffage par bain d'eau a été opéré afin de maintenir les extraits sous reflux (80 °C) pendant 4 h (Mangas *et al.*, 1998). Après refroidissement à température ambiante, 100 ml de saumure saturée préparée dans de l'eau milli-Q-purifiée a été ajoutée dans ce système et le mélange a été extrait à l'aide de dichlorométhane (3 × 50 ml). La phase organique combinée a été séchée par MgSO<sub>4</sub> (préchauffé à 650 °C pendant 12 h) et évaporée à sec sous vide grâce à un évaporateur rotatif dont la température du bain ne dépassait pas 26 °C. Un volume approximatif de 2 ml d'hexane a été ajouté dans ce ballon pour l'étape suivante.

#### **1.2.4.3 Extraction rotative pour échantillons liquides**

Des volumes connus d'échantillons liquides (de préférence 500 ml) ont été transférés dans des bouteilles en verre pré-décontaminées (lavées à l'eau et rincées à l'acétone). Ensuite, des volumes de 200 ml de dichlorométhane et 100 µl de la solution combinée d'étalons de recouvrement (acénaphtène-d<sub>10</sub> et chrysène-d<sub>12</sub> dans l'isooctane) ont été ajoutés dans les bouteilles. Les échantillons liquides ont été extraits sur l'extracteur rotatif à une vitesse de rotation de 6 rpm pendant 24 h à température ambiante. La phase organique a été séparée et séchée par MgSO<sub>4</sub> (préchauffé à 650 °C pendant 12 h). Par la suite, la solution a été évaporée à sec sous vide à l'aide d'un évaporateur rotatif dont la température de bain ne dépassait pas 26 °C. Un volume approximatif de 2 ml d'hexane était ajouté dans ce ballon pour l'étape de purification.

#### **1.2.4.4 Purification des extraits par chromatographie rapide sur colonne de gel de silice**

Les extraits contenant les HAP ont été purifiés par adsorption dans des colonnes de verre ( $\phi$  2,5 cm  $\times$  40 cm pour des échantillons solides;  $\phi$  1,0 cm  $\times$  20 cm pour des échantillons liquides) contenant du gel de silice activé (60 g pour les échantillons solides, 15 g pour les échantillons liquides). Des couches d'environ 1 cm de sulfate de magnésium anhydre ont été placées au-dessus du gel de silice. Après rinçage avec une solution d'hexane, les HAP adsorbés ont été élués par un volume de 150 ml d'une solution d'hexane-dichlorométhane (3 : 2). Après concentration par évaporation, les HAP étaient repris dans des volumes finaux de 2 ml (échantillons liquides) ou 5 ml (échantillons solides) d'isoctane. Les échantillons purifiés ont été conservés dans des vials ambrés à  $\leq -10$  °C avant leur analyse.

#### **1.2.5 Méthodes analytiques**

Les HAP ont été mesurés à l'INRS en utilisant un appareil GC-MS (Perkin Elmer, modèle Clarus 500, colonne de type VF-5MS FS, 30 m  $\times$  0,25 mm  $\times$  0,25  $\mu\text{m}$ ) et chez Bodycote inc. (Québec, QC, Canada) par un appareil GC-MS (Hewlett Packard, modèle 6890 muni d'un MS modèle HP 5973A, colonne de type DB-5, 30 m  $\times$  0,25 mm  $\times$  0,25  $\mu\text{m}$ ).

Le pH a été mesuré à environ 20 °C par un pH-mètre digital Fisher Accumet (modèle 915) équipé d'une électrode Cole-Palmer à référence interne Ag/AgCl et d'une double jonction de verre. Les solides totaux (ST) ont été mesurés par la méthode 2540B de l'APHA *et al.* (1999). Les échantillons liquides ont été filtrés sous vide à l'aide de membranes Whatman 934-AH (Whatman International Ltd, Maidstone, Angleterre), puis acidifiés avec une concentration de 5% (v/v) HCl et conservés à 4 °C avant les mesures de métaux et d'éléments en solution. Les

boues ont été digérées par la méthode 3030 I de l'APHA *et al.* (1999) comprenant une attaque de 0,5 g de boues sèches par HNO<sub>3</sub>, HF et HClO<sub>4</sub> et reprise finalement par 5% HCl. Les métaux et éléments en solution ont été mesurés par spectrophotométrie à émission de plasma induit (ICP-AES) avec un appareil Varian (modèle Vista-AX).

### 1.2.6 Produits chimiques

Les solvants organiques utilisés étaient de grade analytique et provenaient de la compagnie Merck Frosst Canada Limitée (Kirkland, QC, Canada). Le chlorure de sodium, l'hydroxyde de potassium et le sulfate de magnésium anhydre (grade analytique et pré-chauffé à 650 °C pendant 12 h avant utilisation) ont été obtenus de EMD Chemicals Inc (Gibbstown, NJ, USA). L'acide sulfurique (95-98% réactif ACS) a été acheté chez Fisher Scientific Company (Ottawa, ON, Canada). La solution de chlorure ferrique (11% Fe<sup>3+</sup> p p<sup>-1</sup>) a été acquise de Eaglebrook Environment Corporation (Varennes, QC, Canada). Le peroxyde d'hydrogène (33% v v<sup>-1</sup>) a été acheté chez Laboratoire Mat (Beauport, QC, Canada). Le gel de silice 60 (230 ~ 400 mesh) a été acquis de Silicycle (Québec, QC, Canada), alors que le Tween 80 a été acheté de ICI Americas Inc (Wilmington, DE, USA). Les solutions standardisées de HAP comprenant la solution de Mix 44, ainsi que les solutions de benzo(j)fluoranthène, anthracène-d<sub>10</sub>, lpyrène-d<sub>10</sub>, acénaphtène-d<sub>10</sub>, chrysène-d<sub>12</sub>, naphtalène-d<sub>8</sub>, et phénanthrène-d<sub>10</sub> ont été obtenues de Sigma-Aldrich Canada Ltd (Oakville, ON, Canada). L'eau purifiée Milli-Q a été préparée à l'aide d'un système Millipore Milli-Q (Millipore (Canada) Ltd, Nepean, ON, Canada).

### 1.3. Résultats et discussion

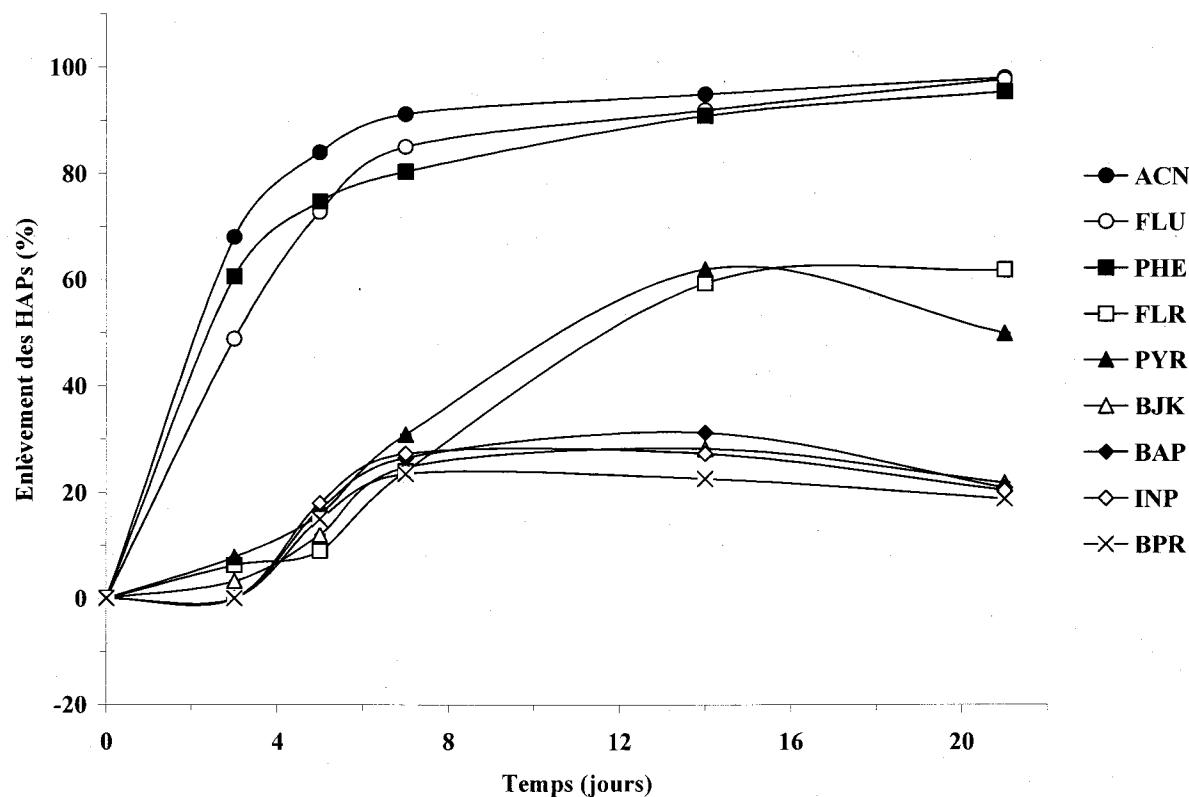
#### 1.3.1 Efficacité d'extraction et de purification des HAPs

L'efficacité de l'extraction Soxhlet avec saponification et purification des HAPs à partir des boues d'épuration a été évaluée par la récupération des étalons de recouvrement. Les pourcentages de récupération ont varié entre 85 et 120% dans la plupart de cas pour chaque étalon de recouvrement et lors de chaque extraction. Par exemple, les étalons de recouvrement des HAPs à deux (acénaphtène-d<sub>10</sub>) et trois (anthracène-d<sub>10</sub>) cycles aromatiques ont été récupérés entre 85 et 110% dans la plupart des cas, alors que les étalons de recouvrement des HAPs à quatre cycles (pyrène-d<sub>10</sub> et chrysène-d<sub>10</sub>) ont été récupérés entre 90 et 120% dans la plupart des cas.

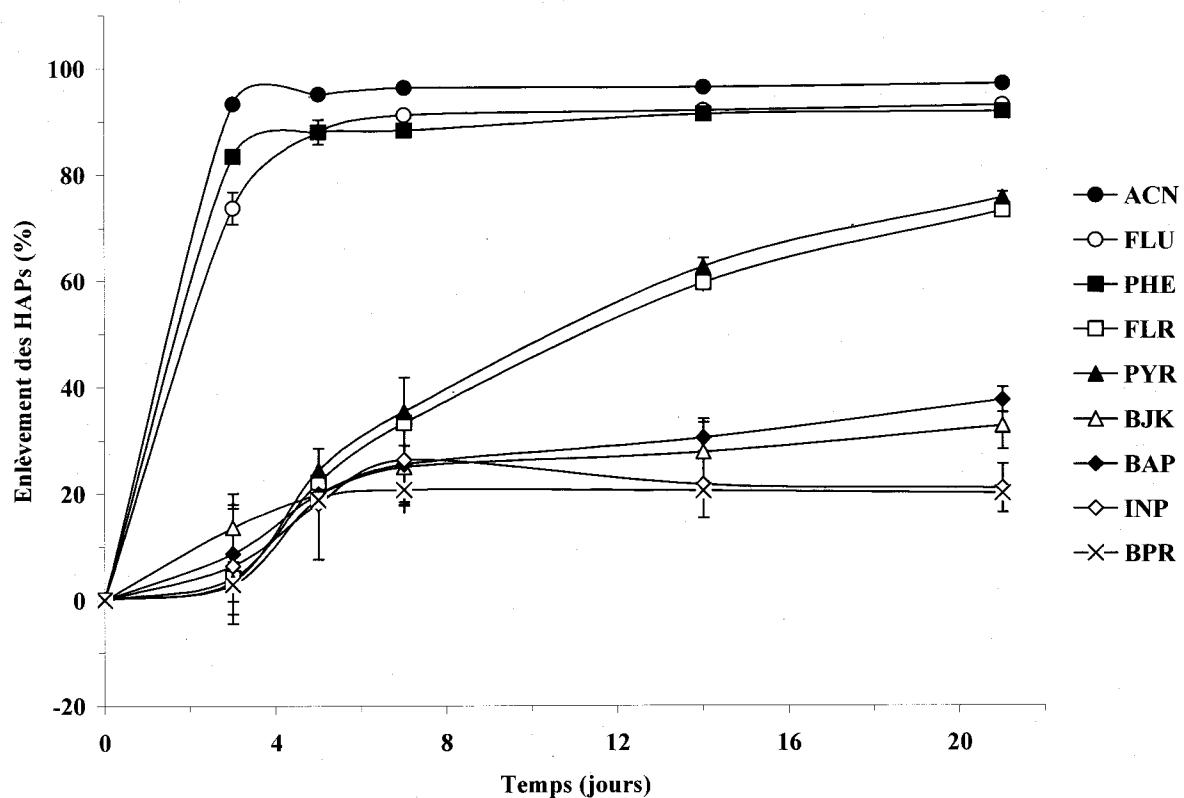
#### 1.3.2 Traitements biologiques (MAD et METIX-BS)

La biodégradation par les bactéries est une méthode qui a été largement étudiée pour l'enlèvement des HAPs à partir de matrices contaminées (Cerniglia, 1992). Les voies métaboliques et les mécanismes de biodégradation ont été bien documentés (Komatsu *et al.*, 1993; Juhasez *et al.*, 1997; Volkering and Breure, 2003). Les recherches ont montré que les HAPs de faible poids moléculaire (2, 3 et 4 cycles aromatiques) sont plus rapidement biodégradés que les composés de haut poids moléculaire (5 cycles et plus) (Heitkamp et Cerniglia, 1989; Cerniglia, 1993). Cependant, il existe très peu de données concernant l'efficacité d'élimination des HAPs par les technologies MAD et METIX-BS. Les Figures 1.1 à 1.3 présentent les cinétiques de biodégradation des HAPs en terme de rendement (%) lors du déroulement des procédés MAD, MAD+Tw80 et METIX-BS. ACN, FLU et PHE sont rapidement biodégradés avec ces trois procédés avec plus de 90% de réduction après

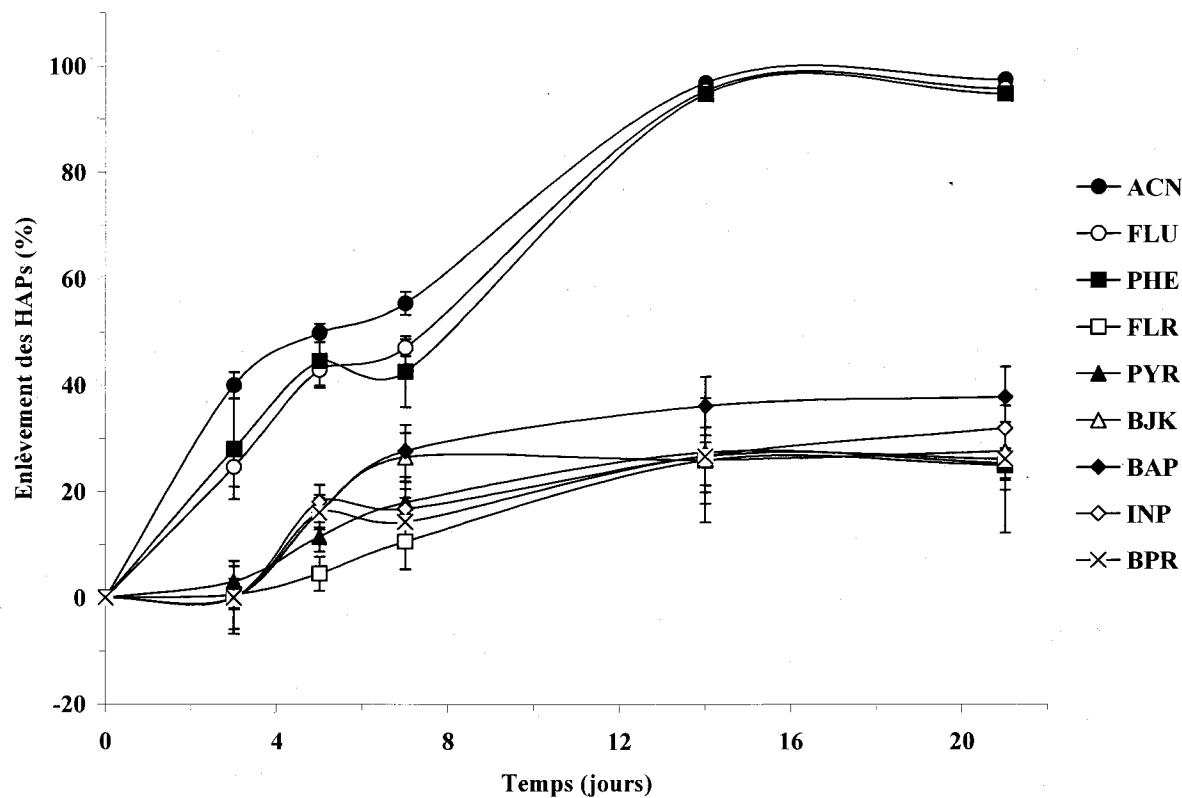
seulement deux semaines de traitement. La biodégradation des autres HAPs débute généralement après une période de 5 jours et les rendements d'élimination sont plus faibles que pour ACN, FLU et PHE.



**Figure 1.1 Variation temporelle de la dégradation des HAPs dans les boues lors du traitement MAD**



**Figure 1.2 Variation temporelle de la dégradation des HAPs dans les boues lors du traitement MAD+Tw80**



**Figure 1.3 Variation temporelle de la dégradation des HAPs dans les boues lors du traitement METIX-BS**

Le Tableau 1.3 montre les concentrations finales de HAPs dans les boues et les rendements correspondants d'enlèvement de ces composés après les différents traitements biologiques. Après 21 jours d'incubation, les boues contrôles stérilisées (DAC) ont montré que la volatilisation des HAPs était modérée pour CAN, FLU et PHE avec des rendements d'enlèvement de respectivement 34,3, 32,8 et 18,7%. La volatilisation était faible (< 15%) dans le cas des autres HAPs analysés.

L'ajout d'un surfactant non-ionique (Tw80) lors du procédé MAD a causé une augmentation substantielle de la biodégradation, particulièrement pour FLR, PYR, BJK et BAP. Cependant, aucun effet notable n'a été révélé dans le cas de INP et BPR. De manière générale, l'ajout de Tw80 a légèrement augmenté l'élimination des HAPs, soit de  $54,4 \pm 2,9\%$  (MAD) à  $60,1 \pm 2,0\%$  (MAD+Tw80).

Le procédé METIX-BS était très efficace pour l'élimination de CAN, FLU et PHE, mais la dégradation de FLR et PYR était plus faible que pour MAD et MAD+Tw80. Cependant, les HAPs de haut poids moléculaire (BAP, INP et BPR) ont été légèrement mieux éliminés que pour le procédé conventionnel MAD. Le rendement global d'enlèvement des HAPs par le procédé METIX-BS ( $51,8 \pm 3,0\%$ ) était pratiquement similaire à celui du procédé MAD. La dégradation des HAPs observée avec le procédé METIX-BS s'explique probablement par des mécanismes biologiques impliquant des microorganismes non-acidophiles (durant les premiers jours de traitement) et acidophiles (lorsque le pH descend sous 4.0), ainsi que par une dégradation chimique attribuable aux conditions acide et oxydante prévalentes dans les derniers jours du traitement (pH 1.42 et POR +461 mV à la fin du traitement). Des études antérieures ont montré que la levure *Blastoschizomyces capitatus* et une moisissure inconnue sont les organismes dominants présents dans les boues à la fin du procédé METIX-BS et seraient, en partie, responsables de la dégradation de la matière organique (Gamache *et al.*, 2001).

Le Tableau 1.4 montre les pourcentages de solubilisation des HAPs dans les boues lors de l'application des différents procédés. Globalement, les résultats révèlent que la proportion de HAPs présente dans la phase liquide est généralement très faible ( $\leq 10\%$  de chaque HAP). En particulier, les 11 composés de HAPs dans la phase liquide ont été presque complètement

éliminés par les traitements MAD et MAD+Tw80, puisque que les concentrations de ces composés étaient aussi faibles que dans le cas des boues contrôles (DOC). Dans le cas du procédé METIX-BS, seulement les composés de faible poids moléculaire (CAN, FLU et PHE) ont été efficacement enlevés de la phase liquide. Les autres HAPs (4, 5 et 6 cycles) ont été partiellement enlevés avec des concentrations finales plus élevées que les boues contrôles. Ces résultats indiquent que les taux de désorption des HAPs lourds à partir des solides des boues sont plus importants que les taux de dégradation correspondants avec le procédé METIX-BS.

La Figure 1.4 illustre l'efficacité des procédés biologiques pour la dégradation des HAPs en fonction du nombre de cycles aromatiques des molécules de HAPs. Premièrement, une proportion modérée ( $28,6 \pm 3,7\%$ ) des HAPs à trois cycles (CAN, FLU et PHE) a été éliminée des boues dopées et autoclavées (DAC), alors que pour les HAPs de quatre à six cycles, les pourcentages d'élimination se situaient à 10-12%. La dégradation des HAPs à trois cycles était presque similaire pour le procédé MAD ( $97,0 \pm 0,3\%$ ) et METIX-BS ( $95,5 \pm 0,7\%$ ), mais les HAPs à quatre cycles (FLR et PYR) étaient significativement mieux éliminés dans le cas de MAD ( $56,6 \pm 1,8\%$ ) en comparaison à METIX-BS ( $29,9 \pm 2,7\%$ ). L'ajout de Tw80 lors du traitement MAD a bien été monté d'enlèvement des HAPs à quatre cycles ( $74,4 \pm 1,1\%$ ). Les HAPs de plus haut poids moléculaire n'étaient pas éliminés efficacement (< 40% enlèvement) par les deux procédés. L'ajout de Tw80 lors du traitement MAD a été seulement favorable pour la dégradation des HAPs à cinq cycles (BJK et BAP) avec un rendement d'enlèvement de  $35,1 \pm 3,4\%$ , en comparaison à  $21,3 \pm 2,7\%$  dans le cas de MAD et  $32,7 \pm 5,6\%$  dans le cas de METIX-BS.

*1. Synthèse française*

**Tableau 1.3 Concentrations (mg kg<sup>-1</sup> MS) et rendements finaux d'enlèvement (%) des HAPs dans les boues après les différents traitements biologiques**

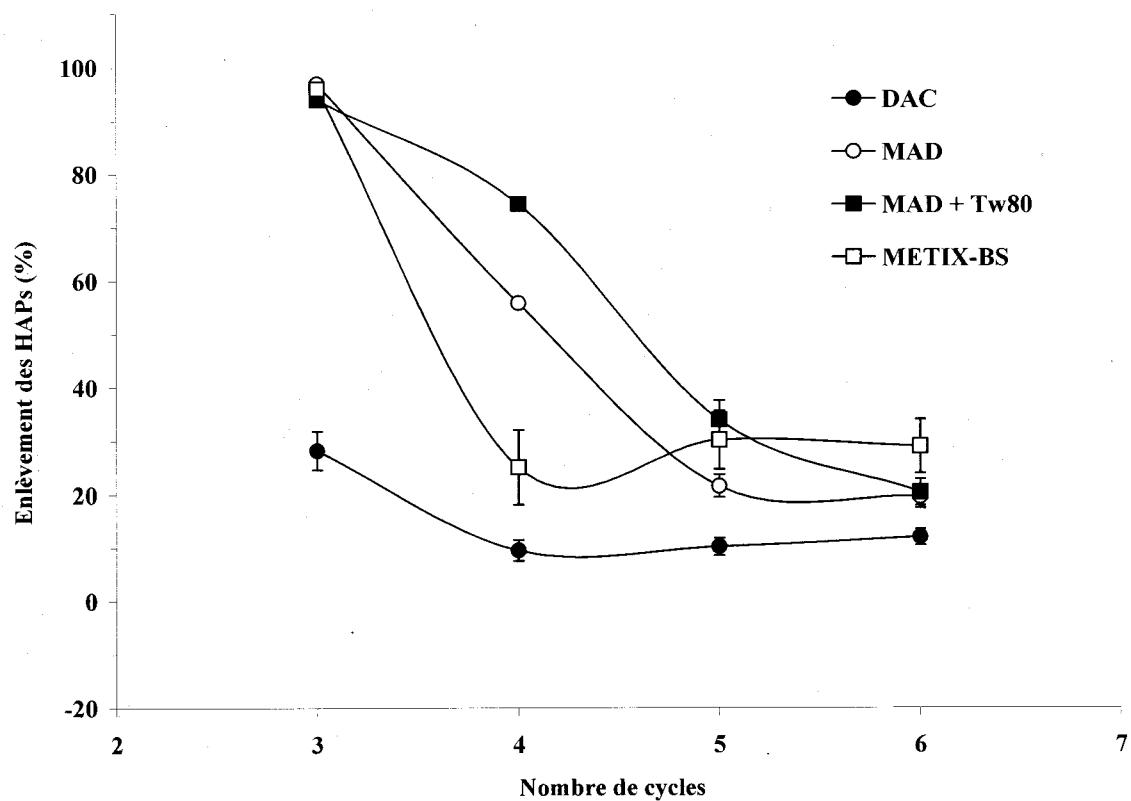
HAPs	DAC		MAD		MAD+Tw80		METIX-BS	
	Conc. (mg kg <sup>-1</sup> )	Enlèvement (%)						
ACN	3,65 ± 0,27	34,3 ± 4,9	0,11 ± 0,01	97,9 ± 0,1	0,18 ± 0,00	96,5 ± 0,1	0,17 ± 0,01	96,9 ± 0,3
FLU	3,74 ± 0,16	32,8 ± 2,8	0,12 ± 0,01	97,8 ± 0,2	0,42 ± 0,03	92,4 ± 0,6	0,27 ± 0,02	95,2 ± 0,4
PHE	4,66 ± 0,19	18,7 ± 3,4	0,28 ± 0,03	95,1 ± 0,6	0,51 ± 0,05	91,1 ± 0,9	0,33 ± 0,08	94,3 ± 1,3
FLR	5,59 ± 0,12	7,2 ± 1,9	2,29 ± 0,07	62,1 ± 1,1	1,65 ± 0,08	72,7 ± 1,4	3,69 ± 0,14	38,8 ± 0,2
PYR	5,28 ± 0,12	12,8 ± 2,1	2,97 ± 0,14	51,1 ± 2,4	1,51 ± 0,07	75,0 ± 1,1	4,79 ± 0,15	21,0 ± 2,4
BJK	15,57 ± 0,39	12,1 ± 2,2	14,05 ± 0,81	20,7 ± 4,6	11,86 ± 0,77	32,1 ± 4,4	13,14 ± 0,51	25,9 ± 2,9
BAP	5,37 ± 0,16	9,4 ± 2,7	4,59 ± 0,34	22,6 ± 5,8	3,72 ± 0,14	37,2 ± 2,3	3,78 ± 0,64	36,2 ± 10,8
INP	5,25 ± 0,37	14,6 ± 6,1	4,64 ± 0,28	24,5 ± 4,6	4,81 ± 0,28	21,7 ± 4,5	4,29 ± 0,11	30,2 ± 1,7
BPR	5,30 ± 0,14	12,6 ± 2,4	4,98 ± 0,42	17,9 ± 7,0	4,77 ± 0,16	21,4 ± 2,7	4,40 ± 0,43	27,5 ± 7,0
Total	54,40 ± 1,22	16,0 ± 3,0	34,01 ± 2,12	54,4 ± 2,9	29,44 ± 1,58	60,1 ± 2,0	34,85 ± 1,95	51,8 ± 3,0

*1. Synthèse française*

**Tableau 1.4 Fraction soluble (%) des HAPs dans les boues après les différents traitements biologiques, chimiques et électrochimiques**

HAPs	DOC	MAD	MAD + Tw80*	METIX-BS	METIX-AC	METIX- AC+Tw80	STABIOX	ELECSTAB
ACN	4,54 ± 0,21	0,48 ± 0,00	0,03 ± 0,00	0,51 ± 0,00	2,33 ± 0,21	1,97 ± 0,17	3,19 ± 0,25	1,53 ± 0,42
FLU	4,74 ± 1,24	0,55 ± 0,10	0,02 ± 0,00	0,69 ± 0,06	3,65 ± 0,65	2,00 ± 0,35	2,84 ± 0,25	1,58 ± 0,37
PHE	4,26 ± 1,79	0,71 ± 0,26	0,02 ± 0,00	0,65 ± 0,01	2,39 ± 0,25	1,48 ± 0,03	1,48 ± 0,19	1,35 ± 0,25
FLR	5,29 ± 1,97	0,79 ± 0,38	0,11 ± 0,01	4,57 ± 0,33	3,20 ± 0,35	7,19 ± 4,98	1,50 ± 0,22	1,96 ± 0,44
PYR	5,23 ± 1,94	0,88 ± 0,39	0,13 ± 0,01	7,13 ± 0,48	3,21 ± 0,36	12,40 ± 0,10	1,53 ± 0,22	2,31 ± 0,24
BJK	3,21 ± 0,55	0,73 ± 0,11	0,05 ± 0,01	7,94 ± 1,23	3,14 ± 0,71	4,22 ± 0,54	0,83 ± 0,05	3,89 ± 0,57
BAP	5,00 ± 0,08	0,74 ± 0,30	0,03 ± 0,00	10,06 ± 1,71	3,57 ± 0,77	1,83 ± 0,36	1,23 ± 0,09	2,70 ± 0,77
INP	5,22 ± 0,54	0,69 ± 0,24	0,04 ± 0,00	10,11 ± 1,49	4,09 ± 0,96	3,06 ± 0,54	1,11 ± 0,05	2,23 ± 0,57
BPR	4,87 ± 0,41	0,72 ± 0,31	4,05 ± 0,44	9,38 ± 1,13	3,94 ± 0,89	2,86 ± 0,68	1,03 ± 0,07	2,43 ± 0,61
Total	4,44 ± 0,70	0,71 ± 0,21	0,42 ± 0,04	6,32 ± 0,84	3,28 ± 0,57	4,18 ± 0,47	1,44 ± 0,10	2,53 ± 0,49

\* Après une période de traitement de 3 jours.



**Figure 1.4 Rendements finaux d'enlèvement des HAPs dans les boues après les différents traitements biologiques en fonction du nombre de cycles aromatiques des HAPs**

### 1.3.3 Traitements chimiques (METIX-AC, STABIOX) et électrochimique (ELECSTAB)

Lors de l'opération des procédés METIX-AC et STABIOX de l'acide sulfurique est ajouté aux boues, suivi par l'ajout séquentiel de peroxyde d'hydrogène ( $H_2O_2$ ) et d'ions ferriques ( $FeCl_3$ ). Ce système est comparable au réactif de Fenton, lequel a été étudié récemment pour le traitement de composés organiques non-biodégradables dans les eaux usées (El-Morsi *et al.*, 2002; Lee *et al.*, 2003). Le système  $Fe^{3+}/H_2O_2$  peut produire des radicaux oxydatifs hydroxyl et perhydroxyl, lesquels sont capables d'oxyder la plupart des substances organiques présentes dans les eaux usées municipales. En plus des espèces radicalaires, le peroxyde d'hydrogène peut aussi former en milieu acide du peroxyde d'hydrogène protoné. Ce composé ( $HOOH_2^+$ ) serait un intermédiaire très instable avec une forte activité oxydante.

Les concentrations et rendements finaux d'enlèvement des HAPs par les différents procédés chimiques et électrochimiques testés sont présentés au Tableau 1.5. De manière générale, la dégradation des HAPs était plus faible que dans le cas des procédés biologiques. Le procédé METIX-AC a tout de même permis d'atteindre des rendements intéressants pour ACN ( $58,1 \pm 6,9\%$ ), FLU ( $47,1 \pm 7,1\%$ ) et PHE ( $41,5 \pm 11,2\%$ ), mais une très faible dégradation des HAPs ayant un haut poids moléculaire (< 35%).

L'ajout d'un surfactant non-ionique (Tw80) lors du procédé METIX-AC n'a pas été bénéfique pour la dégradation des HAPs. En fait, les rendements globaux d'enlèvement des HAPs étaient plus faible pour les procédés METIX-AC ( $29,6 \pm 7,7\%$ ) et METIX-AC+Tw80 ( $11,6 \pm 5,2\%$ ).

Les conditions moins acide et oxydante prévalantes lors de l'opération du procédé STABIOX, en comparaison au traitement METIX-AC, a causé une plus faible dégradation des HAPs avec un rendement global d'enlèvement des 11 HAPs de seulement  $22,2 \pm 3,2\%$ . Une étude de Pignatello

a montré que la transformation des polluants organiques serait optimale à pH 2,7 ~ 2,8 par  $\text{Fe}^{2+}/\text{H}_2\text{O}_2$  parce que la moitié du fer serait présent sous forme de  $\text{Fe}(\text{OH})^{2+}$ , lequel serait un intermédiaire important dans la réaction de Fenton (Pignatello, 1992). Des travaux futurs devraient donc être réalisés afin d'optimiser les valeurs de pH lors de l'opération du procédé METIX-AC pour un enlèvement maximal combiné des métaux et des HAPs.

La dégradation des HAPs de faible poids moléculaire (ACN, FLU et PHE) lors du procédé ELECSTAB était assez bonne avec environ 62% d'enlèvement. Cependant, pratiquement aucune élimination des HAPs de haut poids moléculaire n'a été mesurée suite à l'application de ce traitement électrolytique.

La proportion de HAPs présente dans la phase liquide n'a pas été modifiée de manière notable par les procédés chimiques et électrochimique. (Tableau 1.4). Ainsi, à la fin de chaque traitement, les concentrations des 11 HAPs dans la phase liquide étaient presque semblables à celles mesurées dans les boues contrôles (DOC).

1. Synthèse française

**Tableau 1.5 Concentrations (mg kg<sup>-1</sup> MS) et rendements finaux d'enlèvement (%) des HAPs dans les boues après les différents traitements chimiques et électrochimiques**

HAPs	METIX-AC		METIX-AC+Tw80		STABIOX		ELECSTAB	
	Conc. (mg kg <sup>-1</sup> )	Enlèvement (%)						
ACN	2,33 ± 0,39	58,1 ± 6,9	3,86 ± 0,20	30,5 ± 3,5	3,75 ± 0,09	32,4 ± 1,6	1,78 ± 0,07	67,9 ± 1,2
FLU	2,94 ± 0,40	47,1 ± 7,1	4,53 ± 0,22	18,5 ± 3,7	4,37 ± 0,12	21,4 ± 2,1	2,30 ± 0,07	58,7 ± 1,3
PHE	3,35 ± 0,64	41,5 ± 11,2	4,57 ± 0,18	20,2 ± 3,2	3,26 ± 0,11	43,0 ± 1,9	2,26 ± 0,27	60,5 ± 4,7
FLR	4,01 ± 0,55	33,5 ± 9,4	6,47 ± 0,23	2,0 ± 3,8	4,61 ± 0,32	23,5 ± 5,3	5,81 ± 0,45	3,6 ± 7,4
PYR	3,80 ± 0,53	37,2 ± 8,9	6,04 ± 0,76	1,0 ± 12,6	4,49 ± 0,32	25,9 ± 5,4	5,80 ± 0,29	4,2 ± 4,9
BJK	15,36 ± 0,39	13,3 ± 2,8	15,49 ± 0,23	12,6 ± 1,3	14,04 ± 0,56	20,8 ± 3,2	17,55 ± 0,88	1,0 ± 4,9
BAP	4,39 ± 0,17	25,9 ± 3,4	5,79 ± 0,36	2,3 ± 6,1	5,05 ± 0,10	14,8 ± 1,7	5,66 ± 0,25	4,5 ± 4,1
INP	6,03 ± 0,64	1,9 ± 11,9	5,83 ± 0,49	5,1 ± 8,0	5,44 ± 0,30	11,4 ± 4,8	5,55 ± 0,21	9,6 ± 3,4
BPR	5,57 ± 0,69	8,2 ± 11,4	5,24 ± 0,29	13,7 ± 4,8	5,65 ± 0,19	6,9 ± 3,1	5,46 ± 0,34	10,0 ± 5,6
Total	47,70 ± 4,33	29,6 ± 8,2	57,81 ± 2,99	11,8 ± 5,2	50,67 ± 2,10	22,2 ± 3,2	52,19 ± 1,09	24,4 ± 4,2

### **1.3.4 Traitement au surfactant non-ionique**

Le Tableau 1.6 montre les rendements de solubilisation des HAPs à partir des boues dopées après différents traitements avec l'agent d'extraction Tw80. De bons rendements de solubilisation ont été obtenus pour ACN ( $78,0 \pm 0,2\%$ ), FLU ( $72,3 \pm 0,5\%$ ) et PHE ( $69,3 \pm 1,0\%$ ) après 6 h de traitement en présence of  $0.5 \text{ g Tw80 L}^{-1}$  (assai Tw80-A), alors que les autres HAPs n'ont pas été efficacement extraits (enlèvement  $\leq 25\%$ ) des boues. La baisse du temps de traitement de 6 à 4 h (essai Tw80-B) a eu un effet négatif sur la solubilisation des HAPs avec des rendements inférieurs à 40% et ce, pour tous les HAPs étudiés. L'augmentation de la concentration de surfactant à  $2,0 \text{ g Tw80 L}^{-1}$  a permis, même pour un court temps de traitement (2 h), de hausser les rendements de solubilisation à plus de 90% pour CAN et PHE et 80% pour FLU. Les rendements d'extraction des autres HAPs sont toutefois demeurés faibles ( $< 20\%$ ). Il faut également noter que cette concentration de Tw80 à  $2,0 \text{ g L}^{-1}$ , utilisée à l'échelle laboratoire, ne pourrait être envisagée à l'échelle industrielle pour des raisons économiques.

*1. Synthèse française*

**Tableau 1.6 Fraction solubile (%) des HAPs dans les boues après les différents traitements au surfactant**

HAPs	Méthode de séparation S/L	Tw80-A				Tw80-B	Tw80-C
		Filtration	Centrifugation			Filtration	Filtration
			500 x g	1 000 x g	2 000 x g		
ACN		78,0 ± 0,2	38,8 ± 0,4	33,8 ± 0,0	39,5 ± 2,3	48,3 ± 1,3	36,6 ± 3,6
FLU		72,3 ± 0,5	39,5 ± 3,4	31,9 ± 2,2	37,2 ± 3,9	43,6 ± 1,3	35,2 ± 4,7
PHE		69,3 ± 1,0	39,4 ± 1,8	36,3 ± 0,5	37,0 ± 1,5	47,5 ± 0,7	21,9 ± 5,3
FLR		25,1 ± 1,7	5,9 ± 7,6	14,3 ± 5,9	-1,5 ± 6,2	5,0 ± 7,5	8,6 ± 1,5
PYR		9,4 ± 4,0	12,1 ± 7,4	11,6 ± 11,2	3,5 ± 6,0	7,1 ± 8,1	9,4 ± 3,1
BJK		14,8 ± 11,9	15,0 ± 7,2	22,7 ± 5,6	2,0 ± 8,4	4,5 ± 8,7	11,1 ± 6,0
BAP		9,9 ± 6,3	4,3 ± 4,7	5,4 ± 17,1	1,8 ± 2,7	6,0 ± 3,7	10,1 ± 6,5
INP		13,8 ± 10,4	2,5 ± 7,5	22,8 ± 8,6	5,7 ± 8,4	10,0 ± 5,0	8,2 ± 7,9
BPR		14,6 ± 15,5	9,8 ± 12,9	8,9 ± 19,9	1,7 ± 8,8	-5,2 ± 4,5	11,4 ± 4,1
Total		30,0 ± 1,9	17,6 ± 4,2	21,0 ± 1,9	11,5 ± 1,5	15,5 ± 4,2	15,6 ± 2,6
							26,7 ± 6,6

La comparaison de la filtration et de la centrifugation en tant que méthodes de séparation S/L des boues traitées avec un surfactant a démontré que la première option donne de meilleurs rendements de récupération des HAPs dans la phase liquide. En fait, les HAPs solubilisés dans les micelles de surfactant peuvent être facilement séparés de la phase solide puisque les micelles présents dans la phase liquide traversent aisément la membrane Whatman No. 4. D'un autre côté, la centrifugation, même à faible vitesse, semble entraîner une séparation des micelles de la fraction liquide et retournés ceux-ci dans le culot de centrifugation. Un comportement similaire a également été observé (résultats non présentés) dans le cas de l'essai avec 2.0 g Tw80 L<sup>-1</sup>.

Aucun des traitements avec le surfactant ne s'est révélé efficace pour l'enlèvement des HAPs entre 4 et 6 cycles aromatiques à partir des boues. Ainsi, la combinaison du Tw80, ou d'un autre surfactant, avec un traitement biologique (MAD ou METIX-BS) ou un traitement chimique (METIX-AC) apparaît être une voie plus intéressante que l'utilisation seule de surfactant pour la décontamination des boues d'épuration. Finalement, il est important de prendre en considération que les HAPs extraits des boues par un surfactant se retrouvent dans la phase liquide (filtrat) et doivent être détruits, récupérés, ou gérés à des coûts probablement très élevés.

#### **1.4. Conclusions**

Dans cette étude, l'extraction Soxhlet suivie d'une saponification a été utilisée pour analyser les HAPs présents dans des boues issues du traitement des eaux usées municipales. La purification par gel de silice s'est avérée nécessaire pour éliminer d'autres composés organiques pouvant interférer lors de l'analyse par GC-MS. Tel que discuté précédemment, l'efficacité de l'extraction

Soxhlet suivie de la saponification et de la purification des HAPs à partir des boues d'épuration a été évaluée par la récupération des étalons de recouvrement. Les pourcentages de récupération de ceux-ci se situaient entre 85 et 110% dans la plupart des cas.

