Optimizing removal of arsenic, chromium, copper, pentachlorophenol and polychlorodibenzo-dioxins/furans from the 1-4 mm fraction of polluted soil using an attrition process

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ABSTRACT

The objective of this study was to evaluate, at a pilot plant scale, the performance of an attrition process for removing As, Cr, Cu, pentachlorophenol (PCP) and polychlorodibenzodioxins and furans (PCDDF) from a 1-4 mm soil fraction. Once optimized, this attrition process would be applied to the coarse fractions (> 0.250 mm) whereas the fine particles might be treated using a chemical leaching process. The tests were carried out on 2 kg of soil (fraction 1-4 mm) in a 10 L stainless reactor, using tap water. A Box-Behnken experimental design was utilized to evaluate the influence of several parameters (temperature, surfactant concentration and pulp density) and to optimize the main operating parameters of this attrition process. According to the results, the concentration of surfactant (cocamidopropylbetaine-BW) was the main parameter influencing both PCP and PCDDF removal from the 1-4 mm soil fraction by attrition. The behavior of each dioxin and furan congener during the attrition process was studied. The results indicated that the concentration of surfactant had a significant and positive effect on the removal of almost all of the dioxin and furan. The removal of 56%, 55%, 50%, 67% and 62% of the contaminants were obtained for As, Cr, Cu, PCP and PCDDF, respectively, using the optimized conditions ([BW] = 2% (w.w^{-1}), T = 25°C and PD = 40% (w.w^{-1})). These results showed that attrition in the presence of a surfactant can be efficiently used to remediate the coarse fractions of soil contaminated by As, Cr, Cu, PCP and PCDDF.

KEYWORDS: Attrition; Contaminated soil; Pentachlorophenol; Polychlorodibenzodioxins and furans; Experimental design methodology.

INTRODUCTION

Contamination of soils by PCP, PCDDF and metals

Soil contamination has become a major problem in industrial countries all over the world. The presence of organic and inorganic contaminants at very high levels in soil is the result of industrial and/or agricultural practices, such as pesticides or herbicides production, oil and gas exploration, mineral extraction, the use of chlorinated solvents, and the preservation and commercialization of treated-wood. Chromated copper arsenate (CCA) and pentachlorophenol (PCP) have been used as major wood preservative agents for industrial and residential applications in recent decades. The inappropriate management and/or disposal of treated wood led to the contamination of several sites across the world. The main organic contaminants found in disposal sites of treated wood are PCP and PCDDF, which are by-products of the production and the degradation of PCP when it is used to preserve wood. [1] In the United States (US), 313 sites are contaminated by PCP and are listed on the national priorities intervention list (NPL). [2] Recently, in Canada, PCP and PCDDF concentrations ranging from 11 to 35 mg PCP.kg^{-1} and from 1,210 to 13,100 ng TEQ.kg^{-1} (TEQ: Toxic Equivalent Quantity) for PCDDF were measured in contaminated soils. [3] In addition to these persistent organic pollutants, the presence of arsenic, chromium and copper (inorganic compounds encountered in the formulation of CCA-preservative agents) have been found in soil due to the leaching of these metals from CCA-treated structures. Large concentrations of As, Cr and Cu were measured in different soils near CCA-treated wood disposal sites, with concentrations ranging from 50 to 250 mg As.kg^{-1}, from 2.5 to 220 mg Cr.kg^{-1} and from 80 to 350 mg Cu.kg^{-1}. [4]

Type of contaminants

Due to the harmful effects of PCP to humans and to the environment, this organochlorine compound is listed among the 126 priority pollutants identified by the Environmental Protection Agency of the United States (USEPA) and the European Union. [5-7] PCP is a semi-volatile (vapor pressure of 0.002 Pa) and a highly hydrophobic compound ($K_{o/w} = 5.0$). [8] Due to its chemical properties, this compound can easily persist in the environment. [9] PCP is soluble in most organic solvents, such as alcohol, ether and benzene. [2] However, the solubility of PCP in an aqueous medium depends on the pH, which controls the equilibrium between the PCP anion and its protonated form. [10] In its protonated form, the solubility of PCP is approximately 14 $mg.L^{-1}$, but at basic pH values (pH=8), its solubility increases up to 8,000 $mg.L^{-1}$, where PCP is mainly present in its anionic form. [11] In addition, the temperature has an impact on the solubility of PCP, which seemed to be optimal at 75°C. [12] Moreover, several studies have shown that the adsorption of PCP is very sensitive to temperature and pH. [13] Indeed, at acidic pH values, PCP can form hydrogen bonds with the carboxylic groups (-COOH) of organic matter present in soil; [14] making PCP less bioavailable. [15] However, under its anionic form, PCP is less easily bound to organic matter and is, therefore, more easily leachable by rainwater and runoff. [16]

PCDDF compounds are present as impurities in chlorinated products. The family of PCCDF is composed of 210 congeners subdivided into two sub-families, polychlorinated dibenzo-p-dioxin (75 PCDD) and polychlorinated dibenzofurans (135 PCDF). These congeners have similar molecular structures and physicochemical properties, which explains the similarity of their toxic effects. They are semi-volatile and non-polar compounds. In addition, these compounds are known to be highly hydrophobic. The solubility of PCDDF in water decreases gradually as the number of chlorine atoms present in the molecule increases, which limits the biodegradation process. Furthermore, due to their stability and lipophilicity, PCDDF compounds are accumulated in the environment. [17] The USEPA has also estimated that the half-life of the 2,3,7,8-TCDD in soil is between 10 and 30 years. [18] Indeed, desorption of PCDDF from soil is difficult and the vertical migration of these compounds is limited to 10 cm of the soil surface, which limits the risk of groundwater contamination. [19] However, PCDDF adsorbed by soil particles can be mobilized or transported in the water or in the air through dissolved organic matter or fine soil particles. [20] The toxicity of PCDDF compounds depends on the number and the position of the chlorine atoms. Only 17 congeners substituted in positions 2, 3, 7, and 8 are known as toxic for humans and for many animals. [20]

The oxidation states and mobilities of arsenic, chromium and copper depend on the soil composition. Arsenic is mainly leached under its arsenate form, As(V), and binds to minerals by adsorption to oxides and hydroxides of aluminum, manganese or iron present in soil. It also tends to be adsorbed on clay particles and seems to be present in residual fraction for some contaminated soils. [21-22] In the case of chromium, this element is mainly leached in its trivalent oxidation state from CCA-treated wood structures. However, when chromium is leached in its most toxic and oxidized form, Cr(VI), this contaminant is quickly reduced by iron and manganese oxides present in soil and is immobilized as Cr(III). [23] For Cu, this compound is leached in its cupric form and is then fixed to the organic matter and minerals present in soil. [22;24]

Treatment of soil contaminated by As, Cr, Cu, PCP and PCDDF

The contamination of soils by both organic and inorganic contaminants complicates the rehabilitation of the contaminated sites. [25-26] Usually, successive steps are required for the removal of organic and inorganic contaminants; significantly increasing the cost of treatment. [3;27-30] Incineration and bioremediation are effective techniques for the removal and/or the degradation of organic pollutants such as PCP, polycyclic aromatic hydrocarbons (PAHs) and/or PCDDF, but these techniques are inefficient for the removal of metallic compounds. [31-39] According to Stinson et al. [40], the use of a biodegradation process allowed the removal of more than 94% of PCP initially present at concentrations ranging from 130 to 680 mg.kg^{-1}. Bates et al. [41] showed that more than 82% of PAHs were removed after 400 days of treatment using a bioremediation process. Immobilisation techniques seemed to be efficient to simultaneously remove organic and inorganic contaminants from soils [42]. A study was performed at full scale showing that the solidification/stabilization (S/S) technology can be used to remediate soil contaminated by dioxins, PCP, creosote and metals. However, this technology leads to the immobilization of contaminants rather than their removal [43].

