Different Methods for Polychlorinated Biphenyls Removal from Contaminated Soils

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The paper presents results of laboratory experiments focused on the efficiency of polychlorinated biphenyls (PCBs) removal by electroremediation, pyrolysis and incineration decontamination methods. The experiments were carried out using soil contaminated with heavy metals and PCBs. Different process parameters were changed during the experiments in order to achieve maximum removal efficiency. Concerning the thermal methods (pyrolysis and incineration), the operating temperature and retention time were the two key parameters affecting the processes that were assessed. With regards to the electroremediation alternative for soil decontamination, the redox potential (ORP), pH, electric field and temperature were continuously monitored. Results showed that thermal treatments are the most indicated methods for removing PCBs from contaminated soils. Pyrolysis and incineration remediation methods ensured, with some exceptions, an efficiency of over 99%. The same efficiency level was attended just in case of applying the electrochemical method for removing PCB126.

Keywords: soil pollution, polychlorinated biphenyls, incineration, pyrolysis, electrokinetic remediation

The importance of soil on human life has always been recognized but the anthropogenic activities have often had a negative impact on the quality of the soil. Different types of organic and/or inorganic pollutants were shown to contaminate important surfaces. PCBs are one of the most dangerous pollutants for both human health and the environment. PCBs are a family of man-made chemicals that contain 209 individual compounds with various toxicity levels and have been widely used as coolants and lubricants in capacitors and other electrical equipment because of their insulating and non-flammable properties [1, 2]. Actually, at European level, waste-containing PCBs are carefully monitored and specific regulations are applied. Worldwide, much effort is invested in developing or improving new remediation methods or principles ensuring fast and improved remediation of contaminated soils. Different methods, such as thermal, chemical, microbial or physicochemical, are used alone or in various combinations, i.e. in situ, on site or off site applications [3] for the remediation of contaminated soils. In this context, the authors of this paper compared three types of soil decontamination technologies. Concerning the thermal treatments, the degrees of PCBs soil remediation and PCBs emission levels (for pyrolysis and incineration cases) have been assessed. The behavior of PCB28, PCB77, PCB126, and PCBs has been studied by applying different process parameters. During the incineration, three process temperatures have been considered: 600, 800 and 1000°C. Throughout the pyrolysis process, the applied process temperatures were: 400, 600 and 800°C. For both thermal treatment methodologies, two retention times of the solid material within the incineration and respectively, pyrolysis reactor were taken into account: 30 and 60 min. Related to the electrochemical remediation of soil contaminated with PCBs, the experimental tests monitored the parameters with high influence on the process efficiency: specific voltage (1V/cm), current density, ORP, time and soil *p*H.

Experimetal part

Materials and methods

Soil samples description and analysis method

The soil used during the experimental campaign was collected from one of the most contaminated areas within Central Romania. The soil was historically contaminated with heavy metals (Pb, Cd, Ni, As, Zn, Cu, Fe, Mn, Cr, Co, Hg, As and Be) and artificially contaminated, only for laboratory purposes, with transformer oil. In this way, soil polluted with PCBs has been ensured with an initial concentration for PCBs of 4.4661 mg/kg$_{dw}$. According to the national legislation, Order 756/1997, the intervention threshold for sensible use is established at 1 mg/kg$_{dw}$. So, the initial concentration for PCBs has exceeded four times the legal limit. Table 1 shows the main composition of the contaminated soil before decontamination (where P_{AL} mobile phosphorus content [mg/kg]; K_{AL} is potassium content [mg/kg]; N_t is total nitrogen [%]). The main characteristics of the contaminated soil are: apparent density is 1.2 g/cm^3, while soil humidity is 20%; from the granulometry analysis it could be noticed that more than

Table 1
RAW SOIL COMPOSITION AND HEAVY METALS AND PCBs CONCENTRATION LEVELS

Soil	pH	C organic	Nt*	P_{AL}*	K_{AL}*
		%		mg/kg	
Initial	7,78	4,73	0,182	36	323
Heavy Metals	**Pb**	**Cd**	**Be**	**Cr^{tot}**	**Ni**
[mg/kg$_{dw}$]	2305	75	903	82	72
Polychlorinated biphenyls	**PCB_{28}**	**PCB_{77}**	**PCB_{126}**	**PCBs**	
[mg/k$_{dw}$]	1,3593	0,0576	0,0026	4,4662	

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55% was composed by particle with a diameter smaller than 0.08 mm.