Des résultats prometteurs ont été obtenus pour l'enlèvement des trois HAPs à trois cycles (acénaphthène, phénanthrène et fluorène) et ce, par deux traitements biologiques (digestion aérobiose mésophile (MAD) et METIX-BS) et le traitement de MAD avec un ajout de Tw80. L'addition de Tw80 lors de l'opération du procédé de MAD peut augmenter la vitesse de biodégradation des HAPs à quatre (pyrène) et cinq (benzo(b,j,k)fluoranthène) cycles. Toutefois, l'enlèvement des HAPs à six cycles est demeuré faible pour l'ensemble des procédés testés.

Les HAPs à trois cycles (acénaphthène, phénanthrène et fluorène) peuvent aussi être enlevés des boues d'épuration par deux traitements chimiques (METIX-AC et STABIOX) et le traitement électrochimique (ELECSTAB). Les meilleurs résultats ont toutefois été obtenus par les traitements METIX-AC et ELECSTAB. Les conditions d'opération de ces procédés devraient toutefois être optimisées afin d'accroître les rendements d'élimination des HAPs.

Les recherches devraient se poursuivre afin, notamment, de mettre au point des procédés capables d'extraire ou de dégrader les HAPs les plus lourds ( $\geq 4$  cycles). Les procédés biologiques, tels que la digestion aérobiose et le procédé combiné de digestion des boues et de biolixiviation des métaux (METIX-BS) semblent les voies les plus prometteuses à explorer. Une combinaison de traitements biologiques et chimiques pourrait également représenter une option intéressante.

## 2. INTRODUCTION

Sewage sludge, also known as biosolids, is an end product of wastewater treatment process. For instance, using this material as a fertilizer can benefit the environment with turning the wastes into valuable resources (Duvaud *et al.*, 1999; USEPA, 1999). However, certain organic and inorganic toxics present in the sewage sludge may threaten crop yields, long term soil quality, cattle and wildlife health, and eventually human health (Gardiner *et al.*, 1995; Lee *et al.*, 1996).

Risks associated with pathogens in sewage sludge, such as bacteria, viruses, parasites, can be reduced, for example, by biological or chemical stabilization techniques (USEPA, 1999; Spinoso and Vesilind, 2001). Moreover, many remediation methods for heavy metals removal from sludge have been proposed in the last fifteen years (Mercier *et al.*, 2002; Chan *et al.*, 2003). However, the removal of persistent organic pollutants (POP) is much more difficult because of their stable physical and chemical properties.

Polycyclic aromatic hydrocarbons (PAHs) are an important class of the POP in causing environmental problems due to their high potential of toxicity, mutagenicity, and carcinogenicity to mammals and aquatic organisms (Wilson and Jones, 1993; Mangas *et al.*, 1998). In the late 1980s, the United States Environmental Protection Agency (USEPA) specified 16 main PAHs because of their known toxicity to mammals and aquatic organisms (USEPA, 1987). Currently, the concentration limitations in sewage sludge were not released and enforced in North America but they are in a few European countries. The maximum concentration of 11 main PAHs compounds from acenaphthene (ACN) to benzo(ghi)perylene (BPR) in sewage sludge could not exceed  $6 \text{ mg kg}^{-1}$  DM if the sludge is designated to be applied on agricultural land. In France, the recent regulation released on January, 8<sup>th</sup>, 1998 imposes a maximum acceptable limit for three

most carcinogenic PAHs (fluoranthene (FLR), benzo(b)fluoranthene and benzo(a)pyrene (BAP)) and they are legislatively controlled to be lower than 5, 2.5 and 2 mg kg<sup>-1</sup> DM. Therefore, the accurate determination of PAHs concentrations in sewage sludge has become a closely relevant topic because of the release of the draft by the European Union (EU, 2000).

PAHs are considered among the most difficult POP to be treated because of their highly stable physical-chemical characteristics and frequent occurrences (Volkering and Breure, 2003). In the last years, different approaches have been studied for the removal of PAHs from contaminated soils or solid wastes: biodegradation (Cerniglia, 1992; Chang *et al.*, 2003; Stapleton *et al.*, 1998), chemical oxidation (Masten et Davies, 1997; Flotron *et al.*, 2003a; N'Guessan *et al.*, 2004) and photodegradation (Fukuda *et al.*, 1988; Sigman *et al.*, 1998; Miller and Olejnik, 2001). In addition, some extraction methods using surfactants have been successfully applied for remediation of PAH-contaminated soils or sediments (Volkering *et al.*, 1995; Guha *et al.*, 1998; Kim *et al.*, 2001; Li and Chen 2002; Zhou and Zhu, 2005).

## 2.1. Objectives

The aim of this study is to evaluate the potential of different sludge treatment processes for the removal of PAHs from municipal sewage sludge. This notably includes two biological methods (conventional mesophilic aerobic digestion (MAD) and simultaneous sludge digestion and metal leaching process (METIX-BS or SSDML) (Tyagi *et al.*, 1995; Blais *et al.*, 2004)). In addition, two chemical oxidation processes (chemical stabilization (STABIOX) (Blais *et al.*, 2003) and chemical metal leaching (METIX-AC) (Blais *et al.*, 2005; Mercier *et al.*, 2002)), and one electrochemical stabilization process (ELECSTAB) (Droguic *et al.*, 2005) will be investigated for

PAHs degradation. The effect of a nonionic surfactant (Tween 80) on the solubilization and biodegradation of PAHs in sewage sludge will also be explored.

## **2.2. Overview**

The central theme in this work is quantitative analysis and removal of PAHs from sewage sludge. There are four parts in this English version of the thesis. In Chapter 3, a brief literature review including basic information of PAHs, methodologies for PAHs analysis in pollutants and different PAHs removal technologies are given. In Chapter 4, methodologies and experimental details are described. In Chapter 5, the results and discussion of PAHs determinations (Soxhlet extraction-saponification and purification) and PAHs removal from contaminated sewage sludge were presented. The major conclusions from this work and directions for the future work are presented in Chapter 6.



## 3. LITTERATURE REVIEW

### 3.1. Polycyclic aromatic hydrocarbons (PAHs)

#### 3.1.1 Definition and origin of PAHs

##### 3.1.1.1 *Structures of PAHs*

PAHs are a class of very stable organic molecules made up of only carbon and hydrogen. These molecules are flat, with each carbon having three neighboring atoms much like graphite. They are a relevant group of compounds classified as more than 100 chemicals which stick to solid particles and can be concentrated in sediments, soils or sludges. These compounds have more than 2 benzene rings and molecular weights from  $128 \text{ g mol}^{-1}$  to  $300 \text{ g mol}^{-1}$ . The 11 major PAHs compounds as an example were given in Table 3.1, which presents in most pollution sources and is restricts to a maximum total concentration not exceed  $6 \text{ mg kg}^{-1}$  DM (dry matter) in sewage sludge, if the sludge is designated to be applied on agricultural land (EU, 2000).

##### 3.1.1.2 *Sources of PAHs*

PAHs are emitted into the environments from both natural and anthropogenic sources. Forest fires, volcanic eruptions and decaying organic matters are all natural sources of PAH. Industrial processes such as pharmaceuticals, dyes, plastics, and pesticides making, aluminum production using old technology and iron smelting, petroleum refining, coal coking, thermal power generation, tar paper production, and wood preservation operations using creosote, are all examples of major human sources. Beside a small amount from natural occurrence, most PAHs are resulted from industrial process and other human activities (Neff, 1979; WHO, 1998). Many useful products such as mothballs, blacktop and creosote wood preservatives contain PAHS, and

they are also found at low concentrations in some special-purpose skin creams and anti-dandruff shampoos which contain coal tars (Revised, 2000).

### **3.1.2 Physical and chemical properties of PAHs**

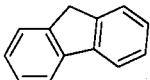
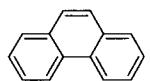
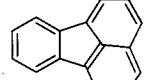
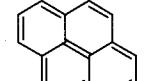
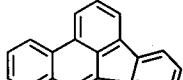
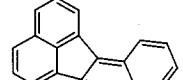
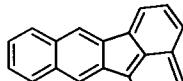
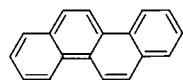
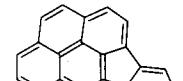
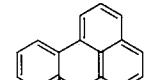
The physical and chemical properties of PAHs are governed by their large number of carbon atoms and shape (ring linkage pattern) of each individual molecule. All of the completely unsaturated PAHs are solid at room temperature and have relatively high melting and boiling points. The pure PAHs usually are colorless, white, or pale yellow-green. They describe the physical assets of a specific compound such as: solubility, vapor pressure, octanol-water coefficient (*K<sub>ow</sub>*) and Henry's Law constants etc. (Mackay *et al.*, 1992).

#### **3.1.2.1 Solubility and vapor pressure of PAHs**

PAHs are generally insoluble in water but can be readily dissolved in organic solvents. Solubility (S) and vapor pressure (V<sub>p</sub>) characteristics of PAHs are the major physical factors that control their distribution between the soluble and particle components of the atmosphere, hydrosphere and biosphere. The aqueous solubility and the vapor pressure of PAHs are inversely proportional to the number of rings it contains and decrease with increasing the molecular size.

3. Literature review

**Table 3.1 11 PAHs in sewage sludge restricted by EU (EU, 2000)**

Chemical names	Abbreviation	Molecular formula	Structure	Molecular weight
Acenaphtene	ACN	C <sub>12</sub> H <sub>10</sub>		154
Fluorene	FLU	C <sub>13</sub> H <sub>10</sub>		166
Phenanthrene	PHE	C <sub>14</sub> H <sub>10</sub>		178
Fluoranthene	FLR	C <sub>16</sub> H <sub>10</sub>		202
Pyrene	PYR	C <sub>16</sub> H <sub>10</sub>		202
Benzo(b)fluoranthene	B(B)F	C <sub>20</sub> H <sub>12</sub>		252
Benzo(j)fluoranthene	B(J)F	C <sub>20</sub> H <sub>12</sub>		252
Benzo(k)fluoranthene	B(K)F	C <sub>20</sub> H <sub>12</sub>		252
Benzo(a)pyrene	BAP	C <sub>20</sub> H <sub>12</sub>		252
Indeno(1,2,3-cd)pyrene	INP	C <sub>22</sub> H <sub>12</sub>		276
Benzo(ghi)perylene	BPR	C <sub>22</sub> H <sub>12</sub>		276

### 3.1.2.2 Octanol-water coefficient

The octanol-water coefficient (*K<sub>ow</sub>*) is the ratio of concentration of a specific chemical in an octanol-water mixture. It is a measure of the hydrophobicity of organic chemical and it can be used to determine the partitioning and fate of PAHs in the environment. The PAHs constituents ranges from moderately to highly lipophilic, having *logK<sub>ow</sub>* of 3.37-6.75.

$$K_{ow} = \frac{\text{concentration of chemical in octanol}}{\text{concentration of chemical in water}}$$

### 3.1.2.3 Henry's Law constants

Henry's Law constants (*H<sub>c</sub>*) in a liquid-vapor system are the ratio of gas pressure to liquid concentration of the chemical in question. Volatilization is potentially important for some PAHs with *H<sub>c</sub>* greater than 10<sup>-4</sup> (Table 3.2) even if it is somehow limited by adsorption onto organic matter particles.

$$H_c = \frac{\text{pressure of chemical in vapor phase}}{\text{concentration of chemical in liquid phase}}$$

**Table 3.2** Physical properties of 11 PAHs (Volkering *et al.* 2003)

PAH	Color	Physical state	Density (g cm <sup>-3</sup> )	MP (°C)	BP (°C)	S <sub>H<sub>2</sub>O</sub> (mg l <sup>-1</sup> )	Vp (Pa)	log K <sub>ow</sub>	H <sub>c</sub> (Pa m <sup>-3</sup> mol <sup>-1</sup> )	Organic solvent
ACN	white	needle	1.225 (0°C)	96	277	3.8	0.3	3.92	8.4	ethanol, methanol, propanol, chloroform benzene, toluene, acetic acid ethanol, methanol, acetone, benzene, toluene, acetic acid ethanol, methanol, acetone chloroform benzene, acetic acid ethanol, ether benzene, acetic acid ethanol, methanol, acetone chloroform benzene, acetic acid benzene acetone ethanol acetic acid ethanol benzene acetone acetic acid methanol benzene, toluene xylene ether ethanol, methanol, acetone chloroform benzene, toluene, acetic acid ethanol benzene CH <sub>2</sub> Cl <sub>2</sub>
FLU	white	plate	1.203 (0°C)	116	295	1.9	0.09	4.18	7.87	
PHE	colorless	plate	0.980 (4°C)	101	339	1.1	0.02	4.57	3.24	
FLR	pale yellow	needle, plate	1.252 (0°C)	111	375	0.26	0.00123	5.22	1.037	
PYR	colorless /pale yellow	plate	1.271 (23°C)	156	360	0.132	0.0006	5.18	0.92	
B(B)F	colorless	needle, plate	-	168	481	0.0015	6.70 x 10 <sup>-5</sup>	5.80	-	
B(J)F	yellow /orange	needle	-	166	-	0.00068	1.50 x 10 <sup>-8</sup>	-	-	
B(K)F	pale yellow	needle	-	217	481	0.0008	5.20 x 10 <sup>-8</sup>	6.00	0.016	
BAP	pale yellow	needle, plate	1.271 (23°C)	175	495	0.0038	7.00 x 10 <sup>-7</sup>	6.04	0.046	
INP	yellow	needle, plate	-	164	533	0.062	1.30 x 10 <sup>-8</sup>	6.60	-	
BPR	pale yellow-green	plate	-	277	550	0.00026	1.03 x 10 <sup>-10</sup>	6.50	0.075	

\*MP, melting point at normal pressure; BP, boiling point at normal pressure; S (water solubility) and Vp at 20 °C.

### 3.1.3 PAHs in the environments

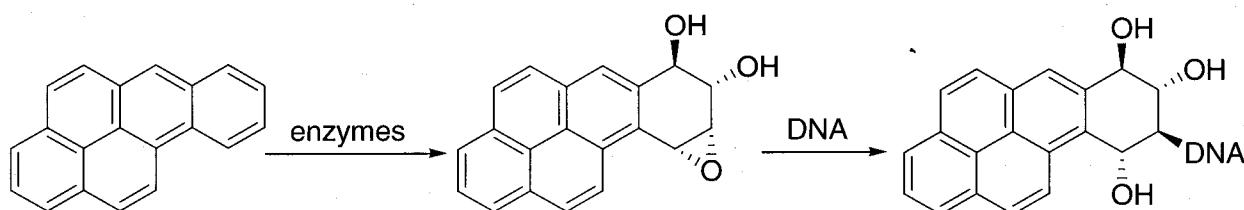
PAHs are monitored worldwide in a wide range of environmental matrices, including atmosphere, drinking water, waste water, furnace emissions, soils and hazardous waste extracts. Because of the characteristics of PAHs, they tend to accumulate in sediments and other organic phases, and the sedimentary concentrations of total PAHs vary depending on the locations in a range from a few  $\text{ng g}^{-1}$  to several  $\mu\text{g g}^{-1}$  (Richardson and Sajwan, 2001). PAHs enter air mostly on their releases from volcanoes, forest fires, burning coal, and automobile exhaust attaching tightly to dust particles. PAHs enter water directly from air with dust and precipitation, or on particles washed from the soils by runoff. The solubility of PAHs in water is inversely proportional to the number of rings it contains. As a result, large molecule (more than 3-ring) PAHs are almost exclusively bound to particulate matter, but for smaller molecular weight (2- or 3- ring) PAHs can be found dissolved in water. Furthermore, PAHs tends to have low vapor pressures, except that some PAHs particles can readily evaporate into the atmosphere from soils or surface waters. The large molecular PAHs are almost adsorbed onto the particular matters in atmosphere, but for the smaller molecular weight PAHs can be found free in the atmosphere and bound to particulates (ToxProbe Inc., 2002).

### 3.1.4 Health effects

By breathing air containing PAHs in the workplace of coking, coal-tar, asphalt production plants, municipal trash incineration facilities, wood smoke and vehicle exhausts, humans may be exposed to PAHs. Today, the effects of breathing high concentrations of PAHs have not been studied. Animal studies have shown that PAHs can cause harmful effects on the skin, body fluids

and ability to fight disease after their both short- and long-term exposure. However, these effects have not been observed in people. PAHs may be attached to dust or ash causing lung irritation, and skin contact with PAHs may cause redness, blistering and peeling (Information on Toxic Chemicals, 2000). These molecules may be highly carcinogenic and metabolically activated by organisms, directing their reactivity towards the nucleophilic groups of cellular macromolecules. Metabolic activation of PAHs to DNA adducts is considered as a crucial event in chemical carcinogenesis, involving covalent binding between the chemical carcinogen (PAHs) and DNA (Wilson and Jones, 1993). BAP is the most intensively studied PAHs in experimental animals and best known to be considered as one of the most toxic PAHs having carcinogenicity (Scheme 3.1) to humans and animals (Miller and Miller, 1981). Currently, few reference on human exposure to any single pure PAHs are known. There is just one report available on accidental exposure to pure naphthalene, and some data are from defined short-term studies of volunteers. All other reports are of exposure to the mixtures of PAHs, which also contained other potentially carcinogenic chemicals, in occupational and environmental situations (NEPM, 2003).

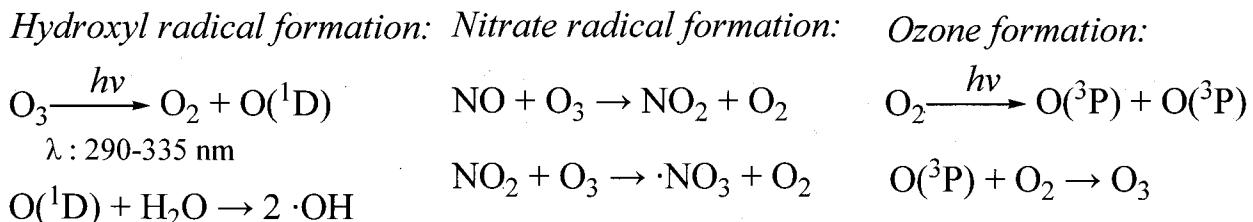
**Scheme 3.1 Induced carcinogenesis by formation of DNA adduct**



### 3.1.5 Directive products of PAHs

A number of researches have demonstrated that PAHs are susceptible to photo-chemical oxidation under simulated atmospheric conditions. The chemistry of the atmosphere activated by ultraviolet wave in the sun light results in formation of oxidant ozone ( $O_3$ ) and in formation of many radical species such as hydroxyl ( $\cdot OH$ ) and nitrate radical ( $\cdot NO_3$ ) etc. (Scheme 3.2) (Graedel and Crutzen, 1993) all of which will react with organic compounds, including PAHs (Atkinson, 1994).

**Scheme 3.2 Photochemical formations of reactive radicals and ozone**

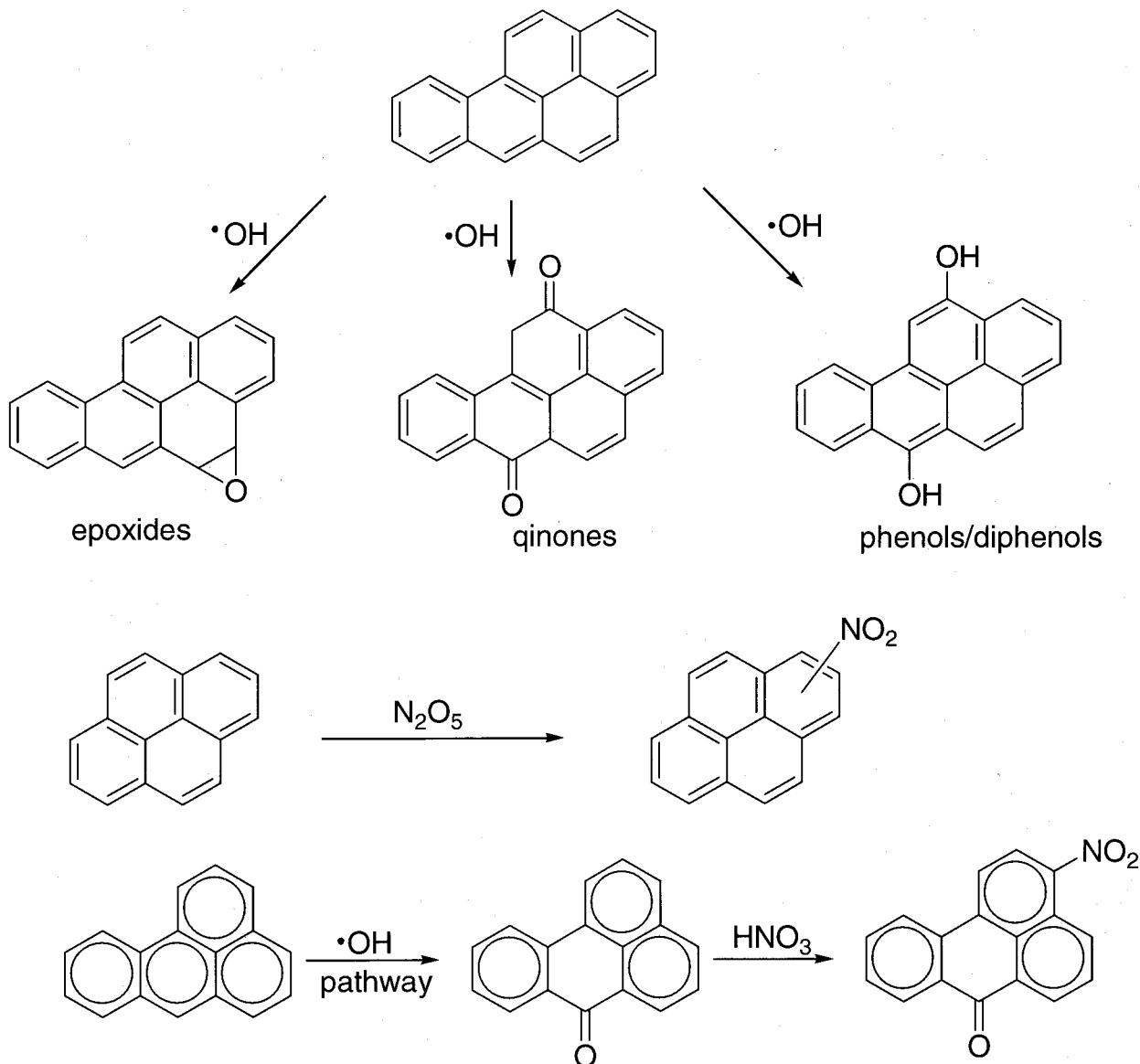


$O(^1D)$ : high energy oxygen atom;  $O(^3P)$ : low energy oxygen atom.

Among the products described herein are the highly mutagenic nitro-PAHs and nitro-PAHs ketones (Scheme 3.3) (Atkinson and Arey, 1994; Arey, 1998). Although the PAHs present in atmosphere have a general lifetime of less than one day which limits their range of impact, the reaction with  $HO\cdot$  will result in products which may have an adverse effect on human health and ecological consequences.

Normally, the directive products of PAHs have larger polarity than the corresponding precursors because of introduction of one or more polar functional groups. Therefore, they are easy to condense and become particle-associated.

**Scheme 3.3 PAHs derived products**



### 3.2. Technologies for analysis of PAHs in pollutants

The hydrophobicity of PAHs make them difficult to analyze in aqueous phase, especially in wastewater samples. Generally, the chemical analysis of PAHs can be described with the following steps:

- Sample preparation (Pretreatment of a sample and preparation of the test portion);
- Extraction from the collected sample;
- Clean up to remove interfering compounds;
- Identification and quantification by instrumental analysis.

#### 3.2.1 Sample pretreatment

Pretreatment is a very important process, which makes the samples homogeneous and even, and in the meantime reducing water to less than 25%. Without sample pretreatment, the results of concentrations obtained in the experiments are irreproducible because the accumulation of PAHs in soils or in sludges through irregular absorption on particles makes the sedimentary concentration of PAHs totally dependent on the sampling location. This procedure depends on the sample to be treated and is necessary for soils, sludges and mixed waster samples prior to the extraction process. Typically, vigorously mechanical stirring and/or grinding can make the sample homogeneous and even. Water can be removed by freeze drying method, or other simple treatments such as centrifugation and filtration etc. (Harmsen *et al.*, 2003).

### 3.2.2 Extract PAHs from the pollutants

Due to that PAHs are always adsorbed on particles in soils or sediments, the extraction efficiency of PAHs from pollutants might be influenced by several factors, such as pollutants moisture, the polar properties of solvents used, the concentration of PAHs in pollutants and the texture of pollutants etc. (Letellier *et al.*, 1999). Hence, to achieve high extraction rate, the choice of a proper extraction technique and organic solvent is important for different samples.

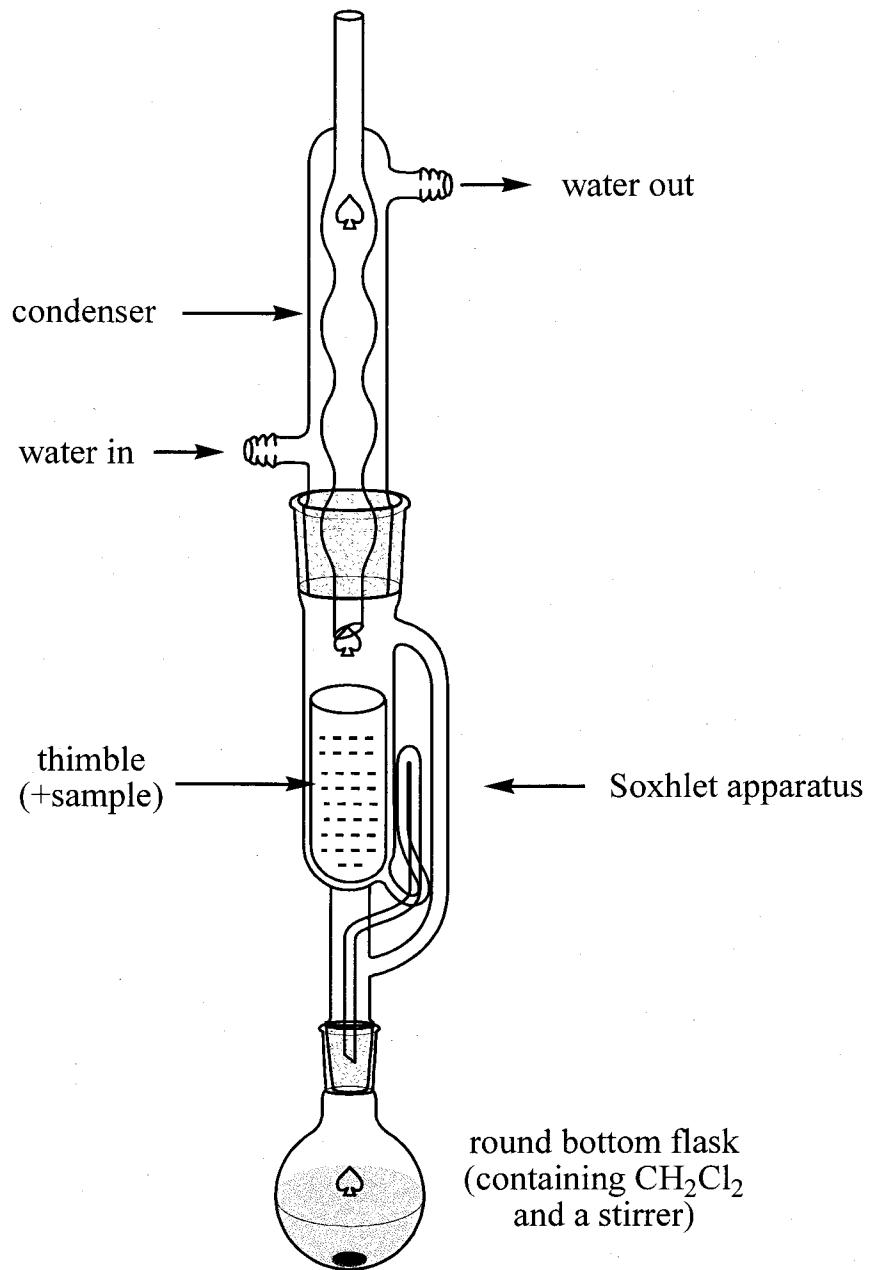
A brief summary of different extraction methods were made according to current literatures as below.

#### 3.2.2.1 *Soxhlet extraction*

PAHs are extracted from various matrices by Soxhlet extraction<sup>2</sup>, a traditional method showed in Methods 8310 (USEPA, 1982). This method is very effective and can achieve high extraction rate with almost quantitative extraction of PAHs. Amongst the currently different extraction methods reported in the literatures, Soxhlet extraction with dichloromethane as solvent is always a better choice for PAHs extraction.

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<sup>2</sup> Soxhelt extractor is a type of laboratory glassware invented in 1879 by Franz von Soxhelt and it was originally designed for the extraction of lipid from a solid test material, but can be used whenever it is difficult to extract any compound from a solid.



**Figure 3.1** Procedure for Soxhlet extraction

Typically, a dry sample is placed in a thimble (Figure 3.1). The extractor is attached to a flask containing organic solvent and a condenser. The solvent is heated to boiling, then, the hot solvent vapor rise through the thimble and into the condenser, where the solvent vapor condenses into liquid and drips down onto the sample. The thimble containing sample slowly fills with warm solvent until almost full, whereupon, it is emptied by siphon action back down to the flask. This cycle should be allowed to repeat as many times as necessary. During each cycle, a portion of the organic compounds dissolves in the organic solvent and the organic compounds can fall down with the solvent into flask. At the end of the extraction, the excess solvent can be removed using a rotary evaporator.

### **3.2.2.2 *Ultrasonic extraction***

An inexpensive and fast method for PAHs extraction was developed by the USEPA Methods 3550A (revision) (USEPA, 1990b) with organic solvent in an ultrasonic bath for 30 min. This method can be divided into two sections based on the expected concentration of organic compounds in sample. The lower concentration method (for each organic compound expected at less than  $20 \text{ mg kg}^{-1}$  DM) uses a larger quantity sample and the sample is extracted with solvent three times using ultrasonic extraction. The higher concentration method (for individual organic compound expected at more than  $20 \text{ mg kg}^{-1}$  DM) uses a small quantity of sample and one extraction. The extract is separated from the sample by vacuum filtration or centrifugation.

However, the result shows that this method is only suitable for the samples with higher concentrations of organic pollutants. Because, this method involves ultrasonic agitation and polar solvents, hence, it can not be applied in the extraction of materials like plastic and bitumen.

### 3.2.2.3 **Microwave-assisted solvent extraction (MAE)**

Microwave-assisted solvent extraction (MAE) was first reported by Ganzler (Ganzler *et al.*, 1986). It is a technique using an organic solvent as the extractant heated with microwave energy. As compared to classical heating, microwave energy heats all the sample simultaneously but without a vessel. This method has been successfully tested for rapid extraction of PAHs from solid samples (Camel, 2000). The extractors are now commercial available and all function at frequencies of 2.45 GHz to avoid serious hazards such as the flaming of organic solvents.

There are two technologies used to extract PAHs from solid environmental matrices: PMAE (pressurized microwave-assisted solvent extraction: with closed vessels: under controlled pressure and temperature) or FMAE (focused microwave-assisted solvent extraction: with open vessels: under atmospheric pressure). These two techniques were compared for the extraction of PAHs from soils. The pressurized MAE of PAHs from spiked soils with hexane-acetone (1:1, v  $v^{-1}$ ) at 115 °C for 5 min were successfully tested (Chee *et al.*, 1996). To compare with the technique of PMAE, FMAE has also been tested for extracting PAHs from environmental matrices with acetone, hexane-acetone (1:1, v  $v^{-1}$ ) or dichloromethane among which acetone is the most efficient (Lopez-Avila and Benedict, 1996).

### 3.2.2.4 Supercritical fluid<sup>3</sup> extraction (SFE)

Supercritical fluid extraction (SFE) has been applied only recently to sample preparation on an analytical scale. It was studied by Hawthorne, and used as a rapid alternative to conventional solvent extraction from polyurethane foam absorbents (Hawthorne *et al.*, 1989). This technique is quite similar to Soxhlet extraction except using a supercritical fluid as solvent. This extraction method could avoid use of organic solvents because the fluid can provide a broad range of useful properties. In addition, the rate of extraction can be enhanced with less degradation of solutes occurs due to the lipids have the high diffusion coefficients in supercritical fluids (Tavlarides *et al.*, 2000).

### 3.2.2.5 Solid phase extraction (SPE)

Solid phase extraction (SPE) is an extraction method that uses a solid phase and a liquid phase to isolate one, or one type of analyte from a solution. The general procedure is to load a solution onto the SPE phase, wash away undesired components, and then wash off the desired analytes with another solvent into a collection tube. A traditional SPE method utilizes bonded silica or resin solid sorbents packed into disposable plastic or glass cartridges or imbedded into Teflon or glass fiber disks. In SPE procedures, a solid sorbent material, typically an alkyl bonded silica, is packed into a cartridge or imbedded in a disk and performs essentially the same function as the organic solvent in liquid-liquid extraction. For example, reverse-phase SPE is employed to

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<sup>3</sup> Supercritical fluid is a substance above its critical temperature and critical pressure. The critical point represents the highest temperature and pressure at which the substance can exist as a vapor and liquid in equilibrium. It is increasingly replacing the organic solvents that are used in industrial purification and recrystallization operations because of regulatory and environmental pressures on hydrocarbon and ozone-depleting emissions.

extract non-polar compounds, pesticides for instance, from polar samples such as water generally utilizing a solid sorbent containing non-polar functional groups such as octadecyl ( $C_{18}$ ) or octyl ( $C_8$ ) bonded silica gels. Aqueous samples are pumped or pulled through a cartridge or disk while organic compounds in samples will interact with non-polar functional groups on the sorbent and thus are effectively extracted from the target sample. Organic compounds in the original aqueous sample, therefore, can be eluted from the cartridge or disk with a small volume of organic solvent (USEPA, 1990c). However, SPE extraction method is usually used for the determination of organic analytes in aqueous samples and it has been developed and approved in monitoring compliance purposes for the Safe Drinking Water Act (SDWA).

### **3.2.2.6 Solid phase micro-extraction (SPME)**

Solid-phase micro-extraction (SPME) utilizes a small segment of fused silica fiber coated with an appropriate material and mounted on syringe-like device for extraction of analyses from various matrices and introduction to a chromatographic system without use of solvents in the process. Analysis of extraction and pre-concentration are combined in a single step. It can eliminate a significant number of undesirable steps associated with conventional sample preparation techniques. It was first described by the group of Pawliszyn in trace analysis with more recent initial work in SPME-HPLC demonstrating the fast and easy determination of PAHs (Chen and Pawliszyn, 1995). The extraction time is typically 15 h with stirring at 1 000 rotations per minute (rpm). However, this method was just developed for the analysis of water samples. The Energy & Environmental Research Center (EERC), University of North Dakota is now combining a subcritical water extraction with SPME to allow a very rapid and organic solvent-free method for determining organic pollutants on soils or sludges.

**3.2.2.7    *Conclusions***

Table 3.3 gives the comparison of different extraction methods. The suitable technique could be chosen depending on the form of samples and recoveries or duration of the extraction.

**Table 3.3 Comparison of different extraction methods**

Method	Sample	Time	Vol. <sub>solvent</sub> (mL)	Advantages	Disadvantages
Soxhlet	Soil Sludge	12-24 h	150-500	<ul style="list-style-type: none"> <li>• Easy to handle</li> <li>• Filtration free</li> <li>• Effective</li> <li>• Higher matrix capacity</li> <li>• Inexpensive equipment</li> </ul>	<ul style="list-style-type: none"> <li>• Long time for extraction</li> <li>• Large volume solvent</li> <li>• Heating to boiling</li> <li>• Non-automatable</li> </ul>
Sonication	Soil Sludge Clay	10-60 min	50-200	<ul style="list-style-type: none"> <li>• Easy to handle</li> <li>• Inexpensive equipment</li> </ul>	<ul style="list-style-type: none"> <li>• Filtration</li> <li>• Large volume solvent</li> <li>• Non-automatable</li> </ul>
PMAE	Soil Sludge	10-30 min	10-40	<ul style="list-style-type: none"> <li>• Fast</li> <li>• Multiple cells</li> <li>• Moderate volume solvent</li> </ul>	<ul style="list-style-type: none"> <li>• Filtration</li> <li>• Polar solvent</li> <li>• Careful handling</li> <li>• Change of rupture membrane for each extraction</li> </ul>
FMAE	Soil Sludge	10-30 min	30-70	<ul style="list-style-type: none"> <li>• Filtration free</li> <li>• Moderate solvent volume</li> <li>• Possibility of using multiple system</li> <li>• Safer than PMAE</li> </ul>	<ul style="list-style-type: none"> <li>• The solvent must absorb microwave (except water)</li> <li>• Careful handling to the use of microwave</li> </ul>
SFE	Sediments	10-60 min	Solid trap: 2-5 Liquid trap: 30-60	<ul style="list-style-type: none"> <li>• Filtration free</li> <li>• Smalll volume solvent</li> <li>• Multiple cells</li> <li>• Possible high selectivity</li> <li>• Possible coupling to instrument</li> </ul>	<ul style="list-style-type: none"> <li>• Expensive equipment</li> <li>• Problem for matrices with high water content</li> <li>• Elevated pressure</li> <li>• Compression of solvent</li> <li>• Lower temperature</li> </ul>
SPE	Solution	10-30 min	30-60	<ul style="list-style-type: none"> <li>• Fast and easy to handle</li> <li>• Less amount of solvent</li> <li>• Higher concentration factor</li> <li>• High selective extraction</li> <li>• Easy for automatisation</li> </ul>	<ul style="list-style-type: none"> <li>• Low recovery</li> <li>• High variability</li> <li>• Poor reproducibility</li> <li>• Change cartridge for each extraction</li> </ul>
SPME	Water	15 h	0	<ul style="list-style-type: none"> <li>• Solvent free</li> <li>• Heating free</li> <li>• Efficient and versatile</li> <li>• Possible coupling to instrument</li> <li>• Easy automatisation</li> </ul>	<ul style="list-style-type: none"> <li>• Careful handling</li> <li>• Currently, only for water sample</li> <li>• Fiber coatings required (fibers are very vulnerable to mechanical damage and relatively expensive)</li> </ul>

### 3.2.3 Clean-up samples

The samples obtained by extraction are required to be purified in order to remove unwanted contaminants, which could interfere with the subsequent analytical results. If there is no or negligible interfering substances in the extract, clean-up is not necessary. In most cases, depending on the interfering substances to be removed, different methods can be used. Before application of the clean-up to real samples, it must be ensured that the recoveries after using this method for clean-up are at least 80% for all of the target PAHs compounds. Washing with water or clean-up by aluminum oxide or silica gel can remove some polar compounds. However, it is difficult to adjust water content by using aluminum oxide (Harmsen *et al.*, 2003). Silica gel has been widely used as column sorbents. The samples are usually purified by flash column chromatography to eliminate interferences from both polar and non-polar compounds. Activated silica gel clean-up and deactivated silica gel clean-up methods were investigated by Mangas, and the results showed that clean-up using activated silica gel gave the better result (Mangas *et al.*, 1998).

### 3.2.4 Instrumental analysis of PAHs

After clean-up, the samples are ready for instrumental analysis. Currently, the literature shows that the analysis of PAHs by different instruments is listed as follows.

#### 3.2.4.1 Gas Chromatography with Mass Spectrometry (GC-MS)

Gas chromatography with mass spectrometry (GC-MS) is a most common method for identification and quantification of PAHs (USEPA, 1998; Khim *et al.*, 1999). The compounds that need to be analyzed are introduced into the GC-MS by an injection of the sample extract into

a GC with a narrow-bore fused-silica capillary column. The temperature-programmed GC column is responsible for the separation of each compound, which is then identified by MS connected to the GC.

With GC-MS, the peak identification is not only achieved through retention time of each compound, but also confirmed by their mass spectrum characteristic peaks with comparison to the data base. So, once separated by chromatography, the mass spectrum of each compound is then compared with an authentic mass spectra in library data base and a match was obtained. Deuterated internal standards have to be used in the quantification with a calibration curve having at least 4 points and a blank. If fewes calibration points are used, the calibration has to be limited to the linear part of the curve.

### **3.2.4.2 Gas Chromatography with Flame Ionization Detection (GC-FID)**

Gas chromatography with flame ionization detection (GC-FID) (USEPA, 1986; Simpson *et al.*, 1995; USEPA, 2003) is another commonly used method for identifying and quantifying PAH. Similar to GC-MS, after separation of each compound by GC column, the compounds are then detected with flame ionization detector (FID). Then PAHs coming out of the GC column, effluent (carrier gas and any organic compounds) is ignited in a flame made from hydrogen and air. The compounds, as they are burning, produce ions which conduct electricity. Changes in current within the flame are measured and sent to the computer to be shown in the form of peaks on the chromatogram. FID is a good and general detector for organic compounds, and is able to detect at the nanogram level.

#### **3.2.4.3 High Performance Liquid Chromatography (HPLC)**

Another commonly used technique for determination of PAH molecules is high performance liquid chromatography (HPLC) with fluorescence detector (USEPA, 1982; Beltran *et al.*, 1996; Kayali-Sayadi *et al.*, 1999) or with ultraviolet (UV)-visible detector (USEPA, 1990a, c). After the sample extraction or purification, a portion of the extract is injected onto a reversed-phase HPLC column. The PAHs eluted with a gradient water/acetonitrile are detected using UV absorbance or fluorescence detector.

#### **3.2.4.4 Conclusions**

With GC-MS, the peak identification is achieved through both retention time and mass spectrum of each peak which corresponds to a single compound if not overlaped. GC-MS with use of selected ion monitoring (SIM), the limits of detection can be reduced to 1/5-1/10, approaching that of HPLC. However, this is a specialized technique which few laboratory can offer. As compared to GC-MS, GC-FID is a more sensitive technique for the determination of PAHs but subject to background interferences from other carbonaceous sources. HPLC is generally the most sensitive but expensive method and subject to interferences. Therefore, it is not as widely used as compared to GC-MS and GC-FID (Dionex, 1994).