According to several studies, soil washing with organic solvents (polar and non-polar) was considered as a viable remediation technology for the removal of organic compounds. [35; 44-46] Indeed, Sahle-Demessie et al. [47] showed that the solubilization of PCP and PCDDF in the presence of dimethyl ether at 48°C is highly efficient; allowing the removal of more than 95% of PCP and PCDDF initially present in the soils. In this study carried out at laboratory scale, the concentrations of metals were not reduced using the solvent extraction treatment. Khodadoust et al. [48] extracted up to 81% of the PCP from contaminated soil in the presence of an aqueous solution of methanol (water/MeOH = 50/50). According to Xiao et al. [49], soil washing in an alkaline medium (pH > 12) seemed to be very effective for the solubilization of PCP, with removal yields reaching 90%. The use of dimethyl ether (soil/dimethyl ether = 1/1), at 48°C was effective for the solubilization of more than 95% of the PCDDF and PCP originally present in the contaminated soils. [50] To enhance the extraction of organic contaminants such as PCP from soil, several studies suggested the addition of a surfactant to the leaching solution due to their amphoteric properties [51-53]. Bates et al. [41] showed that the use of nonionic surfactant enhances the performances of soil washing to remove PAHs, PCP and PCDDF from contaminated soils. Indeed, these authors highlighted that the addition of a surfactant allowed an improvement of contaminants removals from 69 to 78%. Recently, Reynier et al. [4] studied the performances of different surfactants in an acidic leaching/flotation process to remove As, Cr, Cu and PCP from highly contaminated soils. The results showed that an amphoteric biosurfactant cocamidopropylbetaine (BW) showed good removal yields for both metals (30-93%) and PCP (36-78%). A chelating agent ([S,S]-ethylene-di-amine-succinate) and a non-ionic surfactant (Brij 98) seemed to be a very effective method for simultaneously removing 70% of As, 75% of Cr, 80% of Cu, 90% of PCP and 79% of PCDDF contaminants from the soil. However, the costs of the decontamination process developed were excessive (137,000 $.tst^{-1}). [54] According to the study carried out by You and Liu [55], the non-ionic surfactant, Triton X-100, inhibited the solubilization of PCP from contaminated soils. Recently, Reynier et al. [4] successfully treated soil contaminated by As, Cr, Cu, PCP and PCDDF using an amphoteric surfactant (BW) at a pH higher than 13 by chemical leaching. However, the application of this decontamination process on the 0-6 mm soil fraction is very expensive (approximately 600 $.tst^{-1}).

Over the last several decades, various physical techniques have been successfully applied for the remediation of contaminated sites. For soils contaminated by both organic and inorganic compounds, attrition was found to be a promising pre-treatment step followed by chemical or/and physical treatment methods. Indeed, this technology allows the removal of the films around soil particles and/or the detachment of fine particles from the surface of coarse particles, resulting in the release of contaminants. [56] Several parameters, such as the type and the initial concentration of the contaminants, the pulp density, the temperature, the number of attrition steps, the retention time, and the stirring rate, can influence the efficiency of the attrition process. [3; 56-59] Recently, Jobin et al. [60] applied attrition as a pre-treatment step followed by different physical technologies to treat a 0.250-4 mm soil fraction that was contaminated by organic and inorganic compounds. This process allowed the removal of 50% of Cu, 64% of Pb and 40% of the PAHs. Bisone et al. [27] removed more than 90% of the PAHs from contaminated soils by attrition in the presence of a surfactant ([CAS] = 0.2 g.L^{-1}). These authors also observed a significant increase in the removal of Cu and Zn (~ 50%) from soil contaminated by metallurgical slag when attrition upstream of a Wilfley table was used. According to Jobin et al. [58], the attrition process, used as a conditioning treatment, significantly impacted the removal efficiency of the gravity separation for all of the contaminants, with the exception of As. Recently, researchers applied attrition without the presence of a surfactant to successfully treat all the coarse fractions (0.125-12 mm) of different soils contaminated by As, Cr, Cu, PCP and PCDDF, with removal yields varying from 86 to 91% [3].

Aim of the study

The aim of the present study was to evaluate the efficiency of an attrition process as a treatment method to remove simultaneously As, Cr, Cu, PCP and PCDDF from the 1-4 mm soil fraction of a contaminated soil to further applied this treatment to the coarse soil fractions. The use of attrition process on the coarse fraction will simplify the rehabilitation of contaminated site and therefore the costs compared to the use of chemical or biological process on the entire soil. Therefore, this research was conducted to study the attrition parameters (number of attrition steps, concentration of surfactant, solid/liquid ratio) that influence the removal rate of contaminants from the 1-4 mm fraction of contaminated soil in order to apply this attrition process to the coarse fractions (> 0.250 mm) of contaminated soils.

MATERIAL AND METHODS

Soil sampling and characterization

Soil sample contaminated by PCP- and/or CCA-treated wood, called “S3”, was collected from an industrial area. Over 500 kg of soil samples were excavated at a depth of 0 to 15 cm using an excavator and were stored in High-Density Polyethylene (HDPE) containers. The main physical and chemical characteristics of this soil were determined. The soil was wet-sieved using a mechanical Sweco^{TM}. Four different sieves (12-4-1-0.250 mm) were used to analyze the particle size distribution, as described by Mercier et al. [61]. The pH level was determined according to the method described by the Quebec Expertise Center for Environmental Analysis (CEAEQ) (MA. 100-Ph 1.1). A CHNS Leco analyzer was used to determine the total organic carbon, total nitrogen (N) and sulfur (S) content present. Concentrations of As, Cr, Cu, PCP and PCDDF were also measured in the 1-4 mm fraction of the S3 soil sample. To determine the inorganic contaminant content of the soil, the 1-4 mm soil fraction was crushed and digested according to the Method 3030I [62] and then analyzed by ICP-AES.

Attrition Decontamination Process

The attrition process developed in this study represents a small part of a global soil washing process. Indeed, once optimized, the attrition process will be applied to the coarse soil fractions (> 0.250 mm) whereas the fine particle will be treated using a chemical leaching process. The attrition sludge will be recovered from this attrition process and treated using a chemical leaching process or appropriately disposed of whereas the attrition wastewaters will be treated and recycled into the attrition process. All attrition experiments were performed on the 1-4 mm fraction of the S3 soil in a stainless reactor with a capacity of 10 L, equipped with internal baffles, using tap water. During experiments, agitation was conducted using a mechanical stirrer (Light EV1 P25, AXFLOW, New York, NY, USA) and a stainless steel axial propeller (6-cm diameter). Agitation was set at 1,700 rotations per minute (rpm) for a period of 20 minutes. The performance of an amphoteric surfactant (Cocamidopropylbetaine: BW, L.V. Lomas, Dorval, QC, Canada) was evaluated for both organic and inorganic contaminant removal. This surfactant was added, in adequate proportion, before each attrition step. After each attrition step, the soil was sieved through a 0.5 mm sieve and washed with 4 L of tap water before being returned to the process. The process was repeated until a total of five attritions were achieved. The number of attrition steps was selected based on previous studies. [3] After the 5th stage of attrition, soil was placed in a HDPE reactor with a capacity of 20 L to be washed with 4 L of water using a Karcher (Karcher, electric sprayer, 2000 lb.po^{-2}, Quebec, QC, Canada) before being sieved through a 0.5 mm sieve. Then, the attrited soil was washed with 2 L of water. The 1-4 mm soil fractions were then dried at 60°C in an oven and analyzed. The samples were analyzed before and after the attrition treatment to estimate the contaminant removal yields.