In order to identify the PCBs concentration levels in the soil, the following standards were applied: EN 10382:2007, SR EN 15308:2008; EPA Method 3540. The analysis method has been used both for contaminated and decontaminated soil samples, before and after the decontamination treatment. Before weighing, the solid material was sieved using a 10-mesh sieve and approximately 2-mm openings in thickness. The material was first weighed, mixed with sodium sulphate as drying agent and then sieved. The purpose of using this chemical substance was to provide a uniform drying of the sample particles surface. Following that, applying the Soxhlet extraction the solid samples were brought to a form that allowed for their analysis. About 20g of dried solid material were mixed with 250 mL of HPLC grade petroleum ether solvent. The extract was concentrated to a low solvent volume using a Heidolph rotary evaporator and was re-eluted with hexane. A gas chromatograph with a Shimadzu QP2010 type mass detector has been used to quantify the PCBs concentrations within the soil sample. Calibration curves were obtained using a series of standard solutions, prepared by diluting the standard mix (Pesticide Mix 20 from Dr. Ehrenstorfer) to the desired concentrations using hexane (EPA Method 3540).

Experimental part

Thermal treatment plant

Both thermal treatments (incineration and pyrolysis) have been carried out using the same experimental facility. The compact batch facility is presented in figure 1.

The experimental facility consists of a unique stainless steel horizontal reactor, externally heated by a series of inductive electrical resistances. The reactor has an external diameter of about 60 mm and a total capacity of 800 g of solid material. Due to the appropriate position of the electrical resistances, the uniformity of the temperature field inside the pyrolysis/incineration reactor is guaranteed. The furnace is equipped with automatic and integrated control and can be heated over a length of maximum 750 mm. The maximum achievable temperature is 1300°C using the multi-stage time-temperature settings. 4 kW is the power consumed at a nominal rate. Two ceramic fibreboards externally insulate the furnace with positive impact on external surface heat losses. As already stated, the facility can be used both for pyrolysis or incineration processes based on the gaseous fluid introduced in the reactor (nitrogen or air). The solid material is manually loaded into the reactor. In order to determine the existence of PCBs within the flue gas stream, a portable sampling device form TECORA-Isostack Basic has been used. The system allowed the capitation of both solid and gaseous PCBs within the gas stream. The measurement method of this device is based on isokinetic sampling conditions of a gas volume and deposition of toxic substances on PUF and quartz filter element, correspondingly to EN1948 [4].

Electrochemical treatment

Another treatment methodology applied during the present research was the electrochemical one. This method involved the use of an electric current in order to create an electric field in the polluted area by using electrodes placed into the ground. So, the electric based treatment, also known as electrochemical treatment, uses a specific DC electrical signal to mineralize organic compounds. Specific DC converters produce a low-voltage, low-amperage electric field that polarizes the soil or sediments, so that the soil particles charge and discharge electricity. This causes redox reactions that occur at all interfaces within the soil – water – contaminant – electrode system, mineralizing organics. Electric fields, as well as electron transfer processes, have been used for the decontamination of soils and underground water containing unwanted organic or inorganic substances. The main phenomena involved in the experimental tests were: electrolysis, electrophoresis, electro osmosis, and electro migration. When suitable anodes and cathodes are strategically buried in the ground or placed in contact with slurry and an electric field from a DC source is applied, one or more of these phenomena occur and the output effect is used for the removal of polluting substances. Tests were performed using electrochemical remediation; pilot plant equipment was developed in the framework of RECOLAND project and entitled IPER 2 (fig. 2).