### **3.3. Removals of PAHs from polluted matrix**

During the last decade, the number of references on PAHs removal from contaminated substances are in thousands though the PAHs are considered more difficult to be treated because of their stable physical-chemical characteristics and frequent occurrence.

The traditional waste treatment processes such as biological treatments (aerobic and anaerobic), chemical treatments using chlorine, ozone, hydrogen peroxide and high-energy ultraviolet light in removal of PAHs from soils or sediments have been investigated. In addition, the oxidation of PAHs using heterogeneous photocatalysis on metal oxide semiconductor particles and solubilization of PAHs by surfactants have been shown to be an effective means for removing PAHs from soils.

### **3.3.1 Biological treatments**

The hydrophobicity of PAHs limits their solubility in aqueous phase resulting in a low bioavailability to bacterial biodegradation, particularly for the high molecular weight compounds (Aitken *et al.*, 1997). The natural biodegradation rates of PAHs are very slow in soils or in sewage sludges (Genney *et al.*, 2004) as compared to those in the atmosphere with half lives of 5-30 days (Veety Mc and Hites, 1988).

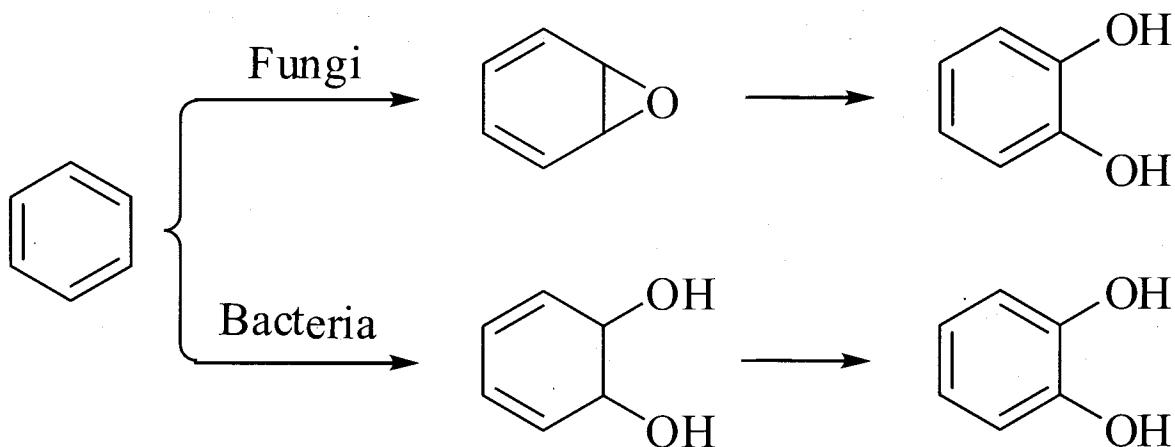
Although PAHs are not easily treated by biological means, they are known to be biodegradable. The bioremediation is often considered as an option in treating PAH-contaminated soils or sewage sludges. A number of different techniques are available for the biodegradation of PAHs by bacteria in contaminated soils and sediments (Sims and Overcash, 1983; Wang *et al.*, 1990; Cerniglia, 1992; Nales *et al.*, 1998; Kanaly and Bartha, 1999; Juhasez and Naidu, 2000; Haeseler *et al.*, 2001). However, the removals of PAHs by microorganisms are incomplete, particularly for the high molecular weight compounds because of their large and complex molecular structure. All but one of these bacteria were able to degrade a wide range of 2-, 3- and 4-ring PAHs and, in some cases, the 5-ring PAHs like compound benzo(a)pyrene (Aitken *et al.*, 1997).

The most popular mechanism for PAHs biodegradation is metabolic dissimilation, which could cause the complete degradation to CO<sub>2</sub> and H<sub>2</sub>O. The microorganism could obtain energy from oxidation of organic compounds like PAH. The biodegradation could occur with two necessary factors: a sufficient quantity of microorganisms and bioavailable substrate.

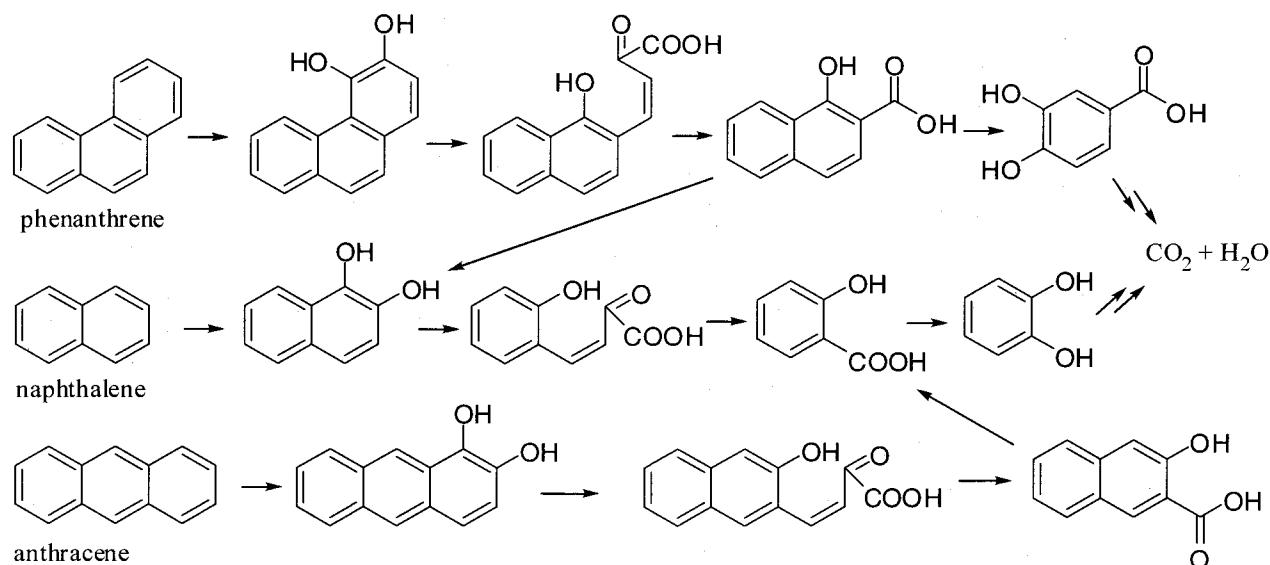
### 3.3.1.1 *Aerobic degradation*

Aerobic degradation is a biological conversion process in which micro-organisms breathe in oxygen to digest/decompose organic compounds. Some researches have been conducted on the aerobic degradation of PAHs in soils focusing on species of mesophilic (room temperature) and neutrophilic (neutral pH: 6 ~ 7) microorganisms. For example, several species of algae, bacteria and fungi are known to have the enzymatic capability to oxidize PAHs (Cerniglia, 1992). To date, a significant number of mesophilic, aerobic and acidophilic bacteria, which can utilize PAHs as energy sources, have also been found in environments (Stapleton *et al.*, 1998). There are two main hydroxylation mechanisms of eukaryotic and prokaryotic microorganisms. As an example given in Scheme 3.4, the first step is the bacterial or fungal benzene catabolism. In the fungi attack of the aromatic ring, epoxide is usually formed and then forming a dihydroxybenzene by epoxy ring opening and further oxidation, however, in the bacterial attack of the aromatic ring, a dihydrodiol is formed and then undergo oxidation to form the same product at the end (Volkering and Breure, 2003).

**Scheme 3.4 Initial steps in the microbial catabolism of homocyclic aromatic compounds**



**Scheme 3.5 Aerobic bacterial biodegradation of phenanthrene, naphthalene and anthracene**



An overview of the biodegradation of some low molecular weight PAHs compounds such as naphthalene, phenanthrene and anthracene are shown in Scheme 3.5 (Komatsu *et al.*, 1993). For high molecular PAHs, the use as sole source of carbon and energy has been reported for FLR, BAP, pyrene, chrysene, coronene, etc (Weissenfels *et al.*, 1991; Boldrin *et al.*, 1993; Juhasez *et al.*, 1997).

### 3.3.1.2 *Anaerobic degradation*

Anaerobic degradation is a biological conversion process performed by microorganisms in the absence of oxygen to digest/decompose biodegradable organic compounds. The biodegradation of PAHs in anaerobic methanogenic systems has only been shown to be able to occur in recent years (Christensen *et al.*, 2004). In contrast to the aerobic degradation, little is known about the mechanism of anaerobic degradation (Chang *et al.*, 2002) because it is difficult to obtain direct proofs from the anaerobic PAHs degradation in sediments. According to the theory of Volkering, under anaerobic conditions, chemorganotrophic bacteria could obtain energy from the transport of electrons from PAHs to inorganic electron acceptors other than oxygen, such as nitrate, Fe(III), Mn(IV), sulfate and carbonate (Volkering and Breure, 2003). A few researchers have found that a number of two or three rings PAHs can be degraded anaerobically (Bregnard *et al.*, 1996; Coates *et al.*, 1996; Langenhoff *et al.*, 1996; Chang *et al.*, 2001). In 2002, Chang investigated the degradation of pyrene (4-ring PAH) under anaerobic conditions (Chang *et al.*, 2002). More results were obtained by Trably and his coworker in 2003. They studied the anaerobic degradation of 13 PAHs with a higher molecular mass (3-6 rings) in continuous stirred tank reactors fed with sewage sludges (Trably *et al.*, 2003). Temperature and pH values influence the removal efficiency of anaerobic degradation of PAHs. The optimal incubation

condition was noted as pH 8.0 and temperature at 30 °C by Chang *et al.* (2002). A subsequent study by Trably and Christensen showed that the biological removal of PAHs was significantly enhanced by increasing the temperature from 35 °C to 55 °C. They also found that the variation of pH value (5.0 ~ 9.0) has a minor influence on the degradation rates. The optimal pH was found at a weak basic condition (pH 8.0) (Trably *et al.*, 2003; Christensen *et al.*, 2004).

In conclusion, the capability to biodegrade PAHs is influenced by the kind of pollutants (e.g., clay soils can adsorb contaminants making them unavailable to the microorganisms); oxygen supply; moisture content (the suitable moisture level is in range of 20 to 80%); nutrient supply required for cell growth (N, P, K, S, Mg, Ca, Mn, Fe, Zn, and Cu etc.); pH value, temperature and the species of microorganisms (having been discussed briefly above); the concentration of the contaminants (high concentration may be toxic to the microorganisms) and the presence of toxic substances to the microorganisms (e.g.: mercury or inhibitors of the metabolism).

### 3.3.2 Chemical treatment

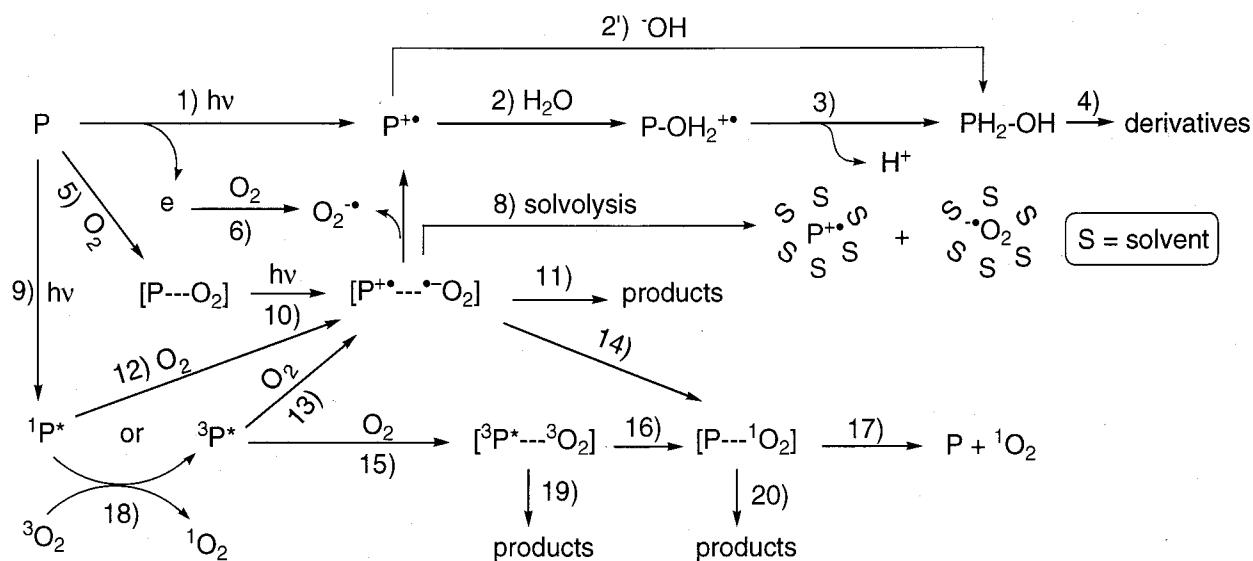
#### 3.3.2.1 Photodegradation of PAHs

The photodegradation of PAHs has been extensively studied over the last several years (Zepp and Schlotzhauer, 1979; Mill *et al.*, 1981; Fukuda *et al.*, 1988; Sigman *et al.*, 1991, 1996, 1998). PAHs can be degraded by direct or sensitized photochemical reactions. Most PAHs can absorb surface solar radiation to allow the possibility of direct photodegradation. Some researchers have proved that a number of PAHs could be degraded when irradiated with 313 and 366 nm light in pure water (Zepp and Schlotzhauer, 1979; Mill *et al.*, 1981; Fukuda *et al.*, 1988; Fasancht and Blough, 2002). However, the mechanism of the direct photodegradation remains unclear.

There are three kinds of primary reactions that have been proposed for photoionization which is the initial step in photodegradation (Scheme 3.6) (Mill *et al.*, 1981; Vialaton *et al.*, 1999; Miller and Olejnik, 2001). The photoionization involves ejection of an electron from PAHs (P) by absorption of a photon (Scheme 3.6, eq 1). In aerated solutions, this electron will be rapidly captured by an O<sub>2</sub> to form a superoxide radical (O<sub>2</sub><sup>•</sup>) (Scheme 3.6, eq 6) (Blough and Zepp, 1995). The resulted PAHs cation radical (P<sup>+</sup>) can react with water (or hydroxide ion, Scheme 3.6, eq 2' & 3') to form a second radical intermediate (Scheme 3.6, eq 2), which, after evolution of a proton (Scheme 3.6, eq 3), can form a stable product (PH<sub>2</sub>-OH). Further oxidation of these products will become easier to give many other derivatives (Scheme 3.6, eq 4) (Steenken *et al.*, 1990; Sigman *et al.*, 1998; Vialaton *et al.*, 1999).

One electron transfer from PAHs to O<sub>2</sub> under light irradiation in a ground state has been proposed for pyrene (Scheme 3.6, eqs 5 and 10) (Sigman *et al.*, 1998). The resultant charge-transferred complex [P<sup>+</sup>--O<sub>2</sub><sup>•</sup>] may undergo solvolysis forming P<sup>+</sup> and O<sub>2</sub><sup>•</sup> (Scheme 3.6, eq 8), or react within the collision complex to form products (Scheme 3.6, eq 11). The excited singlet PAHs (<sup>1</sup>P\*) and triplet <sup>3</sup>P\* may be diffusional quenched by O<sub>2</sub> (Scheme 3.6, eqs 12 and 13) to produce the complex [P<sup>+</sup>--O<sub>2</sub><sup>•</sup>] identical to that formed via eqs 5 and 10.

Finally, the photodegradation could be initiated by the direct reaction of O<sub>2</sub> with <sup>3</sup>P\* to form a [<sup>3</sup>P\*--<sup>3</sup>O<sub>2</sub>] complex (Scheme 3.6, eq 15) which then form a [P--<sup>1</sup>O<sub>2</sub>] complex by energy transfer from <sup>3</sup>P\* to <sup>3</sup>O<sub>2</sub> within the collision complex (Scheme 3.6, eq 16). Due to spin restriction, the reaction of <sup>3</sup>O<sub>2</sub> with <sup>1</sup>P\* to form products directly should not occur (Turro, 1991) but as indicated the above could proceed through a [P<sup>+</sup>--O<sub>2</sub><sup>•</sup>] complex (Scheme 3.6, eq 12).

**Scheme 3.6 Proposed pathways of PAHs photodegradation**

Semiconductors<sup>4</sup> (e.g. TiO<sub>2</sub>, ZnO, Fe<sub>2</sub>O<sub>3</sub>, CdS, and ZnS) can act as sensitizers for light-induced redox processes because of their electronic structure, which is characterized by a filled valence band and an empty conduction band (Hoffmann *et al.*, 1995). Therefore, the semiconductor can catalyze the photodegradation by transformation of one electron in the catalysis cycle under light irradiation. To photocatalyze oxidation of organic pollutants by oxygen, the semiconductor sensitizer must meet the following requirements (Hoffmann *et al.*, 1995):

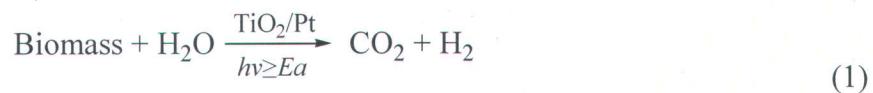
1. Highly photoactive;
2. Be able to utilize visible and/or near UV light;

<sup>4</sup> A semiconductor is a material whose valence band and conduction band are separated by an energy gap or bandgap. When a semiconductor molecule absorbs photons with energy equal to or greater than its bandgap, electrons in the valence band can be excited and jump up into the conduction band and thus charge carriers are generated.

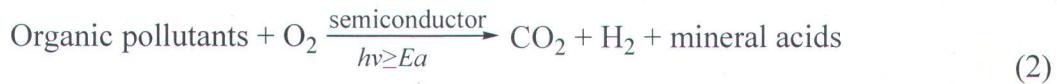
3. Biologically and chemically inert;
4. Photo stable (e.g.: not liable to photo anodic corrosion);
5. Not expensive.

By common consent, the semiconductor titanium dioxide ( $\text{TiO}_2$ ) satisfies the criteria from 1 to 5 and is proved to be one of the best semiconductors for sensitization.  $\text{TiO}_2$  has been studied by a number of researchers as a frequently used photocatalyst (Mills and LeHunte, 1997) because  $\text{TiO}_2$  is non-toxic and chemically stable, and possesses relatively high photocatalytic activity. Studies in laboratory have demonstrated that biomass and organic compounds such as alcohols, carboxylic acid, phenolic derivatives, chlorinated aromatics and PAHs can be readily mineralized by  $\text{TiO}_2$  into harmless carbon dioxide, water, and simple mineral acids, using molecular oxygen as primary oxidant.

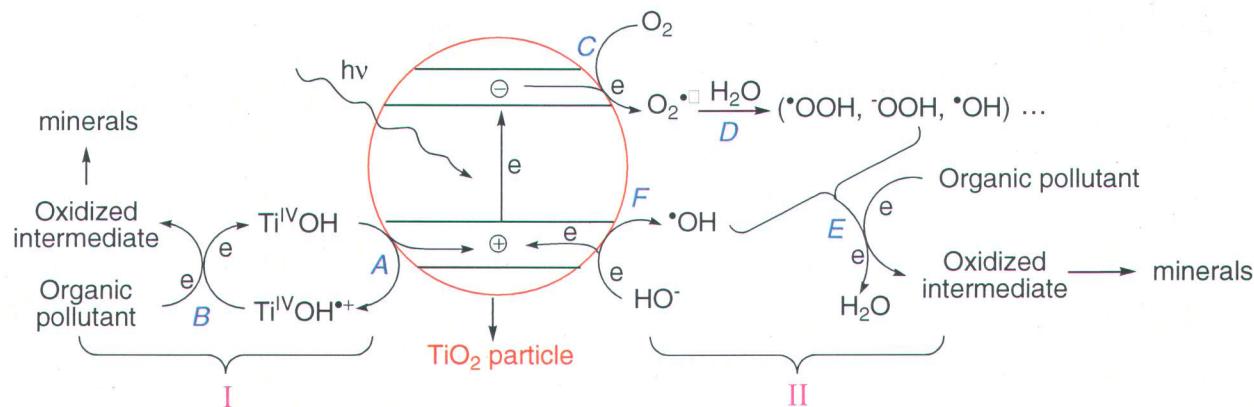
The first example (reaction 1) is the oxidation of biomass (including grass, wood, algae, seaweed, cockroaches, urine and glucose) with the concomitant reduction of water to hydrogen (Sakata and Kawai, 1981):



The second example is the semiconductor-sensitized photo mineralization of organic substrates by molecular oxygen. A variety of organic molecules can be photocatalytically oxidized and eventually mineralized according to the following general reactions. In the last decade, a number of references on toxic and hazardous compounds removal by heterogeneous photocatalytic degradations from water and air (Herrmann, 1999).



The mechanism of photocatalytic oxidation mediated by  $\text{TiO}_2$  is still under discussion, but the most widely accepted mechanism is illustrated in Figure 3.2 (I).



\*Where  $\text{Ti}^{IV}\text{OH}$  represent the primary hydrated surface functionality of  $\text{TiO}_2$

**Figure 3.2 Mechanism of  $\text{TiO}_2$  catalyzed photodegradation process**

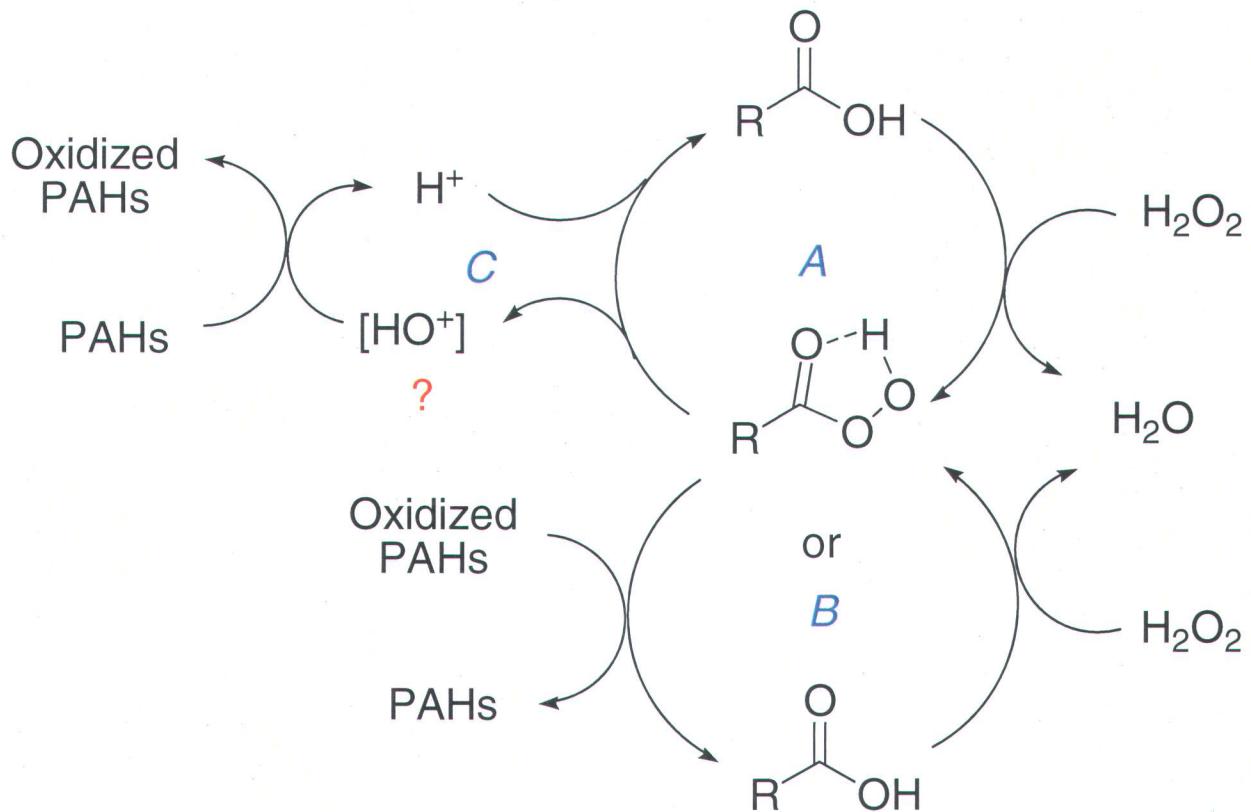
The electrons located in the valence band at ambient temperatures, when illuminated with UV or the sun rays ( $\lambda < 380 \text{ nm}$ ), can be excited from the valence band to the empty conduction band to form current. Prior to recombination with the hole ( $\oplus$ ) by releasing energy, the excited electrons and holes are trapped at the surface of  $\text{TiO}_2$  by their encounter electron acceptors ( $\text{O}_2 \rightarrow \text{O}_2^\cdot$ , C in Figure 3.2) and donors ( $\text{Ti}^{IV}\text{OH} \rightarrow \text{Ti}^{IV}\text{OH}^\cdot$ , A in Figure 3.2) which involve in the redox half-reactions. In the next step, there are two possibilities for the organic molecules to be oxidized (Heller, 1995): I), The  $\text{Ti}^{IV}\text{OH}^\cdot$  formed from the surface-trapping by the valence band hole will oxidize organic molecules by gain of one electron. The organic pollutants can be oxidized by

$\text{Ti}^{\text{IV}}\text{OH}^+$ . And, II), The superoxide radical ( $\text{O}_2^{\cdot-}$ ), formed from trapping the electron excited from the valence band, which may also participate in the degradation reactions of the organic molecules, or will further react with water to form very reactive chemical species such as  $\text{HOO}\cdot$ ,  $\text{HOO}^-$ ,  $\text{H}_2\text{O}_2$ ,  $\text{HO}\cdot$  etc which will oxidize organic pollutants in formation of water and carbon dioxide as final products and  $\text{HCl}$  if the organic molecule contains chlorine ( $C \rightarrow D \rightarrow E$ ). The hydroxyl radical ( $\text{HO}\cdot$ ) in II) can also be generated from transformation of one electron from a hydroxyl group ( $\text{HO}^-$ ) to the electron deficient hole ( $F$ ).

### 3.3.2.2 PAHs oxidation by chemical reagents

PAHs can be oxidized by ozone ( $\text{O}_3$ ) or  $\text{HO}\cdot$  radical, hence,  $\text{O}_3$  and hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) are always used as oxidants for chemical oxidations of PAHs (Yao and Masten, 1992). For instance, pyrene, naphthalene, chrysene and phenanthrene in soils can be oxidized by ozone at a dosage of  $500 \text{ mg kg}^{-1}$ . The studies showed that 81% pyrene was removed (Masten and Davies, 1997).

Since the 1940s, the pulping paper industry has used organic peroxy acids as oxidants to alter chemical structures (Johnson, 1980; Poppius *et al.*, 1989). Organic peroxy acids are formed by insitu mixing organic acids (e.g. acetic acid, propionic acid, etc.) and hydrogen peroxide. They are relatively selective oxidizing agents with activity that can be targeted towards electron dense rich structures found in aromatic rings, double and triple bonds without the fear of unwanted competing reactions such as with sugars.



**Figure 3.3 Proposed oxidation of PAHs by organic peroxy acid**

In the catalytic cycle,  $\text{H}_2\text{O}_2$  reacts with an organic acid in solution to form a peroxy organic acid (A, Figure 3.3). The peracid in turn may react directly with organic compounds (B) or may generate a hydroxyl cation  $[\text{HO}^+]$  which oxidizes the organic pollutants (C). However, the real mechanism is not well understood yet and still under investigation. A preliminary investigation by N'Guessan, in oxidation of benzo(a)pyrene with an organic peroxy acid may be a promising development of new advanced oxidation process as a fast acting, relatively low cost, remediation technique for PAHs contaminated sediments (N'Guessan *et al.*, 2004).

In addition to organic peroxy acids that can oxidize organic pollutants, Fenton's reagent<sup>5</sup> ( $\text{Fe}^{2+}/\text{H}_2\text{O}_2$  system) a combination of hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) and ferrous iron ( $\text{Fe}^{2+}$ ) is capable of oxidizing various organic compounds. This reagent, in aqueous medium, allows formation of hydroxyl radicals, which are very reactive and nonselective oxidants. Flotron and his coworkers showed that Fenton's reagent could oxidize PAHs in sewage sludges, but inefficient in removal of various PAHs because of the oxidation of PAHs is limited by the competition from the organic matter and their strong adsorption on particles. From their experimental results, the removal efficiency of three PAHs degradation was in the order of BAP > FLR > B(J)F (Flotron *et al.*, 2003a,b).

### 3.3.3 Enhanced solubilization of PAHs by surfactants

As known, it is difficult to remove PAHs from subsurface environment using traditional technologies for decontamination of sewage sludges (Li and Chen, 2002; Prak and Pritchard, 2002). Solubilization of PAHs by surfactant can be used for removing PAHs from contaminated substances.

Surfactant (surface active agent) molecules contain hydrophilic (water-loving) head and long hydrophobic (water-hating or oil-loving) tail. The surfactants are able to reduce the surface tension of water by adsorbing at the air-water interface, and can also reduce the interfacial tension between water and organic phase by adsorbing at the liquid-liquid interface. Hence, they are typically soluble in both organic and water phases. Surfactants can form micelles over a

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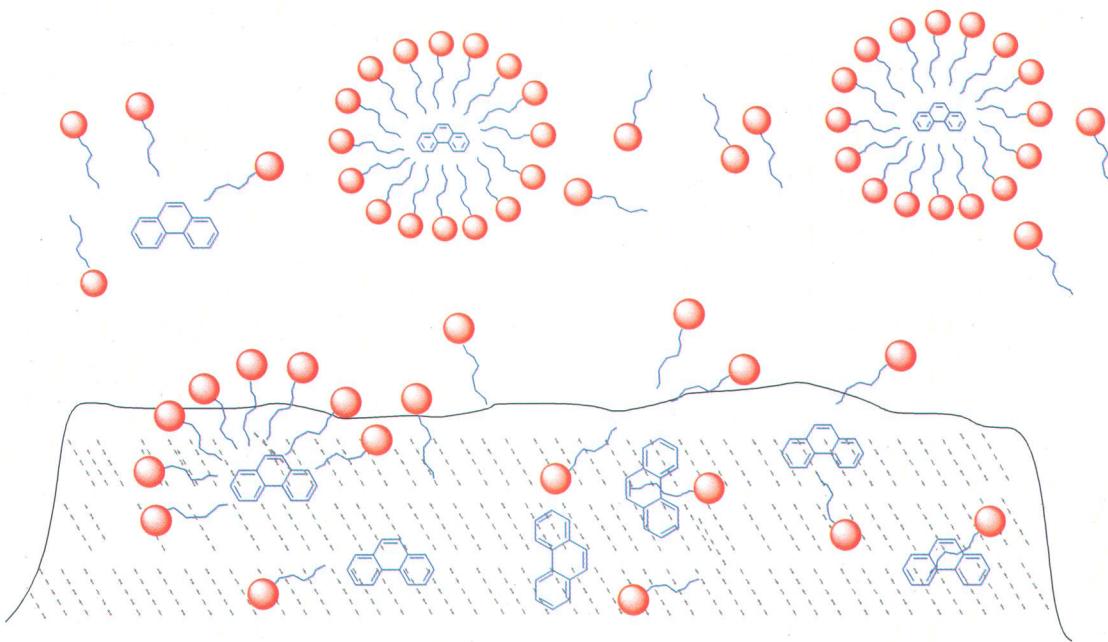
<sup>5</sup> Fenton's reagent: The reactivity of this system was first observed in 1894 by its inventor H.J.H. Fenton, but its utility was not recognized until the 1930's once the mechanisms were identified.

certain aqueous concentration which called the critical micelle concentration (CMC). The formed surfactant micelles can sequester PAHs which are sorbed to soils or sludges.

Surfactants can be classified into three types by the ionic characteristics of hydrophilic head group: anionic, cationic and nonionic. Many studies conducted showed that surfactants can enhance the solubility of PAHs in aqueous phase from soils (Bury and Miller, 1993; Volkering *et al.*, 1995; Doong *et al.*, 1996). When choosing a surfactant for remediation PAHs, a number of factors must be considered such as removal efficiency of recovery and reusage, effect of solubilization, surfactant sorption, biodegradation and effect on biota.

As might be expected, charged surfactants (anionic and cationic) appear to have a greater denaturing effect than nonionic surfactants. Especially, cationic surfactants also appear to be the most toxic to both freshwater and marine species of algae, invertebrates and fish and they have high sorption tendency on the negative charged surface of soils (Lewis, 1991). Anionic surfactants are usually chosen by researchers due to their lower degree of adsorption than cationic and nonionic surfactants. The nonionic surfactants are chosen because they are generally economical and their solubilization capacities are high (West and Harwell, 1992).

The mechanism of using surfactants to enhance the solubility of PAHs has been studied by Edwards (1994). Figure 3.4 shows a solid-aqueous phase system containing surfactant, PAHs (represented by acenaphthene) and a soil particles of moderate organic carbon content. As illustrated in this figure, the surfactants as dissolved monomers can reach the surface of the soil particles with formation of micelles around PAHs molecules exposed on the solid-water interface. The PAHs solubilized in surfactant micelles will be brought to the aqueous phase of the surfactants.



**Figure 3.4 Enhanced solubilization of PAHs by surfactants**

In recent years, various surfactants have been employed with attempts to increase the concentrations of PAHs in aqueous phase in order to enhance the biodegradation of PAHs from soil particles. Three types of surfactants were investigated in solubilizing and mobilizing PAHs from soils by Sun. The results showed that a nonionic surfactant (Brij 30) started transporting PAHs from soil to water phase at concentrations below its apparent CMC. If increasing its concentration, Brij 30 was able to transport more PAHs to the aqueous phase from soil. Therefore, Brij 30 presented a great potential in remediation of PAH-contaminated soils. In contrast, the tested anionic surfactant (SDS: sodium dodecylsulfate) and cationic surfactant (DTAB: dodecyltrimethylammonium bromide) did not show any solubilization effect until the concentrations reached their apparent CMCs (Sun and Puri, 1997). In equilibrium, the concentration of solubilized PAHs linearly depends on the surfactant concentration above the

### *3. Literature review*

CMC (Edwards *et al.*, 1991; Bury and Miller, 1993; Tiehm, 1994; Tsomides *et al.*, 1995; Volkering *et al.*, 1995; Doong *et al.*, 1996; Guha *et al.*, 1998). Concerning the effects of surfactants on the solubility and PAHs biodegradation were studied by Kim and his coworker (Kim *et al.*, 2001). Surfactants have been applied successfully for remediation of sewage sludge or soil-bound PAHs by many researchers (Li and Chen, 2002; Prak and Pritchard, 2002; Chu and Chan, 2003; Zhu and Feng, 2003; Jessica *et al.*, 2004; Zhou and Zhu, 2004).

Now, mixed surfactants are of great interest in scientific and industrial application. Some surfactant combinations exhibit synergistic properties: they show considerable decrease in surface tension and a lower CMC value than each of the individual members (Holland, 1986). Mixed surfactants could be used over a wider range of temperature, salinity and hardness condition than the individual surfactant. A few studies have been conducted on the solubilization of PAHs on mixed surfactants. As an example, Zhou and his co-workers have investigated the solubilization of pyrene in some anionic-nonionic mixed surfactants (Zhou *et al.*, 2004, 2005).

Recently, the removal characteristics of PAHs from soil using a surfactant-enhanced electrokinetic process were investigated. This technology can effectively remove organic pollutants from fine-grained soil (Saichek and Reddy, 2003). Although surfactant-enhanced remediation technology has been developed for relatively permeable subsurface contaminated by PAHs, it is not often directly applicable to fine-grained soil and sediments due to the low permeability and high resistance to hydraulic flow through these types of porous media (Yang *et al.*, 2005).

## **4. MATERIALS AND METHODS**

### **4.1. Sewage sludge sampling and characteristics**

Biological sewage sludge (from sequential batch reactor) was collected from the municipal wastewater treatment plant of Haute-Bécancour (Black Lake, QC, Canada) on May, 2005, and stored in a polypropylene container at 4 °C. Total solids (TS) content of the sludge was adjusted to 18.0 and 30.0 g L<sup>-1</sup> for all experiments. Metal and nutrient contents (mg kg<sup>-1</sup> of dry matter (DM)) of the sludge were as follows: Al (19 000 ± 400), Ca (13 900 ± 200), Cd (3.0 ± 0.1), Cr (139 ± 4), Cu (1 800 ± 40), Fe (37 100 ± 900), K (7 200 ± 200), Mg (17 100 ± 200), Mn (468 ± 7), Na (4 700 ± 100), Ni (134 ± 3), P (19 900 ± 400), Pb (87 ± 1), S (5 000 ± 200), Zn (620 ± 10).

### **4.2. Procedure of sludge HAP-spiking**

Before experiments, the sludge was spiked with 5 mg kg<sup>-1</sup> DM of each of 11 PAHs by adding standard solutions Mix 44 (1 000 mg L<sup>-1</sup> in CH<sub>2</sub>Cl<sub>2</sub>-benzene) and benzo(j)fluoranthene (2 000 mg L<sup>-1</sup> in CH<sub>2</sub>Cl<sub>2</sub>-benzene). These standard solutions have been obtained from Sigma-Aldrich Canada Ltd (Oakville, ON, Canada). The sludge was successively mixed by being vigorously stirred (Mechanical shaker, Lab-Line Environ-Shaker, model 3528) for at least 1 h at room temperature (20 ± 2 °C) and then kept at 4 °C for 24 h.

### **4.3. Sludge treatments**

During and after treatment processes, sludge samples (300 to 600 ml) have been taken and solid and liquid phases have been separated by centrifugation (2 050 x g for 30 min, Beckman

Coulter Inc., model Allegra<sup>TM</sup> 6 centrifuge) or vacuum filtration (Whatman No. 4 membrane, porosity : 15 to 20 µm). HAPs extraction and purification steps have been carried out in triplicate on these solid and liquid phases.

#### **4.3.1 Control assays**

At first, to illustrate the removal efficiency of PAHs by different treatments, three control tests were carried out using three volumes of approximately 2 L of sludge. The first control was the sludge without doping and treatment (NDC). The second (DOC) control was prepared by using the prealably doped sludge. A third control (DAC) was prepared using sterilized (autoclaving under 15 psi at 121 °C for 15 min) sludge. The sterilized sludge was averaged over four 1 L Erlenmeyer flasks and was aseptically doped according to the procedure previously described. The doped sludge in one flask (DAC) was separated into solid and liquid phases by centrifugation. Both phases were used for PAHs extraction and analysis. PAH concentrations in these sludge controls are given in Table 4.1. The other three flasks were kept at room temperature ( $20 \pm 2$  °C) for 7, 14 and 21 days respectively before separation into solid and liquid phases.

#### **4.3.2 Mesophilic aerobic digestion (MAD) treatment**

Mesophilic conventional aerobic digestion process has been evaluated for PAHs biodegradation. A volume of 3 L of doped sludge was digested in batch mode in a stirred tank reactor (5 L capacity) at room temperature. Some important operating parameters are described in Table 4.2. During the whole process, dissolved oxygen was kept  $> 2$  mg L<sup>-1</sup>. Samples were taken after 3, 5, 7, 14 and 21 days for PAHs extraction and analysis. Solid and liquid phases were separated by

4. Methodology and experiment

centrifugation (room temperature, 2 050 x g for 30 min, Beckman Coulter Inc., model Allegra<sup>TM</sup> 6 centrifuge).

**Table 4.1 PAHs concentrations (mg kg<sup>-1</sup> DM) in no doped and doped sewage sludge**

PAHs	No doped control (NDC)	Doped control	
		(DOC)*	(DAC)**
ACN	0.036 ± 0.001	4.58 ± 0.12	5.56 ± 0.01
FLU	0.042 ± 0.002	4.51 ± 0.12	5.56 ± 0.01
PHE	0.207 ± 0.010	5.11 ± 0.17	5.73 ± 0.03
FLR	0.493 ± 0.012	4.92 ± 0.56	6.03 ± 0.07
PYR	0.522 ± 0.028	5.07 ± 0.22	6.06 ± 0.06
BJK	0.910 ± 0.084	16.82 ± 0.30	17.72 ± 0.11
BAP	0.493 ± 0.012	4.94 ± 0.22	5.93 ± 0.06
INP	0.392 ± 0.037	5.65 ± 0.45	6.14 ± 0.05
BPR	0.534 ± 0.045	6.02 ± 0.16	6.07 ± 0.04
Total (11 PAHs)	3.586 ± 0.214	57.84 ± 2.31	64.79 ± 0.43

\* TS = 18.0 g L<sup>-1</sup>.

\*\* TS = 30.0 g L<sup>-1</sup>.

**Table 4.2 Experimental conditions of the different assays of PAHs removal**

Treatments	TS (g L <sup>-1</sup> )	Duration	Tw80 (g L <sup>-1</sup> )	Final pH	Final ORP (mV)
DAC	30.0	21 d	0	7.16	-58
MAD	30.0	21 d	0	7.08	+440
MAD+Tw80	30.0	21 d	0.5	6.43	+446
METIX-BS	30.0	21 d	0	1.42	+461
METIX-AC	18.0	4 h	0	2.08	+326
METIX-AC+Tw80	30.0	4 h	0.5	1.88	+370
STABIOX	18.0	80 min	0	4.00	+290
ELECSTAB	18.0	60 min	0	4.00	+171
Tw80 - A	30.0	6 h	0.5	6.60	-70
Tw80 - B	30.0	4 h	0.5	6.59	-83
Tw80 - C	30.0	2 h	2.0	6.62	-82

#### 4.3.3 MAD+Tw80 treatment

Addition of Tween 80 (Tw80), a nonionic surfactant, was tested in combination with the MAD process. A concentration of 1.5 g Tw80 L<sup>-1</sup> (750 ml) has been added to a volume of 1.5 L of doped sludge and the mixture was digested in a stirred tank reactor (5 L). Samples were taken after 3, 5, 7, 14 and 21 days for PAHs extraction and analysis. Solid and liquid phases have been separated by filtration.