Optimization of the Attrition Parameters using experimental design methodology

A Box-Behnken response surface experimental methodology (*Design Expert 8.0*, Stat-Ease Inc. Minneapolis, USA) was used in this study to evaluate the influence of several parameters on the efficiency of the attrition process on the removal of PCP, PCDDF, As, Cr and Cu from the 1-4 mm soil fractions of the S3 soil. This Box-Behnken design (BBD), performed on three numerical factors (parameters) at three different levels coded -1 (low level), 0 (middle level) and +1 (high level), was employed to study the effect of select attrition parameters on the removal of both organic and inorganic contaminants from the 1-4 mm soil fraction. The independent parameters studied were temperature (T), concentration of surfactant (BW) and pulp density (PD). In BBD, the low and high levels were chosen because previous studies have shown their impact on contaminant removal by attrition. [3-4] The experimental region and the levels of each factor are presented in Table 1. The number of assays required for the development of a BBD can be expressed by the following equation (Eq. 1).

$$N = [2\ k\ (k-1)] + C_0 \qquad [1]$$

where k is the number of numeric factors (k = 3) and C_0 is the number of central points (5 replicates).

The experimental design may be represented by a cube (for each contaminant), where the experiments (12) are located in the middle of the edges and the replicates (5) are located in the center of the cube. [63] The results were interpreted using the software, *Design Expert 8.0*. The Box-Behnken methodology was then used to determine the optimal operating conditions in terms of PCP and PCDDF removal efficiencies (as the mathematical models defined for As, Cr and Cu removal efficiencies were not significant).

Analytical Techniques

The pH was determined by using a pH-meter (Accumet Research AR25 Dual Channel pH/Ion meter, Fischer Scientific Ltd., Nepean, Canada) equipped with a double junction Cole-Parmer electrode with an Ag/AgCl reference cell. The pH-meter was calibrated using certified buffer solutions (pH = 2, 4, 7 and 10) before each series of measurements.

Metals analysis

Nitric and hydrchloric acids (HNO_3 and HCl) were used to digest 0.5 g of dry soils samples according to the Method 3030I. [62] Each digestion was performed in triplicates. The concentrations of inorganic contaminants were then measured with an ICP-AES (inductively coupled plasma - atomic emission spectroscopy, Vista Ax CCO simultaneous ICP-AES from Varian, Mississauga, Canada) in our laboratories. The analytical quality controls were performed using certified standard solutions (Multi-elements Standard 900-Q30-100, SCP Science, Lasalle, QC, Canada) and certified soil samples (SQC 001-Lot 011233).

PCP analysis

PCP was extracted from soil samples with methylene chloride using an ultrasonic bath for 30 min at ambient temperature. Then, a liquid/liquid extraction step was performed in the presence of sodium hydroxide to transfer PCP from the organic phase to the aqueous phase. Next, a derivatization step of PCP was performed overnight using anhydrous acetate, and finally, a liquid/liquid extraction step was carried out using methylene chloride. In each step, a recovery standard was added (^{13}C-pentachlorophenol and tribromophenol). The sample is spiked with an internal standard (Phenanthren-D_{10}) prior to analysis by gas chromatography coupled with mass spectrometry (GC-MS) (Perkin Elmer, model Clarus 500, column type RXi-17, 30 m × 0.25 mm × 0.25 µm) according to the CEAEQ method MA. 400-Phe. 1.0. [64] Certified soil samples (CMR 143, BNAs-Sandy Loam) were also analyzed. The concentration of PCP present in the soil before and after treatment was determined by analysis performed in our laboratories.

PCDDF analysis

The analysis of the 17 toxic congeners of PCDDF was performed in our laboratories according to the CEAEQ method MA. 400-D.F. 1.1. [65] First, a Soxhlet extraction of PCDDF from the soil was carried out overnight using toluene. Then, the samples were purified and concentrated using a multilayer silica column, followed by an alumina column. The multilayer silica column was then eluted with hexane, whereas the alumina column was eluted with three different mixtures of hexane and dichloromethane. The eluate that contains PCDDF congeners was then evaporated under nitrogen stream. A volume of 100 µL of internal standard (13C-labelled analogues purchased at Wellington laboratories) was then added and the sample was measured by GC-MS (Thermo Scientific, model Trace 1310 Gas Chromatograph coupled with mass spectrometer detector ISQ, column type ZB Semi-volatile, 60 m × 0.25 mm × 0.25 µm).

RESULTS AND DISCUSSION

Characterization of the soil S3

Table 2 presents the concentrations of As, Cr, Cu, PCP and PCDDF measured as a fraction of the entire S3 soil sample. According to the particle size distribution, the texture class of the entire soil sample is classified as sand. The percentage of clay and silt (particle size < 250 µm) present in the entire S3 soil is less than 10%. The entire soil pH was approximately 7.0 and the total organic carbon content was estimated at 1.3%. The nitrogen and sulfur contents were estimated at 0.07% and 0.05%, respectively.

The distribution of contaminants present in the S3 soil showed that the finer fraction (< 250 µm) was the most contaminated with concentrations reaching 539 mg As.kg^{-1}, 380 mg Cr.kg^{-1}, 681 mg Cu.kg^{-1}, 42.3 mg PCP.kg^{-1} and 45,770 ng TEQ.kg^{-1} for PCDDF. In the coarse fraction

(> 250 µm), the concentration of contaminants varied between 18 and 55 mg.kg^{-1} for As, between 28 and 64 mg.kg^{-1} for Cr, between 37 and 160 mg.kg^{-1} for Cu, between 1.40 and 15.4 mg.kg^{-1} for PCP and between 1,801 and 6,194 ng TEQ.kg^{-1} for PCDDF. The concentrations of Cr and Cu in the 1-4 mm soil fraction (which was used for all assays) were estimated at 63.8 mg Cr.kg^{-1} and 160 mg Cu.kg^{-1}. However, this soil fraction was highly contaminated by As, PCP and PCDDF with concentrations of 54.7 mg.kg^{-1}, 8.7 mg.kg^{-1} and 5,719 ng TEQ.kg^{-1}, respectively.

Table 3 shows the initial concentrations, the toxicity equivalency factors and the associated TEQ for each dioxin and furan initially present in the fraction 1-4 mm of the S3 soil. According to the results obtained, it appeared that 12 of the 17 congeners considered as toxic were present in the fraction 1-4 mm. The concentration of 2,3,7,8 TCDF; 2,3,7,8 TCDD; 1,2,3,7,8 PCDF; 2,3,4,7,8 PCDF and 1,2,3,6,7,8 HCDF were below the detection limit and were, therefore, considered as negligible. Among the 17 congeners followed, the associated TEQ values of the congeners 1,2,3,6,7,8 HCDD (615 ng TEQ.kg^{-1}), 1,2,3,4,6,7,8 HPCDD (1,674 ng TEQ.kg^{-1}) and OCDD (1,797 ng TEQ.kg^{-1}) were very high. Moreover, the concentration of hexa-chlorinated, hepta-chlorinated and octa-chlorinated dioxins and furans were much higher than the concentrations measured for tetra-chlorinated dioxins and furans, which are more toxic to humans.

Evaluation of attrition parameters on contaminant removal performances

Influence of operating parameters on the removal of As, Cr, Cu, PCP and PCDDF

Table 4 presents the As, Cr, Cu, PCP and PCDDF removal yields obtained during the different attrition assays recommended by the *Design Expert 8.0* software. According to these results, contaminant removal yields varied from 32 to 65% for As, from 27 to 52% for Cr, from 36 to 47% for Cu, from 40 to 67% for PCP and from 43 to 69% for PCDDF, depending on the experimental conditions used. According to the variations in contaminant removal yields observed, the experimental design was well-defined to evaluate the influence of attrition parameters on inorganic and organic contaminants and to determine the optimal conditions of attrition to remove contaminants from the 1-4 mm soil fraction of the S3 soil.

Experimental results were then analyzed using *Design Expert 8.0* software and second order polynomial equations were established, as presented in Eq. 2, to predict the contaminant removal yields based on different operating conditions during attrition (temperature, concentration of surfactant and pulp density). The linear, second-order and quadratic coefficients were calculated by the *Design Expert 8.0* software using the least square method.