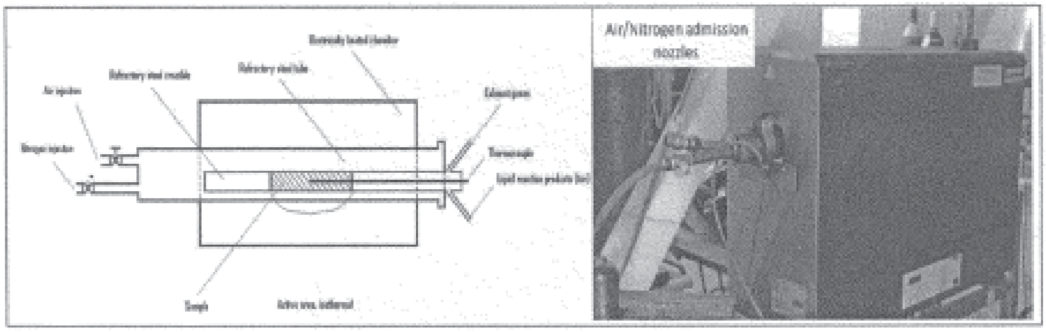

Fig.1. Thermal treatment experimental facility

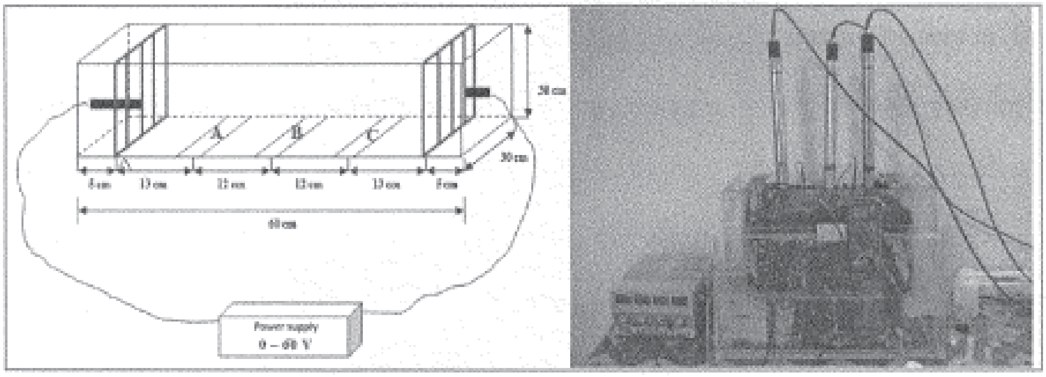

Fig. 2. Experimental setup IPER 2 – electrochemical cell, power supply and the sensors used for pH and redox measurements

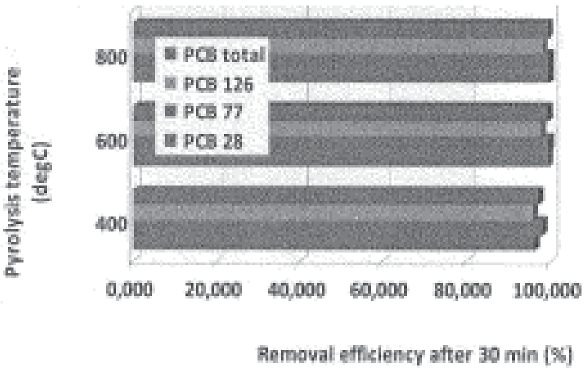

Fig. 3a. Pyrolysis of contaminated soil; PCBs removal efficiency for a 30 min. retention time

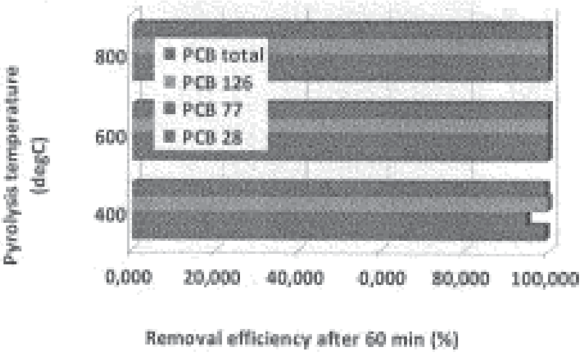

Fig. 3b. Pyrolysis of contaminated soil; PCBs removal efficiency for a 60 min. retention time