#### 4.3.4 METIX-BS treatment

METIX-BS technology is a biological process for simultaneous sludge digestion and metal bioleaching with addition of elemental sulfur. This process can remove toxic metals, eliminate odor and destroy pathogens. The oxidation of elemental sulfur, which is a energy source of the indigenous sludge thiobacilli, into sulfuric acid causes a sludge acidification with lowering the pH value below 2.0. A volume of 3 L of doped sludge was divided into fifteen 200 ml fractions (in 500 ml Erlenmeyer flasks with stainless cap). To each flask was added 1.0 g elemental sulfur and then the Erlenmeyer flasks were placed on a rotary shaker at 150 rotations per minute (rpm) at 28 ± 2°C. Samples were taken and centrifuged after 3, 5, 7, 14 and 21 days for PAHs extraction and analysis. Solid and liquid phases have been separated by centrifugation.

#### 4.3.5 METIX-AC treatment

METIX-AC is a chemical process to remove toxic metals from sludge. This process is also able to eliminate odors and pathogens by using oxidizing agents such as ferric chloride and hydrogen peroxide. A volume of 2 L of doped sludge was acidified to pH 2 by addition of sulfuric acid (5 ml, 5 M) at room temperature in a 5 L stirred tank reactor. Solutions of ferric chloride (12 ml, Fe<sup>3+</sup> content of 11% w w<sup>-1</sup> basis) and hydrogen peroxide (10 ml, 3% v v<sup>-1</sup>) were sequentially

added in order to maintain an oxidoreduction potential (ORP) value ( $> 400$  mV). The sludge was vigorously stirred for 4 h at room temperature. Then solid and liquid phases were separated by centrifugation.

#### **4.3.6 METIX-AC+Tw80 treatment**

Addition of Tw80 was also tested in combination with the METIX-AC process. A volume of 0.6 L of doped sludge was acidified to pH 2 in a beaker (2 L) by addition of sulfuric acid (3 ml, 5 M) prior to the addition of solutions of ferric chloride (6 ml, 11%  $\text{Fe}^{3+}$  w w<sup>-1</sup>) and hydrogen peroxide (5 ml, 3% v v<sup>-1</sup>) in sequence. After the sludge was successfully mixed by stirring at room temperature for 2 h, a concentration Tw80 in 1.5 g L<sup>-1</sup> (300 ml) was added. After adjustment the pH to 2 by addition of sulfuric acid (1 ml, 5 M), and the sludge was agitated for another 2 h at room temperature. The sludge solid and liquid phases were separated by filtration.

#### **4.3.7 STABIOX treatment**

STABIOX is a sludge stabilization and conditioning process allowing to reduce odor, destroy pathogens and to increase the dewatering properties of municipal and industrial sludge. A volume of 2 L of doped sludge was acidified to pH 4 by addition of sulfuric acid (3.5 ml, 5 M) in a 5 L stirred tank reactor. Solutions of ferric chloride (1.5 ml, 11%  $\text{Fe}^{3+}$  w w<sup>-1</sup>) and hydrogen peroxide (10 ml, 3% v v<sup>-1</sup>) were then added in sequence. The sludge was then mixed for 80 min at room temperature. The sludge solid and liquid phases were separated by centrifugation.

#### **4.3.8 ELECSTAB treatment**

ELECSTAB is an electrochemical stabilization and conditioning process having an efficient antiseptic effect and allowing to improve the dewatering properties of sludge. A volume of 5 L of doped sludge was charged into a 12-L capacity electro-reactor (42 cm height, 25 cm diameter)

consisting of a cell, two concentrical electrodes (Anode: Ti/RuO<sub>2</sub>, Cathode: Ti) and a DC power supply (XFR 40-70, 0-40 V, 1~70 A). The interelectrode distance was 4 cm. Sodium sulphate (12.5 g, electrolyte), sulfuric acid (13.5 ml, 5 M) and hydrogen peroxide (45 ml, 3% v v<sup>-1</sup>) were added sequentially. Electrical treatment was applied (8 A and 16 V) at room temperature for 60 min. The treated sludge was separated into solid and liquid phases by centrifugation.

#### **4.3.9 Non-ionic surfactant treatment**

Three different procedures (Tw80-A, -B, -C) were used for the treatment of sewage sludge using Tw80 as PAH-extracting agent. These experiments were carried out using volumes of 200 ml of doped sludge (in 500 ml Erlenmeyer flasks with stainless cap). The flasks were placed on a rotary shaker at 150 rpm at room temperature. A concentration of Tw80 in 1.5 g L<sup>-1</sup> (100 ml) was added to the sludge and the sludge was agitated for 6 h during the assay Tw80-A. After treatment, the solid phase and liquid phase were separated by centrifugation at different speeds (500, 1 000, 2 000 and 3 000 × g for 30 min) and by vacuum filtration. Final concentrations of Tw80 in 0.5 g L<sup>-1</sup> and Tw80 in 2.0 g L<sup>-1</sup> were used to treat the sewage sludge for 4 h and 2 h during assays Tw80-B and Tw80-C. After treatment, the solid and liquid phases were separated by filtration.

### **4.4. PAHs extraction, purification and analysis**

#### **4.4.1 Soxhlet extraction for solid samples**

In this study, PAHs were extracted from sludge samples using a modified method of Centre d'expertise en analyse environnementale du Québec (Gouvernement du Québec, 2002). Dichloromethane was used as solvent for all extractions of PAHs from both solid and liquid

samples. A solution of a surrogate standard spiking (as recovery; acenaphtene-d<sub>10</sub> and chrysene-d<sub>12</sub> in isoctane 100 mg L<sup>-1</sup>) was used during extraction.

The solid samples were dried with anhydrous MgSO<sub>4</sub> (pre-heated at 650 °C for 12 h in oven before using). The powder-like samples were transferred into pre-decontaminated cellulose extraction thimbles (43 × 123 mm, Advance MFS Inc.) dipped in dichloromethane at least for 24 h before using. The solid samples were Soxhlet extracted (Organamation Glassware with three flasks (500 ml) Heaters, Electrothermal EME 30500/EX1) for 24 h at rate of 4-6 cycles h<sup>-1</sup> (MA. 400-HAP 1.1). After the extraction was complete, the extracts were allowed to cool to room temperature and then concentrated to approximately 5 ml by rotary evaporation (Büchi, model Rotavapor-R) at lower than 26 °C.

#### **4.4.2 Saponification**

Potassium hydroxide solution (0.5 M in methanol) was added to the extracts from the Soxhlet extraction. The mixtures were refluxed for 4 h at 80 °C in a water-bath (Mangas *et al.*, 1998). After cooling to room temperature, volume of 100 ml of saturated brine in milli-Q-purified water was added and the mixture was exacted with dichloromethane (3 × 50 ml). The combined organic phase was dried by filtration over anhydrous MgSO<sub>4</sub> (pre-heated at 650 °C for 12 h in oven before using) and then evaporated to near dryness on a rotary evaporator at lower than 26 °C. The residues were dissolved in 2 ml hexane for the purification step.

#### **4.4.3 Rotating extraction for liquid samples**

The liquid samples were extracted using dichloromethane by rotating extraction at a rate of 6 cycles min<sup>-1</sup> for 24 h at room temperature. The separated extracts were then concentrated nearly

#### *4. Methodology and experiment*

to dryness by rotary evaporation at lower than 26 °C, and the residues were dissolved in hexane (1 ml) for the purification step.

##### **4.4.4 PAHs purification by silica gel column flash chromatography**

In the clean-up step, a recovery solution of anthracene-d<sub>10</sub> and pyrene-d<sub>10</sub> (100 mg l<sup>-1</sup> in isoctane), a glass column and a certain quantity (1/6, w<sub>Si</sub>/w<sub>dry solid sample</sub>, 10 g for the extracts from liquid samples) activated silica gel were used. A typical procedure was shown below.

Activated silica gel (60 g) was packed in a glass column with a pad of anhydrous magnesium sulfate (*ca* 1 cm) on top for clean-up extracts. The silica gel was rinsed with hexane (100 ml) before loading the extract and the recovery onto the column and eluted with hexane-dichloromethane (3:0, 100 ml → 3:2, 150 ml). The second elute containing PAHs was collected and concentrated to approximately dryness by rotary evaporation at lower than 26 °C. Then the purified sample was quantitatively transferred into a 5 ml volumetric flask isoctane and this purified samples in isoctane must be protected from light and stored in sealed amber vials at ≤ -10 °C until analysis.

#### **4.5. Analytical methods**

PAHs were quantified using GC-MS (Perkin Elmer, model Clarus 500, with column type of VF-5MS FS, 30 m × 0.25 mm × 0.25 µm) at Institut National de la Recherche Scientifique (INRS) and GC-MS-SIM (selected ion monitoring; Hewlett Packard 6890 series GC equipped with a HP 5973A series mass spectrometer detector; with column type of DB-5, 30 m × 0.25 mm × 0.25 µm) at Bodcote (Québec, QC, Canada).

The pH was determined using a pH-meter (Fisher Acumet model 915) equipped with a double-junction Cole-Palmer electrodes with Ag/AgCl reference cell. Total solids were measured according to the method 2540B (APHA *et al.*, 1999). To determine metal concentrations, the samples were first filtered on Whatman 934-AH membrane (Whatman International Ltd, Maidstone, England) under vacuum, then the filtrates were acidified with concentrated nitric acid (5% v v<sup>-1</sup>) and kept at 4 °C until analysed. The digestion method of the sludge was executed by digesting 0.5 g dry samples in presence of HNO<sub>3</sub>, HF and HClO<sub>4</sub>, in a final solution of 5% HNO<sub>3</sub> (method 3031 I; APHA *et al.*, 1999). The metal concentrations were determined by plasma emission spectroscopy with a simultaneous ICP-AES (Inductively Coupled Plasma, Varian company, Vista model).

#### **4.6. Chemicals**

Organic solvents were analytical grade reagents from Merck. Sodium chloride, potassium hydroxide and anhydrous magnesium sulfate (analytical grade and pre-heated at 650 °C for 12 h in oven before using) were supplied by EMD Chemicals Inc. Sulfuric acid (95%-98% ACS reagent) was purchased from Fisher Scientific. Ferric chloride solution (Fe<sup>3+</sup> content of 11% w w<sup>-1</sup> basis) was provided by Eaglebrook Environment Corporation (Varennes, QC, Canada). Hydrogen peroxide (33% v v<sup>-1</sup> basis) was obtained from Laboratoire Mat (Beauport, QC, Canada). Silica gel 60 (230 ~ 400 mesh) was bought from Silicycle (Québec, QC, Canada), and Tween 80 was purchased from ICI Americas Inc. Sewage sludge doping reagents, standard references for analysis including Mix 44 (44 PAHs mixture), benzo(j)fluorantene, anthracene-d<sub>10</sub>, pyrene-d<sub>10</sub>, acenaphtene-d<sub>10</sub>, chrysene-d<sub>12</sub>, naphtalene-d<sub>8</sub>, and phenanthrene-d<sub>10</sub> were commercially available from Sigma-Aldrich Canada Ltd (Oakville, ON, Canada). Milli-Q

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purified water was prepared by further purification of de-ionized water on a Millipore Milli-Q system.

## 5. RESULTS AND DISCUSSION

### 5.1. Efficiency of PAHs extraction and purification

The efficiency of sequential Soxhlet extraction, saponification and purification for the recovery of PAHs from solid sludge samples was assessed with a surrogate spiking standard. The recovery percentages obtained were between 85 and 120% in most cases for each surrogate spiking recovery standard in each extraction with different number of aromatic rings and molecule weights of PAHs. For example, the recoveries of 3-ring acenaphtene-d<sub>10</sub> and anthracene-d<sub>10</sub> ranged from 85 to 110% in most cases, and the recoveries of 4-ring pyrene-d<sub>10</sub> and chrysene-d<sub>10</sub> ranged from 90 to 120% in most cases.

#### 5.1.1 Biological treatments (MAD and METIX-BS)

Research has been focused on the investigation of the microorganisms which have the ability to metabolize PAHs (Cerniglia, 1993). The biochemical pathways and the mechanisms of biodegradation have been well documented (Komatsu *et al.*, 1993; Juhasez *et al.*, 1997; Volkering and Breure, 2003). The lower weight PAHs (2, 3 and 4 aromatic rings) were found to be biodegraded more rapidly than the higher weight (5 and more aromatic rings) compounds (Heitkamp and Cerniglia, 1989; Cerniglia, 1993). However, there is very little information available for the removal efficiency of PAHs by MAD or METIX-BS from sewage sludge.

Figures 5.1 to 5.3 present the kinetic of the PAHs biodegradation in term of removal (%) during the MAD, MAD + Tw80 and METIX-BS processes. ACN, FLU and PHE are rapidly biodegraded in all three cases with more than 90% of reduction after only two weeks of

### 5. Results and discussion

treatment. The biodegradation of others PAHs begins generally after a 5-day period and the final removal yields are weaker than for ACE, FLU and PHE.

Table 5.1 gives the final PAH concentrations in the sludge and the corresponding removal yields after the different biological treatments. After 21 days of incubation, the sterilized control (DAC) showed that volatilization of PAH was moderated for ACN, FLU and PHE with 34.3, 32.8 and 18.7% of removal, respectively. Volatilization was not important (< 15%) in the case of the others PAHs analyzed.

Addition of nonionic surfactant (Tw80) during MAD treatment has caused a significant increase of the biodegradation, particularly for FLR, PYR, benzo(b,j,k)fluoranthene (BJK) and BAP. However, no effect was detected in the case of INP and BPR. Overall, the addition of Tw80 has slightly increased the total PAHs removal from  $54.4 \pm 2.9\%$  (MAD) to  $60.1 \pm 2.0\%$  (MAD + Tw80).

METIX-BS process was very efficient for ACN, FLU and PHE removal, but degradation of FLR and PYR were lower than for MAD or MAD + Tw80. However, high molecular weight PAHs (BAP, INP and BPR) were slightly more eliminated than for conventional MAD. Total PAHs removal yield by METIX-BS ( $51.8 \pm 3.0\%$ ) was almost similar to MAD. PAHs degradation observed during METIX-BS process is probably explained by biological mechanisms involving non-acidophilic (during the first days of treatment) and acidophilic microorganisms (when the sludge pH decreases under 4.0) and by chemical degradation due to the acidophilic and oxidative conditions under the last days of treatment (pH 1.42 and ORP +461 mV at the end of the experiment). Previous studies have shown that the yeast *Blastoschizomyces capitatus* and a non-identified fungus are the dominant organisms present in the sludge at the end of the METIX-BS

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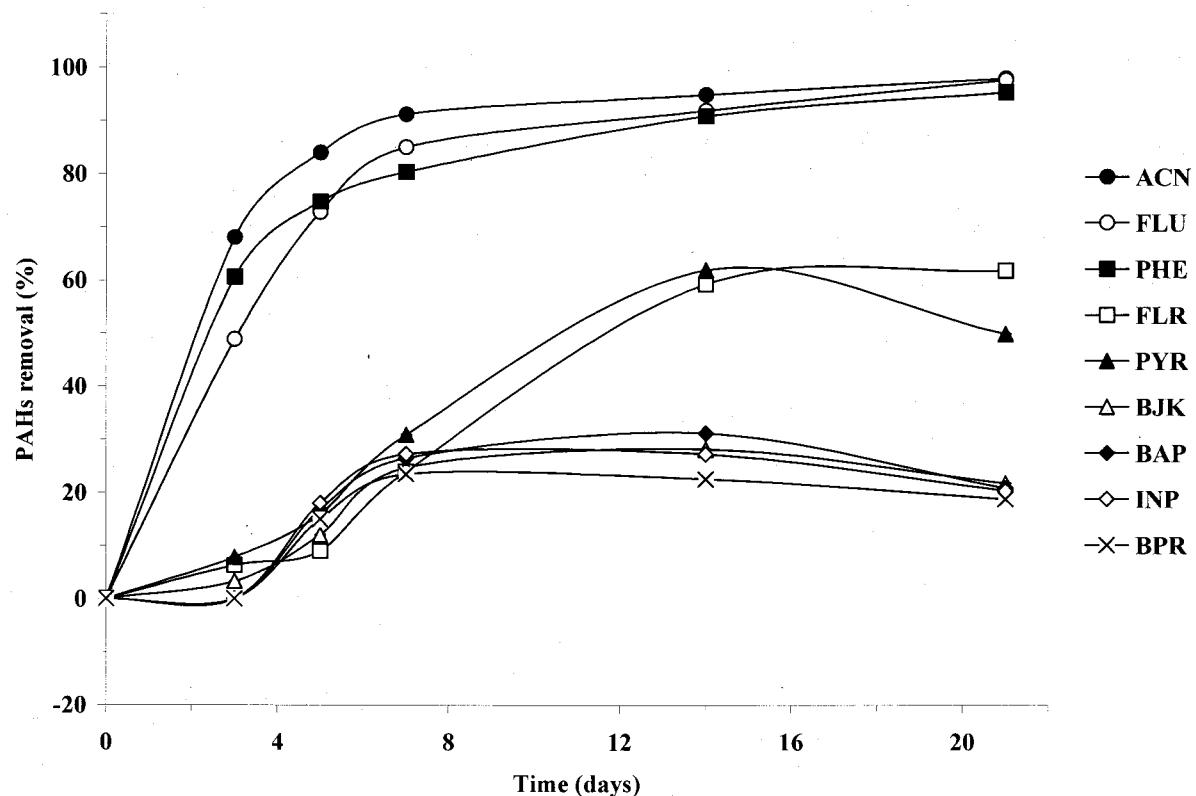
process and would be, in part, responsible of the organic matter degradation (Gamache *et al.*, 2001).

Table 5.2 shows the percentages of PAHs solubilized from the sludge during the different processes tested. Overall, the results reveal that the portion of the PAHs present in the liquid phase is generally very weak ( $\leq 10\%$  of each PAH). In particular, 11 PAH compounds in the liquid phase have been almost completely removed by MAD and MAD + Tw80 treatments because the concentrations of these compounds were as low as in the control sludge (DOC). In the case of METIX-BS, only low weight compounds (ACN, FLU and PHE) have been efficiently removed from the liquid phase. Others PAHs (4-, 5- and 6-ring PAH) were partially removed because the concentrations of these PAHs were higher than the control. These results indicate that the desorption rates of the larger PAHs from the solid sludge was faster than their corresponding degradation rates in METIX-BS.

Figure 5.4 illustrates the efficiency of the biological processes for the degradation of PAHs in relation to the number of aromatic rings. First, a moderate proportion ( $28.6 \pm 3.7\%$ ) of the 3-ring PAHs (ACN, FLU and PHE) was eliminated in the doped and autoclaved control sludge (DAC), whereas for 4 to 6-ring PAHs, the percentage of removal was stabilized to approximately 10-12%. Degradation of the 3-ring PAHs was almost similar for MAD ( $97.0 \pm 0.3\%$ ) and METIX-BS ( $95.5 \pm 0.7\%$ ), but 4-ring PAHs (FLR and PYR) was significantly more removed in the case of MAD ( $56.6 \pm 1.8\%$ ) in comparison to METIX-BS ( $29.9 \pm 2.7\%$ ). Higher molecular weight PAHs were not efficiently eliminated (< 40% removal) by both processes. Addition of Tw80 during the MAD treatment was only beneficial for the degradation of the 5-ring PAHs (BJK and

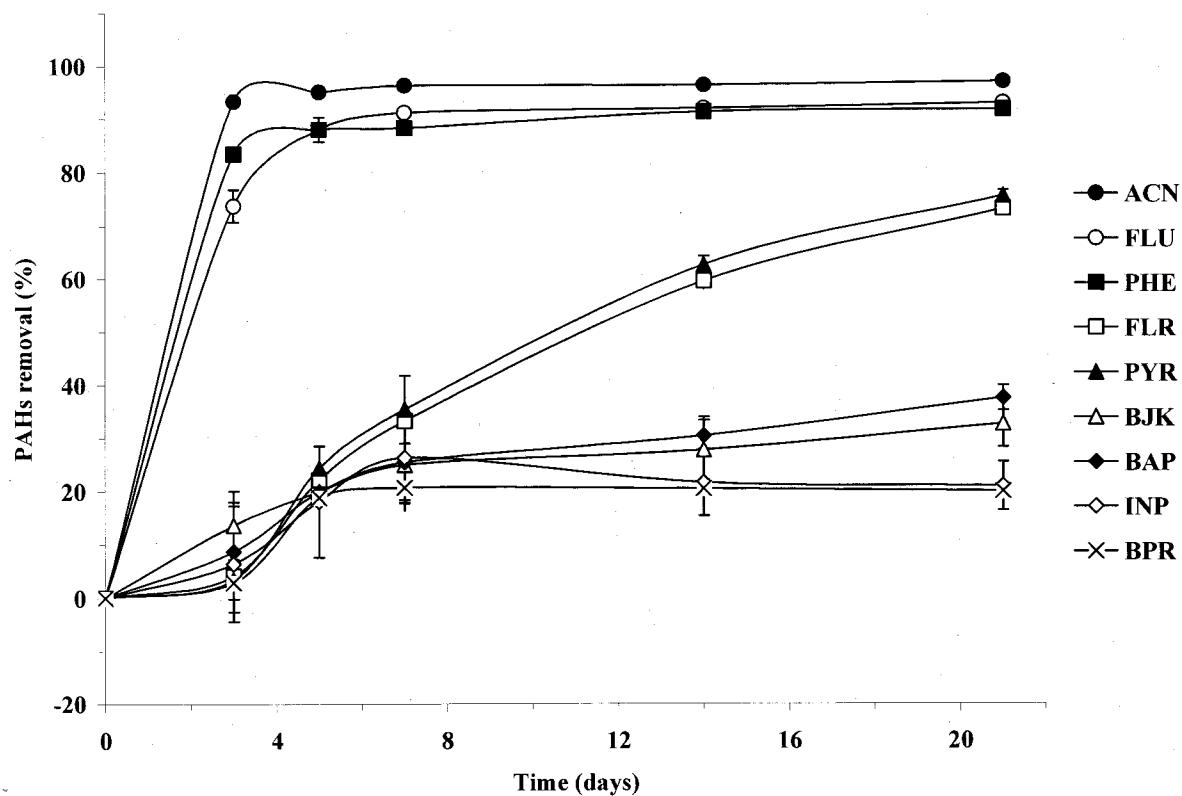
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BAP) with a removal yield of  $35.1 \pm 3.4\%$ , in comparison to  $21.3 \pm 2.7\%$  in the case of MAD and  $32.7 \pm 5.6\%$  in the case of METIX-BS.



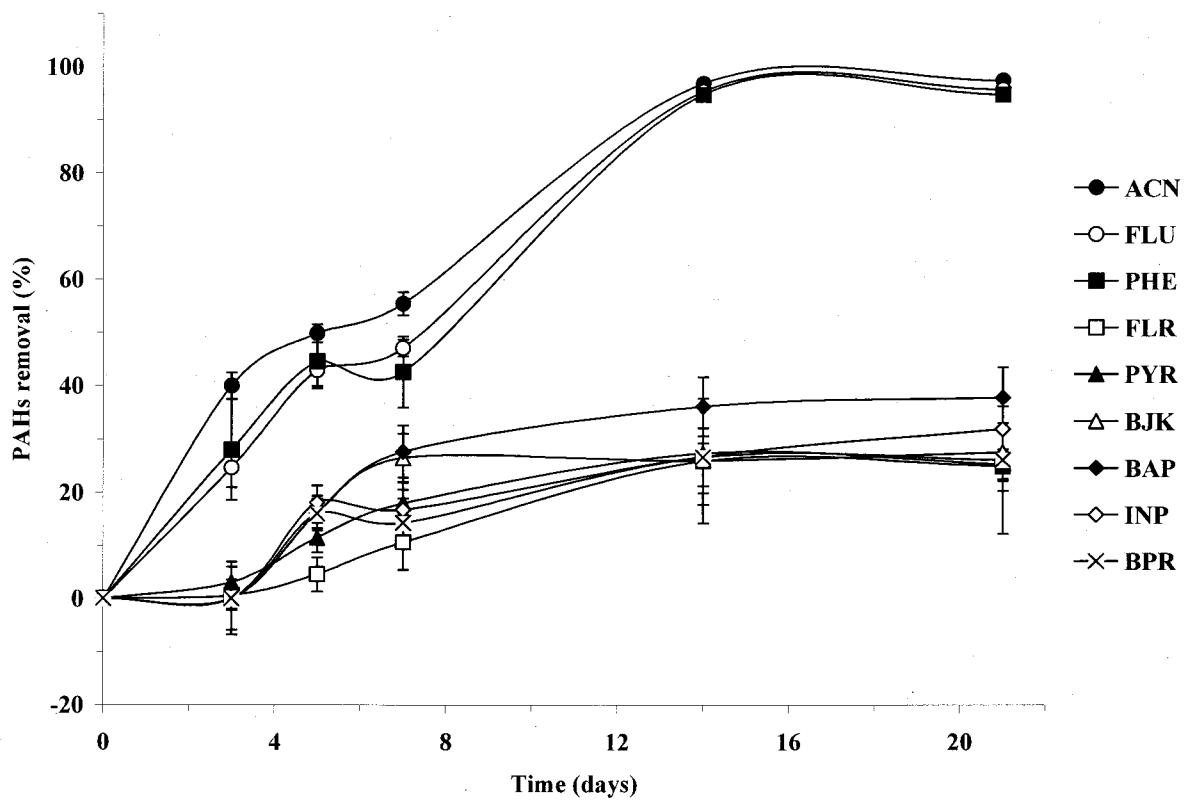
**Figure 5.1** Time variation of the PAHs degradation in the sludge during MAD treatment

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**Figure 5.2** Time variation of the PAHs degradation in the sludge during MAD+Tw80 treatment

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**Figure 5.3** Time variation of the PAHs degradation in the sludge during METIX-BS treatment

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**Table 5.1 Final PAHs concentrations ( $\text{mg kg}^{-1}$  DM) and removal yields (%) in the sludge after different biological treatments**

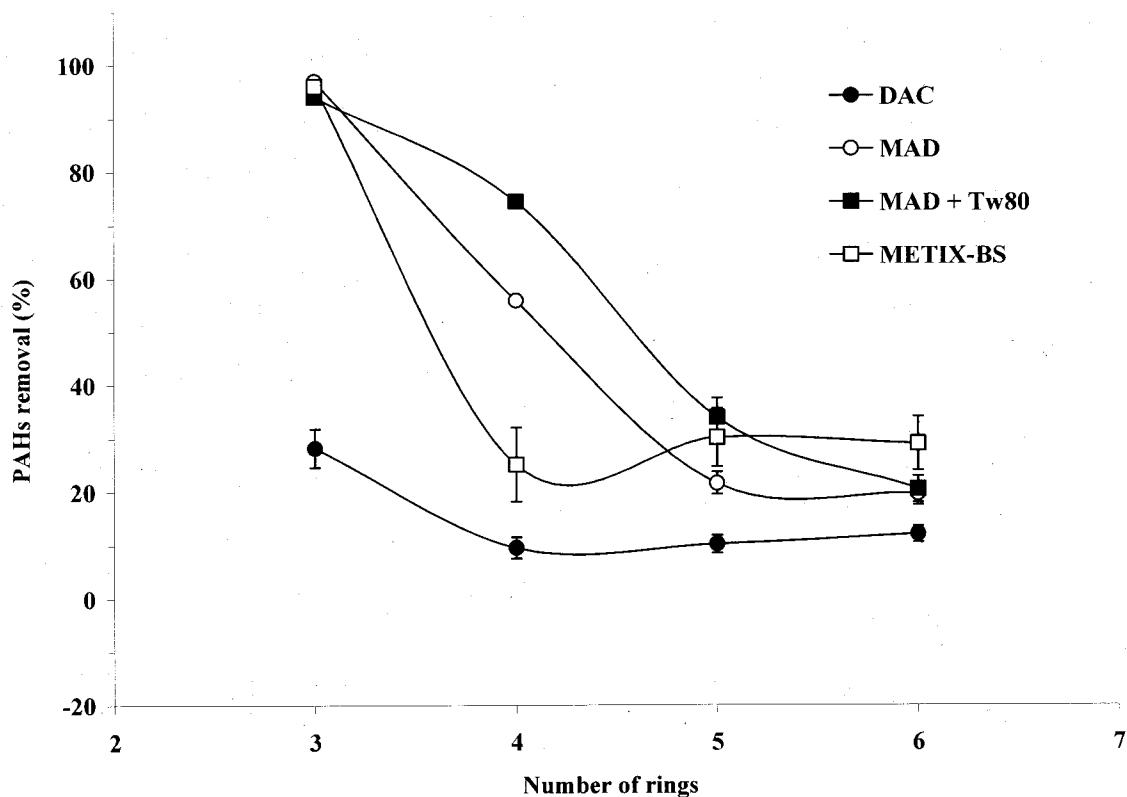
PAHs	DAC		MAD		MAD+Tw80		METIX-BS	
	Conc. ( $\text{mg kg}^{-1}$ )	Removal (%)						
ACN	$3.65 \pm 0.27$	$34.3 \pm 4.9$	$0.11 \pm 0.01$	$97.9 \pm 0.1$	$0.18 \pm 0.00$	$96.5 \pm 0.1$	$0.17 \pm 0.01$	$96.9 \pm 0.3$
FLU	$3.74 \pm 0.16$	$32.8 \pm 2.8$	$0.12 \pm 0.01$	$97.8 \pm 0.2$	$0.42 \pm 0.03$	$92.4 \pm 0.6$	$0.27 \pm 0.02$	$95.2 \pm 0.4$
PHE	$4.66 \pm 0.19$	$18.7 \pm 3.4$	$0.28 \pm 0.03$	$95.1 \pm 0.6$	$0.51 \pm 0.05$	$91.1 \pm 0.9$	$0.33 \pm 0.08$	$94.3 \pm 1.3$
FLR	$5.59 \pm 0.12$	$7.2 \pm 1.9$	$2.29 \pm 0.07$	$62.1 \pm 1.1$	$1.65 \pm 0.08$	$72.7 \pm 1.4$	$3.69 \pm 0.14$	$38.8 \pm 0.2$
PYR	$5.28 \pm 0.12$	$12.8 \pm 2.1$	$2.97 \pm 0.14$	$51.1 \pm 2.4$	$1.51 \pm 0.07$	$75.0 \pm 1.1$	$4.79 \pm 0.15$	$21.0 \pm 2.4$
BJK	$15.57 \pm 0.39$	$12.1 \pm 2.2$	$14.05 \pm 0.81$	$20.7 \pm 4.6$	$11.86 \pm 0.77$	$32.1 \pm 4.4$	$13.14 \pm 0.51$	$25.9 \pm 2.9$
BAP	$5.37 \pm 0.16$	$9.4 \pm 2.7$	$4.59 \pm 0.34$	$22.6 \pm 5.8$	$3.72 \pm 0.14$	$37.2 \pm 2.3$	$3.78 \pm 0.64$	$36.2 \pm 10.8$
INP	$5.25 \pm 0.37$	$14.6 \pm 6.1$	$4.64 \pm 0.28$	$24.5 \pm 4.6$	$4.81 \pm 0.28$	$21.7 \pm 4.5$	$4.29 \pm 0.11$	$30.2 \pm 1.7$
BPR	$5.30 \pm 0.14$	$12.6 \pm 2.4$	$4.98 \pm 0.42$	$17.9 \pm 7.0$	$4.77 \pm 0.16$	$21.4 \pm 2.7$	$4.40 \pm 0.43$	$27.5 \pm 7.0$
Total	$54.40 \pm 1.22$	$16.0 \pm 3.0$	$34.01 \pm 2.12$	$54.4 \pm 2.9$	$29.44 \pm 1.58$	$60.1 \pm 2.0$	$34.85 \pm 1.95$	$51.8 \pm 3.0$

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**Table 5.2 PAHs solubilization yields (%) from the sludge after different biological, chemical and electrochemical treatments**

PAHs	DOC	MAD	MAD + Tw80*	METIX-BS	METIX-AC	METIX- AC+Tw80	STABIOX	ELECSTAB
ACN	4.54 ± 0.21	0.48 ± 0.00	0.03 ± 0.00	0.51 ± 0.00	2.33 ± 0.21	1.97 ± 0.17	3.19 ± 0.25	1.53 ± 0.42
FLU	4.74 ± 1.24	0.55 ± 0.10	0.02 ± 0.00	0.69 ± 0.06	3.65 ± 0.65	2.00 ± 0.35	2.84 ± 0.25	1.58 ± 0.37
PHE	4.26 ± 1.79	0.71 ± 0.26	0.02 ± 0.00	0.65 ± 0.01	2.39 ± 0.25	1.48 ± 0.03	1.48 ± 0.19	1.35 ± 0.25
FLR	5.29 ± 1.97	0.79 ± 0.38	0.11 ± 0.01	4.57 ± 0.33	3.20 ± 0.35	7.19 ± 4.98	1.50 ± 0.22	1.96 ± 0.44
PYR	5.23 ± 1.94	0.88 ± 0.39	0.13 ± 0.01	7.13 ± 0.48	3.21 ± 0.36	12.40 ± 0.10	1.53 ± 0.22	2.31 ± 0.24
BJK	3.21 ± 0.55	0.73 ± 0.11	0.05 ± 0.01	7.94 ± 1.23	3.14 ± 0.71	4.22 ± 0.54	0.83 ± 0.05	3.89 ± 0.57
BAP	5.00 ± 0.08	0.74 ± 0.30	0.03 ± 0.00	10.06 ± 1.71	3.57 ± 0.77	1.83 ± 0.36	1.23 ± 0.09	2.70 ± 0.77
INP	5.22 ± 0.54	0.69 ± 0.24	0.04 ± 0.00	10.11 ± 1.49	4.09 ± 0.96	3.06 ± 0.54	1.11 ± 0.05	2.23 ± 0.57
BPR	4.87 ± 0.41	0.72 ± 0.31	4.05 ± 0.44	9.38 ± 1.13	3.94 ± 0.89	2.86 ± 0.68	1.03 ± 0.07	2.43 ± 0.61
Total	4.44 ± 0.70	0.71 ± 0.21	0.42 ± 0.04	6.32 ± 0.84	3.28 ± 0.57	4.18 ± 0.47	1.44 ± 0.10	2.53 ± 0.49

\* After a 3-d period of treatment.



**Figure 5.4** Final PAHs removal yields in the sludge after different biological treatments in relation to the number of aromatic rings

### 5.1.2 Chemical (METIX-AC and STABIOX) and electrochemical (ELECSTAB) treatments

During METIX-AC and STABIOX processes, sulfuric acid was added to acidify the sewage sludge followed by sequential addition of hydrogen peroxide ( $H_2O_2$ ) and ferric ion ( $FeCl_3$ ), a system similar to Fenton's reagent, which had been investigated recently in treatment of toxic non-biodegradable organic compounds in wastewater (El-Morsi *et al.*, 2002; Lee *et al.*, 2003). The  $Fe^{3+}/H_2O_2$  system could produce highly oxidative hydroxyl, perhydroxyl radicals, which are

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able to oxidize most of the complex organic materials, presented in municipal wastewaters. Beside the reactive radical species, hydrogen peroxide could also form protonated hydrogen peroxide in acidic milieu. The protonated  $\text{HOOH}_2^+$  is believed to be a very unstable intermediate with highly oxidative activity.

Final PAH concentrations and removal yields by chemical and electrochemical treatments are presented in Table 5.3. In general, PAH degradation was lower than for biological processes. METIX-AC process gave interesting removal yields for ACN ( $58.1 \pm 6.9\%$ ), FLU ( $47.1 \pm 7.1\%$ ) and PHE ( $41.5 \pm 11.2\%$ ), but very weak degradation of high molecular weight PAHs (< 20%).

Addition of nonionic surfactant (Tw80) during METIX-AC treatment was not beneficial to PAHs degradation. In fact, the removed amounts of PAHs by this combinative treatment METIX-AC + Tw80 ( $11.6 \pm 5.2\%$ ) were less than the independent treatment of METIX-AC ( $29.6 \pm 7.7\%$ ). Because the double bond in the lipid chain of Tw80 would be easily cleaved by Fenton-like reagent since the oxidation of an isolate double bond occurs more easily than the conjugated  $\pi$  bond of PAHs on one hand. On the other hand, once the double bond was cleaved, Tw80 completely lost its function as a surfactant. To avoid this defection, similar surfactants with saturated lipophilic side chain could be a possible solution in future work.

Less acidic and oxidative conditions prevailing during STABIOX in comparison to METIX-AC treatment gave weaker degradation of PAHs with a global removal yield for the 11 PAHs of only  $22.2 \pm 3.2\%$ . Pignatello's study supports that the transformation of organic pollutants can give the best result at pH  $2.7 \sim 2.8$  by  $\text{Fe}^{3+}/\text{H}_2\text{O}_2$  because half of the iron could be transformed into  $\text{Fe(OH)}^{2+}$  form which was the pivotal intermediate to keep the Fenton reaction at the optimal pH

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value (Pignatello, 1992). Future work should be conducted on optimization of pH value during METIX-AC for combined removal of both heavy metals and PAHs.

Degradation of low weight PAHs (ACN, FLU and PHE) during ELECSTAB process were quiet good with approximately 62% of removal. However, practically no reduction was observed in the concentration of high molecular weight PAHs.

Proportion of PAHs presents in the liquid phase was not greatly affected by the chemical and electrochemical process (Table 5.2). At the end of each treatment, the concentrations in liquid phase of the 11 PAHs, were almost similar to the control sludge (DOC).

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**Tableau 5.3 Final PAHs concentrations ( $\text{mg kg}^{-1}$  DM) and removal yields (%) in the sludge after different chemical and electrochemical treatments**

HAPs	METIX-AC		METIX-AC+Tw80		STABIOX		ELECSTAB	
	Conc. ( $\text{mg kg}^{-1}$ )	Removal (%)	Conc. ( $\text{mg kg}^{-1}$ )	Removal (%)	Conc. ( $\text{mg kg}^{-1}$ )	Removal (%)	Conc. ( $\text{mg kg}^{-1}$ )	Removal (%)
ACN	$2.33 \pm 0.39$	$58.1 \pm 6.9$	$3.86 \pm 0.20$	$30.5 \pm 3.5$	$3.75 \pm 0.09$	$32.4 \pm 1.6$	$1.78 \pm 0.07$	$67.9 \pm 1.2$
FLU	$2.94 \pm 0.40$	$47.1 \pm 7.1$	$4.53 \pm 0.22$	$18.5 \pm 3.7$	$4.37 \pm 0.12$	$21.4 \pm 2.1$	$2.30 \pm 0.07$	$58.7 \pm 1.3$
PHE	$3.35 \pm 0.64$	$41.5 \pm 11.2$	$4.57 \pm 0.18$	$20.2 \pm 3.2$	$3.26 \pm 0.11$	$43.0 \pm 1.9$	$2.26 \pm 0.27$	$60.5 \pm 4.7$
FLR	$4.01 \pm 0.55$	$33.5 \pm 9.4$	$6.47 \pm 0.23$	$2.0 \pm 3.8$	$4.61 \pm 0.32$	$23.5 \pm 5.3$	$5.81 \pm 0.45$	$3.6 \pm 7.4$
PYR	$3.80 \pm 0.53$	$37.2 \pm 8.9$	$6.04 \pm 0.76$	$1.0 \pm 12.6$	$4.49 \pm 0.32$	$25.9 \pm 5.4$	$5.80 \pm 0.29$	$4.2 \pm 4.9$
BJK	$15.36 \pm 0.39$	$13.3 \pm 2.8$	$15.49 \pm 0.23$	$12.6 \pm 1.3$	$14.04 \pm 0.56$	$20.8 \pm 3.2$	$17.55 \pm 0.88$	$1.0 \pm 4.9$
BAP	$4.39 \pm 0.17$	$25.9 \pm 3.4$	$5.79 \pm 0.36$	$2.3 \pm 6.1$	$5.05 \pm 0.10$	$14.8 \pm 1.7$	$5.66 \pm 0.25$	$4.5 \pm 4.1$
INP	$6.03 \pm 0.64$	$1.9 \pm 11.9$	$5.83 \pm 0.49$	$5.1 \pm 8.0$	$5.44 \pm 0.30$	$11.4 \pm 4.8$	$5.55 \pm 0.21$	$9.6 \pm 3.4$
BPR	$5.57 \pm 0.69$	$8.2 \pm 11.4$	$5.24 \pm 0.29$	$13.7 \pm 4.8$	$5.65 \pm 0.19$	$6.9 \pm 3.1$	$5.46 \pm 0.34$	$10.0 \pm 5.6$
Total	$47.70 \pm 4.33$	$29.6 \pm 8.2$	$57.81 \pm 2.99$	$11.8 \pm 5.2$	$50.67 \pm 2.10$	$22.2 \pm 3.2$	$52.19 \pm 1.09$	$24.4 \pm 4.2$

### 5.1.3 Non-ionic surfactant treatments

Table 5.4 shows the PAH solubilization yields from sewage sludge after different treatments using Tw80, as an extracting agent. ACN ( $78.0 \pm 0.2\%$ ), FLU ( $72.3 \pm 0.5\%$ ) and PHE ( $69.3 \pm 1.0\%$ ) were efficiently solubilized after 6 h of treatment in presence of Tw80 in  $0.5 \text{ g L}^{-1}$  (assay Tw80 – A), whereas others PAHs were not extracted well (removal  $\leq 25\%$ ) from the sludge. The decrease of treatment time from 6 to 4 h (assay Tw80 - B) resulted in the significative reduction of the PAHs removal with less than 40% removal for each PAH. The increase of surfactant concentration to Tw80 in  $2.0 \text{ g L}^{-1}$  allowed, even for a short treatment time (2 h), to increase the solubilization yields to more than 90% for ACN and PHE and 80% for FLU. However, extraction yields for others PAHs remain weak ( $< 20\%$ ). However, this concentration of Tw80 in  $2.0 \text{ g L}^{-1}$  can be only used in laboratory study and not at industrial scale because of the economical aspect. Moreover, too much Tween 80 used can also cause environmental problems.