[2]

where Y represents the response, x_i and x_j are the variables, b_0 is the interception coefficient, b_i are linear coefficients, b_{ij} are quadratic coefficients, b_{ii} are second-order coefficients, k is the number of parameters studied (k=3) and e_0 is the error. [67]

Analysis of variance (ANOVA) tests were carried out by the software to check the adequacy of the mathematical models established, to identify the influence of each parameter on the attrition process efficiency and to optimize the attrition conditions (temperature, surfactant concentration and pulp density). According to the results obtained by the *Design Expert 8.0* software (not shown), the studied parameters (temperature, pulp density, BW concentration) had no significant influence on the As, Cr and Cu removal efficiencies from the 1-4 mm soil fraction by attrition.

This could be because attrition was performed at pH = 7 and that the solubilization of metals is not favorable at a neutral pH.

Table 5 shows the results of the ANOVA for the mathematical models established for the prediction of the PCP and PCDDF removal efficiencies from the 1-4 mm soil fraction. The values "Prob> F (p)" allowed us to determine if the model or the studied variables are significant or not. If "Prob> F (p)" is higher than 0.10, the mathematical model or the studied factor is insignificant, while a value lower than 0.05 indicates that the mathematical model or the studied factor is significant. The results presented in Table 5 showed that the mathematical models defined for the removal of PCP and PCDDF (general TEQ) from the 1-4 mm soil fraction by attrition were significant, thus signifying that the experimental data are appropriate for the quadratic models established. To assess the adequacy of the quadratic models established, it is also important to evaluate the accurate estimation of the experimental error and to determine if there is a lack of fit of the model or not. This information is determined by the experiments made in the center of the experimental region in replicates ($n = 5$). According to the ANOVA results, no significant lack of fit was observed for either the PCP or PCDDF quadratic models. Additionally, the values of the correlation coefficient (R^2) were estimated at 0.92 for PCP removal and 0.99 for PCDDF removal. A R^2 value of 0.99 for the PCDDF and of 0.92 for the PCP quadratic models indicated that only 1% and 8% of the experimental values obtained were unable to be explained by the defined mathematical models, respectively. These R^2 values were higher than 0.80, indicating that the mathematical models developed adequately predicted the behavior of the attrition process observed experimentally to remove PCP and PCDDF from the 1-4 mm soil fraction [68]. The values of the variation coefficient (CV) for the quadratic models established for PCP and PCDDF were lower than 10%, thus confirming the reliability of the models established [69]. Additionally, the adequate precision values of the signal-to noise ratio were estimated at 14.5 and 42.3 for the PCP and PCDDF quadratic models, respectively. These values, higher than 4, supported the suitability of the mathematical models established to predict PCP and PCDDF removals from the 1-4 mm soil fraction by attrition. [70-71]

The analysis of variance (ANOVA) showed that among the factors considered (temperature, surfactant concentration, pulp density), only the concentration of BW had a significant influence on the removal of the PCP (“Prob > F” < 0.0001), whereas the temperature had no significant influence on the removal of PCP through attrition (“Prob > F” = 0.5253). For PCDDF removal by attrition, all parameters had a significant influence (“Prob > F” = < 0.0001 for both temperature and surfactant concentration and “Prob > F” = 0.0253 for pulp density) on PCDDF removal from the 1-4 mm soil fraction by attrition. Fig. 1 shows the variations between the values predicted by the *Design Expert 8.0* software and the values obtained experimentally for the removal of PCP and PCDDF for the 17 assays conducted on the 1-4 mm soil fraction by attrition. This representation showed a satisfactory correlation between the predicted and the experimental values for both PCP and PCDDF removals, confirming the adequacy of the quadratic models established.

Mathematical equations in terms of coded factors were established using the *Design Expert 8.0* software to predict the percentage of PCP (Eq. 3) and PCDDF (expressed as %TEQ- Eq. 4) removed from the 1-4 mm soil fraction by attrition for all of the conditions studied in the experimental region.

$$\textbf{PCP Removal (\%)} = 64.3 - 0.65\,A + 7.32\,B + 2.10\,C - 4.85\,A^2 - 9.39\,B^2 + 3.70\,C^2 \qquad \textbf{[3]}$$

$$\textbf{PCDDF Removal (\%)} = 60.6 + 2.05\,A + 9.48\,B + 0.81\,C + 0.82\,AB + 1.67\,AC + 3.14\,A^2 - 6.23\,B^2 + 0.02\,C^2 \qquad [4]$$

where A is the temperature, B is the concentration of surfactant (BW) and C is the pulp density.

The sign of the coefficient of each term indicated whether the influence of the parameter on the PCP and PCDDF removal yields from the 1-4 mm soil fraction was positive or negative. According to Eq. 3 and Eq. 4, the concentration of BW had a significant and positive effect on the removal of both PCP and PCDDF. This result indicated that increasing the concentration of BW significantly improved the removal of PCP and PCDDF from the 1-4 mm soil fraction. Indeed, the increase in the amount of BW added to the solution of attrition resulted in an increase in the number of micelles formed, thus favoring the removal of hydrophobic compounds such as PCP and PCDDF from contaminated soil by attrition. Additionally, the temperature and pulp density had a significant and positive effect on the removal of PCDDF. These results indicated that increasing the temperature of the solution of attrition or the pulp density led to a significant increase in the removal of PCDDF from the 1-4 mm soil fraction. When comparing the mathematical coefficients defined for each parameter, it appeared that the concentration of surfactant (+ 7.32 for PCP and + 9.48 for PCDDF) is the main parameter influencing the removal of both PCP and PCDDF from the 1-4 mm soil fraction by attrition, followed by the temperature (+ 2.05 for PCDDF) and finally the pulp density (+ 0.81 for PCDDF), which had a very small influence.

Fig. 2 and 3 show the influence of the interactions of AB (temperature and BW concentration) and AC (temperature and pulp density) on the removal of the PCP and PCDDF, respectively.

According to Fig. 2a and 3a, for a pulp density fixed at 30% (middle level), the influence of the concentration of BW had the same effect on PCP and PCDDF removals even if the temperature varied between 25 and 75°C. Additionally, for a pulp density between 20 and 40%, the temperature variation between 25 and 75°C had no significant effect on PCP removal from the 1-4 mm soil fraction by attrition (Fig. 2b, 3b).

Effect of attrition conditions on the removal efficiency of each dioxin and furan congener

The behavior of each dioxin and furan congener during the attrition process was studied using *Design Expert 8.0* software. According to the removal yields obtained during the 17 assays carried out (not shown), it appeared that the removal was more favorable for dioxins than for furans, independent of the number of chlorine atoms substituted on the molecule (penta-, hexa-, hepta- and octa-). Thus, it seemed that the removal of dioxins and furans from the 1-4 mm soil fraction by attrition can be influenced by the conformation of the molecule and the number of oxygen atoms in the molecule (1 atom of oxygen on furans and 2 atoms on dioxins). For the 1,2,3,7,8 PCDD, the 1,2,3,6,7,8 HCDD and the 1,2,3,4,6,7,8 HPCDD molecules, the resulting extraction yields were higher than 50% and even reached more than 90% for most of the operating conditions tested. The results obtained showed that the removal of furans varied between 9.6 and 71%, whereas the removal of dioxins varied between 12 and 89.7%.