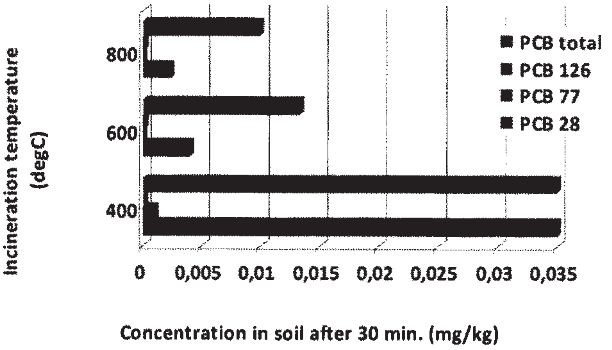

Fig. 4. PCBs concentration level in 30 min. pyrolized soil

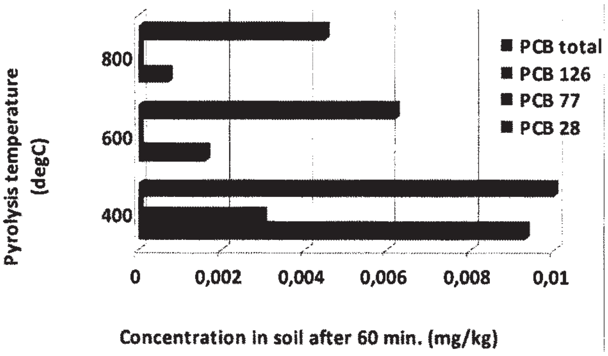

Fig. 5. PCBs concentration level in 60 min. pyrolized soil

Results and discussions

Pyrolysis of the artificially contaminated samples

Soil used during the experimental campaigns has the same characteristics as illustrated in table 1. For every single test, a sample of contaminated soil with PCBs was used. The results of the pyrolysis process showed PCBs removal efficiency from soil between 97% and 99% as a consequence of process temperatures (400, 600°C and 800°C) and different retention times of the solid material inside the reactor (30 min and, respectively 60 minutes). Figures 3.a and 3.b illustrate removal efficiencies for every single contaminant (PCB28, PCB77 and PCB126), but also for PCBs.

Concerning PCB28, except the case of 400°C pyrolysis temperature and 30 min retention time for which the removal efficiency is of 96.38%, all other experiments revealed efficiencies of more than 99% for PCB28. Furthermore, no major difference can be noticed between the lowest and the highest values of the PCB28 removal efficiencies: a lesser amount than 1%. This is valid for both retention times during soil pyrolization. For a process temperature of 400°C, the influence of the retention time is higher: increasing the retention time from 30 to 60 min lead to the increase with 2.94% of the PCB28 elimination efficiency. The highest PCB28 removal efficiency was of more than 99.949% at a retention time of 60 min and 600°C process temperature. If in terms of the 400°C process temperature, the retention time has no influence on the PCB28 removal efficiency, the same situation does not apply in terms of soil pyrolysis at 600°C (fig. 4 and fig. 5).