Comparison of filtration and centrifugation as S/L separation methods for surfactant-treated sludge has shown that the first one gives better recovery of the PAHs in the liquid phase. In fact, solubilized PAHs in surfactant micelles can be easily separated from the solid phase since the micelles in aqueous phase can pass through Whatman No. 4 filter paper. On the other hand, centrifugation, even at low speed, pelleted out micelles from aqueous phase, and makes the micelles back into solid phase giving a decrease of separation efficiency. A similar behavior has also been observed (results not shown) in the case of the experiment carried out with a concentration of Tw80 in  $2.0 \text{ g L}^{-1}$ .

### *5. Results and discussion*

None of the surfactant treatments tested were effective in removal of 4- to 6-ring PAHs from the sludge solids. These results coincide with what Ko and his co-workers found that surfactants are not directly applicable in treatment of fine-grained soils and sediments due to the low permeability and high resistance to hydraulic flow through these types of porous media (Ko *et al.*, 2000). Therefore, the combination of Tw80, or another surfactant, with biological treatment (MAD or METIX-BS) or chemical treatment (METIX-AC) appears more promising than the use of only this type of chemical for sewage sludge decontamination. Finally, it is important to take into consideration that extracted PAHs from the sludge and found in liquid phase (filtrate) should be destroyed, recovered or managed at probably high costs.

*5. Results and discussion*

**Table 5.4 PAHs solubilization yields (%) from the sludge after different surfactant treatments**

PAHs S/L separation method	Filtration	Tw80-A				Filtration	Tw80-B	Tw80-C			
		Centrifugation									
		500 x g	1 000 x g	2 000 x g	3 000 x g						
ACN	78.0 ± 0.2	38.8 ± 0.4	33.8 ± 0.0	39.5 ± 2.3	48.3 ± 1.3	36.6 ± 3.6	91.1 ± 0.8				
FLU	72.3 ± 0.5	39.5 ± 3.4	31.9 ± 2.2	37.2 ± 3.9	43.6 ± 1.3	35.2 ± 4.7	79.2 ± 0.4				
PHE	69.3 ± 1.0	39.4 ± 1.8	36.3 ± 0.5	37.0 ± 1.5	47.5 ± 0.7	21.9 ± 5.3	90.6 ± 0.8				
FLR	25.1 ± 1.7	5.9 ± 7.6	14.3 ± 5.9	-1.5 ± 6.2	5.0 ± 7.5	8.6 ± 1.5	16.0 ± 4.0				
PYR	9.4 ± 4.0	12.1 ± 7.4	11.6 ± 11.2	3.5 ± 6.0	7.1 ± 8.1	9.4 ± 3.1	14.8 ± 3.7				
BJK	14.8 ± 11.9	15.0 ± 7.2	22.7 ± 5.6	2.0 ± 8.4	4.5 ± 8.7	11.1 ± 6.0	0.2 ± 13.8				
BAP	9.9 ± 6.3	4.3 ± 4.7	5.4 ± 17.1	1.8 ± 2.7	6.0 ± 3.7	10.1 ± 6.5	-0.5 ± 13.5				
INP	13.8 ± 10.4	2.5 ± 7.5	22.8 ± 8.6	5.7 ± 8.4	10.0 ± 5.0	8.2 ± 7.9	6.0 ± 9.6				
BPR	14.6 ± 15.5	9.8 ± 12.9	8.9 ± 19.9	1.7 ± 8.8	-5.2 ± 4.5	11.4 ± 4.1	5.1 ± 11.0				
Total	30.0 ± 1.9	17.6 ± 4.2	21.0 ± 1.9	11.5 ± 1.5	15.5 ± 4.2	15.6 ± 2.6	26.7 ± 6.6				



## **6. CONCLUSIONS**

In this study, a typical procedure involving Soxhlet extraction (dichloromethane) plus saponification was modified to be suitable for achieving a high efficient extraction of PAHs from sewage sludge samples. The purification with activated silica gel flash chromatography was necessary to remove other organic compounds which can interfere with the result of GC-MS analysis.

Biological, chemical and electrochemical treatments have been compared for PAHs removal from prealably doped sewage sludge. The PAHs removal efficiency from sewage sludge by different treatments varies with the properties of different PAHs. The tendency is that low molecular weight PAHs were more easily removed than larger molecule in all treatments.

Excellent results were obtained for 3-ring (ACN, FLU and PHE) PAHs removal by two biological treatments (MAD and METIX-BS) and one combinative treatment MAD + Tw80. The use of Tw80 with MAD enhances the biodegradation rate of low weight PAHs. Moreover, the combination of MAD + Tw80 was the most efficient process for the removal of 4-ring PAHs (PYR) and 5-ring PAHs (BJK), but no gains were observed for the removal of 6-ring PAHs.

The 3-ring PAHs can be also partially removed from sewage sludge by two chemical (METIX-AC and STABIOX) and one electrochemical (ELECSTAB) treatments. However, best conditions and addition of the suitable surfactant in process METIX-AC or ELECSTAB for treatment of PAHs are needed in the future.

Further study on more effective removal of larger PAHs ( $\geq$  4-ring) is still necessary. Biological methods, like the conventional aerobic digestion or the new simultaneous sewage sludge digestion and metal leaching process (METIX-BS/SSDML), seem the most encouraging ways to

### *6. Conclusions*

explore. A combination of chemical and biological treatments could also be an advantageous alternative.

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## 8. ANNEXES

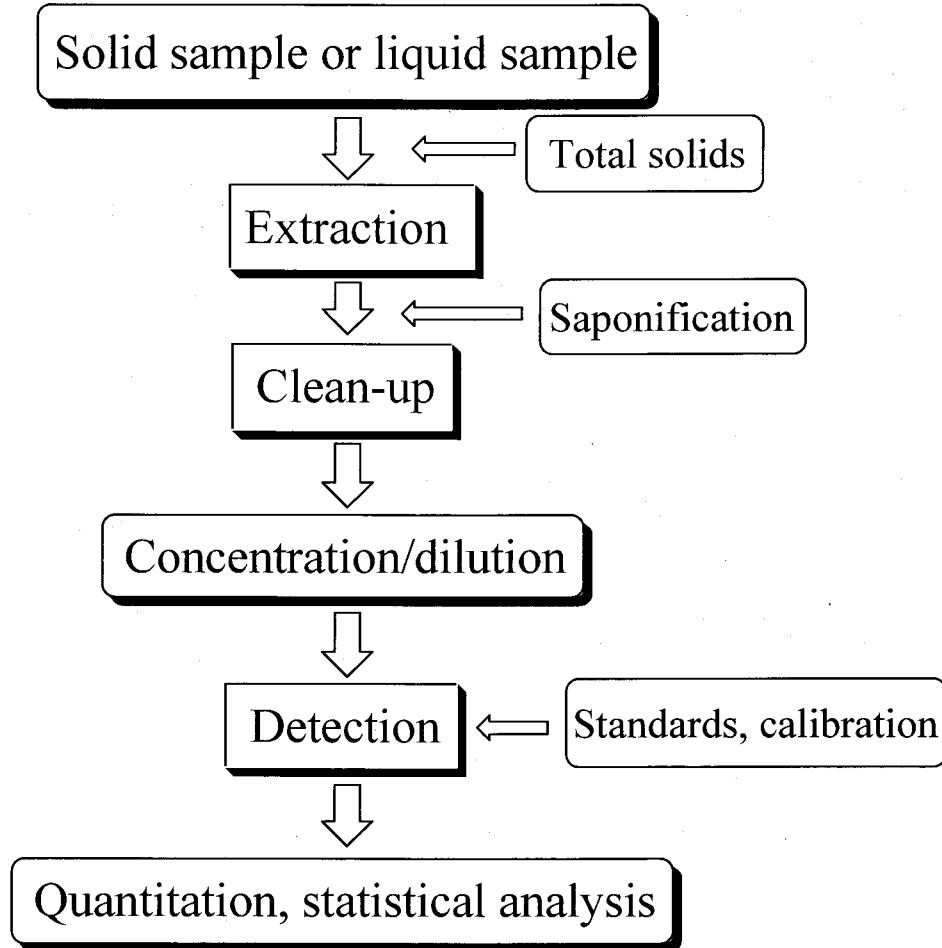
### 8.1. Metal concentrations determination

To determine metal concentrations, the samples were first filtered on Whatman 934-AH membrane (Whatman International Ltd, Maidstone, England) under vacuum, then the filtrates were acidified with concentrated HCl (5% v v<sup>-1</sup>) and kept at 4 °C until analysed. The digestion method of the soil was executed by digesting 0.5 g dry samples in presence of HNO<sub>3</sub>, HF and HClO<sub>4</sub>, in a final solution of 5% HCl (method 3031 I; APHA *et al.*, 1999).

The metal concentrations were determined by plasma emission spectroscopy with a simultaneous ICP-AES (Inductively Coupled Plasma, Varian company, Vista model).

### 8.2. Extractions, purification and analysis of PAHs

Generally, the experiment of extraction, purification and analysis of PAHs after different treatments which mentioned above were designed as shown in Figure 8.1.



**Figure 8.1** Experiments designed for extraction, purification and analysis of PAH

### **8.2.1 Soxhlet extraction plus saponification for solid samples**

#### **8.2.1.1 *Dry weight determination of the solid samples***

Before any extraction of PAHs from solid samples, dry weight must be determined followed this general procedure. A small amount (300–600 mg) of solid sample was put into labeled and pre-weighed aluminum weighing dishes in triplicate. These dishes containing the solid sample were weighed and then placed into a drying oven at 105 °C for 24 h. All of the samples were allowed to cool to room temperature (~ 10 min) in a desiccator before they were weighed. When the sample results are to be calculated on a dry weight basis, the same residual samples should be weighed at the same time as the portion used for extraction in next step.

#### **8.2.1.2 *Soxhlet extraction***

In this study, the PAHs were extracted from sludge samples using a modification of method of

Centre d'expertise en analyse environnementale du Québec (Gouvernement du Québec, 2002).

Dichloromethane was used as solvent for all extractions of PAHs from both solid and liquid samples.

A solution of surrogate standard spiking (as recovery; acenaphtene-d<sub>10</sub> and chrysene-d<sub>12</sub> in iso-octane 100 mg L<sup>-1</sup>, 250 µl) was added to a beaker (200 ml) containing a pre-weighed solid sample (from 1 to 10 g scale) for extraction. The solid sample was then dried with *ca* 10-fold anhydrous MgSO<sub>4</sub> (pre-heated at 650 °C for 12 h in oven before using) with cooling in a cold water bath. The powder-like sample was transferred into a pre-decontaminated cellulose extraction thimble (43 x 123 mm, Advance MFS Inc.) dipped in dichloromethane at least for 24 h before using. The solid sample was Soxhlet extracted (Organamation Glassware with three

flasks (500 ml) Heaters, Electrothermal EME 30500/EX1) for 24 h using 300 ml of dichloromethane at a rate of 4-6 rings  $\text{h}^{-1}$  (MA. 400-HAP 1.1). After the extraction was complete, the extracts were allowed to cool to room temperature and then concentrated to approximately 5 ml by rotary evaporation (Büchi, model Rotavapor-R) under reduced pressure in a water bath at lower than 26 °C.

### 8.2.1.3 *Saponification*<sup>6</sup>

Besides PAHS, various organic compounds coexisted in sludge samples could also be extracted with dichloromethane by Soxhlet extraction. These interfering organic compounds will, when subject to GC-MS analysis, overlap with the targeted PAHs peaks because they have similar or the same retention times on GC capillary column. Of all these organic compounds, triacylglycerols<sup>7</sup> are a major group of interfering esters since it is very difficult to remove these compounds from the extract by silica gel column flash chromatography. To efficiently remove these triacylglycerols from the extract in the clean-up step, it was necessary to transfer them into more polar glycerol and carboxylates by carrying out a saponification step prior to the clean-up.

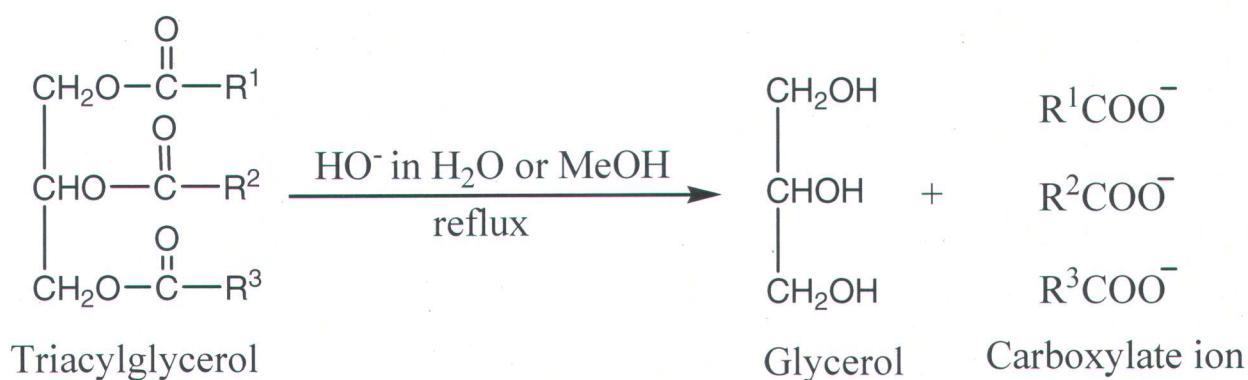
To a solution of potassium hydroxide (0.5 M in methanol, 150 ml) in a flask (500 ml) was charged the extract from the Soxhlet extraction. The mixture was refluxed for 4 h at 80 °C in a

<sup>6</sup> Saponification is a alkaline hydrolysis of the fatty acid esters (triacylglycerols included) to corresponding alcohols and carboxylates. The carboxylate ion produced is very uncreative toward nucleophilic substitution because it is negatively charged. Base-promoted hydrolysis of an ester, as a result, is an essentially irreversible reaction.

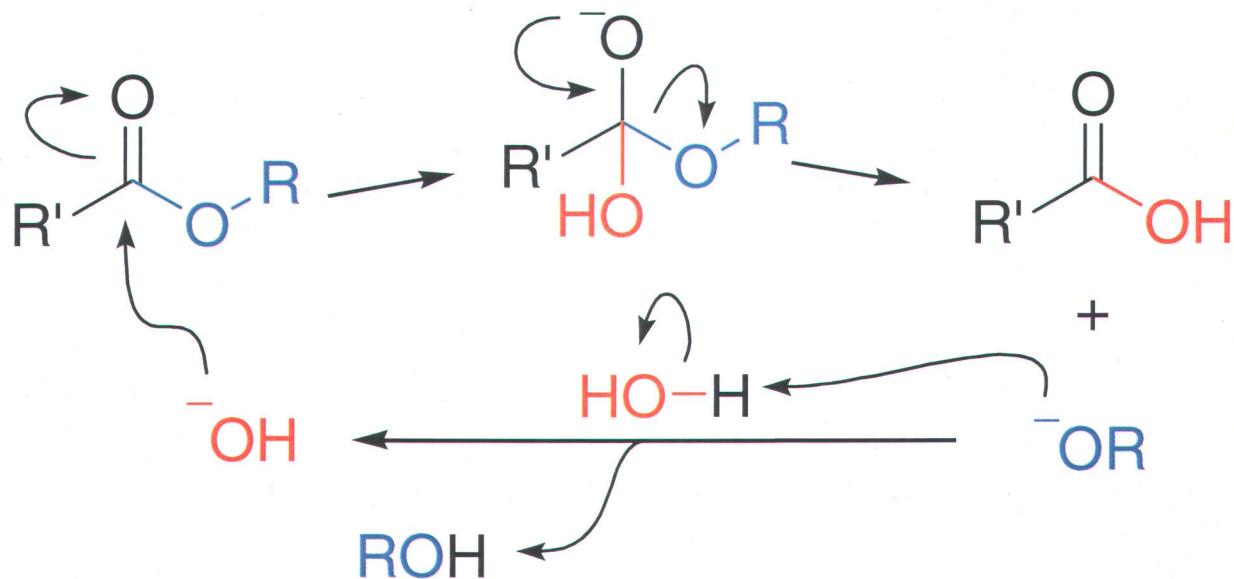
<sup>7</sup> Triacylglycerols are the oils of plants or the fats of animal origin. They include such common substances as peanut oil, soybean oil, butter, lard and tallow etc. They are liquids at room temperature are generally called oil, those that are solids are called fats. These matters can very easily enter sewage sludges.

water-bath (Mangas *et al.*, 1998). After cooling to room temperature, 100 ml of saturated brine in milli-Q-purified water were added. The mixture was exacted with dichloromethane ( $3 \times 50$  ml). The combined organic phase was dried by filtration over anhydrous  $\text{MgSO}_4$  (pre-heated at 650 °C for 12 h in oven before using) and then evaporated to near dryness on a rotary evaporator under reduced pressure at lower than 26 °C. The residue was dissolved in 2 ml hexane for next clean-up. The saponification reaction was shown in Scheme 8.1 and the mechanism was presented in Scheme 8.2.

**Scheme 8.1 Hydrolysis of fats or oils<sup>8</sup>**

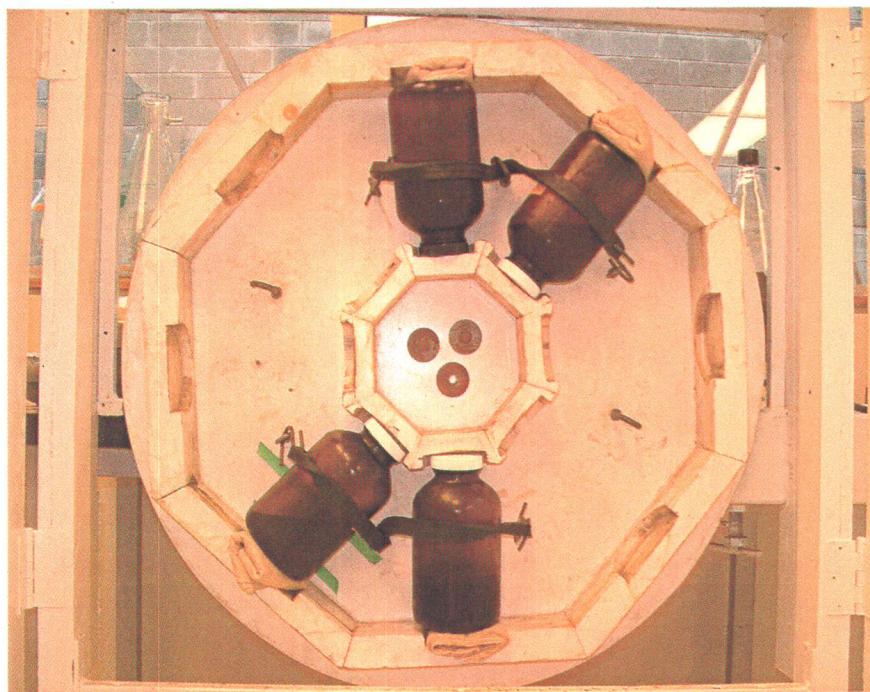


<sup>8</sup> The groups  $\text{R}^1$ ,  $\text{R}^2$  and  $\text{R}^3$  in Scheme 8.1 are usually long-chain alkyl groups containing one or more carbon-carbon double bonds, and may be different from one another.

**Scheme 8.2 Mechanism for base-promoted hydrolysis of lipids**

### 8.2.2 Rotating extraction for liquid samples

After transformation of liquid phase (500 ml) to a pre-decontaminated glass bottle (washed with water and rinsed with acetone), dichloromethane (200 ml) and a solution of recoveries standard (acenaphtene-d<sub>10</sub> and chrysene-d<sub>12</sub> in isoctane 100 mg L<sup>-1</sup>, 100 µl) were added. The liquid sample was extracted by rotating extraction at a rate of 6 rings/min for 24 h at room temperature (Figure 8.2). The separated extracts were then concentrated nearly to dryness by rotary evaporation under reduced pressure (lower than 26 °C), and the residue was dissolved in hexane (1 ml) for the next clean-up step.



**Figure 8.2    Rotating extraction for liquid samples**

### 8.2.3    Clean-up of the PAHs

#### 8.2.3.1    *Identification of PAHs using thin layer chromatography (TLC)*

Thin layer chromatography<sup>9</sup> (TLC, silica gel 60 pre-coated plate, 0.25 mm in thickness) as one of the widely used simple means in identification of organic compounds was used to identify PAHs

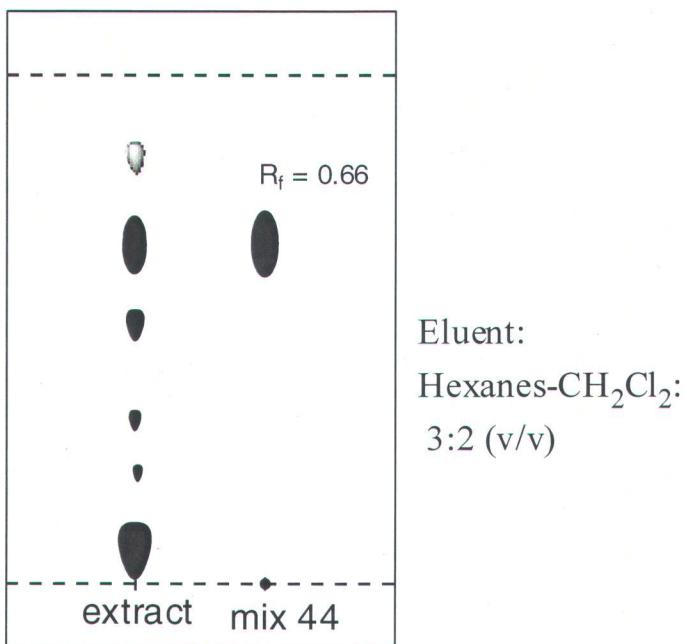
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<sup>9</sup> Thin layer chromatography (TLC) as a one of the powerful analytical tools. It is a simple, quick, and inexpensive that gives us a quick answer as to how many components are in a mixture and it is also used to support the identity of a compound in a mixture when the  $R_f$  of a compound is compared with the  $R_f$  of a known compound (preferably both run on the same TLC plate).

$$R_f = \frac{\text{Distance from start to centre of substance spot}}{\text{Distance from start to solvent front}}$$

in this study, and so as to determine the polarity of the eluent used for the next chromatography clean-up step (Figure 8.3).

In order to obtain an imaginary start line, make two notches with pencil on each side of the TLC plate. The start line should be 0.5 cm from the bottom of the plate. Using a drawn-out capillary tube, spot the samples on the plate so that they line up with the notches you etched and then the plate is developed in hexane-dichloromethane (3:2, v v<sup>-1</sup>) in a development chamber. When the solvent has reached the top of the notch, the plate is removed from the developing chamber, dried, and the separated components of the mixture are visualized with CAM<sup>10</sup> (Ceric-sulfate Ammonium Molybdate).



**Figure 8.3 Identification of PAHs using TLC**

<sup>10</sup> CAM: 4 g CeSO<sub>4</sub> (Ceric sulfate), 75g (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>·4H<sub>2</sub>O (Ammonium Molybdate) in aqueous H<sub>2</sub>SO<sub>4</sub> (10%, 500 ml).

### 8.2.3.2 Purification of PAHs by silica gel flash chromatography

In the clean-up step, the extracts from solid samples, a recovery solution of anthracene-d<sub>10</sub> and pyrene-d<sub>10</sub> (100 mg l<sup>-1</sup> in isoctane, 250 µl), a glass column ( $\phi$  2.5 cm × 40 cm) and a certain quantity (1/6, w<sub>Si</sub>/w<sub>dry solid sample</sub>) activated silica gel were used. One typical procedure was shown below.

Activated silica gel (60 g) was packed in a glass column with a pad of anhydrous magnesium sulfate (*ca* 1 cm) on top for clean-up extract from dry solid sample (10 g). The silica gel was rinsed with hexanes (100 ml) before loading the extract and the recovery onto the column and eluted with hexane-dichloromethane (3:0, 100 ml → 3:2, 150 ml). The second elute containing PAHs was collected and concentrated to approximately dryness by rotary evaporation at lower than 26 °C. Then the purified sample was quantitatively transferred into a 5 ml volumetric flask (2 ml for liquid samples) with isoctane. The volumetric flask was filled to final volume with isoctane. The flask was stopped, swirled and inverted several times to ensure thorough mixing of the solution. The purified sample in isoctane must be protected from light and stored in sealed amber vials at  $\leq -10$  °C until analysis.

### 8.2.4 Quantitative analysis of PAHs by GC-MS

#### 8.2.4.1 Conditions of the instruments

PAHs were quantified using GC-MS (Perkin Elmer, model Clarus 500, with column type of VF-5MS FS, 30 m × 0.25 mm × 0.25 µm) at Institut National de la Recherche Scientifique (INRS) and GC-MS-SIM (selected ion monitoring; Hewlett Packard 6890 series GC equipped with a HP 5973A series mass spectrometer detector; with column type of DB-5, 30 m × 0.25 mm ×

0.25 µm) at Bodycote (Québec, Québec). The temperature programs involved are listed in Table 8.1.

**Table 8.1      Optimized conditions for the analysis of PAHs by GC-MS**

GC-MS	Characteristics	INRS	Bodycote
Injector	Temperature (°C)	200	250
	Mode	spitless	spitless
	Purge (min)	1.0	1.0
	EPC Const Flow	ON	ON
	Flow (ml min <sup>-1</sup> )	1.1	1.1
Column	Volume for injection (µl)	1	1
	Temperature (°C)	80 (2 min)	75 (1 min)
	Program 1 (°C min <sup>-1</sup> )	15	20
	Temperature (°C)	220 (0 min)	220 (0 min)
	Program 2 (°C min <sup>-1</sup> )	5	30
Detector	Temperature (°C)	320 (5 min)	280 (0 min)
	Program 3 (°C min <sup>-1</sup> )	-	10
	Temperature (°C)	-	325 (5.25 min)
	Gas vector	Helium	Helium
	Flux (ml min <sup>-1</sup> )		1.1
	Type	MS	MS
	Temperature (°C)	280	280
	Solvent Delay (min)	4.00	4.00
	Acquiring mode	SIM	SIM

#### **8.2.4.2      Preparation of sample solutions and calibration standards**

##### **8.2.4.2.1    Preparation of sample solutions**

The internal standards (25 mg l<sup>-1</sup>, 50 µl) were added to each sample (450 µl). Each purified sample from the solid samples in isoctane were prepared in triplicate for GC-MS analysis as shown in Table 8.2: (F<sub>1</sub>), the purified sample in isoctane (450 µL) was measured directly; (F<sub>2</sub>),

the purified sample in isoctane (225 µl) was diluted with isoctane (225 µl) and, ( $F_5$ ) the purified sample in isoctane (90 µl) was diluted with isoctane (360 µl). Each purified sample from the liquid samples in isoctane was prepared similarly to that of the solid samples in duplicate as shown in Table 8.2: ( $F_1$ ) and ( $F_2$ ).

**Table 8.2 Preparation of sample solutions**

Sample	Factor of dilution ( $F$ )	Purified sample (µl)	Isooctane (µl)	25 mg L <sup>-1</sup> Internal standards (µl)
Solid	1 ( $F_1$ )	450	0	50
	2 ( $F_2$ )	225	225	50
	5 ( $F_5$ )	90	360	50
Liquid	1 ( $F_1$ )	450	0	50
	2 ( $F_2$ )	225	225	50

#### 8.2.4.2.2 Preparation of calibration standards

The standard solutions were prepared in volumetric flask (100 mg l<sup>-1</sup>, 5 ml) as below in Table 8.3 and stored at  $\leq -10$  °C. They must be prepared and used within 6 months.

**Table 8.3 Preparation of standard solutions**

PAH	Mix 44	B(J)F	Anthracene-d <sub>10</sub>	PYR-d <sub>10</sub>	ACN-d <sub>10</sub>	Chrysene-d <sub>12</sub>	Isooctane
[Conc] <sub>i</sub> *	1000	2000	1000	1000	1000	1000	-
(mg L <sup>-1</sup> )							
Volume (μl)	500	250	500	500	500	500	~ 4250
[Conc] <sub>f</sub>	100	100	100	100	100	100	-
(mg L <sup>-1</sup> )							

\*solutions in CH<sub>2</sub>Cl<sub>2</sub>-benzene (1:1, v v<sup>-1</sup>).

The quantitations are accomplished by comparing the response of a major (quantitative) ion relative to an internal standard (naphthalene-d<sub>8</sub> and phenanthrene-d<sub>10</sub>) using a five-point calibration curve for each target PAH. The calibration standards were prepared at 0 (blank), 0.1, 0.5, 1.0, 2.5 and 5.0 mg L<sup>-1</sup> from the standard solution (100 mg L<sup>-1</sup>). The internal standards (25 mg L<sup>-1</sup>, 50 μl) were added to each standard sample (450 μl) to result a mid-level (2.5 mg L<sup>-1</sup>) standard solution for calibration concluding the analysis sequence (Table 8.4). The calibration verification standards should be prepared weekly and stored at ≤ 4°C.

The concentrations based on individually resolved peaks were summed to obtain the total PAHs concentration. Standards and samples are warmed to room temperature prior to the injection for GC-MS analysis. Blank, calibration standards and the samples are injected sequentially.

**Table 8.4 Preparation of calibration standard solutions**

Calibration standards (mg L <sup>-1</sup> )	5 mg/L standards (µl)	Isooctane (µl)	25 mg/L internal standards (µl)
5.0	450	0	50
2.5	225	225	50
1.0	90	360	50
0.5	45	405	50
0.1	9	441	50
0	0	450	50

The calibration curves were constructed for all target compounds analyzed in the samples. When the conditions of instrument (GC-MS) were changed, a calibration verification of the curve is necessary.

#### **8.2.4.3 Measurement of limits of detection (LD)<sup>11</sup>**

LD of GC-MS analysis in SIM mode were obtained (Table 8.5) allowing to illustrate the lowest concentration of each monitored PAHs presented in the sewage sludge samples.

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<sup>11</sup> The limits of detection (LD) are estimates of concentrations at which we can be fairly certain that the compound is present. They are usually determined on "clean" samples. Concentrations below this limit may not be detected. Concentrations above this limit are almost certainly detected in the analysis. Using statistics, the certainty of detection can be quantified as 99%. Therefore, "not detected" indicates that the analyte may be present below the LD.

**Table 8.5 Target compounds and their parameters**

PAH	Nature	Ions of quantification (m/z)	Retention time (min)	Limits of detection		Observe windows (min)
				Solid sludge mg kg <sup>-1</sup>	Liquid sludge (mg L <sup>-1</sup> )	
Naphthalene-d <sub>8</sub>	Internal standard	134, 136	5.30	-	-	4'1-6'8
Acenaphtene-d <sub>10</sub>	Recovery	162, 164	8.13	-	-	6'8-8'5
Acenaphtene	PAH	153, 154	8.17	0.055	0.013	
Fluorene	PAH	165, 166	9.02	0.091	0.022	8'5-10'3
Phenanthrene-d <sub>10</sub>	Internal standard	184, 188	10.47	-	-	
Phenanthrene	PAH	176, 178	10.51	0.091	0.022	10'2-11'0
Anthracene-d <sub>10</sub>	Recovery	184, 188	10.55	-	-	
Fluoranthene	PAH	200, 202	12.57	0.116	0.028	
Pyrene-d <sub>10</sub>	Recovery	208, 212	12.99	-	-	12'4-13'2
Pyrene	PAH	200, 202	13.04	0.127	0.031	
Chrysene-d <sub>12</sub>	Recovery	236, 240	16.86	-	-	16'5-17'5
Benzo(b)fluoranthene	PAH	250, 252	21.57	0.119	0.028	
Benzo(j)fluoranthene	PAH	250, 252	21.57	0.119	0.028	21'4-22'5
Benzo(k)fluoranthene	PAH	250, 252	21.66	0.119	0.028	
Benzo(a)pyrene	PAH	126, 250, 252	23.03	0.125	0.030	22'5-23'5
Indeno(1,2,3-cd)pyrene	PAH	138, 274, 276	28.59	0.163	0.039	28'0-30'0
Benzo(ghi)perylene	PAH	138, 274, 276	29.72	0.272	0.065	

### 8.2.5 Calculation of the PAHs concentrations in samples

The result of analysis was obtained by using the method of internal standard. Relative response factors (*RRFs*) for each analyte are calculated from the calibration standard peak areas using equation (3):

$$RRF_{std} = \left( \frac{C_a}{X_a} \right)_{std} \div \left( \frac{C_{istd}}{X_{istd}} \right)_{std} \quad (3)$$

Where:

$C_a$ : The known concentration of calibration standard injected ( $\text{mg l}^{-1}$ )

$X_a$ : The calibration standard's peak area

$C_{istd}$ : The known concentration of the appropriate internal standard ( $\text{mg l}^{-1}$ )

$X_{istd}$ : The internal standard's peak area

The concentration of each PAHs contained in dose sample was checked by internal standards and can be calculated from the  $RRF_{std}$  and the internal standard response in the sample by the following equation (4). The quantitation report can be calculated by the software kit in the GC-MS workstation. The data files and quantitation results can be stored automatically.

$$A = X_{sample} \times RRF_{std} \times \left( \frac{C_{istd}}{X_{istd}} \right)_{sample} \quad (4)$$

Where:

$A$ : The concentration of each target PAHs contained in sample ( $\text{mg l}^{-1}$ );

$X_{sample}$ : The peak area of each target PAH's in the sample;

$C_{std}$ : The concentration of internal standard spiked into the sample;

$X_{std}$ : The internal standard's peak area in the sample.

The concentration of PAHs in  $\text{mg l}^{-1}$  was calculated by the followed equation:

$$c = \frac{A \times v}{V} \quad (5)$$

Where:

$c$ : The concentration of each target PAHs contained in sewage sludge ( $\text{mg l}^{-1}$ );

$A$ : The concentration of each PAHs contained in dose sample ( $\text{mg l}^{-1}$ , a value can be calculated by GC-MS workstation);

$v$ : The total volume of the sample solution for GC-MS after dilution (450  $\mu\text{l}$ );

$V$ : The volume of the sample from purified sample solution for GC-MS before dilution ( $\mu\text{l}$ ).

$$\frac{v}{V} = \text{the factor of dilution } (F) \quad (6)$$

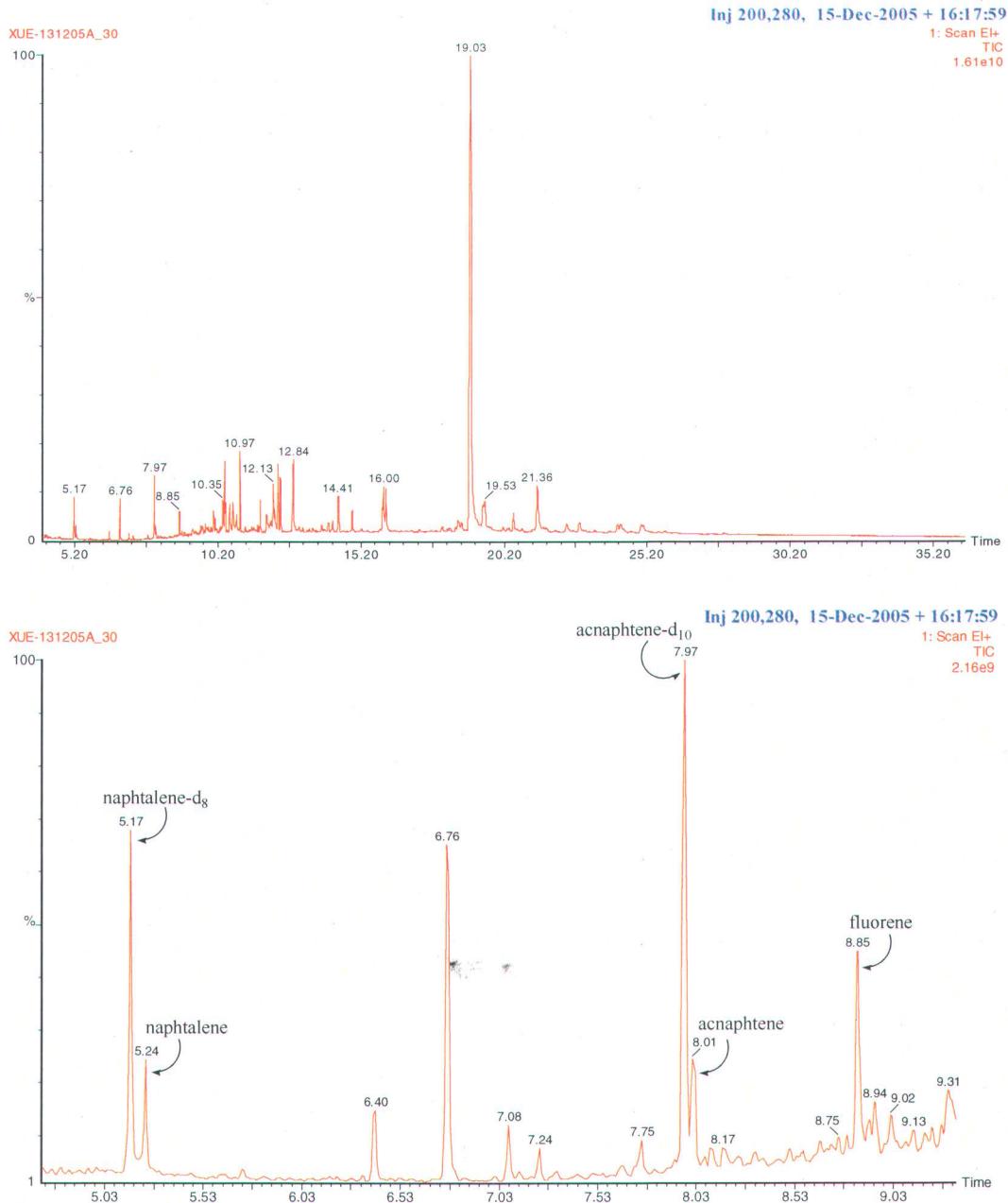
At last, the value of concentration ( $\text{mg l}^{-1}$ ) changed the dimension with  $\text{mg/kg dry matter}$ :

$$C = \frac{c \times V_0}{m} \quad (7)$$

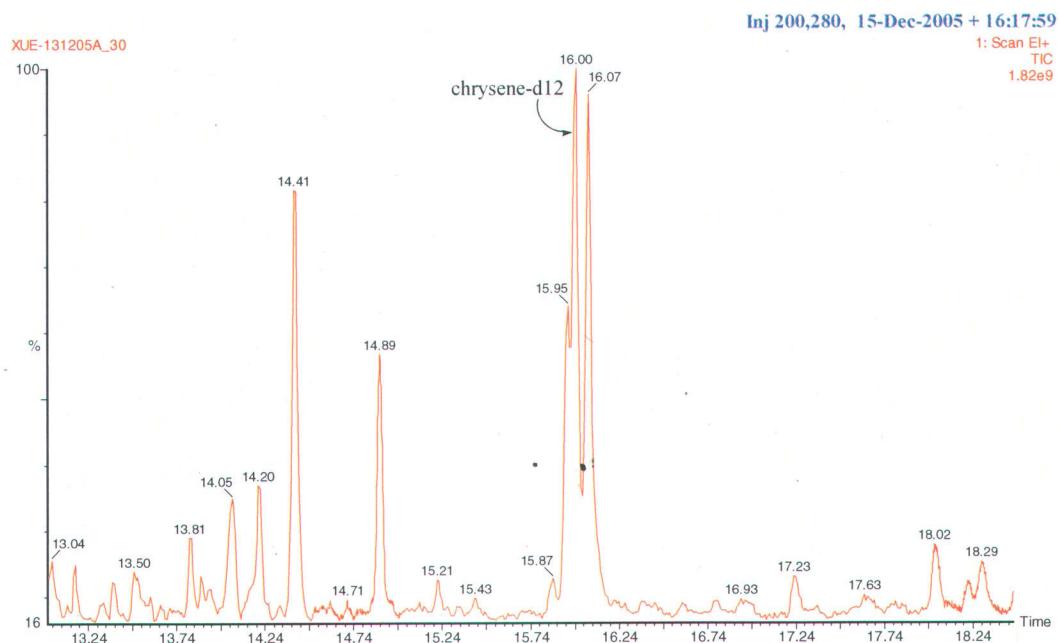
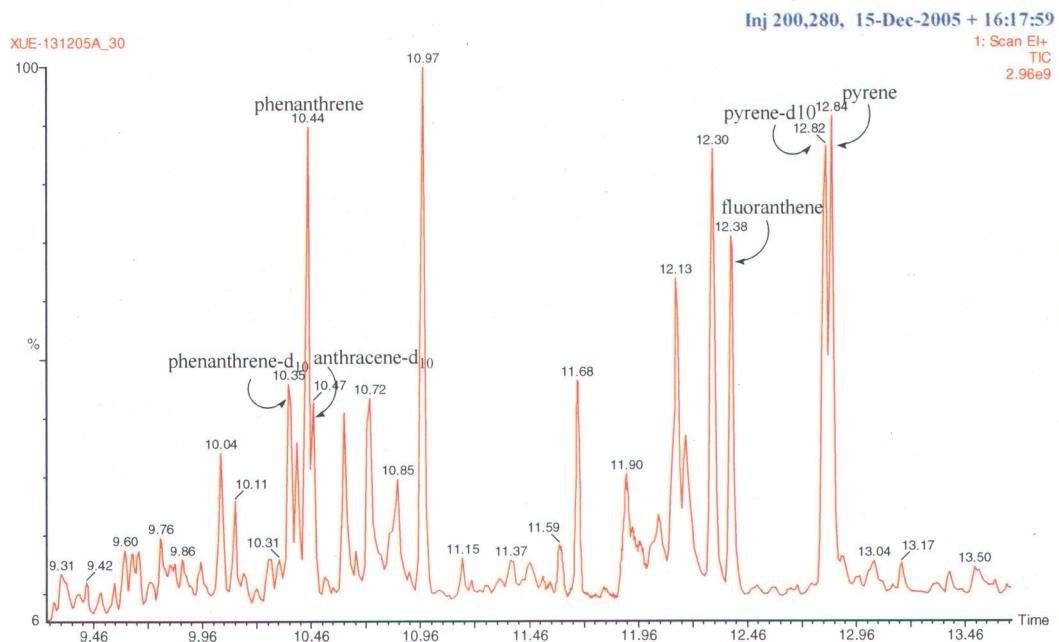
Where:

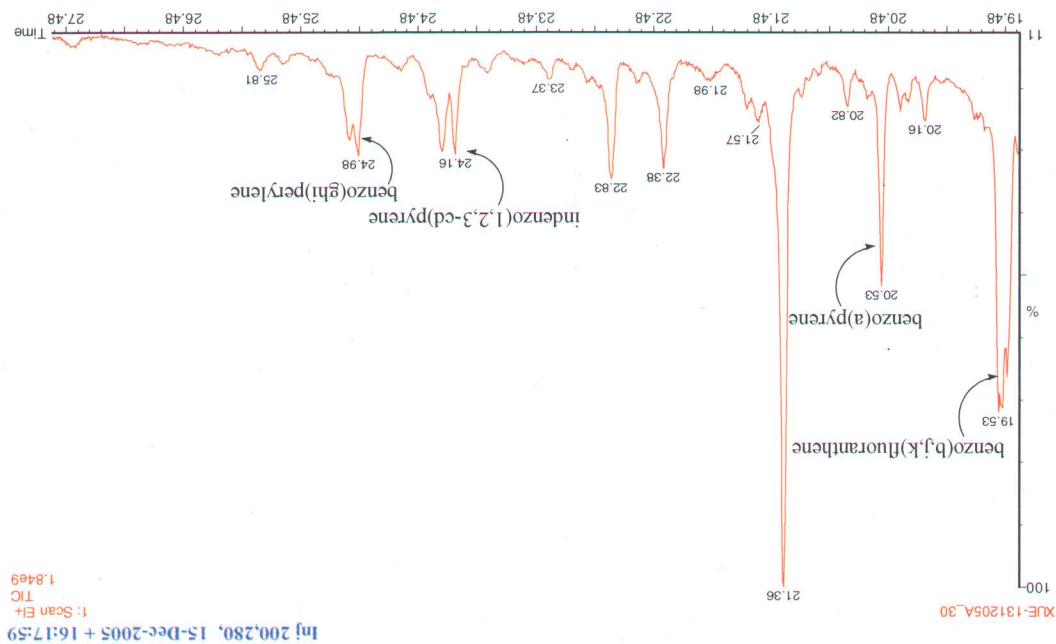
- C: The concentration of each PAHs contained in sewage sludge ( $\text{mg kg}^{-1}$ );
- c: The concentration of each target PAHs contained in sewage sludge ( $\text{mg L}^{-1}$ );
- $V_0$ : The volume of the sample concentrated after clean-up (solid: 5 ml, liquid: 2 ml);
- m: The mass of the total solids in each sludge sample (g) for Soxhlet extraction.