Among the 12 congeners of dioxins and furans followed during the attrition experiments (five dioxins and furans were not detectable), the mathematical models established were significant for eleven dioxins and furans (Table 6). However, the mathematical model obtained was not significant for only one congener (1,2,3,4,7,8 HCDD), indicating that no trend between the responses (removal yields) and the operating conditions (temperature, BW concentration, PD) could be found. In other words, the studied parameters had no significant influence on the removal of this congener from the 1-4 mm soil fraction by attrition on the experimental region studied. According to the significant mathematical models established for the eleven dioxins and furans, it appeared that the concentration of BW (B) had a significant effect on the eleven molecules listed earlier, but the temperature (A) had a significant impact on the removal of only six molecules of dioxins and furans (1,2,3,4,7,8 HCDF; 1,2,3,6,7,8 HCDD; 1,2,3,7,8,9 HCDD; 1,2,3,4,7,8,9 HPCDF; 1,2,3,4,6,7,8 HPCDD and OCDF) while the pulp density (C) had no significant effect on the removal of ten dioxins and furans listed earlier, except for the 1,2,3,4,6,7,8 HPCDD. It was also observed that both the temperature and surfactant concentration studied had a significant effect on the removal of the 1,2,3,4,7,8 HCDF; 1,2,3,6,7,8 HCDD; 1,2,3,7,8,9 HCDD; 1,2,3,4,7,8,9 HPCDF; 1,2,3,4,6,7,8 HPCDD and OCDF compounds.

Optimization of the operating conditions for the removal of PCP and PCDDF

The second objective of this study was to determine the optimal operating conditions for the removal of organic contaminants present in the 1-4 mm fraction of the soil S3 using a response surface methodology. The *Design Expert 8.0* software allowed us to define the optimal operating conditions based on the maximization of the removal of PCP and PCDDF (expressed in TEQ) from the 1-4 mm soil fraction by attrition, while minimizing the temperature of the attrition step to reduce operational costs. The defined criteria were then combined by the software in terms of "function desirability" to determine the optimum operating conditions. Indeed, when multiple responses are considered in the optimization of the operating conditions of a process, a compromise must be defined. [72] Desirability is an objective function whose values ranged from 0 (low satisfaction) to 1 (high satisfaction), depending on the level at which the criteria imposed were met or not met. [73]

Based on the defined criteria (maximization of the removal of PCP and PCDDF and minimization of temperature), the *Design Expert 8.0* software proposed fourteen solutions, which are presented in Table 7 and are ordered according to the level of desirability obtained (ability to perfectly meet the imposed criteria). All solutions proposed by the software recommended performing the attrition steps at 25°C, with predicted removal yields ranging from 61.6 to 67.3% for PCP and from 62.7 to 65.5% for PCDDF. This decontamination process, carried out at room temperature, will be easier to operate on an industrial scale. Therefore, among the solutions proposed by the software, the selected conditions are: pulp density = 40% (w.w^{-1}), T = 25°C and BW concentration = 2% (w.w^{-1}) (we preferred to round 2.30% to 2.00%). According to the optimal attrition conditions defined, PCP and PCDDF removal yields predicted were calculated using Eq. 3 and Eq. 4 and were estimated at 67.2% and 63.7%, respectively.

Attrition tests were performed in triplicate to validate the optimal attrition conditions determined by the *Design Expert 8.0* software. Table 8 presents the amount of PCP and PCDDF content measured in the soil after treatment, the corresponding removal yields, as well as the removal yields predicted by the software. The experiments carried out in triplicate showed that the average removal of PCP was approximately 66.8% and that the final concentration of PCP was 2.89 ± 0.05 mg.kg^{-1}. In the case of PCDDF, the results showed that the average removal of PCDDF was 61.7%. At the optimal attrition conditions, the PCP and PCDDF (expressed as %TEQ) removal yields predicted by the software were estimated at 67.2% and 63.7%, respectively. Comparing the experimental and predicted PCP and PCDDF removal yields, it is possible to confirm the validity and the adequacy of the mathematical model established for the removal of PCP and PCDDF from the 1-4 mm soil fraction by attrition.

CONCLUSION

This research evaluated the parameters influencing the removal of arsenic, chromium, copper, pentachlorophenol and polychlorodibenzodioxins and furans from contaminated soil using an attrition process. An experimental design methodology was employed to evaluate the influence of several parameters (temperature, concentration of surfactant, pulp density) on the removal of contaminants and to optimize the main parameters of attrition to maximize organic contaminant removals from the 1-4 mm soil fraction. The attrition process developed was better at removing organic contaminants than metal contaminants from the soil. Attrition was applied at a pH = 7 and the solubilization of metals is limited at this neutral pH. The results showed that attrition allowed the reduction of organic contaminant concentrations in the 1-4 mm soil fraction. According to our results, the concentration of surfactant is the main parameter influencing the removal of both PCP and PCDDF from the 1-4 mm soil fraction by attrition. Additionally, it seemed that the removal of dioxins and furans from the 1-4 mm soil fraction by attrition can be influenced by the conformation of the molecule and the number of oxygen atoms in the molecule. This study proved that attrition (V = 5 L, PD = 40% ($w.w^{-1}$), T = 25°C, [cocamidopropylbetaine] = 2% ($w.w^{-1}$), t = 20 min, five attrition steps) can be considered as an efficient soil treatment method and leads to the removals of 56.1% of As, 55.2% of Cr, 49.9% of Cu, 66.8% of PCP and 61.7% of PCDDF from the 1-4 mm fraction of soil. The attrition sludge recovered from this process will be treated using a chemical leaching process or appropriately disposed of whereas the attrition wastewaters will be treated and recycled into the attrition process. Future studies should attempt to evaluate the potential of the attrition process for the removal of inorganic and organic contaminants from different soil fractions and from soils containing different initial concentrations of organic contaminants. This process should also be tested on different types of contaminated soil.

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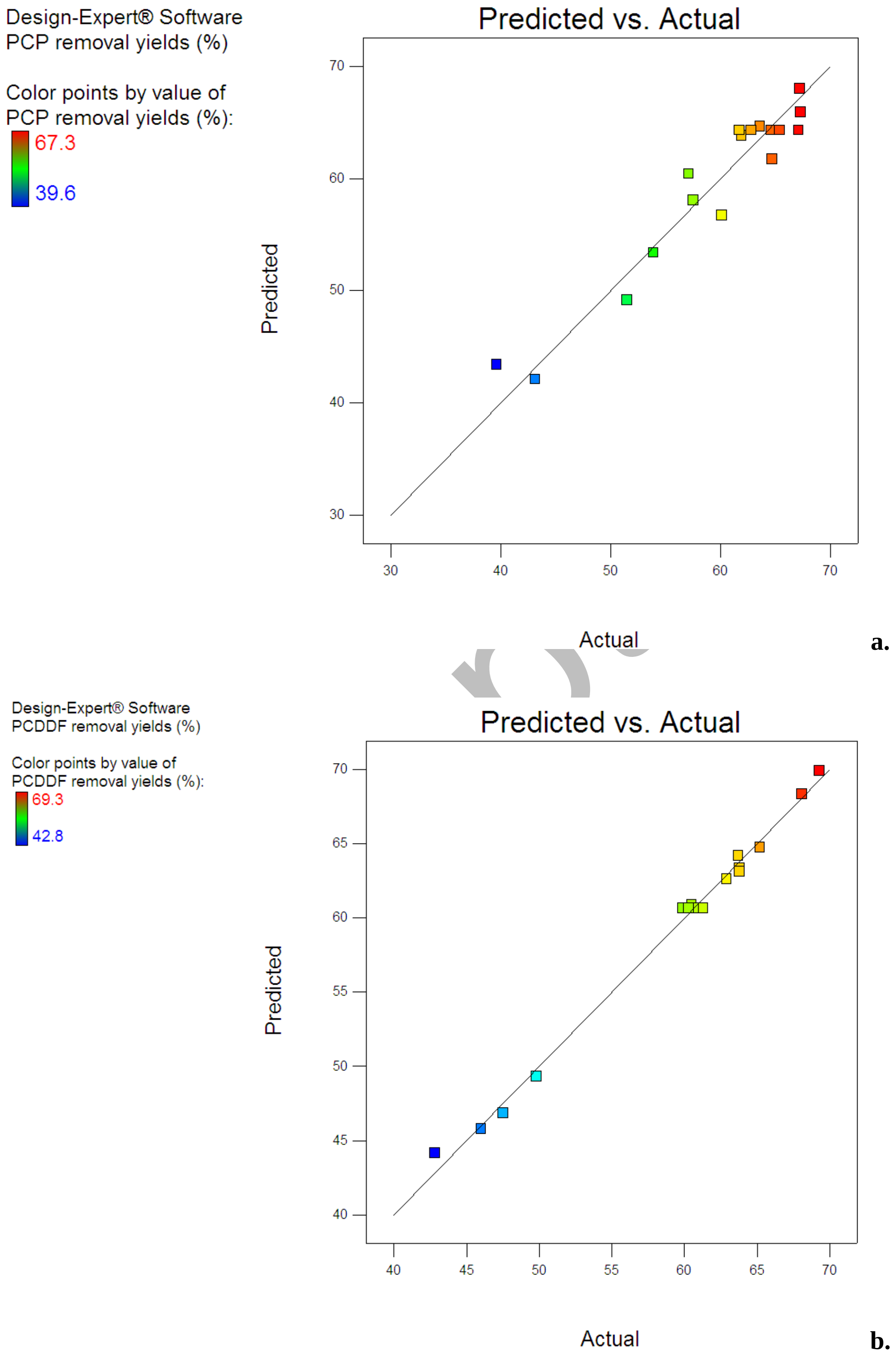
Figure 1 Variation between the experimental and the predicted removal yields for PCP (Fig 1a) and PCDDF (Fig. 1b) from the 1-4 mm soil fraction