### 8.3. Chromatography of GC-MS for the extract



8. Annexes





#### **8.4. Results of the PAHs removal assays from sewage sludge**



Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Total (II HAP)						Enlèvement (%)	
						initial	final	initial	final	A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
Contrôle non-dope et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	3,765	3,630	3,286	3,560	0,247	6,95%	8,91%	1,17%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	54,940	57,997	51,368	54,768	3,318	6,06%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	33,519	33,559	33,595	33,558	0,038	0,11%	38,73%	0,07%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	33,705	35,627	36,474	35,269	1,419	4,02%	35,60%	2,59%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	46,470	43,651	47,912	46,011	2,167	4,71%	15,99%	3,96%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	50,613	49,903	49,652	49,889	0,639	1,28%	8,91%	1,17%
ELECTSTAB	ELECTSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	51,220	49,577	30,912	50,570	0,873	1,73%	7,67%	1,59%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					44,769	43,704	46,163	44,879	1,233	2,75%	30,03%	1,92%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					49,773	54,952	53,738	52,814	2,701	5,11%	17,66%	4,21%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	50,483	49,500	51,948	50,643	1,231	2,43%	21,04%	1,92%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					55,822	56,594	57,706	56,707	0,947	1,67%	11,59%	1,48%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					52,939	52,251	57,219	54,136	2,692	4,97%	15,60%	4,20%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					42,417	47,617	50,835	46,956	4,248	9,05%	26,79%	6,62%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	47,875	52,161	54,236	51,424	3,244	6,31%	19,83%	5,06%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					51,425	54,297	55,839	53,854	2,240	4,16%	16,04%	3,49%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					52,775	59,789	56,795	56,453	3,520	6,23%	11,99%	5,49%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	52,429	55,748	54,049	54,075	1,660	3,07%	15,69%	2,59%
Metic-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	55,939	55,062	53,998	54,999	0,973	1,77%	14,25%	1,52%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	64,295	63,607	64,519	64,140	0,476	0,74%	0,00%	0,74%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	62,540	63,398	64,332	63,590	0,935	1,47%	0,86%	1,46%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	55,120	57,099	59,847	57,356	2,374	4,14%	10,58%	3,70%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58	56,627	54,811	53,866	55,101	1,404	2,55%	14,09%	2,19%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,08	5			53,289	50,389	52,609	52,096	1,517	2,91%	18,78%	2,36%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	46,417	45,107	40,767	44,097	2,957	6,71%	31,25%	4,61%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	40,683	37,917	35,233	37,944	2,725	7,18%	40,84%	4,25%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	34,683	32,928	32,235	33,282	1,262	3,79%	48,11%	1,97%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	34,087	33,572	34,277	33,979	0,365	1,07%	47,02%	0,57%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	45,604	45,899	44,134	45,212	0,946	2,09%	29,51%	1,47%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	41,417	38,300	37,800	39,172	1,580	5,00%	38,92%	3,69%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	39,233	35,754	34,643	35,877	2,941	8,20%	44,07%	4,59%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	353	33,984	31,317	30,589	31,963	1,787	5,59%	50,17%	2,79%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	31,167	31,326	29,185	30,559	1,194	3,91%	52,36%	1,86%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	57,235	56,904	58,924	57,688	1,083	1,88%	10,06%	1,69%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	51,417	49,144	48,752	49,771	1,439	2,89%	22,40%	2,24%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	47,816	43,444	45,034	45,433	2,213	4,87%	29,17%	3,45%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	36,230	33,198	35,050	34,826	1,528	4,39%	45,70%	2,38%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	35,390	33,338	31,153	33,294	2,119	6,36%	48,09%	3,30%

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		HAP à 3 cycles (ACN, FLU, PHE)							
						initial	final	initial	final	Concentration - fraction solide (mg/kg)			Enlèvement (%)				
										A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,308	0,286	0,276	0,290	0,016	5,59%		
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	13,487	13,871	12,861	13,406	0,510	3,80%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	0,403	0,404	0,431	0,413	0,016	3,79%	96,92%	0,12%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	0,554	0,648	0,669	0,624	0,061	9,80%	95,35%	0,46%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	8,162	8,134	8,456	8,251	0,179	2,16%	38,45%	1,33%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	11,292	11,032	10,833	11,052	0,230	2,08%	17,56%	1,72%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	6,396	5,735	6,153	6,095	0,335	5,49%	54,54%	2,50%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					4,566	4,400	4,542	4,503	0,090	1,99%	73,17%	0,53%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					9,933	10,550	10,125	10,203	0,316	3,09%	39,21%	1,88%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6					10,908	11,183	11,125	11,072	0,145	1,31%	34,03%	0,86%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					10,500	10,783	10,000	10,428	0,397	3,80%	37,87%	2,36%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					9,067	8,833	9,042	8,981	0,128	1,43%	46,50%	0,76%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					2,108	2,150	2,292	2,183	0,096	4,40%	86,99%	0,57%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2					6,850	6,517	6,583	6,650	0,176	2,65%	60,38%	1,05%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					9,817	10,250	10,292	10,119	0,263	2,60%	39,71%	1,57%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					10,508	10,416	10,500	10,475	0,051	0,49%	37,59%	0,30%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	12,286	11,610	10,786	11,561	0,751	6,50%	31,12%	4,47%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	12,825	12,796	12,361	12,661	0,260	2,05%	24,57%	1,55%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	16,831	16,745	16,778	16,785	0,043	0,26%	0,00%	0,26%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	16,857	16,033	16,438	16,443	0,412	2,51%	2,04%	2,46%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	12,963	13,354	13,458	13,258	0,261	1,97%	21,01%	1,56%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	94	6,657	6,826	7,184	6,859	0,310	4,52%	59,14%	1,85%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	4,000	3,833	3,600	3,844	0,214	5,57%	77,10%	1,28%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	2,517	2,417	2,400	2,444	0,063	2,58%	85,44%	0,38%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	0,983	1,417	1,367	1,256	0,237	18,88%	92,52%	1,41%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	7,11	7,16	-79	-58	12,651	12,042	11,444	12,046	0,603	5,01%	28,23%	3,59%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	94	6,657	6,826	7,184	6,859	0,310	4,52%	59,14%	1,85%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	3,000	2,725	2,584	2,770	0,212	7,64%	83,50%	1,26%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	1,750	1,467	1,633	1,617	0,142	8,81%	90,37%	0,85%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	1,400	1,350	1,333	1,361	0,035	2,55%	91,89%	0,21%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	1,150	1,158	1,125	1,144	0,017	1,50%	93,18%	0,10%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	1,050	1,091	0,929	1,023	0,084	8,25%	93,90%	0,50%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	11,675	11,033	12,126	11,611	0,549	4,73%	30,82%	3,27%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	9,667	9,000	8,667	9,111	0,509	5,58%	45,72%	3,03%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	9,286	8,167	8,594	8,682	0,565	6,50%	48,27%	3,36%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	0,778	0,698	0,717	0,731	0,042	5,69%	95,65%	0,25%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	0,723	0,672	0,653	0,683	0,036	5,32%	95,93%	0,22%

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		HAP à 4 cycles (FLR, PYR)						Enlèvement (%)	
						initial	final	initial	final	Concentration - fraction solide (mg/kg)			Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
										A	B	C					
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	1,045	1,041	0,987	1,024	0,933	3,18%	0,00%	0,00%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	9,026	10,014	8,436	9,159	0,796	8,70%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	5,024	5,314	5,115	5,151	0,148	2,88%	43,76%	1,62%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	7,639	7,473	8,187	7,766	0,374	4,81%	15,21%	4,08%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	7,085	6,731	8,749	7,522	1,077	14,32%	17,86%	11,76%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	8,976	8,881	9,033	8,963	0,077	0,66%	2,14%	0,84%
ELECTSTAB	ELECTSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	11,964	10,620	11,503	11,362	0,683	6,01%	-24,05%	7,46%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					9,550	10,117	10,167	9,944	0,343	3,44%	17,23%	2,85%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					9,917	11,250	11,625	10,931	0,698	8,21%	9,02%	7,47%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	10,883	10,947	9,545	10,459	0,792	7,57%	12,95%	6,59%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					11,050	12,300	12,333	11,894	0,732	6,15%	1,00%	6,09%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					10,258	11,533	12,083	11,292	0,936	8,29%	6,02%	7,79%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					9,667	10,250	10,583	10,167	0,464	4,56%	15,38%	3,86%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	10,225	10,983	11,500	10,903	0,641	5,88%	9,26%	5,34%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					10,175	11,283	11,458	10,972	0,696	6,34%	8,66%	5,79%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					10,633	11,183	9,917	10,578	0,635	6,00%	11,96%	5,29%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	10,857	11,122	10,821	10,933	0,164	1,50%	9,00%	1,37%
Metic-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	12,285	10,536	10,771	11,197	0,949	8,48%	6,81%	7,90%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	12,138	12,029	11,877	12,015	0,131	1,09%	0,00%	1,09%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	12,286	13,043	13,356	12,895	0,550	4,27%	-7,33%	4,58%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	11,420	11,801	13,333	12,185	0,103	8,31%	-1,41%	8,43%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	7,11	7,16	-79	-58	11,145	10,733	10,741	10,873	0,235	2,17%	9,51%	1,96%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	11,331	11,164	11,017	11,171	0,157	1,41%	7,03%	1,31%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	10,700	11,200	9,667	10,522	0,782	7,43%	12,42%	6,51%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,69	5	275	8,167	10,500	7,500	8,722	1,575	18,06%	27,40%	13,11%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	6,200	4,833	5,000	5,344	0,746	13,95%	55,52%	6,21%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	5,200	5,314	5,115	5,210	0,100	1,92%	56,64%	0,83%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	12,071	11,533	11,033	11,546	0,519	4,50%	3,90%	4,32%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	9,833	8,633	9,000	9,222	0,536	5,81%	23,24%	4,48%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	8,667	7,500	7,500	7,889	0,674	8,54%	34,34%	5,61%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	4,833	4,737	4,464	4,678	0,191	4,09%	61,06%	1,59%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	3,183	2,909	3,143	3,078	0,148	4,81%	74,38%	1,23%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	11,725	11,854	11,823	11,801	0,067	0,57%	1,78%	0,56%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	11,333	11,167	10,667	11,056	0,347	3,14%	7,98%	2,89%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	10,714	10,500	9,688	10,301	0,542	5,26%	14,27%	4,51%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	7,937	9,000	9,500	8,812	0,798	9,08%	26,66%	6,65%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	8,167	8,167	8,000	0,289	3,61%	33,42%	2,40%	

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		HAP à 5 cycles (BJK, BAP)					
						initial	final	initial	final	Concentration - fraction solide (mg/kg)			Enlèvement (%)		
										A	B	C	Moyenne	Écart-type	C.V. (%)
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	1,423	1,375	1,162	1,320	0,139	10,51%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	20,835	22,140	19,750	20,908	1,197	5,72%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	16,859	17,842	18,687	18,463	0,544	2,96%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	14,881	16,625	17,304	16,270	1,250	7,68%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	18,848	18,710	19,734	19,097	0,555	2,91%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	19,651	18,501	18,561	18,904	0,647	3,42%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	21,686	22,732	22,712	22,376	0,598	2,67%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					22,056	18,368	20,014	20,146	1,847	9,17%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					18,852	20,644	21,784	20,426	1,478	7,24%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6					17,958	18,453	20,686	19,032	1,453	7,63%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					23,524	21,206	23,796	22,842	1,423	6,23%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					22,324	20,409	23,766	22,166	1,684	7,60%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					20,117	23,233	26,500	23,283	3,192	13,71%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2					20,317	23,450	24,963	22,910	2,370	10,34%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					20,450	21,250	22,200	21,300	0,876	4,11%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					20,817	24,957	24,151	23,308	2,195	9,42%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	19,143	21,713	21,439	20,765	1,411	6,80%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	20,736	20,225	20,333	20,431	0,270	1,32%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	23,370	23,395	23,130	23,298	0,146	0,63%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	22,266	23,641	23,288	23,072	0,703	3,05%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	20,679	21,429	22,083	21,397	0,703	3,28%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58	21,386	20,681	20,741	20,936	0,391	1,87%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	23,675	21,584	23,767	23,008	1,235	5,37%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	21,567	20,273	18,833	20,224	1,367	6,76%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	20,000	16,500	16,833	17,778	1,932	10,87%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	18,000	17,500	17,000	17,500	0,500	2,86%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	17,800	17,542	18,487	17,943	0,488	2,72%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	20,533	20,500	20,217	20,417	0,174	0,85%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	19,167	18,667	18,167	18,667	0,500	2,68%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	19,333	16,167	16,833	17,444	1,669	9,57%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	18,000	16,316	15,714	16,677	1,185	7,10%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	15,667	16,061	14,464	15,397	0,832	5,40%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	21,733	22,767	23,342	22,614	0,815	3,61%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	20,333	19,167	19,556	19,674	3,44%	16,06%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	18,143	16,000	17,031	17,058	1,072	6,28%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	18,095	15,333	16,500	16,643	1,386	8,33%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	17,667	16,000	15,167	16,278	1,273	7,82%

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		HAP à 6 cycles (INP, BPR)						Enlèvement (%)	
						initial	final	initial	final	Concentration - fraction solide (mg/kg)			Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
										A	B	C					
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,980	0,929	0,861	0,926	0,065	6,98%	0,00%	0,00%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	11,592	11,973	10,319	11,295	0,866	7,67%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	9,233	9,068	9,363	9,531	0,409	4,30%	15,61%	3,63%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	10,632	10,881	10,314	10,609	0,284	2,68%	6,07%	2,52%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	12,374	10,076	10,973	11,141	1,158	10,40%	1,36%	10,26%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	10,694	10,989	11,225	10,969	0,266	2,42%	2,88%	2,35%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	11,174	10,491	10,545	10,736	0,380	3,54%	4,94%	3,38%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					8,597	10,619	11,440	10,285	1,495	14,54%	14,55%	12,41%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					11,071	12,489	10,204	11,255	1,153	10,25%	6,54%	9,58%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	10,733	8,917	10,591	10,080	1,010	10,02%	16,30%	8,39%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					10,749	12,305	11,577	11,543	0,779	6,75%	4,15%	6,47%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					11,290	11,475	12,328	11,698	0,554	4,73%	2,86%	4,60%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					10,525	11,983	11,460	11,323	0,739	6,62%	5,98%	6,13%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	10,483	11,211	11,189	10,961	0,414	3,78%	8,98%	3,44%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					10,983	11,513	11,890	11,462	0,455	3,97%	4,82%	3,78%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					10,816	13,232	12,227	12,092	1,214	10,04%	-0,41%	10,08%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	10,143	11,303	11,003	10,816	0,602	5,57%	10,18%	5,00%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	10,093	11,506	10,531	10,710	0,723	6,75%	11,07%	6,01%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	11,956	11,438	12,734	12,043	0,652	5,42%	0,00%	5,42%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	11,111	11,180	11,250	11,180	0,669	6,62%	7,16%	0,58%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	10,059	10,515	10,972	10,515	0,457	4,34%	12,68%	3,79%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	7,11	7,16	-79	-58	11,447	11,355	10,940	11,247	0,270	2,40%	6,60%	2,24%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	11,717	10,816	10,641	11,058	0,577	5,22%	8,18%	4,79%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	10,150	9,700	8,667	9,506	0,761	8,00%	21,07%	6,32%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	10,000	8,500	9,000	8,666	9,62%	25,27%	7,19%	
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	9,500	9,178	8,868	9,182	0,316	3,44%	23,75%	2,62%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	10,667	10,311	10,245	10,408	0,227	2,18%	13,58%	1,88%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	10,000	11,141	10,300	10,480	0,592	5,65%	12,97%	4,91%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	10,667	9,333	9,000	9,667	0,882	9,12%	19,73%	7,32%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	9,833	8,737	8,976	9,182	0,577	6,28%	23,75%	4,79%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	10,000	9,107	9,286	9,464	0,473	5,00%	21,41%	3,53%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	11,267	11,285	10,647	11,060	0,357	3,23%	8,16%	2,97%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	12,102	11,250	11,633	11,662	0,427	3,66%	3,16%	3,54%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	10,083	9,810	10,252	10,048	0,223	2,22%	16,56%	1,85%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	9,673	8,778	9,725	9,392	0,533	5,67%	22,01%	4,42%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	9,421	8,167	8,333	8,840	0,681	7,88%	26,25%	5,65%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	8,833	8,500	7,667	8,333	0,601	7,21%	30,80%	4,98%

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Acénaphthène (ACN)							
						initial	final	initial	final	Concentration - fraction solide (mg/kg)			Enlèvement (%)				
										A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,035	0,036	0,036	0,036	0,000	1,14%		
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	4,265	4,261	4,450	4,325	0,108	2,50%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	0,075	0,090	0,080	0,082	0,008	9,48%	98,11%	0,18%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	5,57	-277	398	0,093	0,103	0,108	0,101	0,008	7,76%	97,66%	0,18%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	2,566	1,815	2,310	2,230	0,382	17,12%	48,43%	8,83%
STABIOX	STABIOX	Centrifugation	0	18	133	6,78	4,00	-41	290	3,688	3,606	3,532	3,609	0,078	2,16%	16,55%	1,81%
ELECTAB	ELECTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	1,716	1,650	1,727	1,698	0,042	2,45%	60,74%	0,96%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					1,233	1,217	1,208	1,219	0,013	1,04%	77,57%	0,23%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					3,375	3,417	3,375	3,386	0,024	0,71%	38,78%	0,43%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6					3,667	3,667	3,667	3,667	0,000	0,00%	33,77%	0,00%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					3,392	3,450	3,208	3,350	0,126	3,77%	39,49%	2,28%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					2,883	2,783	2,917	2,861	0,069	2,43%	48,32%	1,25%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					0,458	0,483	0,542	0,494	0,043	8,66%	91,07%	0,77%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2					2,075	1,900	1,917	1,964	0,097	4,92%	64,52%	1,74%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					2,900	3,000	3,125	3,008	0,113	3,75%	45,66%	2,04%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					3,342	3,333	3,958	3,544	0,359	10,12%	35,98%	6,48%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	3,714	3,500	3,321	3,512	0,197	5,61%	36,57%	3,56%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	3,842	3,892	3,532	3,755	0,195	5,19%	32,16%	3,52%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	5,535	5,531	5,542	5,536	0,006	0,10%	0,00%	0,10%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	5,429	5,163	5,137	5,243	0,161	3,08%	5,30%	2,91%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	3,704	4,037	3,667	3,803	0,204	5,37%	31,31%	3,69%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	-5	-58	3,916	3,665	3,370	3,650	0,273	7,48%	34,06%	4,93%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	-5	-96	1,633	1,787	1,917	1,772	0,142	8,02%	67,90%	2,57%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	-5	-225	0,950	0,900	0,817	0,889	0,067	7,56%	83,94%	1,22%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	-5	-361	0,217	0,333	0,300	0,283	0,060	21,21%	94,88%	1,09%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	-5	-440	0,100	0,090	0,080	0,090	0,010	11,10%	98,37%	0,18%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	-5	-106	0,408	0,358	0,342	0,369	0,034	9,32%	93,33%	0,62%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	-5	-153	0,267	0,250	0,300	0,272	0,025	9,35%	95,08%	0,46%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	-5	-190	0,217	0,200	0,200	0,206	0,010	4,68%	96,29%	0,17%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	-5	-333	0,200	0,193	0,214	0,202	0,011	5,36%	96,34%	0,20%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	-5	-446	0,167	0,167	0,161	0,165	0,003	2,09%	97,03%	0,06%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	-294	3,275	3,463	3,217	3,325	0,140	4,21%	39,94%	2,53%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	-310	2,833	2,833	2,667	2,778	0,096	3,46%	49,62%	1,74%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	-397	2,571	2,333	2,500	2,468	0,122	4,95%	55,41%	2,21%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	-425	0,190	0,165	0,167	0,174	0,014	8,19%	96,86%	0,26%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	-461	0,157	0,138	0,144	0,011	7,71%	97,40%	0,20%	

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Fluorène (FLU)						Enlèvement (%)	
						initial	final	initial	final	A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-263	0,056	0,041	0,044	0,047	0,008	16,40%	0,00%	0,00%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	4,448	4,122	4,084	4,218	0,200	4,75%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,51	6,13	-250	1	0,086	0,092	0,092	0,090	0,003	3,66%	97,86%	0,08%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	0,165	0,191	0,180	0,179	0,013	7,28%	95,76%	0,31%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	2,849	2,355	3,148	2,784	0,400	14,38%	34,01%	9,49%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	4,299	4,320	4,129	4,249	0,104	2,46%	-0,74%	2,48%
ELECTSTAB	ELECTSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	2,233	2,149	2,246	2,209	0,053	2,38%	47,63%	1,25%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					1,558	1,500	1,542	1,533	0,030	1,95%	72,33%	0,54%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					3,209	3,567	3,292	3,353	0,191	5,69%	39,50%	3,44%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	3,633	3,850	3,833	3,772	0,121	3,20%	31,94%	2,18%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					3,442	3,717	3,292	3,483	0,216	6,19%	37,15%	3,89%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					3,208	3,083	3,125	0,072	2,31%	43,61%	1,30%	
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					1,125	1,167	1,167	1,153	0,024	2,09%	79,20%	0,43%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	2,467	2,433	2,375	2,425	0,046	1,91%	56,24%	0,84%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					3,200	3,500	3,458	3,386	0,163	4,80%	38,90%	2,93%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					3,417	3,383	3,000	3,267	0,232	7,09%	41,06%	4,18%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	3,857	3,576	3,342	3,592	0,258	7,18%	35,19%	4,65%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	4,408	4,629	4,221	4,419	0,204	4,62%	20,26%	3,69%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	5,551	5,536	5,540	5,542	0,008	0,14%	0,00%	0,14%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	5,429	5,163	5,479	5,357	0,170	3,17%	3,34%	3,07%
Contrôle dopé et autoclave (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	4,012	4,037	3,958	4,003	0,040	1,01%	27,78%	0,73%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	7,11	7,16	-79	-58	3,916	3,665	3,630	3,737	0,156	4,17%	32,57%	2,81%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	2,767	2,767	2,975	2,836	0,120	4,23%	48,82%	2,17%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	1,667	1,600	1,267	1,511	0,214	14,18%	72,73%	3,87%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,69	5	275	0,883	0,800	0,817	0,833	0,044	5,29%	84,96%	0,80%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	0,333	0,517	0,500	0,450	0,101	22,53%	91,88%	1,83%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	0,120	0,092	0,092	0,101	0,016	15,98%	98,17%	0,29%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	1,850	1,350	1,367	1,456	0,169	11,58%	73,73%	3,04%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	0,650	0,633	0,700	0,661	0,035	5,25%	88,07%	0,63%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	0,517	0,500	0,450	0,489	0,035	7,10%	91,16%	0,63%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	0,433	0,456	0,446	0,445	0,011	2,57%	91,97%	0,21%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	0,383	0,424	0,357	0,388	0,034	8,71%	92,99%	0,61%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	4,058	4,058	4,417	4,178	0,207	4,96%	24,62%	3,74%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	3,333	3,167	3,000	3,167	0,167	5,26%	42,86%	3,01%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	3,000	2,833	2,969	2,934	0,089	3,02%	47,06%	1,60%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	0,270	0,250	0,250	0,257	0,011	4,46%	95,37%	0,21%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	0,250	0,233	0,239	0,010	4,03%	95,69%	0,17%	

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Phénanthrène (PHE)							
						initial	final	initial	final	Concentration - fraction solide (mg/kg)			Enlèvement (%)				
										A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,217	0,209	0,196	0,207	0,010	5,05%		
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	4,774	5,486	4,327	4,863	0,586	12,04%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	0,241	0,222	0,259	0,241	0,019	7,75%	95,05%	0,38%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	0,296	0,354	0,381	0,344	0,043	12,51%	92,93%	0,88%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	2,747	3,964	2,999	3,237	0,642	19,84%	33,44%	13,20%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	3,305	3,106	3,171	3,194	0,101	3,17%	34,32%	2,08%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	2,448	1,936	2,180	2,188	0,256	11,70%	55,01%	5,26%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					1,775	1,683	1,792	1,750	0,058	3,34%	69,33%	1,02%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					3,358	3,567	3,458	3,461	0,104	3,01%	39,35%	1,83%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6					3,608	3,667	3,625	3,633	0,030	0,83%	36,33%	0,53%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6	6,84	6,60	-120	-70	3,667	3,617	3,500	3,594	0,086	2,38%	37,01%	1,50%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					2,975	2,967	3,042	2,994	0,041	1,37%	47,53%	0,72%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					0,525	0,500	0,583	0,536	0,043	7,97%	90,61%	0,75%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2					2,308	2,183	2,292	2,261	0,068	3,00%	60,38%	1,19%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2	6,84	6,62	-120	-82	3,717	3,750	3,708	3,725	0,022	0,59%	34,73%	0,39%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					3,750	3,700	3,542	3,664	0,109	2,96%	35,79%	1,90%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	4,714	4,534	4,123	4,457	0,303	6,80%	21,90%	5,31%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	4,575	4,275	4,608	4,486	0,183	4,09%	21,39%	3,22%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	5,745	5,679	5,696	5,707	0,034	0,60%	0,00%	0,60%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	6,000	5,707	5,822	5,843	0,148	2,53%	-2,39%	2,59%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	5,247	5,280	5,833	5,453	0,330	6,04%	4,44%	5,78%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58	4,819	4,712	4,444	4,659	0,193	4,14%	18,37%	3,38%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	2,167	2,292	2,250	2,072	3,21%	60,57%	1,28%	
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	1,383	1,433	1,517	1,444	0,067	4,66%	74,69%	1,18%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	1,117	1,150	1,100	1,122	0,025	2,27%	80,33%	0,45%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	0,433	0,567	0,567	0,522	0,077	14,74%	90,85%	1,36%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	0,200	0,222	0,259	0,227	0,030	13,15%	96,02%	0,52%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	0,942	1,017	0,875	0,945	0,071	7,52%	83,45%	1,24%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	0,833	0,583	0,633	0,683	0,132	19,36%	88,03%	2,32%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	0,667	0,650	0,683	0,667	0,017	2,50%	88,32%	0,29%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	0,517	0,509	0,464	0,497	0,028	5,69%	91,30%	0,49%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	0,500	0,500	0,411	0,470	0,052	10,96%	91,76%	0,90%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	4,342	3,492	4,492	4,109	0,539	13,12%	28,00%	9,45%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	3,500	3,000	3,000	3,167	0,289	9,12%	44,51%	5,06%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	3,714	3,000	3,125	3,280	0,381	11,63%	42,53%	6,68%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	0,317	0,283	0,300	0,300	0,017	5,68%	94,74%	0,30%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	0,317	0,283	0,300	0,300	0,017	5,56%	94,74%	0,29%

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Fluoranthène (FLR)							
						initial	final	initial	final	Concentration - fraction solide (mg/kg)		Enlèvement (%)					
										A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,505	0,505	0,482	0,497	0,013	2,68%	0,00%	0,00%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	4,491	5,114	4,200	4,602	0,467	10,15%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	2,186	2,262	2,267	2,238	0,045	2,02%	51,36%	0,98%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	3,649	3,608	3,772	3,676	0,085	2,32%	20,11%	1,86%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	3,621	3,475	4,497	3,864	0,553	14,30%	16,03%	12,01%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	4,192	4,659	4,775	4,542	0,308	6,79%	1,30%	6,70%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	6,126	5,282	5,681	5,696	0,422	7,41%	-23,78%	9,17%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					4,367	4,550	4,542	4,486	0,104	2,31%	25,13%	1,73%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					5,117	5,833	5,958	5,636	0,454	8,06%	5,94%	7,58%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	5,542	4,964	4,905	5,137	0,352	6,65%	14,27%	5,87%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					5,650	6,300	6,292	6,081	0,373	6,13%	-1,48%	6,22%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					5,200	5,800	6,083	5,694	0,451	7,92%	4,97%	7,53%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					4,775	5,083	5,250	5,036	0,241	4,79%	15,95%	4,02%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	5,092	5,533	5,875	5,500	0,393	7,14%	8,21%	6,55%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					5,303	5,783	5,875	5,656	0,304	5,38%	5,62%	5,08%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					5,500	5,667	4,792	5,320	0,464	8,73%	11,22%	7,75%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	5,571	5,468	5,389	5,476	0,091	1,67%	8,61%	1,53%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	6,114	5,657	5,947	5,906	0,231	3,91%	1,43%	3,85%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	6,062	5,993	5,921	5,992	0,071	1,18%	0,00%	1,18%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	6,000	6,793	6,507	6,433	0,402	6,25%	-7,37%	6,71%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	326	7,11	7,21	-79	-62	5,884*	6,211	6,667	6,247	0,402	6,44%	-4,26%	6,72%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58	5,723	5,497	5,556	5,502	0,117	2,09%	6,68%	1,95%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	5,714	5,589	5,550	5,618	0,066	1,53%	6,25%	1,43%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	5,533	5,833	5,000	5,456	0,422	7,74%	8,95%	7,04%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	4,167	5,667	3,833	4,556	0,977	21,44%	23,97%	16,30%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	3,160	2,000	2,167	2,439	0,621	25,49%	50,30%	10,37%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	2,200	2,262	2,267	2,243	0,037	1,68%	62,57%	0,62%
Digestion aérobie mésophile (MAD)	MAD	Filtration	0,5	30	72	8,16	8,12	5	106	6,038	5,733	5,450	5,740	0,294	5,12%	4,20%	4,91%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	5,000	4,500	4,500	4,667	0,289	6,19%	22,12%	4,82%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	4,333	3,833	3,833	4,000	0,289	7,22%	33,24%	4,82%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	2,500	2,456	2,321	2,426	0,093	3,64%	59,52%	1,55%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	1,667	1,615	1,643	1,608	0,081	5,07%	73,16%	1,36%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	5,900	5,979	5,998	5,959	0,052	0,87%	0,55%	0,87%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	5,833	5,833	5,500	5,722	0,192	3,36%	4,50%	3,21%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	5,571	5,500	5,000	5,357	0,311	5,81%	10,60%	5,20%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	3,651	4,667	5,000	4,439	0,703	15,83%	25,92%	11,73%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	3,667	3,500	3,333	3,500	0,167	4,76%	41,59%	2,78%

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Pyrine (PYR)						
						initial	final	initial	final	Concentration - fraction solide (mg/kg)			Enivrement (%)			
										A	B	C	Moyenne	Écart-type	Moyenne	Écart-type
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,540	0,536	0,505	0,527	0,019	3,66%	
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	4,534	4,900	4,238	4,557	0,332	7,28%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	2,838	3,052	2,848	2,913	0,121	4,15%	36,09%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	3,989	3,865	4,415	4,090	0,288	7,05%	10,26%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	3,464	3,256	4,252	3,658	0,525	14,36%	19,74%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	4,783	4,222	4,259	4,421	0,314	7,10%	2,99%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	5,838	5,338	5,822	5,666	0,284	5,02%	24,32%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					5,183	5,567	5,625	5,458	0,240	4,40%	9,37%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					4,800	5,417	5,667	5,294	0,446	8,43%	12,09%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6					5,342	5,983	4,640	5,322	0,672	12,63%	11,64%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					5,400	6,000	6,042	5,814	0,359	6,18%	3,47%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					5,058	5,733	6,000	5,597	0,485	8,67%	7,07%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					4,892	5,167	5,333	5,131	0,223	4,35%	14,81%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2					5,133	5,450	5,625	5,403	0,249	4,61%	10,29%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					4,867	5,500	5,583	5,317	0,392	7,37%	11,72%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					5,133	5,517	5,125	5,258	0,224	4,26%	12,69%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	5,286	5,854	5,432	5,457	0,185	3,40%	9,39%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	6,171	4,878	4,824	5,291	0,763	14,41%	12,15%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	6,076	6,036	5,957	6,023	0,060	1,00%	
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	6,286	6,250	6,849	6,462	0,336	5,20%	7,29%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	5,556	5,590	6,667	5,937	0,632	10,64%	1,42%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	-5	96	5,617	5,575	5,467	5,553	0,077	1,39%	7,60%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	-5	223	5,167	5,367	4,667	5,067	0,361	7,12%	15,88%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	-5	275	4,000	4,833	3,667	4,167	0,601	14,42%	30,82%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	-5	361	3,050	2,833	2,833	2,906	0,125	4,31%	51,76%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	-5	440	3,000	3,052	2,848	2,967	0,106	3,57%	50,74%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	-5	106	6,033	5,800	5,583	5,805	0,225	3,88%	3,61%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	-5	153	4,833	4,333	4,500	4,556	0,255	5,59%	24,36%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	-5	190	4,333	3,667	3,667	3,889	0,385	9,90%	35,43%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	-5	333	2,333	2,281	2,143	2,252	0,098	4,37%	62,60%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	-5	446	1,517	1,394	1,500	1,470	0,067	4,53%	75,59%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	5,825	5,875	5,825	5,842	0,029	0,49%	3,01%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	5,500	5,333	5,167	5,333	0,167	3,13%	11,45%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	5,143	5,000	4,688	4,943	0,233	4,71%	17,92%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	4,286	4,333	4,500	4,373	0,113	2,57%	27,39%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	4,500	4,667	4,333	4,500	0,167	3,70%	25,28%

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Benzo[bk]fluoranthènes (BJK)						Enlèvement (%)	
						initial	final	initial	final	Concentration - fraction solide (mg/kg)			Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
										A	B	C					
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	1,007	0,963	0,813	0,928	0,102	10,95%		
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	16,068	17,367	15,343	16,259	1,026	6,31%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	14,691	13,109	13,058	13,919	0,792	5,69%	14,39%	4,87%
METIX-B5	METIX-B5	Centrifugation	0	18	240	6,37	1,57	-277	398	10,962	12,376	12,628	11,989	0,898	7,49%	26,26%	5,52%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	14,709	14,584	15,316	14,870	0,301	2,63%	8,54%	2,41%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	14,550	13,566	13,620	13,912	0,553	3,98%	14,44%	3,40%
ELECTSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,09	-172	171	15,986	17,433	17,201	16,874	0,777	4,61%	-3,7%	4,78%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					17,117	13,057	14,330	14,835	2,076	14,00%	14,77%	11,83%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					13,529	14,801	16,033	14,788	1,252	8,47%	15,04%	7,19%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	12,342	13,900	14,125	13,466	0,971	7,22%	22,70%	5,58%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					17,592	15,406	18,176	17,058	1,460	8,56%	2,00%	8,39%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					16,625	15,118	18,130	16,627	1,511	9,09%	4,47%	8,68%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					15,000	17,300	19,792	17,364	2,396	13,80%	0,24%	13,77%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	15,175	18,283	18,750	17,403	1,943	11,17%	0,02%	11,16%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					15,292	15,083	17,083	15,819	1,099	6,95%	9,12%	6,32%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					15,175	18,283	18,958	17,472	2,018	11,55%	-0,36%	11,59%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	14,286	16,239	15,876	15,467	1,039	5,50%	11,14%	5,97%
METIX-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	14,686	14,558	15,009	14,751	0,233	1,58%	15,25%	1,34%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	17,455	17,483	17,281	17,406	0,109	0,63%	0,00%	0,63%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	16,571	17,391	17,123	17,029	0,418	2,45%	2,17%	2,40%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	15,432	15,839	16,250	15,840	0,409	2,58%	9,00%	2,35%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	7,11	7,16	-79	-58	15,964	15,183	15,556	15,568	0,390	2,51%	10,56%	2,24%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	17,458	15,392	17,667	16,839	1,257	7,47%	3,26%	7,22%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	16,517	15,273	14,167	15,319	1,176	7,67%	11,99%	6,75%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	15,167	12,333	12,833	13,444	1,512	11,25%	22,76%	8,69%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	13,333	12,833	13,000	13,056	0,255	1,95%	24,99%	1,46%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	13,400	13,109	13,958	13,489	0,431	3,20%	22,50%	2,48%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	14,775	15,892	14,442	15,036	0,760	5,05%	13,61%	4,36%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	14,333	14,000	13,600	13,844	0,419	3,01%	19,89%	2,41%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	14,500	12,167	12,500	13,056	1,262	9,67%	24,99%	7,25%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	13,667	12,281	11,786	12,578	0,975	7,75%	27,74%	5,60%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	11,833	12,424	10,893	11,717	0,772	6,59%	32,69%	4,44%
METIX-B5	METIX-B5	Centrifugation	0	30	72	7,19	3,13	-51	294	15,975	17,492	17,567	17,011	0,698	5,28%	2,27%	5,16%
METIX-B5	METIX-B5	Centrifugation	0	30	120	7,19	2,71	-51	310	15,167	14,333	14,333	14,611	0,481	3,29%	16,06%	2,76%
METIX-B5	METIX-B5	Centrifugation	0	30	168	7,19	2,15	-51	397	13,571	12,000	12,813	12,795	0,786	6,14%	26,49%	4,51%
METIX-B5	METIX-B5	Centrifugation	0	30	336	7,19	1,83	-51	425	13,968	11,833	12,833	12,878	1,068	8,29%	26,01%	6,14%
METIX-B5	METIX-B5	Centrifugation	0	30	504	7,19	1,42	-51	461	13,667	12,333	11,833	12,811	0,948	7,51%	27,55%	5,44%

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Benzo[a]pyrène (BAP)						
						initial	final	initial	final	Concentration - fraction solide (mg/kg)			Enlèvement (%)			
										A	B	C	Moyenne	Écart-type	Moyenne	Écart-type
Contrôle non-dupé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,416	0,412	0,349	0,392	0,037	9,54%	
Contrôle dupé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	4,767	4,773	4,407	4,649	0,210	4,51%	0,00% 0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	4,168	4,733	4,729	4,544	0,325	7,15%	2,27% 6,99%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	3,918	4,249	4,675	4,281	0,380	8,87%	7,92% 8,17%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	4,138	4,126	4,417	4,227	0,165	3,90%	9,07% 3,54%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	5,101	4,935	4,941	4,992	0,094	1,89%	-7,38% 2,02%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	5,700	5,299	5,510	5,503	0,201	3,65%	-18,36% 4,32%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					4,939	5,312	5,684	5,312	0,372	7,01%	9,85% 6,32%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					5,323	5,842	5,751	5,639	0,277	4,92%	4,30% 4,71%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6					5,617	4,553	6,560	5,577	1,005	18,01%	5,36% 17,05%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					5,932	5,800	5,620	5,784	0,157	2,71%	1,84% 2,66%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					5,699	5,292	5,627	5,539	0,217	3,92%	5,99% 3,69%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					5,117	5,933	6,708	5,919	0,796	13,45%	-0,46% 13,51%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2					5,142	5,167	6,213	5,507	0,611	11,10%	6,54% 10,38%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					5,158	6,167	5,116	5,480	0,595	10,85%	6,99% 10,08%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					5,642	6,674	5,193	5,836	0,759	13,01%	0,95% 12,89%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	4,857	5,474	5,563	5,298	0,384	7,26%	10,08% 6,52%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	6,050	5,667	5,324	5,680	0,363	6,40%	3,69% 6,17%
Contrôle dupé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	5,916	5,912	5,849	5,892	0,037	0,63%	0,00% 0,63%
Contrôle dupé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	5,714	6,250	6,164	6,043	0,288	4,76%	-2,56% 4,88%
Contrôle dupé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	5,247	5,590	5,833	5,557	0,295	5,30%	5,69% 5,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	7,11	7,16	-79	-58	5,422	5,497	5,185	5,368	0,163	3,03%	6,89% 2,76%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	6,217	6,192	6,100	6,169	0,061	1,00%	-4,71% 1,04%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	5,050	5,000	4,667	4,906	0,208	4,25%	16,74% 3,54%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	4,833	4,167	4,000	4,333	0,441	10,18%	26,46% 7,48%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	4,667	4,667	4,000	4,444	0,385	8,66%	24,57% 6,53%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	4,400	4,433	4,529	4,454	0,067	1,50%	24,41% 1,14%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	5,758	4,608	5,775	5,380	0,669	12,43%	8,69% 11,35%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	4,833	4,667	4,667	4,722	0,096	2,04%	19,86% 1,63%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	4,833	4,000	4,333	4,389	0,419	9,56%	25,51% 7,12%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	4,333	4,035	3,929	4,099	0,210	5,12%	30,43% 3,56%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	3,833	3,636	3,571	3,680	0,136	3,71%	37,54% 2,31%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	5,758	5,275	5,775	5,603	0,284	5,07%	4,91% 4,82%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	5,167	4,833	4,833	4,944	0,192	3,89%	16,08% 3,27%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	4,571	4,000	4,219	4,263	0,288	6,76%	27,64% 4,89%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	4,127	3,500	3,667	3,765	0,325	8,63%	36,11% 5,51%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	4,000	3,667	3,333	3,667	0,333	9,09%	37,77% 5,66%

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH				POR (mV)				Indéno[1,2,3-cd]pyrène (INP)						Enlèvement (%)	
						initial		final		initial		final		Concentration - fraction solide (mg/kg)			Moyenne			Écart-type	
						A	B	C	Moyenne	Écart-type	C.V. (%)	A	B	C	Moyenne	Écart-type	C.V. (%)	A	B	C	Moyenne
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-276	-283	0,534	0,503	0,461	0,498	0,036	7,29%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	5,261	6,007	4,730	5,333	0,642	12,03%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	4,299	4,653	4,628	4,593	0,270	5,87%	13,67%	5,06%	0,00%	0,00%	0,00%	0,00%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	5,125	5,254	5,183	5,187	0,065	1,24%	2,73%	1,21%	0,00%	0,00%	0,00%	0,00%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	6,314	5,934	6,082	5,810	0,682	11,74%	8,94%	12,79%	0,00%	0,00%	0,00%	0,00%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	5,050	5,609	5,492	5,384	0,295	5,48%	0,96%	5,53%	0,00%	0,00%	0,00%	0,00%
ELECTSTAB	ELECTSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	5,531	5,215	5,510	5,419	0,177	3,26%	-1,61%	3,31%	0,00%	0,00%	0,00%	0,00%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					4,483	5,631	5,491	5,202	0,627	12,05%	13,79%	10,38%	0,00%	0,00%	0,00%	0,00%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					6,034	6,244	5,380	5,866	0,451	7,66%	2,45%	7,47%	0,00%	0,00%	0,00%	0,00%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	4,125	4,683	5,167	4,658	0,521	11,19%	22,80%	8,64%	0,00%	0,00%	0,00%	0,00%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					5,487	6,267	5,314	5,689	0,508	8,93%	5,71%	8,42%	0,00%	0,00%	0,00%	0,00%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					5,165	5,375	5,756	5,432	0,300	5,52%	9,98%	4,97%	0,00%	0,00%	0,00%	0,00%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					5,542	6,300	5,168	5,670	0,577	10,17%	6,04%	9,56%	0,00%	0,00%	0,00%	0,00%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	5,375	5,144	5,348	5,289	0,126	2,39%	12,35%	2,09%	0,00%	0,00%	0,00%	0,00%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					5,375	5,447	5,348	5,390	0,051	0,95%	10,68%	0,84%	0,00%	0,00%	0,00%	0,00%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					5,458	6,667	5,685	5,937	0,642	10,82%	1,62%	10,65%	0,00%	0,00%	0,00%	0,00%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	5,000	5,751	5,876	5,542	0,474	8,55%	8,15%	7,85%	0,00%	0,00%	0,00%	0,00%
Matic-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	5,125	6,109	5,693	5,642	0,494	8,76%	6,49%	8,19%	0,00%	0,00%	0,00%	0,00%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	6,086	6,003	6,013	6,034	0,045	0,75%	0,00%	0,75%	0,00%	0,00%	0,00%	0,00%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	5,556	5,280	5,417	5,417	0,138	2,55%	10,22%	2,29%	0,00%	0,00%	0,00%	0,00%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	4,938	5,280	5,417	5,211	0,246	4,73%	13,63%	4,08%	0,00%	0,00%	0,00%	0,00%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	504	7,11	7,16	-79	-58	5,422	5,497	4,815	5,245	0,374	7,13%	13,08%	6,20%	0,00%	0,00%	0,00%	0,00%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	6,250	5,925	5,805	5,994	0,229	3,82%	0,68%	3,80%	0,00%	0,00%	0,00%	0,00%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	5,150	5,367	4,333	4,950	0,545	11,01%	17,97%	9,03%	0,00%	0,00%	0,00%	0,00%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	5,167	4,000	4,000	4,389	0,674	15,35%	27,27%	11,16%	0,00%	0,00%	0,00%	0,00%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,89	5	361	4,500	4,333	4,388	4,096	2,19%	2,19%	27,27%	1,59%	0,00%	0,00%	0,00%	0,00%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	4,800	4,653	4,828	4,760	0,094	1,97%	21,11%	1,56%	0,00%	0,00%	0,00%	0,00%
Digestion aérobie mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	5,000	6,308	5,633	5,647	0,654	11,58%	6,42%	10,84%	0,00%	0,00%	0,00%	0,00%
Digestion aérobie mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	5,667	4,667	4,500	4,944	0,631	12,76%	18,06%	10,49%	0,00%	0,00%	0,00%	0,00%
Digestion aérobie mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	5,000	4,000	4,333	4,444	0,509	11,46%	26,34%	8,44%	0,00%	0,00%	0,00%	0,00%
Digestion aérobie mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	5,167	4,561	4,464	4,731	0,381	8,05%	21,60%	6,31%	0,00%	0,00%	0,00%	0,00%
Digestion aérobie mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	5,000	4,848	4,464	4,771	0,276	5,79%	20,93%	4,58%	0,00%	0,00%	0,00%	0,00%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	5,936	6,417	6,633	6,329	0,357	5,64%	4,88%	5,92%	0,00%	0,00%	0,00%	0,00%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	5,167	4,833	4,833	4,944	0,192	3,89%	18,06%	3,19%	0,00%	0,00%	0,00%	0,00%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	5,070	4,778	5,225	5,024	0,227	4,52%	16,73%	3,76%	0,00%	0,00%	0,00%	0,00%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	4,921	3,833	4,500	4,418	0,648	12,41%	26,78%	9,09%	0,00%	0,00%	0,00%	0,00%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	4,333	4,167	3,833	4,111	0,255	6,19%	31,87%	4,22%	0,00%	0,00%	0,00%	0,00%

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Benzog(hi)perylène (BPR)							
						initial	final	initial	final	Concentration - fraction solide (mg/kg)			Enlèvement (%)				
										A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,456	0,425	0,399	0,427	0,028	6,65%	0,00%	0,00%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	6,331	5,965	5,589	5,962	0,371	6,22%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	4,934	5,345	4,535	4,938	0,405	8,20%	17,17%	6,79%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	5,507	5,626	5,131	5,421	0,258	4,77%	9,07%	4,34%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	6,061	5,043	4,891	5,331	0,636	11,93%	10,57%	10,67%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	5,644	5,380	5,733	5,586	0,183	3,28%	6,31%	3,08%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	5,642	5,275	5,035	5,317	0,306	5,76%	10,81%	5,14%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					4,114	5,188	5,949	5,084	0,922	18,13%	15,40%	15,34%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					5,037	6,244	4,824	5,368	0,766	14,27%	10,65%	12,75%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6					6,608	4,233	5,424	5,422	1,188	21,90%	9,76%	19,76%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					5,261	6,038	6,263	5,854	0,525	8,98%	2,57%	8,74%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					6,125	6,100	6,572	6,266	0,266	4,24%	4,28%	4,42%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					4,963	5,683	6,292	5,653	0,655	11,56%	5,92%	10,90%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2					5,108	6,067	5,841	5,672	0,501	8,83%	5,60%	8,34%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					5,608	6,067	6,542	6,072	0,467	7,69%	-1,06%	7,77%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					5,358	6,566	6,542	6,155	0,690	11,22%	-2,44%	11,49%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	5,143	5,552	5,127	5,274	0,241	4,57%	12,23%	4,01%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	4,968	5,397	4,838	5,067	0,292	5,77%	15,66%	4,67%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,59	-79	-80	5,870	5,435	6,721	6,009	0,654	10,89%	0,00%	10,89%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	5,556	5,901	5,833	5,763	0,183	3,17%	4,08%	3,04%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	5,120	5,236	5,556	5,304	0,225	4,25%	11,73%	3,76%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58	6,025	5,858	6,125	6,003	0,135	2,25%	0,10%	2,25%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	98	5,467	4,891	4,833	5,064	0,350	6,92%	15,73%	5,03%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	223	5,000	4,333	4,333	4,556	0,395	8,49%	24,18%	6,41%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	4,833	4,500	4,500	4,611	0,192	4,17%	23,26%	3,20%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	5,000	4,845	4,535	4,793	0,237	4,94%	20,23%	3,94%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	5,867	5,658	5,417	5,647	0,225	3,99%	6,01%	3,75%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	5,000	4,833	4,667	4,750	0,118	2,48%	20,95%	1,96%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	5,000	4,667	4,500	4,722	0,255	5,39%	21,41%	4,24%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	4,833	4,737	4,643	4,738	0,096	2,01%	21,15%	1,59%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	4,833	4,545	4,821	4,733	0,163	3,44%	21,22%	2,71%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	6,267	6,417	6,183	6,289	0,119	1,89%	-4,67%	1,97%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	6,167	4,833	5,000	5,333	0,726	13,62%	11,24%	12,09%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	4,917	4,977	5,419	5,104	0,274	5,37%	15,05%	4,56%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	4,603	4,000	4,500	4,368	0,323	7,39%	27,31%	5,37%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	4,500	4,333	3,833	4,222	0,347	8,22%	29,73%	5,77%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	4,500	4,333	3,833	4,222	0,347	8,22%	29,73%	5,77%

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Recouvrement Acénaphthène-D10 (%) - fraction solide					
						initial	final	initial	final	A	B	C	Moyenne	Écart-type	C.V. (%)
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	71,90%	68,50%	61,10%	67,17%	5,52%	8,22%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	114,00%	78,30%	98,50%	96,93%	17,90%	18,47%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	102,20%	105,00%	112,40%	106,53%	5,27%	4,95%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	102,50%	104,20%	109,30%	105,33%	3,54%	3,36%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	119,00%	91,10%	105,90%	105,33%	13,80%	13,25%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	84,50%	110,30%	102,10%	92,30%	24,42%	26,46%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	95,40%	107,00%	102,10%	101,50%	5,82%	5,74%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					133,80%	132,80%	127,00%	131,20%	3,67%	2,80%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					106,20%	112,80%	111,00%	110,00%	3,41%	3,10%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	99,00%	103,20%	106,00%	102,73%	3,52%	3,43%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					94,00%	100,80%	92,00%	95,60%	4,61%	4,83%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					126,40%	128,00%	126,00%	126,80%	1,06%	0,63%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					61,60%	62,00%	60,00%	61,20%	1,06%	1,73%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	81,00%	80,00%	78,00%	79,67%	1,53%	1,92%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					90,80%	101,20%	106,00%	99,33%	7,77%	7,82%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					81,80%	99,20%	93,00%	91,33%	8,82%	9,66%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	71,00%	83,00%	79,00%	77,67%	6,11%	7,87%
METIX-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	105,00%	112,00%	118,10%	111,70%	6,56%	5,87%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	73,90%	68,50%	69,10%	70,50%	2,96%	4,20%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	99,40%	86,84%	80,33%	88,86%	9,69%	10,91%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	99,00%	79,23%	82,00%	86,74%	10,71%	12,34%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58	80,86%	94,33%	90,80%	88,66%	6,89%	7,88%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	-5	96	86,00%	89,00%	98,00%	91,00%	6,24%	6,86%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	-5	223	80,60%	75,60%	83,40%	79,87%	3,95%	4,95%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	-5	275	95,00%	116,00%	94,00%	101,67%	12,42%	
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	236	8,16	5,80	-5	361	119,08%	99,00%	99,20%	105,76%	11,54%	10,91%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	-5	440	95,00%	91,28%	90,32%	92,20%	2,47%	2,68%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0,5	30	72	8,16	8,12	-5	106	99,00%	79,00%	102,10%	90,55%	16,33%	18,04%
Digestion aérobie mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	-5	153	77,24%	73,08%	72,60%	74,30%	3,65%	3,56%
Digestion aérobie mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	-5	190	84,68%	86,30%	91,40%	87,45%	3,82%	4,02%
Digestion aérobie mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	-5	333	91,20%	98,91%	92,00%	94,04%	4,24%	4,51%
Digestion aérobie mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	-5	446	88,63%	88,14%	86,63%	88,20%	1,60%	
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	106,00%	88,00%	85,00%	93,00%	11,38%	12,21%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	91,10%	88,00%	91,98%	90,36%	2,09%	2,31%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	86,67%	84,60%	92,67%	88,64%	4,03%	4,65%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	92,48%	80,88%	74,25%	82,53%	9,22%	11,18%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	84,50%	76,10%	66,38%	75,66%	9,07%	11,99%

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Recouvrement Chrysène-D12 (%) - fraction solide					
						initial	final	initial	final	A	B	C	Moyenne	Écart-type	C.V. (%)
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	93,20%	96,00%	78,30%	89,17%	9,51%	10,67%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	117,00%	80,20%	82,10%	93,10%	20,72%	22,26%
Digestion aérobique mesophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	129,33%	123,80%	138,60%	130,58%	7,48%	5,73%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	113,00%	144,10%	149,10%	135,40%	19,56%	14,45%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	122,40%	126,80%	153,70%	134,33%	16,92%	12,60%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	126,80%	121,13%	103,40%	118,78%	9,42%	7,93%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	104,93%	113,67%	105,00%	108,20%	4,76%	4,40%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					109,00%	104,00%	110,00%	107,67%	3,21%	2,99%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					111,20%	109,20%	121,00%	113,80%	6,32%	5,55%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6					117,00%	129,60%	121,00%	122,53%	6,44%	5,25%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					130,00%	105,20%	121,00%	118,73%	12,55%	10,57%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					130,20%	108,00%	124,00%	120,73%	11,45%	9,49%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					113,20%	124,80%	134,00%	124,00%	10,42%	8,41%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2					114,00%	124,80%	116,00%	118,27%	5,75%	4,86%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					110,80%	99,20%	92,00%	100,67%	9,49%	9,42%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					114,00%	92,40%	94,00%	100,13%	12,04%	12,02%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	97,00%	113,00%	97,00%	102,33%	9,24%	9,03%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	114,00%	128,00%	116,10%	119,70%	8,12%	6,76%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	93,20%	96,00%	88,30%	92,50%	3,90%	4,21%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	107,08%	82,96%	86,63%	92,22%	13,00%	14,09%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	107,00%	87,58%	97,07%	97,21%	9,71%	9,99%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58	97,00%	97,00%	89,35%	94,45%	4,42%	4,68%
Digestion aérobique mesophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	124,00%	119,00%	129,00%	122,67%	3,21%	2,62%
Digestion aérobique mesophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	111,60%	101,60%	74,80%	96,00%	19,03%	19,82%
Digestion aérobique mesophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	71,08%	72,60%	84,00%	75,89%	7,06%	9,30%
Digestion aérobique mesophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	92,66%	69,18%	68,80%	76,88%	13,67%	17,78%
Digestion aérobique mesophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	91,28%	103,80%	104,60%	101,23%	8,84%	8,83%
Digestion aérobique mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	121,00%	121,00%	128,10%	124,55%	5,02%	4,03%
Digestion aérobique mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	91,20%	83,10%	78,24%	84,18%	6,55%	7,78%
Digestion aérobique mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	102,80%	87,62%	87,44%	92,62%	8,81%	9,52%
Digestion aérobique mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	103,36%	100,44%	88,16%	97,32%	8,07%	8,29%
Digestion aérobique mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	95,92%	79,70%	77,76%	84,46%	9,97%	11,81%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	122,00%	107,00%	100,00%	109,67%	11,24%	10,25%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	96,85%	80,00%	90,50%	89,12%	8,51%	9,55%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	93,75%	77,50%	91,18%	87,48%	8,73%	9,98%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	107,56%	83,24%	82,44%	91,08%	14,28%	15,68%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	98,72%	87,89%	83,00%	89,87%	8,05%	8,95%

Essais (Fraction solide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Recouvrement Anthracène-D10 (%) - fraction solide					
						initial	final	initial	final	A	B	C	Moyenne	Écart-type	C.V. (%)
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	6	18	0	6,40	6,47	-270	-283	90,20%	91,00%	85,30%	88,83%	3,09%	3,47%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	133,00%	106,70%	100,50%	113,40%	17,25%	15,22%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	98,30%	97,10%	101,30%	98,80%	2,16%	2,19%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	95,50%	98,70%	102,50%	98,80%	3,50%	3,54%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	104,30%	91,90%	101,40%	99,20%	6,49%	6,54%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	104,90%	107,20%	99,30%	103,80%	4,06%	3,91%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	100,30%	100,50%	99,30%	100,03%	0,64%	0,64%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					107,40%	102,80%	102,00%	104,07%	2,91%	2,80%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					106,20%	110,00%	109,00%	108,40%	1,97%	1,82%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	104,20%	106,00%	108,00%	106,07%	1,90%	1,79%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6					104,80%	110,40%	104,00%	106,40%	3,49%	3,28%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					104,40%	107,60%	108,00%	106,67%	1,97%	1,85%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					101,40%	103,20%	104,00%	102,87%	1,33%	1,29%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	102,00%	106,40%	112,00%	106,80%	5,01%	4,69%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2					89,20%	98,80%	96,00%	94,67%	4,94%	5,22%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					102,00%	98,80%	112,00%	104,27%	6,89%	6,60%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	109,00%	121,00%	98,00%	109,33%	11,50%	10,52%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	107,00%	115,00%	112,00%	111,33%	4,04%	3,63%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	90,20%	98,00%	87,30%	91,83%	5,53%	6,03%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	105,20%	89,30%	85,18%	93,23%	10,57%	11,34%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	105,00%	92,96%	92,02%	96,66%	7,24%	7,49%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58	105,60%	105,60%	96,78%	102,66%	5,09%	4,96%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	95,00%	78,00%	110,00%	94,33%	16,01%	16,97%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	101,40%	108,00%	96,00%	101,80%	6,01%	5,90%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	100,60%	91,06%	90,80%	94,15%	5,58%	5,93%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	236	8,16	5,80	5	361	102,36%	85,70%	93,04%	93,70%	8,35%	8,91%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	90,32%	97,10%	101,30%	96,24%	5,54%	5,76%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	105,00%	110,00%	109,00%	109,50%	0,71%	0,65%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	88,10%	91,76%	88,60%	93,15%	4,42%	4,74%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	99,00%	98,44%	93,78%	97,07%	2,87%	2,95%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	102,36%	94,46%	88,30%	95,04%	7,05%	7,42%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	102,62%	86,92%	87,62%	92,39%	8,87%	9,60%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	110,00%	95,00%	106,00%	103,67%	7,77%	7,49%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	85,60%	87,00%	85,64%	86,15%	0,74%	0,66%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	92,98%	83,72%	80,74%	85,81%	6,38%	7,44%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	102,60%	87,80%	83,90%	91,43%	9,87%	10,79%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	104,70%	98,84%	86,76%	96,77%	9,15%	9,45%

Essais (Fraction solide)	Code	Déshydratation	Tw80	ST	Temps	pH		POR (mV)		Recouvrement Pyrène-D10 (%) - fraction solide					
						initial	final	initial	final	A	B	C	Moyenne	Écart-type	C.V. (%)
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	91,90%	92,50%	87,40%	90,60%	2,79%	3,08%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,57	-270	-277	136,00%	117,40%	111,50%	121,63%	12,79%	10,51%
Digestion aérobio mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	107,50%	113,80%	120,30%	113,87%	6,40%	5,62%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	102,70%	112,00%	113,30%	109,33%	5,78%	5,29%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	120,40%	101,80%	118,90%	113,70%	10,33%	9,09%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	124,10%	125,70%	116,40%	122,07%	4,97%	4,07%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	135,80%	136,70%	116,40%	129,63%	11,47%	8,85%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30	6					121,80%	133,20%	138,00%	130,33%	7,52%	5,77%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30	6					121,00%	120,00%	119,00%	120,00%	1,00%	0,83%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6					112,40%	127,60%	128,00%	122,67%	8,89%	7,25%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30	6	6,84	6,60	-120	-70	121,80%	126,80%	128,00%	125,53%	3,29%	2,62%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30	6					116,00%	122,80%	110,00%	116,27%	6,40%	5,51%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30	2					115,80%	124,80%	129,00%	123,20%	6,74%	5,47%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2					118,60%	121,60%	114,00%	118,07%	3,83%	3,24%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2	6,84	6,62	-120	-82	116,80%	122,40%	125,00%	121,40%	4,19%	3,45%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30	2					118,60%	125,60%	115,00%	119,73%	5,39%	4,50%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	110,00%	113,00%	121,00%	114,67%	5,69%	4,96%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	117,00%	121,00%	121,40%	119,80%	2,43%	2,03%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	99,00%	92,50%	81,40%	90,97%	8,90%	8,78%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	105,00%	89,58%	83,94%	82,84%	10,00%	11,74%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	105,00%	88,56%	91,40%	84,99%	8,79%	9,25%
Digestion aérobio mésophile (MAD)	MAD	Centrifugation	0	30	504	7,11	7,16	-79	-58	104,40%	104,40%	97,62%	102,14%	3,91%	3,83%
Digestion aérobio mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	105,00%	113,00%	120,00%	112,67%	7,51%	6,66%
Digestion aérobio mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	112,40%	121,60%	91,32%	108,44%	15,52%	14,32%
Digestion aérobio mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	104,94%	93,50%	92,78%	97,07%	6,82%	7,03%
Digestion aérobio mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	106,28%	87,10%	96,20%	96,53%	9,59%	9,94%
Digestion aérobio mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	93,42%	113,80%	120,30%	109,17%	14,02%	12,85%
Digestion aérobio mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,12	5	108	118,00%	118,80%	119,40%	119,10%	0,42%	0,36%
Digestion aérobio mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	153	99,80%	90,76%	85,94%	92,17%	7,04%	7,63%
Digestion aérobio mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	190	104,86%	96,56%	92,78%	98,06%	6,18%	6,30%
Digestion aérobio mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	333	103,62%	96,40%	90,20%	96,74%	6,72%	6,94%
Digestion aérobio mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	446	102,90%	90,40%	92,00%	95,10%	6,80%	7,15%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	119,00%	105,00%	115,00%	113,00%	7,21%	6,38%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	96,00%	90,00%	89,00%	91,67%	3,79%	4,13%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	98,70%	86,66%	85,56%	90,31%	7,28%	8,07%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	107,80%	91,16%	84,88%	94,61%	11,84%	12,52%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	105,32%	99,94%	86,26%	97,17%	9,83%	10,11%



























Essais (Fraction liquide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Benzogéni <p>perylène (BAP)</p>							
						initial	final	initial	final	Concentration - fraction liquide (mg/kg)			Solubilisation (%)				
										A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,103	0,119	0,120	0,114	0,010	8,67%	1,83%	0,16%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	0,294	0,312	0,263	0,290	0,025	8,53%	4,83%	0,41%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	0,064	0,033	0,031	0,043	0,019	43,41%	0,68%	0,30%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	0,487	0,598	0,610	0,565	0,068	12,03%	9,03%	1,09%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	0,201	0,299	0,212	0,237	0,054	22,55%	3,80%	0,86%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	0,066	0,063	0,058	0,062	0,004	6,44%	1,00%	0,06%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	0,187	0,122	0,124	0,144	0,037	25,33%	2,40%	0,61%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30													
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30													
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70								
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30													
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30													
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30													
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30													
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2	6,84	6,62	-120	-82								
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30													
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	0,450	0,465	0,458	0,011	2,36%	7,62%	0,18%	
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	0,193	0,123	0,193	0,170	0,040	23,77%	2,83%	0,67%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80								
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66								
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62								
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58								
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	9,08	5	98								
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225								
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275								
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361								
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	0,064	0,033	0,031	0,043	0,019	43,41%	0,71%	0,31%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	0,233	0,219	0,270	0,241	0,026	10,86%	4,01%	0,44%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153								
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190								
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333								
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	0,039	0,035	0,031	0,035	0,004	11,63%	0,58%	0,07%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294								
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310								
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397								
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425								
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	0,167	0,193	0,167	0,176	0,015	8,77%	2,92%	0,26%

Essais (Fraction liquide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Recouvrement Acénaphthène-D10 (%) - fraction liquide					
						initial	final	initial	final	A	B	C	Moyenne	Écart-type	C.V. (%)
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6.40	6.47	-270	-283	79,00%	41,80%	82,30%	67,70%	22,49%	33,22%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6.40	6.37	-270	-277	88,00%	81,00%	86,00%	85,00%	3,61%	4,24%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6.31	6.13	-250	1	79,50%	80,80%	82,00%	80,77%	1,25%	1,55%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6.37	1,57	-277	398	69,00%	75,20%	82,30%	75,50%	6,66%	8,81%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6.69	2,08	-345	326	68,00%	104,40%	87,60%	86,67%	18,22%	21,02%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	96,80%	88,50%	104,70%	96,67%	8,10%	8,38%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	88,60%	71,00%	75,20%	78,27%	9,19%	11,74%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30											
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30											
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70						
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30											
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30											
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30											
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82						
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30											
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30											
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83						
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	80,80%	88,50%	84,65%	5,44%	6,43%	
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	85,20%	90,00%	85,20%	86,60%	2,77%	3,19%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66						
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62						
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	79,50%	80,80%	82,00%	80,77%	1,25%	1,55%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	75,00%	92,00%	81,00%	82,67%	8,62%	10,43%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153						
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190						
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	8,06	5	333						
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	89,50%	78,08%	78,20%	81,93%	6,56%	8,01%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294						
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310						
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397						
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425						
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	85,50%	90,00%	81,00%	85,33%	4,51%	5,28%

Essais (Fraction liquide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Recouvrement Chrysène-D12 (%) - fraction liquide					
						initial	final	initial	final	A	B	C	Moyenne	Écart-type	C.V. (%)
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	126,20%	97,80%	105,70%	109,90%	14,66%	13,34%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	120,20%	121,87%	112,10%	118,06%	5,23%	4,43%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	89,20%	84,70%	95,70%	89,87%	5,53%	6,15%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	80,40%	108,30%	105,70%	98,13%	15,41%	15,71%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	115,00%	126,20%	99,40%	113,53%	13,46%	11,86%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	93,60%	87,50%	103,50%	94,87%	8,07%	8,51%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	98,00%	77,10%	86,50%	87,20%	10,47%	12,00%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30											
Tween 80 (essai A)	Tw80 - A	Centrifugation	0,5	30											
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30											
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30											
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30											
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30											
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30											
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30											
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30											
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30											
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,60	-120	-70						
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	95,60%	97,60%	96,60%	1,41%	1,46%	
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	112,00%	101,40%	112,40%	108,60%	6,24%	5,74%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66						
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62						
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	-5	-96						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	-5	-225						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	-5	-275						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	-5	-361						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	-5	-440	89,20%	84,70%	95,70%	89,87%	5,53%	6,15%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	-5	-106	106,00%	102,00%	100,00%	102,67%	3,06%	2,98%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	-5	-153						
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	-5	-190						
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	-5	-333						
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	-5	-446	88,20%	78,47%	86,70%	84,48%	5,24%	6,20%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	-294						
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	-310						
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	-397						
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	-425						
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	-461	98,00%	82,00%	90,00%	90,00%	8,00%	8,89%

Essais (Fraction liquide)	Code	Déshydration	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Recouvrement Anthracène-D10 (%) - fraction liquide					
						initial	final	initial	final	A	B	C	Moyenne	Écart-type	C.V. (%)
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	109,90%	115,40%	104,30%	109,87%	5,55%	5,05%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	113,30%	110,40%	102,90%	108,87%	5,37%	4,93%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	93,20%	87,20%	89,90%	90,10%	3,00%	3,34%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	5,57	-277	398	132,10%	125,80%	104,30%	120,73%	14,58%	12,07%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	120,40%	121,50%	123,90%	121,93%	1,79%	1,47%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	96,20%	94,50%	95,90%	95,53%	0,91%	0,95%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	123,70%	124,80%	101,00%	116,50%	13,43%	11,53%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30											
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30											
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70						
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30											
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30											
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30											
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82						
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30											
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30											
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	107,20%	121,00%		114,10%	9,76%	8,56%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	96,20%	94,90%	95,90%	95,67%	0,68%	0,71%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80						
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66						
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62						
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	93,20%	87,20%	89,90%	90,10%	3,00%	3,34%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	95,00%	93,00%	100,00%	96,00%	3,61%	3,76%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153						
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190						
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333						
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	90,20%	89,20%	118,90%	99,43%	16,87%	16,96%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294						
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310						
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397						
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425						
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	89,50%	83,00%	98,00%	90,17%	7,52%	8,34%

Essais (Fraction liquide)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Recouvrement Pyrène-D10 (%) - fraction liquide					
						initial	final	initial	final	A	B	C	Moyenne	Écart-type	C.V. (%)
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-263	101,20%	105,60%	94,40%	100,40%	5,64%	5,62%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	102,60%	103,40%	94,90%	100,30%	4,69%	4,88%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	95,60%	91,30%	90,70%	92,53%	2,67%	2,89%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	132,20%	132,70%	94,40%	119,77%	21,97%	18,34%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	112,40%	121,10%	126,40%	119,97%	7,07%	5,89%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	97,40%	100,70%	99,80%	99,30%	1,71%	1,72%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	137,70%	109,80%	117,80%	121,80%	14,35%	11,78%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30											
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30											
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70						
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30											
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30											
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30											
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30											
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30	2	6,84	6,62	-120	-82						
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30											
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83						
METIX-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	98,60%	102,00%	100,30%	2,40%	2,40%	
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	110,00%	110,00%	110,00%	110,00%	0,00%	0,00%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66						
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62						
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,60	5	361						
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	85,60%	91,30%	90,70%	92,53%	2,67%	2,89%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	107,00%	107,00%	109,00%	107,67%	1,15%	1,07%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153						
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190						
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333						
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	109,60%	84,30%	90,70%	94,87%	13,15%	13,87%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294						
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310						
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397						
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425						
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	97,00%	86,00%	89,00%	90,67%	5,69%	6,27%

Essais (Boue totale)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Total (11 HAP)					
						initial	final	initial	final	Concentration totale (mg/kg)			Enlèvement (%)		
										A	B	C	Moyenne	Écart-type	Moyenne
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6.40	6.47	-270	-283	4,573	4,283	3,936	4,264	0,319	7,48%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6.40	6.37	-270	-277	57,543	61,360	53,942	57,615	3,709	6,44%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6.31	6.13	-250	1	34,128	33,832	33,970	34,010	0,104	0,31%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6.37	1,57	-277	398	36,604	39,277	40,185	38,689	1,861	4,81%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6.69	2,08	-345	326	49,154	45,768	49,428	47,784	1,858	3,89%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	51,449	50,128	50,419	50,666	0,694	1,37%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7.00	4,00	-172	171	53,200	51,029	52,338	52,169	1,093	2,09%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30						44,769	43,704	46,163	44,879	1,233	2,75%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30						49,773	54,932	53,738	52,814	2,701	5,11%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	50,483	49,500	51,946	50,643	1,231	2,43%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30						55,822	56,594	57,706	56,707	0,947	1,67%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30						52,939	52,251	57,219	54,136	2,692	4,97%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30						42,417	47,617	50,835	46,956	4,248	9,05%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	47,875	52,161	54,236	51,424	3,244	6,31%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30						51,425	54,297	55,839	53,854	2,240	4,16%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30						52,775	59,789	56,795	56,453	3,520	6,23%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	57,887	61,607	54,049	57,848	3,779	6,53%
METIX-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7.53	1,88	-58	370	58,852	57,405	56,776	57,678	1,064	1,85%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	64,295	63,607	64,519	64,140	0,476	0,74%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	62,540	63,898	64,332	63,590	0,935	1,47%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	55,120	57,099	59,847	57,356	2,374	4,14%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	7,11	7,16	-79	-58	56,627	54,811	53,866	55,101	1,404	2,55%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	53,289	50,388	52,609	52,096	1,517	2,91%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	46,417	45,107	40,767	44,097	2,957	6,71%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	40,683	37,917	35,233	37,944	2,725	7,18%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	34,683	32,928	32,235	33,282	1,262	3,79%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	34,696	33,945	34,652	34,431	0,421	1,22%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0,5	30	72	8,16	8,12	5	106	45,866	46,153	44,433	45,484	0,922	2,03%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,46	5	153	41,417	38,300	37,900	38,172	1,060	5,00%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	39,233	33,754	34,643	35,977	2,941	8,20%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	33,864	31,317	30,589	31,963	1,787	5,59%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	31,205	31,362	29,214	30,594	1,198	3,91%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	57,235	56,904	58,924	57,688	1,083	1,88%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	51,417	49,144	48,752	49,771	1,439	2,89%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	47,816	43,444	45,038	45,433	2,213	4,87%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	36,230	33,198	35,050	34,826	1,528	4,39%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	35,557	33,532	31,320	33,469	2,119	6,33%

Essais (Boue totale)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		HAP à 3 cycles (ACN, FLU, PHE)							
						initial	final	initial	final	Concentration totale (mg/kg)			Enlèvement (%)				
										A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,373	0,345	0,336	0,352	0,019	5,36%	0,00%	0,00%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	14,147	14,796	13,547	14,163	0,625	4,41%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	0,524	0,491	0,516	0,510	0,017	3,37%	96,40%	0,12%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	0,637	0,732	0,749	0,706	0,060	8,52%	95,01%	0,42%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	8,552	8,533	8,783	8,623	0,138	1,62%	39,12%	0,98%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	11,643	11,330	11,178	11,384	0,237	2,08%	19,63%	1,67%
ELECTSTAB	ELECTSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	6,712	5,944	6,375	6,344	0,385	6,07%	55,21%	2,72%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30						4,566	4,400	4,542	4,503	0,090	1,99%	73,17%	0,53%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30						9,933	10,550	10,125	10,203	0,316	3,09%	39,21%	1,88%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	10,908	11,183	11,125	11,072	0,145	1,31%	34,03%	0,86%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30						10,500	10,783	10,000	10,428	0,397	3,80%	37,87%	2,36%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30						9,067	8,833	9,042	8,981	0,128	1,43%	46,50%	0,76%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30						2,108	2,150	2,292	2,183	0,096	4,40%	86,99%	0,57%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	6,850	6,517	6,583	6,650	0,176	2,65%	60,38%	1,05%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30						9,817	10,250	10,292	10,119	0,263	2,60%	39,71%	1,57%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30						10,508	10,416	10,500	10,475	0,051	0,49%	37,59%	0,30%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	14,211	13,501	10,786	12,832	1,808	14,09%	23,55%	10,77%
Métx-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	13,112	13,136	12,648	12,965	0,275	2,12%	22,76%	1,64%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	16,831	16,745	16,778	16,785	0,043	0,26%	0,00%	0,26%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	16,857	16,033	16,438	16,443	0,412	2,51%	2,04%	2,46%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	12,963	13,354	13,458	13,268	0,261	1,97%	21,01%	1,59%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58	12,651	12,042	11,444	12,046	0,603	5,01%	28,23%	3,59%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	98	6,567	6,026	7,184	6,659	0,310	4,52%	59,14%	1,85%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	4,000	3,933	3,600	3,844	0,214	5,57%	77,10%	1,28%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	2,517	2,417	2,400	2,444	0,063	2,58%	85,44%	0,38%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	0,983	1,417	1,367	1,256	0,237	18,88%	92,52%	1,41%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	0,541	0,491	0,516	0,516	0,025	4,87%	96,93%	0,15%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	3,004	2,728	2,588	2,773	0,212	7,63%	83,48%	1,26%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	1,750	1,467	1,633	1,617	0,142	8,81%	90,37%	0,85%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	1,400	1,350	1,333	1,361	0,035	2,55%	91,89%	0,21%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	1,150	1,158	1,125	1,144	0,017	1,50%	93,18%	0,10%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	1,050	1,091	0,929	1,023	0,084	8,25%	93,90%	0,50%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	11,675	11,033	12,126	11,611	0,549	4,73%	30,62%	3,27%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	9,667	9,000	8,667	9,111	0,509	5,59%	45,72%	3,03%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	9,286	8,167	8,594	8,682	0,565	6,50%	48,27%	3,36%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	0,776	0,698	0,717	0,731	0,042	5,69%	95,65%	0,25%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	0,723	0,672	0,653	0,683	0,036	5,32%	95,93%	0,22%

Essais (Boue totale)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH	POR (mV)	HAP à 4 cycles (FLR, PYR)						Enlèvement (%)			
								Concentration totale (mg/kg)		A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
								initial	final								
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6.40	6.47	-270	-283	1,166	1,089	1,031	1,095	0,068	6,19%		
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6.40	6.37	-270	-277	9,550	10,852	8,971	9,791	0,963	9,84%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	5,177	5,393	5,184	5,251	0,122	2,33%	46,37%	1,25%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	388	8,130	8,032	8,738	8,300	0,983	4,61%	15,23%	3,51%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	328	7,371	7,059	9,013	7,814	1,050	13,44%	20,19%	10,72%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	9,137	9,003	9,165	9,102	0,087	0,95%	7,04%	0,89%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	12,267	10,856	11,733	11,619	0,713	6,13%	-18,66%	7,28%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30						9,550	10,117	10,167	9,944	0,343	3,44%	17,23%	2,85%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30						9,917	11,250	11,625	10,931	0,898	8,21%	9,02%	7,47%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,64	6,60	-120	-70	10,883	10,847	9,545	10,459	0,792	7,57%	12,95%	6,59%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30						11,050	12,300	12,333	11,894	0,732	6,15%	1,00%	6,09%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30						10,258	11,533	12,083	11,292	0,936	8,29%	6,02%	7,79%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30						9,667	10,250	10,583	10,167	0,464	4,56%	15,38%	3,86%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	10,225	10,983	11,500	10,963	0,641	5,88%	9,26%	5,34%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30						10,175	11,283	11,458	10,972	0,696	6,34%	8,68%	5,79%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30						10,633	11,183	9,917	10,578	0,635	6,00%	11,96%	5,29%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	12,340	12,494	10,821	11,885	0,925	7,78%	1,08%	7,70%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	13,631	11,376	12,118	12,375	1,150	9,29%	-3,00%	9,57%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	12,138	12,028	11,877	12,015	0,131	1,09%	0,00%	1,09%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	12,286	13,043	13,356	12,895	0,550	4,27%	-7,33%	4,58%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	11,420	11,801	13,333	12,185	1,013	8,31%	-1,41%	8,43%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58	11,145	10,733	10,741	10,873	0,235	2,17%	9,51%	1,96%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	11,331	11,164	11,017	11,171	0,157	1,41%	7,03%	1,31%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	10,700	11,200	9,667	10,522	0,782	7,43%	12,42%	6,51%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	8,167	10,500	7,500	8,722	1,575	18,06%	27,40%	13,11%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	8,200	4,933	5,000	5,344	0,746	13,95%	55,52%	6,21%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	5,353	5,393	5,184	5,310	0,111	2,09%	55,80%	0,92%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	105	12,084	11,549	11,047	11,560	0,519	4,49%	3,76%	4,32%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	9,833	8,833	9,000	9,222	0,536	5,81%	23,24%	4,46%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	8,667	7,500	7,500	7,889	0,674	8,54%	34,34%	5,61%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	4,833	4,737	4,464	4,678	0,191	4,09%	61,06%	1,59%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	3,163	2,909	3,143	3,078	0,146	4,81%	74,38%	1,23%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	11,725	11,854	11,823	11,801	0,067	0,57%	1,76%	0,56%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	11,333	11,167	10,667	11,056	0,347	3,14%	7,98%	2,89%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	10,714	10,500	9,688	10,301	0,542	5,26%	14,27%	4,51%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	7,937	9,000	9,500	8,812	0,798	9,06%	26,68%	6,65%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	8,167	8,167	8,000	8,289	3,61%	33,42%	2,40%	