Figure 2 Surface response of the PCP removal efficiency as a function of the temperature and the concentration of BW (ratio of the solid/liquid = 30% (w.w^{-1})) (Fig. 2a) and as a function of the temperature and ratio of the solid/liquid (concentration of BW = 1.5% (w.w^{-1})) (Fig. 2b)

Figure 3 Surface response of the PCDDF removal efficiency as a function of the temperature and the concentration of BW (ratio of the solid/liquid = 30% (w.w^{-1})) (Fig. 3a) and as a function of the temperature and ratio of the solid/liquid (concentration of BW = 1.5% (w.w^{-1})) (Fig. 3b)


Design-Expert® Software
PCP removal yields (%)
Color points by value of
PCP removal yields (%):
67.3
39.6
Predicted vs. Actual
Predicted
Actual
30
40
50
60
70
a.
Design-Expert® Software
PCDDF removal yields (%)
Color points by value of
PCDDF removal yields (%):
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42.8
Predicted vs. Actual
Predicted
Actual
40
45
50
55
60
65
70
b.

Fig. 1

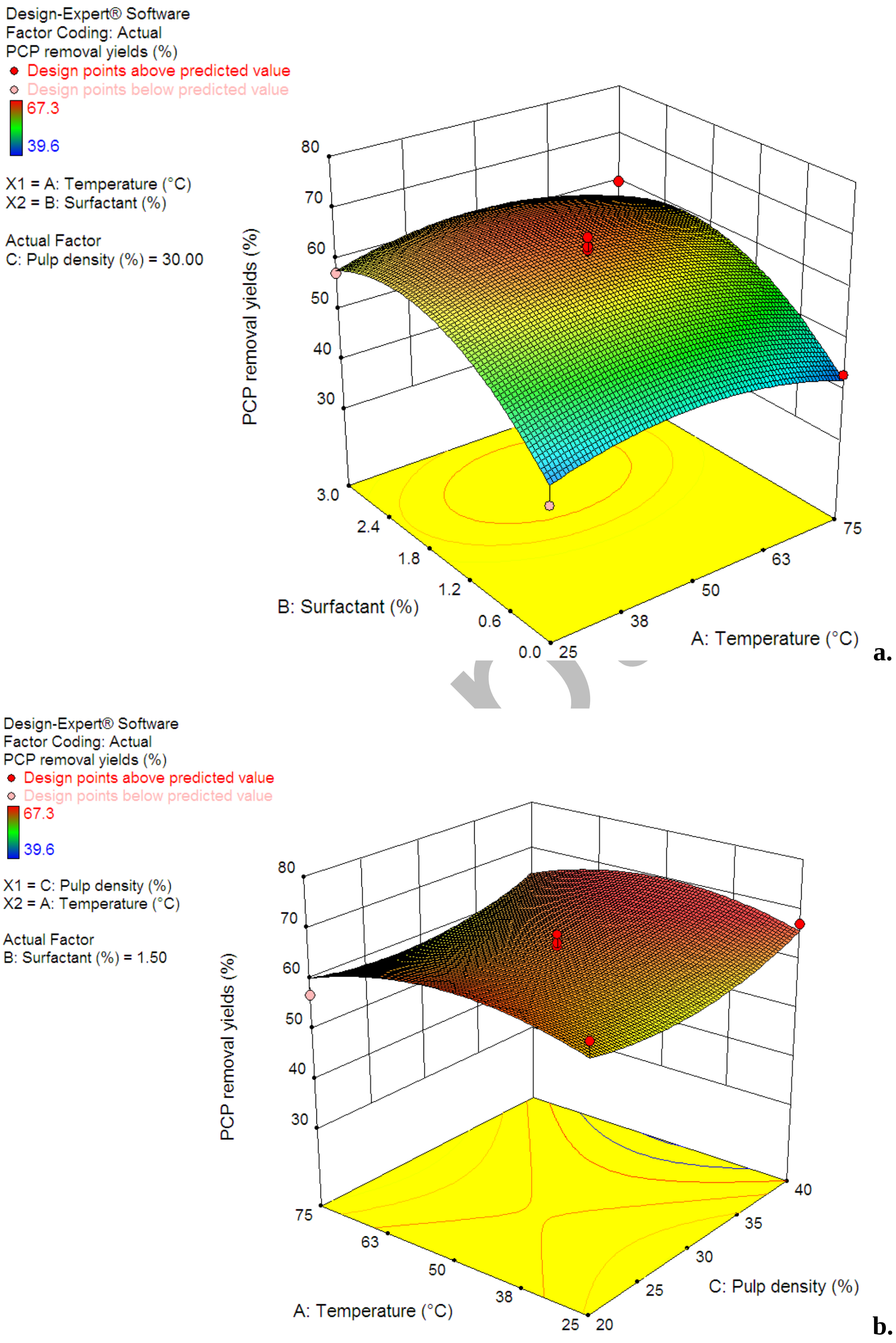
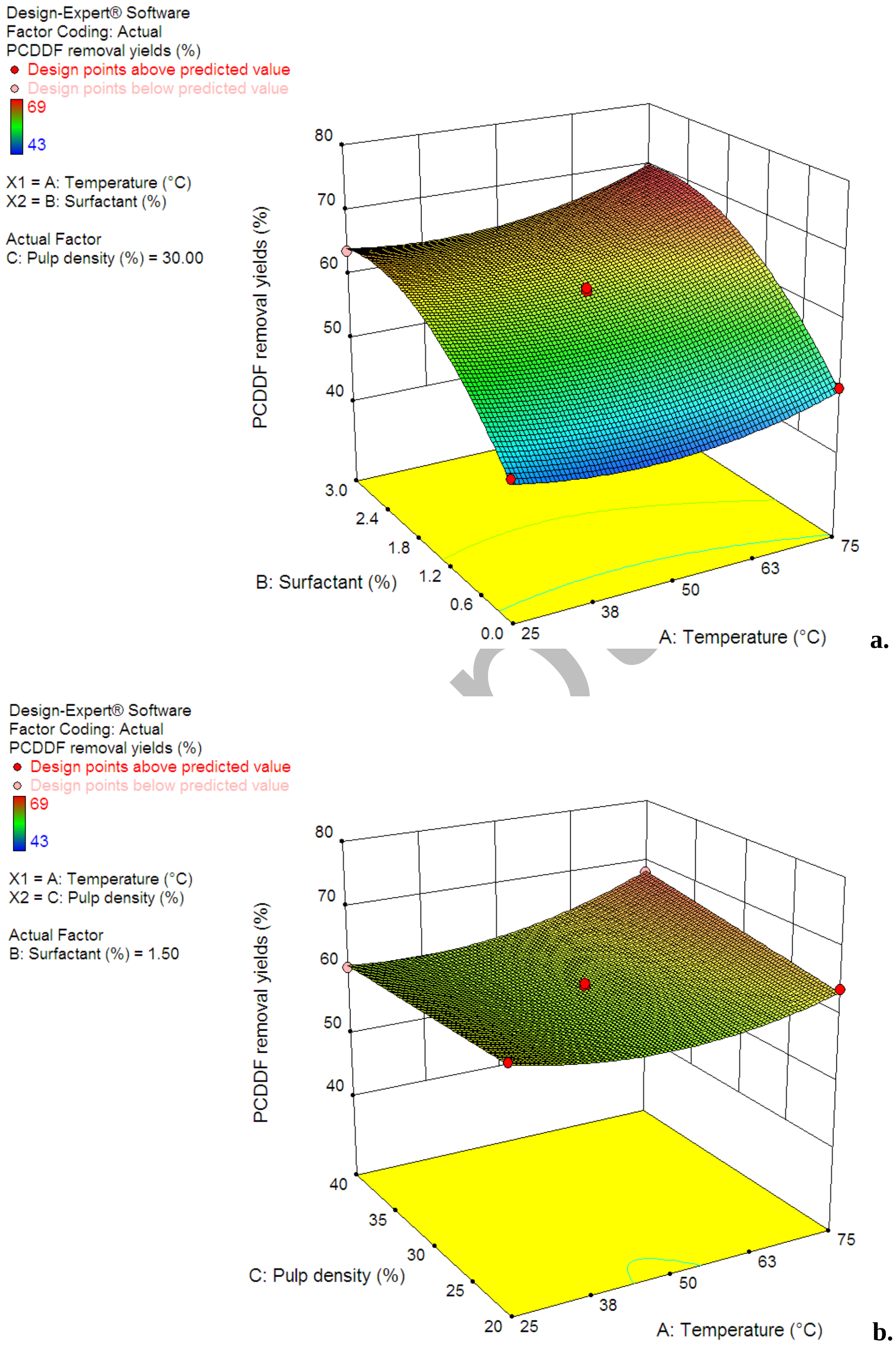