Essais (Boue totale)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		HAP à 5 cycles (BJK, BAP)					
						initial	final	Initial	final	A	B	C	Moyenne	Écart-type	C.V. (%)
															Moyenne
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	1,636	1,681	1,485	1,668	0,176	10,56%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	21,633	23,091	20,560	21,761	1,270	5,84%
Digestion aérobio mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	19,071	17,984	18,643	18,633	0,574	3,06%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	16,275	18,475	19,182	17,977	1,516	8,43%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	19,457	19,530	20,273	19,753	0,451	2,28%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	19,844	18,685	18,738	19,094	0,657	3,44%
ELECTSTAB	ELECTSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	22,686	23,503	23,446	23,212	0,456	1,97%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30						22,056	18,368	20,014	20,146	1,847	9,17%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30						18,852	20,644	21,784	20,426	1,478	7,24%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	17,956	18,453	20,686	19,032	1,453	7,63%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30						23,524	21,206	23,796	22,842	1,423	6,23%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30						22,324	20,409	23,766	22,164	1,684	7,60%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30						20,117	23,233	26,500	23,283	3,192	13,71%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	20,317	23,450	24,963	22,910	2,370	10,34%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30						20,450	21,250	22,200	21,300	0,876	4,11%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30						20,817	24,957	24,151	23,308	2,195	9,42%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	20,385	23,387	21,439	21,737	1,523	7,01%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	21,620	21,118	21,083	21,274	0,300	1,41%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	23,370	23,395	23,130	23,298	0,146	0,63%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	22,286	23,641	23,286	23,072	0,703	3,05%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	20,679	21,429	22,083	21,397	0,703	3,28%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58	21,386	20,681	20,741	20,936	0,391	1,87%
Digestion aérobio mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	23,675	21,584	23,767	23,068	1,235	5,37%
Digestion aérobio mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	21,567	20,273	18,833	20,224	1,367	6,76%
Digestion aérobio mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	20,000	16,500	16,833	17,778	1,932	10,87%
Digestion aérobio mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	18,000	17,500	17,000	17,500	0,500	2,86%
Digestion aérobio mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	18,012	17,684	18,643	18,113	0,488	2,65%
Digestion aérobio mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	20,541	20,512	20,225	20,426	0,175	0,85%
Digestion aérobio mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	19,167	18,667	18,167	18,667	0,500	2,68%
Digestion aérobio mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	19,333	16,167	16,833	17,444	1,669	9,57%
Digestion aérobio mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	18,000	16,316	15,714	16,677	1,185	7,10%
Digestion aérobio mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	15,667	16,081	14,464	15,397	0,832	5,40%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	21,733	22,767	23,342	22,614	0,815	3,61%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	20,333	19,167	19,556	19,674	3,44%	16,06%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	18,143	16,000	17,031	17,058	1,072	6,28%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	18,095	15,333	16,500	16,643	1,386	8,33%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	17,667	16,000	15,167	16,278	1,273	7,82%

Essais (Boue totale)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		HAP à 6 cycles (INP, BPR)					
						initial	final	initial	final	Concentration totale (mg/kg)			Enlèvement (%)		
						A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type	C.V. (%)	Moyenne
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	1,197	1,167	1,083	1,149	0,059	5,16%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	12,214	12,620	10,865	11,889	0,919	7,72%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	-1	9,356	10,065	9,427	9,616	0,391	4,06%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	11,562	12,037	11,516	11,705	0,269	2,47%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	12,774	10,646	11,360	11,593	1,083	1,63%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	10,821	11,110	11,338	11,090	0,250	2,34%
ELECTSTAB	ELECTSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	11,535	10,726	10,785	11,015	0,451	4,09%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30						8,597	10,819	11,440	10,285	1,495	14,54%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30						11,071	12,489	10,204	11,255	1,153	10,25%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	10,733	8,917	10,591	10,080	1,010	10,02%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30						10,749	12,305	11,577	11,543	0,779	6,75%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30						11,290	11,475	12,328	11,698	0,554	4,73%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30						10,525	11,983	11,460	11,323	0,739	6,52%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	10,483	11,211	11,189	10,961	0,414	3,78%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30						10,983	11,513	11,890	11,462	0,455	3,97%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30						10,816	13,232	12,227	12,092	1,214	10,04%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	10,951	12,225	11,003	11,393	0,721	6,33%
Metic-AC + Tween 80	METIK-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	10,489	11,776	10,928	11,064	0,654	5,91%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	11,956	11,438	12,734	12,043	0,652	5,42%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	11,111	11,180	11,250	11,180	0,069	0,62%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	10,059	10,515	10,972	10,515	0,457	4,34%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58	11,447	11,355	10,940	11,247	0,270	2,40%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	11,171	10,816	10,641	11,058	0,577	6,60%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	10,150	9,700	8,667	9,506	0,761	8,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	10,000	8,500	8,500	9,000	0,866	9,62%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	9,500	9,178	8,868	8,182	0,316	3,44%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	10,790	10,378	10,309	10,492	0,260	2,48%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	10,436	11,363	10,572	10,724	0,579	5,40%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	10,667	9,333	9,000	9,607	0,892	9,12%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	9,833	8,737	8,976	9,162	0,577	6,28%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	10,000	9,107	9,286	9,464	0,473	5,00%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	11,305	11,301	10,678	11,095	0,361	3,25%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	12,102	11,250	11,633	11,662	0,427	3,66%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	10,083	9,810	10,252	10,048	0,223	2,22%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	9,673	8,778	9,725	9,392	0,533	5,67%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	9,421	8,167	8,333	8,640	0,681	7,88%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	9,000	8,893	7,833	8,509	0,605	7,11%

Essais (Boue totale)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Acénaphthène (ACN)							
						initial	final	initial	final	Concentration totale (mg/kg)			Enlèvement (%)				
										A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,057	0,055	0,059	0,057	0,002	3,99%		
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	4,507	4,521	4,701	4,576	0,108	2,37%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	0,102	0,117	0,107	0,108	0,008	7,15%	97,63%	0,17%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	0,115	0,125	0,130	0,123	0,008	6,37%	97,30%	0,17%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	2,678	1,908	2,409	2,332	0,390	16,74%	49,05%	8,53%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	3,832	3,732	3,678	3,747	0,078	2,08%	18,11%	1,71%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	1,827	1,717	1,803	1,782	0,058	3,25%	61,05%	1,27%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30						1,233	1,217	1,208	1,219	0,013	1,04%	77,97%	0,23%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30						3,375	3,417	3,375	3,389	0,024	0,71%	38,78%	0,43%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	3,667	3,667	3,667	3,667	0,000	0,00%	33,77%	0,00%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30						3,392	3,450	3,208	3,350	0,126	3,77%	39,49%	2,28%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30						2,883	2,763	2,917	2,861	0,069	2,43%	48,32%	1,25%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30						0,458	0,483	0,542	0,494	0,043	8,66%	91,07%	0,77%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	2,075	1,900	1,917	1,964	0,097	4,92%	64,52%	1,74%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30						2,900	3,000	3,125	3,008	0,113	3,75%	45,66%	2,04%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30						3,342	3,333	3,958	3,544	0,359	10,12%	35,98%	6,48%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	4,281	4,124	3,321	3,909	0,515	13,17%	29,40%	9,30%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	3,945	4,012	3,636	3,864	0,201	5,20%	30,20%	3,63%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	5,535	5,531	5,542	5,536	0,006	0,10%	0,00%	0,10%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	5,429	5,163	5,137	5,243	0,161	3,08%	5,30%	2,91%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	3,704	4,037	3,667	3,803	0,204	5,37%	31,31%	3,69%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	7,11	7,16	-79	-58	3,916	3,665	3,370	3,650	0,273	7,49%	34,06%	4,93%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	98	1,633	1,767	1,917	1,772	0,142	8,02%	67,89%	2,57%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	0,950	0,600	0,817	0,889	0,067	7,58%	83,94%	1,22%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,80	5	275	0,517	0,467	0,483	0,469	0,025	5,21%	91,17%	0,46%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	0,217	0,333	0,300	0,263	0,060	21,21%	94,88%	1,09%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	0,127	0,117	0,107	0,117	0,010	8,57%	97,89%	0,18%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	0,410	0,359	0,344	0,371	0,034	9,30%	93,30%	0,62%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	0,267	0,250	0,300	0,272	0,025	9,35%	95,08%	0,46%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	0,217	0,200	0,200	0,206	0,010	4,68%	86,29%	0,17%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	0,200	0,193	0,214	0,202	0,011	5,36%	96,34%	0,20%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	0,167	0,167	0,161	0,165	0,003	2,09%	97,03%	0,06%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	3,275	3,483	3,217	3,325	0,140	4,21%	39,94%	2,53%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	2,833	2,667	2,778	2,096	0,346%	49,82%	1,74%	
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	2,571	2,333	2,500	2,468	0,122	4,95%	55,41%	2,21%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	0,190	0,165	0,167	0,174	0,014	8,19%	96,86%	0,26%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	0,157	0,138	0,137	0,144	0,011	7,71%	97,40%	0,20%

Essais (Boue totale)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Fluoréne (FLU)						Enlèvement (%)				
						initial	final	initial	final	Concentration totale (mg/kg)			A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	16	0	6,40	6,47	-270	-283	0,079	0,060	0,063	0,067	0,010	15,42%	0,00%	0,00%			
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	16	0	6,40	6,37	-270	-277	4,676	4,447	4,320	4,481	0,181	4,03%	97,31%	0,06%			
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	16	504	6,31	6,13	-256	1	0,124	0,120	0,118	0,121	0,003	2,25%	6,24%	0,29%			
METIX-BS	METIX-BS	Centrifugation	0	16	240	6,37	1,57	-277	398	0,196	0,222	0,207	0,208	0,013	6,24%	0,35%	34,36%			
METIX-AC	METIX-AC	Centrifugation	0	16	4	6,69	2,08	-345	326	3,013	2,836	3,274	2,841	0,374	12,73%	8,35%				
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	260	4,426	4,430	4,259	4,372	0,097	2,23%	2,43%	2,18%			
ELECTSTAB	ELECTSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	2,344	2,225	2,321	2,297	0,063	2,76%	48,75%	1,42%			
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30						1,558	1,500	1,542	1,533	0,030	1,95%	72,33%	0,54%			
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30						3,200	3,567	3,292	3,363	0,191	5,69%	39,80%	3,44%			
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	3,633	3,850	3,833	3,772	0,121	3,20%	31,94%	2,18%			
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30						3,442	3,717	3,292	3,483	0,216	6,19%	37,15%	3,89%			
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30						3,208	3,083	3,083	3,125	0,072	2,31%	43,61%	1,30%			
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30						1,125	1,167	1,167	1,153	0,024	2,09%	79,20%	0,43%			
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	2,467	2,433	2,375	2,425	0,046	1,91%	56,24%	0,84%			
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30						3,200	3,500	3,458	3,386	0,163	4,80%	38,80%	2,93%			
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30						3,417	3,383	3,000	3,267	0,232	7,09%	41,06%	4,18%			
Tween 80 (essai C)	Tw80 - C	Centrifugation (3000 x g)	2,0	30						4,012	4,037	3,958	4,003	0,040	1,01%	27,78%	0,73%			
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	6,84	6,59	-120	-83	4,499	4,160	3,342	4,000	0,595	14,87%	27,82%	10,73%			
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	4	7,53	1,88	-58	370	4,508	4,762	4,321	4,530	0,222	4,89%	18,25%	4,00%			
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	5,551	5,536	5,540	5,542	0,008	0,14%	0,00%	0,14%			
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	5,429	5,163	5,479	5,357	0,170	3,17%	3,34%	3,07%			
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	4,012	4,037	3,958	4,003	0,040	1,01%	27,78%	0,73%			
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	7,11	7,16	-79	-58	3,916	3,665	3,630	3,737	0,156	4,17%	32,57%	2,81%			
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	2,767	2,767	2,975	2,836	0,120	4,23%	48,82%	2,17%			
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	1,667	1,609	1,267	1,511	0,214	14,18%	72,73%	3,87%			
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	0,883	0,860	0,817	0,833	0,044	5,29%	84,96%	0,80%			
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	0,333	0,517	0,500	0,450	0,101	22,53%	91,88%	1,83%			
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	0,157	0,120	0,118	0,132	0,022	16,66%	97,62%	0,40%			
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	1,851	1,351	1,368	1,457	0,169	11,57%	73,71%	3,04%			
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	0,650	0,633	0,700	0,661	0,035	5,25%	88,07%	0,63%			
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	0,517	0,500	0,450	0,469	0,035	7,10%	91,18%	0,63%			
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	0,433	0,456	0,446	0,445	0,011	2,57%	91,97%	0,21%			
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	0,363	0,424	0,357	0,368	0,034	8,71%	92,99%	0,61%			
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	4,058	4,058	4,417	4,178	0,207	4,96%	24,62%	3,74%			
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	3,333	3,167	3,000	3,167	0,167	5,26%	42,86%	3,01%			
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	3,000	2,833	2,969	2,934	0,089	3,02%	47,06%	1,60%			
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	0,270	0,250	0,250	0,257	0,011	4,46%	95,37%	0,21%			
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	0,250	0,233	0,238	0,230	0,010	4,03%	95,69%	0,17%			

Essais (Boue totale)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Phénanthrène (PHE)							
						initial	final	initial	final	Concentration totale (mg/kg)			Enlèvement (%)				
										A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,237	0,231	0,215	0,228	0,012	5,13%	12,99%	0,00%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	4,964	5,629	4,526	5,106	0,663	12,99%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	0,299	0,254	0,291	0,281	0,024	8,55%	94,49%	0,47%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	0,326	0,385	0,412	0,374	0,044	11,65%	92,67%	0,85%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	2,862	4,088	3,100	3,350	0,650	19,41%	34,40%	12,73%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	3,385	3,168	3,240	3,264	0,110	3,38%	36,07%	2,16%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	2,541	2,002	2,251	2,265	0,270	11,90%	55,65%	5,28%
Tween 80 (essai A)	TW80 - A	Filtration	0,5	30						1,775	1,683	1,792	1,750	0,058	3,34%	69,33%	1,02%
Tween 80 (essai A)	TW80 - A	Centrifugation (500 x g)	0,5	30						3,358	3,567	3,458	3,461	0,104	3,01%	39,35%	1,83%
Tween 80 (essai A)	TW80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	3,609	3,667	3,625	3,633	0,030	0,83%	36,33%	0,53%
Tween 80 (essai A)	TW80 - A	Centrifugation (2000 x g)	0,5	30						3,667	3,617	3,500	3,594	0,086	2,36%	37,01%	1,50%
Tween 80 (essai A)	TW80 - A	Centrifugation (3000 x g)	0,5	30						2,975	2,967	3,042	2,994	0,041	1,37%	47,53%	0,72%
Tween 80 (essai C)	TW80 - C	Filtration	2,0	30						0,525	0,500	0,583	0,536	0,043	7,97%	90,61%	0,75%
Tween 80 (essai C)	TW80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	2,308	2,183	2,292	2,261	0,068	3,00%	60,38%	1,19%
Tween 80 (essai C)	TW80 - C	Centrifugation (1000 x g)	2,0	30						3,717	3,750	3,708	3,725	0,022	0,56%	34,73%	0,39%
Tween 80 (essai C)	TW80 - C	Centrifugation (2000 x g)	2,0	30						3,750	3,700	3,542	3,664	0,109	2,96%	35,79%	1,90%
Tween 80 (essai B)	TW80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	5,431	5,217	4,123	4,924	0,702	14,25%	13,72%	12,29%
METIX-AC + Tween 80	METIX-AC + TW80	Filtration	0,5	30	4	7,53	1,88	-58	370	4,658	4,362	4,691	4,570	0,182	3,97%	19,91%	3,18%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	5,745	5,678	5,696	5,707	0,034	0,60%	0,00%	0,60%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	6,000	5,707	5,822	5,843	0,148	2,53%	-2,39%	2,59%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	5,247	5,280	5,833	5,453	0,330	6,04%	4,44%	5,78%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	7,11	7,16	-79	-58	4,919	4,712	4,444	4,659	0,193	4,14%	18,37%	3,38%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	98	2,167	2,292	2,292	2,250	0,072	3,21%	60,57%	1,26%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	1,383	1,433	1,517	1,444	0,067	4,66%	74,69%	1,18%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	1,117	1,150	1,100	1,122	0,025	2,27%	80,33%	0,45%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	8,50	5	361	0,433	0,567	0,567	0,522	0,077	14,74%	90,85%	1,35%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	0,257	0,254	0,291	0,267	0,021	7,69%	95,32%	0,36%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + TW80	Filtration	0,5	30	72	8,16	8,12	5	106	0,943	1,018	0,876	0,946	0,071	7,50%	83,43%	1,24%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + TW80	Filtration	0,5	30	120	8,16	8,48	5	153	0,833	0,583	0,633	0,683	0,132	19,36%	68,03%	2,32%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + TW80	Filtration	0,5	30	168	8,16	8,45	5	190	0,667	0,650	0,683	0,667	0,017	2,50%	88,32%	0,29%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + TW80	Filtration	0,5	30	336	8,16	8,06	5	333	0,517	0,509	0,464	0,497	0,028	5,69%	91,30%	0,49%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + TW80	Filtration	0,5	30	504	8,16	8,43	5	446	0,600	0,500	0,411	0,470	0,052	10,96%	91,76%	0,90%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	4,342	3,492	4,492	4,109	0,539	13,12%	28,00%	9,45%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	3,500	3,000	3,000	3,167	0,289	9,12%	44,51%	5,06%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	3,714	3,000	3,125	3,280	0,381	11,63%	42,53%	6,66%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	0,317	0,283	0,300	0,300	0,017	5,66%	94,74%	0,30%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	0,317	0,300	0,283	0,300	0,017	5,56%	94,74%	0,29%

Essais (Boue totale)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Fluoranthène (FLR)						Enlèvement (%)	
						initial	final	initial	final	Concentration totale (mg/kg)			Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
										A	B	C					
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,568	0,527	0,504	0,533	0,032	6,05%	0,00%	0,00%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	4,754	5,535	4,468	4,919	0,553	11,23%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	2,269	2,299	2,286	0,023	1,00%	53,53%	0,47%	
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	3,841	3,828	3,989	3,886	0,088	2,30%	21,00%	1,81%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	3,763	3,639	4,630	4,011	0,540	13,46%	18,46%	10,97%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	4,272	4,719	4,841	4,611	0,299	6,49%	6,26%	6,09%
ELECTESTAB	ELECTESTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	6,274	5,384	5,783	5,814	0,445	7,66%	-18,19%	9,06%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30						4,367	4,550	4,542	4,486	0,104	2,31%	25,13%	1,73%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30						5,117	5,833	5,958	5,636	0,454	8,06%	5,94%	7,58%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	5,542	4,964	4,905	5,137	0,352	6,85%	14,27%	5,87%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30						5,650	6,300	6,292	6,081	0,373	6,13%	-1,48%	6,22%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30						5,200	5,800	6,083	5,694	0,451	7,92%	4,97%	7,53%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30						4,775	5,083	5,250	5,036	0,241	4,79%	15,95%	4,02%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	5,092	5,533	5,875	5,500	0,393	7,14%	8,21%	6,55%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30						5,308	5,783	5,875	5,656	0,304	5,38%	5,62%	5,08%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30						5,500	5,667	4,792	5,320	0,402	8,73%	11,22%	7,75%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	6,338	6,154	5,389	5,961	0,503	8,45%	0,53%	8,40%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	6,717	5,744	6,550	6,337	0,520	8,21%	-5,76%	8,68%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	6,062	5,993	5,921	5,992	0,071	1,18%	0,00%	1,18%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	6,000	6,793	6,507	6,433	0,402	6,25%	-7,37%	6,71%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	5,864	6,211	6,667	6,247	0,402	6,44%	-4,26%	6,72%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58	5,723	5,497	5,556	5,592	0,117	2,09%	6,68%	1,95%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	5,714	5,589	5,550	5,618	0,086	1,53%	6,25%	1,43%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	5,533	5,833	5,000	5,456	0,422	7,74%	8,95%	7,04%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	4,167	5,867	3,833	4,556	0,977	21,44%	23,97%	16,30%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	3,150	2,000	2,167	2,438	0,621	25,48%	56,30%	10,37%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	2,273	2,299	2,290	0,016	0,65%	61,77%	0,25%	
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	6,044	5,740	5,456	5,747	0,294	5,12%	4,09%	4,91%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	5,000	4,500	4,500	4,867	0,289	6,19%	22,12%	4,82%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	4,333	3,833	3,833	4,000	0,289	7,22%	33,24%	4,82%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	2,500	2,456	2,321	2,426	0,093	3,84%	59,52%	1,55%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	1,667	1,515	1,643	1,608	0,081	5,07%	73,16%	1,36%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	5,900	5,979	5,998	5,959	0,052	0,87%	0,55%	0,87%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	5,833	5,833	5,500	5,722	0,192	3,36%	4,50%	3,21%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	5,571	5,500	5,000	5,357	0,311	5,81%	10,60%	5,20%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	3,651	4,667	5,000	4,439	0,703	15,83%	25,92%	11,73%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	3,667	3,500	3,333	3,500	0,167	4,76%	41,59%	2,78%

Essais (Boue totale)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Pyrène (PYR)							
						initial	final	initial	final	Concentration totale (mg/kg)			Enlèvement (%)				
										A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,598	0,562	0,527	0,562	0,036	6,36%		
Contrôle dopé et non-traité (DCC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	4,797	5,317	4,504	4,872	0,412	8,45%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	2,918	3,093	2,865	2,966	0,112	3,77%	39,13%	2,30%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	4,288	4,204	4,750	4,414	0,294	6,65%	9,41%	6,03%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	3,608	3,420	4,363	3,804	0,511	13,43%	21,93%	10,48%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	4,865	4,284	4,324	4,491	0,324	7,22%	7,83%	6,65%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	5,994	5,471	5,950	5,805	0,290	4,99%	-19,14%	5,95%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30						5,183	5,567	5,625	5,458	0,240	4,40%	9,37%	3,98%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30						4,800	5,417	5,667	5,294	0,446	8,43%	12,09%	7,41%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	5,342	5,983	4,640	5,322	0,672	12,63%	11,64%	11,16%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30						5,400	6,000	6,042	5,814	0,359	6,18%	3,47%	5,96%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30						5,058	5,733	6,000	5,597	0,485	8,67%	7,07%	8,06%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30						4,892	5,167	5,333	5,131	0,223	4,35%	14,81%	3,70%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	5,133	5,450	5,625	5,403	0,249	4,61%	10,29%	4,14%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30						4,867	5,500	5,583	5,317	0,392	7,37%	11,72%	6,51%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30						5,133	5,517	5,125	5,258	0,224	4,26%	12,69%	3,72%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	6,002	6,340	5,432	5,925	0,459	7,74%	1,63%	7,62%
Metix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	6,914	5,631	5,567	6,038	0,760	12,59%	-0,25%	12,62%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	6,076	6,036	5,957	6,023	0,060	1,00%	0,00%	1,00%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	6,286	6,250	6,849	6,462	0,336	5,20%	-7,29%	5,58%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	5,556	5,590	6,667	5,937	0,622	10,64%	1,42%	10,49%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	7,11	7,16	-79	-58	5,422	5,298	5,185	5,241	0,125	2,36%	12,32%	2,07%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	5,617	5,575	5,467	5,553	0,077	1,39%	7,80%	1,28%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	5,167	5,367	4,667	5,067	0,361	7,12%	15,68%	5,99%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	4,000	4,833	3,667	4,167	0,601	14,42%	30,82%	9,98%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	3,050	2,833	2,833	2,906	0,125	4,31%	51,76%	2,08%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	3,080	3,093	2,885	3,020	0,116	3,86%	49,86%	1,93%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	6,040	5,809	5,591	5,813	0,225	3,87%	3,48%	3,73%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	4,833	4,333	4,500	4,556	0,255	5,59%	24,36%	4,23%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	4,333	3,667	3,667	3,889	0,385	9,90%	35,43%	6,39%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	2,333	2,281	2,143	2,252	0,098	4,37%	62,60%	1,63%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	1,517	1,394	1,500	1,470	0,067	4,53%	75,59%	1,11%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	5,825	5,875	5,825	5,842	0,029	0,49%	3,01%	0,48%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	5,500	5,333	5,167	5,333	0,167	3,13%	11,46%	2,77%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	5,143	5,000	4,688	4,943	0,233	4,71%	17,92%	3,87%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	4,286	4,333	4,500	4,373	0,113	2,57%	27,39%	1,87%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	4,500	4,667	4,333	4,500	0,167	3,70%	25,28%	2,77%

Essais (Boue totale)	Code	Déshydration	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Benzo[b]fluoranthènes (BJK)						Enlèvement (%)	
						initial	final	initial	final	Concentration totale (mg/kg)			Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
										A	B	C					
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	1,362	1,246	1,111	1,240	0,126	10,14%	0,00%	0,00%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	16,567	18,025	15,861	16,817	1,104	6,56%	0,00%	0,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	14,839	13,218	14,079	14,046	0,811	5,77%	16,48%	4,82%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	11,984	13,725	13,988	13,232	1,089	8,23%	21,32%	6,48%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,68	2,08	-345	326	15,165	15,200	15,718	15,361	0,310	2,02%	8,66%	1,84%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	280	14,686	13,697	13,741	14,041	0,559	3,98%	16,51%	3,32%
ELECTSTAB	ELECTSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	16,775	18,073	17,801	17,550	0,685	3,90%	-4,35%	4,07%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30						17,117	13,057	14,330	14,835	2,076	14,00%	14,77%	11,93%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30						13,529	14,891	16,033	14,788	1,252	8,47%	15,04%	7,19%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	12,342	13,900	14,125	13,456	0,971	7,22%	22,70%	5,58%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30						17,692	15,406	16,176	17,058	1,460	8,56%	2,00%	8,39%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30						16,625	15,118	16,139	16,627	1,511	9,09%	4,47%	8,68%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30						15,000	17,300	19,792	17,364	2,396	13,80%	0,24%	13,77%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	15,175	18,283	18,750	17,403	1,943	11,17%	0,02%	11,16%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30						15,292	15,083	17,083	15,819	1,099	6,95%	9,12%	6,32%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30						15,175	18,283	18,958	17,472	2,018	11,55%	-0,38%	11,59%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	15,202	17,477	15,876	16,185	1,168	5,50%	7,01%	6,71%
Metic-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	15,449	15,368	15,639	15,485	0,139	0,90%	11,04%	0,80%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	17,455	17,483	17,281	17,406	0,109	0,63%	0,00%	0,63%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	16,571	17,391	17,123	17,029	0,418	2,45%	2,17%	2,40%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	15,432	15,839	16,250	15,840	0,409	2,58%	9,00%	2,35%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	7,11	7,16	-79	-58	15,964	15,183	15,556	15,568	0,390	2,51%	10,56%	2,24%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	17,458	15,392	17,667	16,839	1,257	7,47%	3,26%	7,22%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	16,517	15,273	14,167	15,319	1,176	7,67%	11,99%	6,75%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	15,167	12,333	12,833	13,444	1,512	11,25%	22,76%	8,69%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	13,333	12,833	13,000	13,056	0,255	1,95%	24,99%	1,46%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	13,548	13,219	14,079	13,615	0,434	3,19%	21,78%	2,50%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	14,782	15,902	14,449	15,044	0,761	5,06%	13,57%	4,37%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	14,233	14,000	12,600	13,044	0,419	3,01%	19,89%	2,41%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	14,500	12,167	12,500	13,056	1,262	9,67%	24,99%	7,25%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	13,667	12,281	11,766	12,576	0,975	7,75%	27,74%	5,60%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	15,075	17,492	17,567	17,011	0,898	5,28%	2,27%	5,16%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	14,833	12,424	10,893	11,717	0,772	6,59%	32,69%	4,44%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	15,167	14,333	14,333	14,611	0,481	3,29%	16,06%	2,76%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	13,571	12,000	12,813	12,795	0,786	6,14%	26,49%	4,51%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	13,968	11,833	12,833	12,878	1,068	8,29%	26,01%	6,14%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	13,667	12,333	11,833	12,611	0,948	7,51%	27,55%	5,44%

Essais (Boue totale)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Benzo[a]pyrène (BAP)					
						initial	final	initial	final	Concentration totale (mg/kg)			Enlèvement (%)		
										A	B	C	Moyenne	Écart-type	C.V. (%)
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,474	0,435	0,374	0,428	0,050	11,7%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	5,066	5,066	4,699	4,944	0,212	4,29%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	4,232	4,765	4,764	4,587	0,307	6,70%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	4,291	4,750	5,193	4,745	0,451	9,50%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	4,292	4,330	4,554	4,392	0,142	3,22%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	5,162	4,988	4,997	5,049	0,098	1,94%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	5,911	5,430	5,645	5,662	0,241	4,26%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30						4,939	5,312	5,684	5,312	0,372	7,01%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30						5,323	5,842	5,751	5,639	0,277	4,92%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	5,617	4,553	6,560	5,577	1,005	16,01%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30						5,932	5,800	5,620	5,784	0,157	2,71%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30						5,699	5,292	5,627	5,539	0,217	3,92%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30						5,117	5,933	6,708	5,919	0,796	13,45%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	5,142	5,167	6,213	5,507	0,811	11,10%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30						5,158	6,167	5,116	5,480	0,595	10,85%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30						5,642	6,674	5,193	5,836	0,759	13,01%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	5,182	5,910	5,563	5,552	0,364	6,56%
Matix-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	6,170	5,750	5,444	5,788	0,365	6,30%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	5,916	5,912	5,849	5,892	0,037	0,63%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	5,714	6,250	6,164	6,043	0,288	4,76%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	5,247	5,590	5,833	5,557	0,295	5,30%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-56	5,422	5,497	5,185	5,368	0,163	3,03%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	6,217	6,192	6,100	6,169	0,061	1,00%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	5,050	5,000	4,667	4,906	0,208	4,25%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	4,833	4,167	4,000	4,333	0,441	10,18%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	4,667	4,667	4,000	4,444	0,385	8,66%
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	4,464	4,465	4,564	4,498	0,057	1,27%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	5,760	4,610	5,777	5,382	0,669	12,43%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	4,833	4,667	4,667	4,722	0,096	2,04%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	4,833	4,000	4,333	4,389	0,419	9,56%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	4,333	4,035	3,929	4,099	0,210	5,12%
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	3,833	3,636	3,571	3,680	0,136	3,71%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	5,758	5,275	5,775	5,603	0,284	5,07%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	5,167	4,833	4,833	4,944	0,192	3,89%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	4,571	4,000	4,219	4,263	0,288	6,76%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	4,127	3,500	3,667	3,765	0,325	8,63%
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	4,000	3,667	3,333	3,667	0,333	9,09%

Essais (Boue totale)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH				POR (mV)				Indéno[1,2,3-cd]pyrène (INP)						Enlèvement (%)	
						pH		POR (mV)		Concentration totale (mg/kg)											
						initial	final	initial	final	A	B	C	Moyenne	Ecart-type	C.V. (%)	Moyenne	Ecart-type				
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6.40	6.47	-270	-283	0,638	0,623	0,563	0,608	0,040	6,54%	0,00%	0,00%	0,00%	0,00%		
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6.40	6.37	-270	-277	5,588	6,343	5,012	5,648	0,667	11,82%	0,00%	0,00%	17,93%	4,53%		
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	4,357	4,687	4,861	4,635	0,256	5,52%	2,30%	1,26%	2,33%	1,27%		
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	5,569	5,813	5,775	5,719	0,131	0,131	2,30%	1,26%	1,26%	1,26%		
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	6,513	5,305	6,256	6,025	0,636	10,56%	6,67%	11,27%	11,27%	11,27%		
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	5,111	5,867	5,548	5,442	0,293	5,38%	3,64%	5,18%	5,18%	5,18%		
ELECTSTAB	ELECTSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	5,706	5,329	5,626	5,553	0,199	3,58%	1,67%	3,52%	3,52%	3,52%		
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30						4,483	5,631	5,491	5,202	0,627	12,05%	13,79%	10,38%	10,38%	10,38%		
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30						6,034	6,244	5,380	5,886	0,451	7,66%	2,45%	7,47%	7,47%	7,47%		
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	4,125	4,683	5,167	4,658	0,521	11,19%	22,80%	8,64%	8,64%	8,64%		
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30						5,487	6,267	5,314	5,689	0,508	8,93%	5,71%	8,42%	8,42%	8,42%		
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30						5,165	5,375	5,756	5,432	0,300	5,52%	9,98%	4,97%	4,97%	4,97%		
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30						5,542	6,300	5,168	5,670	0,577	10,17%	6,04%	9,56%	9,56%	9,56%		
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	5,375	5,144	5,348	5,289	0,126	2,39%	12,35%	2,09%	2,09%	2,09%		
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30						5,375	5,447	5,348	5,390	0,051	0,95%	10,66%	0,84%	0,84%	0,84%		
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30						5,458	6,667	5,685	5,937	0,642	10,82%	1,62%	10,65%	10,65%	10,65%		
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	5,358	6,208	5,676	5,814	0,428	7,36%	3,65%	7,09%	7,09%	7,09%		
METIX-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	5,328	6,256	5,896	5,827	0,468	8,03%	3,44%	7,75%	7,75%	7,75%		
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	6,088	6,003	6,013	6,034	0,045	0,75%	0,00%	0,75%	0,75%	0,75%		
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	5,556	5,280	5,417	5,417	0,138	2,55%	10,22%	2,29%	2,29%	2,29%		
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	4,938	5,280	5,417	5,211	0,246	4,73%	13,63%	4,08%	4,08%	4,08%		
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58	5,422	5,497	4,815	5,245	0,374	7,13%	13,08%	6,20%	6,20%	6,20%		
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	96	6,250	5,925	5,808	5,984	0,229	3,82%	0,66%	3,80%	3,80%	3,80%		
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	5,150	5,367	4,333	4,950	0,545	11,01%	17,97%	9,03%	9,03%	9,03%		
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	5,167	4,000	4,389	4,674	0,674	15,35%	27,27%	11,16%	11,16%	11,16%		
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	4,500	4,333	4,333	4,389	0,098	2,19%	27,27%	1,59%	1,59%	1,59%		
Digestion aérobie mésophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	4,859	4,687	4,661	4,802	0,100	2,08%	20,42%	1,66%	1,66%	1,66%		
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	108	5,003	6,311	5,636	5,650	0,654	11,58%	6,37%	10,84%	10,84%	10,84%		
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,48	5	153	5,667	4,667	4,590	4,944	0,631	12,76%	18,06%	10,46%	10,46%	10,46%		
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	5,000	4,000	4,333	4,444	0,505	11,46%	26,34%	8,44%	8,44%	8,44%		
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	5,167	4,561	4,464	4,731	0,381	8,05%	21,60%	6,31%	6,31%	6,31%		
Digestion aérobie mésophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	5,000	4,848	4,464	4,771	0,276	5,79%	20,93%	4,58%	4,58%	4,58%		
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	5,936	6,417	6,633	6,329	0,357	5,64%	4,8%	5,52%	5,52%	5,52%		
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	5,167	4,833	4,833	4,944	0,192	3,89%	18,06%	3,19%	3,19%	3,19%		
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	5,070	4,778	5,225	5,024	0,227	4,52%	16,73%	3,76%	3,76%	3,76%		
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	4,921	3,833	4,500	4,418	0,548	12,41%	26,76%	9,09%	9,09%	9,09%		
METIX-BS	METIX-BS	Centrifugation	0	30	504	7,19	1,42	-51	461	4,333	4,167	3,833	4,111	0,255	6,19%	31,87%	4,22%	4,22%	4,22%		

Essais (Boue totale)	Code	Déshydratation	Tw80 (g/L)	ST (g/L)	Temps (h)	pH		POR (mV)		Benzo[ghi]pérylène (BPR)							
						initial	final	initial	final	Concentration totale (mg/kg)			Enlèvement (%)				
										A	B	C	Moyenne	Écart-type	C.V. (%)	Moyenne	Écart-type
Contrôle non-dopé et non-traité (NDC)	NDC	Centrifugation	0	18	0	6,40	6,47	-270	-283	0,559	0,545	0,520	0,541	0,020	3,66%	0,00%	0,00%
Contrôle dopé et non-traité (DOC)	DOC	Centrifugation	0	18	0	6,40	6,37	-270	-277	6,625	6,278	5,852	6,252	0,387	6,19%	0,00%	0,00%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	18	504	6,31	6,13	-250	1	4,998	5,378	4,566	4,981	0,407	8,16%	20,33%	6,50%
METIX-BS	METIX-BS	Centrifugation	0	18	240	6,37	1,57	-277	398	5,993	6,224	5,741	5,986	0,242	4,04%	4,25%	3,87%
METIX-AC	METIX-AC	Centrifugation	0	18	4	6,69	2,08	-345	326	6,262	5,341	5,103	5,569	0,612	10,99%	10,92%	9,79%
STABIOX	STABIOX	Centrifugation	0	18	1,33	6,78	4,00	-41	290	5,710	5,443	5,790	5,648	0,182	3,22%	9,66%	2,90%
ELECSTAB	ELECSTAB	Centrifugation	0	18	1	7,00	4,00	-172	171	5,829	5,397	5,159	5,462	0,340	6,22%	12,64%	5,43%
Tween 80 (essai A)	Tw80 - A	Filtration	0,5	30						4,114	5,188	5,949	5,084	0,922	18,13%	15,40%	15,34%
Tween 80 (essai A)	Tw80 - A	Centrifugation (500 x g)	0,5	30						5,037	6,244	4,824	5,368	0,766	14,27%	10,65%	12,75%
Tween 80 (essai A)	Tw80 - A	Centrifugation (1000 x g)	0,5	30	6	6,84	6,60	-120	-70	6,608	4,233	5,424	5,422	1,188	21,90%	9,76%	19,76%
Tween 80 (essai A)	Tw80 - A	Centrifugation (2000 x g)	0,5	30						5,261	6,038	6,263	5,854	0,525	8,98%	2,57%	8,74%
Tween 80 (essai A)	Tw80 - A	Centrifugation (3000 x g)	0,5	30						6,125	6,100	6,572	6,266	0,266	4,24%	4,28%	4,42%
Tween 80 (essai C)	Tw80 - C	Filtration	2,0	30						4,983	5,683	6,292	5,653	0,655	11,58%	5,92%	10,90%
Tween 80 (essai C)	Tw80 - C	Centrifugation (500 x g)	2,0	30	2	6,84	6,62	-120	-82	5,108	6,067	5,841	5,672	0,501	8,83%	5,60%	8,34%
Tween 80 (essai C)	Tw80 - C	Centrifugation (1000 x g)	2,0	30						5,608	6,067	6,542	6,072	0,467	7,69%	1,06%	7,77%
Tween 80 (essai C)	Tw80 - C	Centrifugation (2000 x g)	2,0	30						5,358	6,566	6,542	6,155	0,690	11,22%	2,44%	11,49%
Tween 80 (essai B)	Tw80 - B	Filtration	0,5	30	4	6,84	6,59	-120	-83	5,593	6,017	5,127	5,579	0,445	7,98%	7,15%	7,41%
Metic-AC + Tween 80	METIX-AC + Tw80	Filtration	0,5	30	4	7,53	1,88	-58	370	5,161	5,520	5,031	5,237	0,253	4,83%	12,83%	4,21%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	0	7,11	6,99	-79	-80	5,870	5,435	6,721	6,009	0,654	10,89%	0,00%	10,89%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	168	7,11	7,01	-79	-66	5,556	5,901	5,833	5,763	0,183	3,17%	4,08%	3,04%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	336	7,11	7,21	-79	-62	5,120	5,236	5,566	5,304	0,225	4,25%	11,73%	3,75%
Contrôle dopé et autoclavé (DAC)	DAC	Centrifugation	0	30	504	7,11	7,16	-79	-58	6,025	5,858	6,125	6,003	0,135	2,25%	0,10%	2,25%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	72	8,16	8,08	5	66	5,467	4,891	4,833	5,064	0,350	6,92%	15,73%	5,03%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	120	8,16	8,56	5	225	5,000	4,333	4,333	4,566	0,385	8,48%	24,18%	6,41%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	168	8,16	8,60	5	275	4,833	4,500	4,500	4,611	0,192	4,17%	23,26%	3,20%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	336	8,16	5,80	5	361	5,000	4,845	4,535	4,793	0,237	4,94%	20,23%	3,94%
Digestion aérobie mesophile (MAD)	MAD	Centrifugation	0	30	504	8,16	7,08	5	440	5,931	5,691	5,448	5,690	0,242	4,25%	5,30%	4,02%
Digestion aérobie mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	72	8,16	8,12	5	106	5,233	5,053	4,937	4,995	0,082	1,64%	16,87%	1,37%
Digestion aérobie mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	120	8,16	8,46	5	153	5,000	4,667	4,500	4,722	0,255	5,39%	21,41%	4,24%
Digestion aérobie mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	168	8,16	8,45	5	190	4,833	4,737	4,643	4,738	0,095	2,01%	21,15%	1,59%
Digestion aérobie mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	336	8,16	6,06	5	333	4,833	4,545	4,821	4,733	0,163	3,44%	21,22%	2,71%
Digestion aérobie mesophile (MAD) + Tween 80	MAD + Tw80	Filtration	0,5	30	504	8,16	6,43	5	446	6,305	6,452	6,214	6,324	0,120	1,90%	-5,25%	2,00%
METIX-BS	METIX-BS	Centrifugation	0	30	72	7,19	3,13	-51	294	6,167	4,833	5,000	5,333	0,726	13,62%	11,24%	12,09%
METIX-BS	METIX-BS	Centrifugation	0	30	120	7,19	2,71	-51	310	4,917	4,977	5,419	5,104	0,274	5,37%	15,05%	4,56%
METIX-BS	METIX-BS	Centrifugation	0	30	168	7,19	2,15	-51	397	4,603	4,000	4,500	4,368	0,323	7,39%	27,31%	5,37%
METIX-BS	METIX-BS	Centrifugation	0	30	336	7,19	1,83	-51	425	4,500	4,333	3,833	4,222	0,347	8,22%	29,73%	5,77%
METIX-BS	METIX-BS	Centrifugation	-	30	504	7,19	1,42	-51	461	4,667	4,527	4,000	4,398	0,352	7,99%	26,81%	5,85%