Design-Expert® Software
Factor Coding: Actual
PCP removal yields (%)
● Design points above predicted value
● Design points below predicted value
67.3
39.6
X1 = A: Temperature (°C)
X2 = B: Surfactant (%)
Actual Factor
C: Pulp density (%) = 30.00
PCP removal yields (%)
80
70
60
50
40
30
3.0
2.4
1.8
1.2
0.6
0.0
B: Surfactant (%)
25
38
50
63
75
A: Temperature (°C)
a.
Design-Expert® Software
Factor Coding: Actual
PCP removal yields (%)
● Design points above predicted value
● Design points below predicted value
67.3
39.6
X1 = C: Pulp density (%)
X2 = A: Temperature (°C)
Actual Factor
B: Surfactant (%) = 1.50
PCP removal yields (%)
80
70
60
50
40
30
75
63
50
38
25
A: Temperature (°C)
20
25
30
35
40
C: Pulp density (%)
b.

Fig. 2



Design-Expert® Software
Factor Coding: Actual
PCDDF removal yields (%)
● Design points above predicted value
○ Design points below predicted value
69
43
X1 = A: Temperature (°C)
X2 = B: Surfactant (%)
Actual Factor
C: Pulp density (%) = 30.00
PCDDF removal yields (%)
80
70
60
50
40
3.0
2.4
1.8
1.2
0.6
0.0
B: Surfactant (%)
25
38
50
63
75
A: Temperature (°C)
a.
Design-Expert® Software
Factor Coding: Actual
PCDDF removal yields (%)
● Design points above predicted value
○ Design points below predicted value
69
43
X1 = A: Temperature (°C)
X2 = C: Pulp density (%)
Actual Factor
B: Surfactant (%) = 1.50
PCDDF removal yields (%)
80
70
60
50
40
40
35
30
25
20
C: Pulp density (%)
25
38
50
63
75
A: Temperature (°C)
b.

Fig. 3

Table 1 **Experimental region and levels of independent factors defined for the optimization of the attrition process on the 1 – 4 mm soil fraction**

Coded factor (X_i)	Parameters	Unit	Minimum value (-1)	Middle value (0)	Maximum value (+1)
X_1	A: Temperature (T)	°C	25	50	75
X_2	B: Surfactant (BW)	% ($w.w^{-1}$)	0	1.5	3.0
X_3	C: Pulp density (PD)	% ($w.w^{-1}$)	20	30	40

Table 2 Particle size and element concentrations measured in S3

Soil characteristics	Soil proportion (%)	Arsenic (mg.kg^{-1})	Chromium (mg.kg^{-1})	Copper (mg.kg^{-1})	PCP (mg.kg^{-1})	PCDDF (ng TEQ.kg^{-1}) ~~(NATO, 1988)~~[66]
Particle size						
x > 12 mm	15.6	18.0	27.7	36.9	1.40	1,801
4 < x < 12 mm	23.4	33.0	38.8	70.8	2.88	3,080
1 < x < 4 mm	27.9	54.7	63.8	160	8.70	5,719
0.250 < x < 1 mm	23.3	53.5	41.5	116	15.4	6,194
x < 0.250 mm	9.73	539	380	681	42.3	45,770

Mis en forme : Surlignage

Table 3 **Initial levels, toxicity equivalency factor and associated TEQ for each dioxin or furan present in the fraction 1 – 4 mm of the soil S3**

Dioxins or furans	**Toxicity Equivalency Factor (TEF) [66]**	**Initial levels ($ng.kg^{-1}$)**	**Associated TEQ ($ng.kg^{-1}$)**
2,3,7,8 TCDF	0.1	<0.5	<0.05
2,3,7,8 TCDD	1.0	<1.6	<1.6
1,2,3,7,8 PCDF	0.05	<1.2	<0.06
2,3,4,7,8 PCDF	0.5	<1.0	<0.5
1,2,3,7,8 PCDD	0.5	285	143
1,2,3,4,7,8 HCDF	0.1	830	83
1,2,3,6,7,8 HCDF	0.1	<1.6	<0.2
1,2,3,7,8,9 HCDF	0.1	843	84
1,2,3,4,7,8 HCDD	0.1	1 796	180
1,2,3,6,7,8 HCDD	0.1	6 146	615
1,2,3,7,8,9 HCDD	0.1	1 589	159
2,3,4,6,7,8 HCDF	0.1	2 551	255
1,2,3,4,6,7,8 HPCDF	0.01	27 633	276
1,2,3,4,6,7,8 HPCDD	0.01	167 365	1 674
1,2,3,4,7,8,9 HPCDF	0.01	4 885	49
OCDD	0.001	1 797 286	1 797
OCDF	0.001	404 458	404
Total (TEQ)			**5 719**

Table 4 **Experimental design and contaminant removal yields from the fraction 1 – 4 mm of the soil S3 obtained after treatment by attrition (Initial contaminant levels As : 55 ± 2 mg.kg^{-1}, Cr : 64 ± 7 mg.kg^{-1}, Cu : 160 ± 10 mg.kg^{-1} and PCP : 8.7 ± 2.3 mg.kg^{-1}, PCDDF : 5 719 ± 180 ng TEQ.kg^{-1})**

Assay	Temperature (°C)	BW content (% -w.w^{-1})	Solid/liquid ratio (% -w.w^{-1})	Contaminant removal yields (%)				
				As	Cr	Cu	PCP	PCDDF
1	75	1.5	20	64	50	47	57	64
2	75	1.5	40	52	36	38	64	68
3	50	3.0	30	64	47	46	67	65
4	75	3.0	30	32	27	41	60	69
5	50	3.0	20	47	46	42	62	64
6	25	1.5	20	49	45	46	65	63
7	25	1.5	40	51	47	46	67	61
8	25	3.0	30	52	42	45	58	64
9	50	1.5	30	60	52	45	63	61
10	50	1.5	30	55	50	42	65	60
11	50	1.5	30	62	46	36	62	61
12	50	1.5	30	54	48	44	65	61
13	50	1.5	30	51	46	42	67	60
14	50	0.0	20	56	40	43	52	43
15	25	0.0	30	55	44	39	40	48
16	50	0.0	40	65	49	45	54	46
17	75	0.0	30	57	46	40	43	50

Table 5 **Variance analysis (ANOVA) for quadratic models established to predict PCP and PCDDF removals from the 1 – 4 mm contaminated soil fraction by attrition**

Contaminant	Source	Sum of square	Degree of freedom	Mean square	F value	p-value Prob > F	Conclusion
PCP	Model	996	6	166	21.3	< 0.0001	Significant
	A-Temperature	3.38	1	3.38	0.43	0.5253	
	B-Surfactant	429	1	429	55.0	< 0.0001	
	C- Pulp density	35	1	35	4.52	0.0594	
	A^2	98.9	1	98.9	12.7	0.0052	
	B^2	371.8	1	372	47.7	< 0.0001	
	C^2	57.7	1	57.7	7.40	0.0216	
	Residual	78.0	10	7.80			
	Lack of fit	59.8	6	10.0	2.20	0.2327	Not Significant
	Pure error	18.1	4	4.5			
PCDDF	Model	970	8	121	173	< 0.0001	Significant
	A-Temperature	33.6	1	33.6	47.9	<0.0001	
	B-Surfactant	720	1	720	1 027	< 0.0001	
	C- Pulp density	5.28	1	5.28	7.53	0.0253	
	AB	2.72	1	2.72	3.88	0.0843	
	AC	11.2	1	11.2	16.0	0.0039	
	A^2	41.6	1	41.6	59.4	< 0.0001	
	B^2	163	1	163	233	< 0.0001	
	C^2	0.002	1	0.002	0.002	0.9621	
	Residual	5.61	8	0.70			
	Lack of fit	4.30	4	1.07	3.28	0.1385	Not Significant
	Pure error	1.31	4	0.33			

PCP : $R^2 = 0.92$; *adjusted* $R^2 = 0.88$; *adequation precision = 14.5; CV = 4.7%*

PCDDF : $R^2 = 0.99$; *adjusted* $R^2 = 0.99$; *adequation precision = 42.3; CV = 1.4%*

Table 6 **Variance analysis (ANOVA) for significant quadratic models established for the removals of 11 PCDDF from the 1 – 4 mm contaminated soil fraction by attrition (A: Temperature – B: Surfactant – C: Pulp density)**

	1,2,3,7,8 PCDD	1,2,3,4,7,8 HCDF	1,2,3,7,8,9 HCDF	1,2,3,6,7,8 HCDD	1,2,3,7,8,9 HCDD	2,3,4,6,7,8 HCDF	1,2,3,4,6,7,8 HPCDD	1,2,3,4,7,8,9 HPCDF	1,2,3,4,6,7,8 HPCDF	OCDD	OCDF
Source	**Prob > F**	**Prob > F**	**Prob > F**	**Prob > F**	**Prob > F**	**Prob > F**	**Prob > F**	**Prob > F**	**Prob > F**	**Prob > F**	**Prob > F**
Model	< 0.0001*	0.0005	0.0314	<0.0001	<0.0001	0.0155	0.0001	0.0003	0.0279	< 0.0001	< 0.0001
A	0.3328	0.0011	0.1853	0.0028	0.0008	0.2705	0.0002	0.0018	0.7455	0.5144	0.0134
B	< 0.0001	0.0001	0.0089	< 0.0001	< 0.0001	0.0033	0.0004	0.0006	0.0037	< 0.0001	< 0.0001
C	0.7590	0.5138	0.2392	0.8514	0.3729	0.6568	0.0130	0.4843	0.6566	0.9588	0.7943
AB	-	0.0485	-	0.0948	-	-	-	0.0826	-	-	-
AC	-	-	-	-	-	-	-	0.2371	-	-	-
BC	-	0.1369	-	-	-	-	-	-	-	0.0724	-
A^2	0.1123	0.0133	-	0.0144	-	-	-	<0.0001	-	0.0197	-
B^2	< 0.0001	0.0054	-	0.0013	-	-	-	0.6019	-	< 0.0001	0.0070
C^2	0.9125	0.0688	-	-	-	-	-	-	-	0.0516	0.0413
Lack of fit	0.7646	0.9889	0.0171	0.9541	0.7131	0.2570	0.9166	0.78845	0.7834	0.6295	0.3781
Conclusion for the Lack of fit	Not Significant	Not Significant	Significant	Not Significant	Not Significant	Not Significant	Not Significant	Not Significant	Not Significant	Not Significant	Not Significant

** Significant models and parameters were highlighted in grey*

Table 7 **Solutions suggested by the Design-Expert 8.0 software to maximize the PCP and PCDDF removal efficiencies from the fraction 1-4 mm of the soil S3 by attrition**

Solution number	**Temperature (°C)**	**Surfactant (%)**	**Pulp density (%)**	**Predicted removal yields (%)**		**Desirability function**
				PCP	**PCDDF**	
1	25.0	2.30	40.0	67.2	63.8	0.923
2	25.0	2.31	40.0	67.1	63.8	0.923
3	25.0	2.27	40.0	67.2	63.7	0.923
4	25.0	2.40	40.0	66.9	63.9	0.922
5	25.0	2.19	40.0	67.3	63.6	0.922
6	25.0	2.42	40.0	66.9	63.9	0.922
7	25.4	2.31	40.0	67.3	63.7	0.922
8	25.9	2.38	40.0	67.3	63.8	0.920
9	26.1	2.41	40.0	67.3	63.8	0.918
10	25.0	1.87	40.0	67.2	62.7	0.907
11	25.0	2.23	37.5	65.1	63.9	0.901
12	25.0	2.27	20.0	63.0	65.4	0.897
13	25.0	2.29	20.0	63.0	65.5	0.897
14	25.0	2.31	20.0	62.9	65.5	0.897
15	25.0	2.33	20.0	62.9	65.5	0.897
16	25.1	2.17	20.0	63.2	65.2	0.896
17	25.0	2.21	20.2	63.0	65.3	0.895
18	25.6	2.29	20.0	63.2	65.3	0.895
19	25.6	2.20	20.0	63.3	65.2	0.894
20	26.0	2.51	20.0	62.8	65.4	0.888
21	25.0	2.42	35.6	63.4	64.2	0.886
22	26.9	2.04	20.0	63.8	64.5	0.883
23	25.0	2.33	31.4	61.6	64.5	0.867

Table 8 **Comparison of the predicted and experimental PCP and PCDDF removal yields obtained by attrition using the optimal operating conditions (T = 25°C, BW = 2% (w/v) and DP = 40% (w/w) – $[PCP]_i$ = 8.7 ± 2.9 mg.kg^{-1} and $[PCDDF]_i$ = 5,719 ± 180 ng TEQ.kg^{-1})**

Assay	***PCP content in treated soil (mg.kg^{-1})***	***PCP removal yields (%)***	***PCDDF content in treated soil (ng TEQ.kg^{-1})***	***PCDDF removal yields (%)***
Experimental values				
1	2.88	66.5	2,156	63.2
2	2.86	67.8	2,244	59.3
3	2.95	66.1	2,171	62.5
Average	**2.89**	**66.8**	**2,190**	**61.7**
Standard deviation	0.05	0.92	47	2
Predicted values	**2.87**	**67.2**	**2,059**	**64.0**