Due to the fact that PCB28 and PCBs concentration levels in soil are regulated by the Romanian MWFEP Order no. 756/1997, the levels of PCB28 and PCBs from pyrolized soil were compared with the national reference levels. In this case, for both alert and intervention levels for less sensitive use (industrial), soil pyrolization (at every single temperatures and retention times) ensured for PCB28 and PCBs concentrations in soil below the reference levels. Exception to this was the case when a process temperature of 400°C and a retention time of 30 min where applied. In this case the PCB28 concentration in soil was of about 0.0492 mg/kg$_{d.w.}$ (4.92 higher than the intervention concentration – soil for sensitive use). In addition to the congeners considered by the Romanian MWFEP Order no. 756/1997 [5], during the experimental activities, the most toxic PCBs [6], PCB77 and PCB126 were also analysed. In terms of PCB77 removal from contaminated soil, the pyrolysis technology showed to be an important alternative. A high efficiency was obtained even for the lowest temperature (400°C), at two pyrolysis retention times: 98.26% for 30 min. and 94.79% for 60 min. Higher pyrolysis temperatures (600 and 800°C) ensured maximum efficiency for PCB77 removal (99.999 %) for both retention times. Figure 4 and figure 5 illustrate the PCBs concentration after the pyrolysis of the contaminated soils. Concerning the PCB126 removal from contaminated soil functions, the influence of the retention time on the removal efficiency was observed. For the 60 min retention time, 99.999% removal efficiency was observed in case of the 400, 600 and 800°C process temperatures. Contrary to this, the retention time of 30 minutes ensured different efficiencies: from 96.154 % for 400 °C to 98.462 % for 800 °C (fig. 3.a. and fig. 3.b.). Related to pyrolysis tests on PCBs removal from similar soil matrix suggested that, considering the economics point of reducing energy requirements, the lowest retention time at 800°C may be sufficient for ensuring high pyrolysis efficiency (99.99%) [7]. In case of the soil artificially contaminated with PCB-containing transformer oil from the present experimental study, in terms of the pyrolysis process, a high efficiency of 99.778% for PCBs removal was obtained. Other important aspects that must be considered in terms of the thermal treatments methods for soil decontamination are the emissions of PCBs from soil pyrolysis. For this reason, emissions of PCB28, PCB77, PCB126 and PCBs were also analyzed (fig. 6).

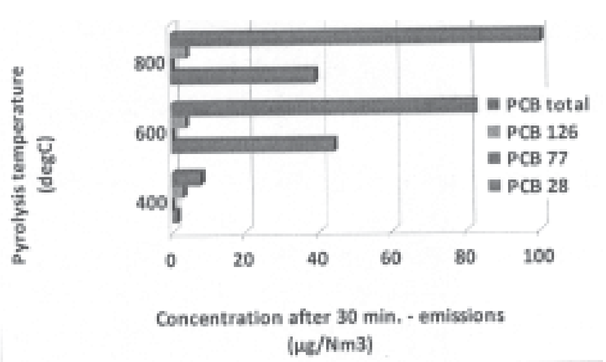

Fig. 6. Emission concentration level of PCBs from soil pyrolization (30 min. retention time)

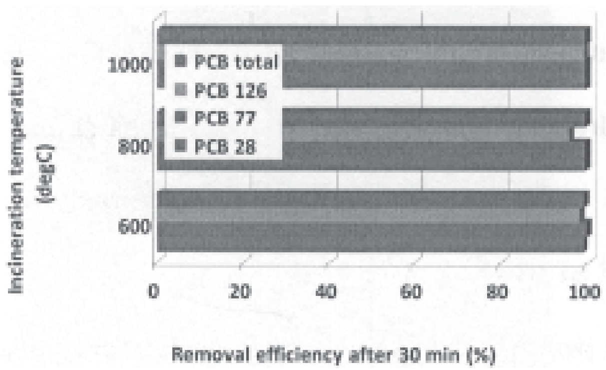

Fig. 7a. Incineration of contaminated soil; PCBs removal efficiency for 30 min. retention time

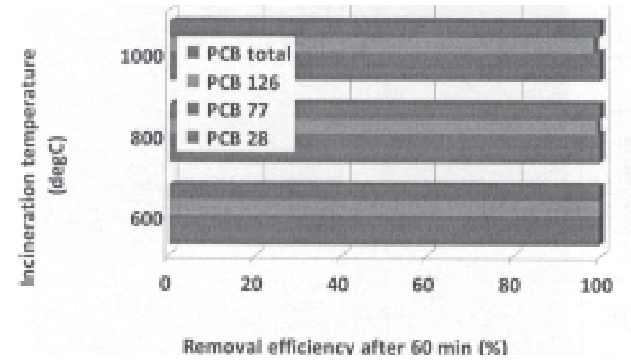

Fig. 7b. Incineration of contaminated soil; PCBs removal efficiency for 60 min. retention time

The results concerning the emissions showed that temperature and retention time have an important influence on PCBs concentration in the flue gases generated during the thermal treatment in a nitrogen atmosphere. However, this is not valid for all thermal treatments developed in the present experimental study. Consequently, the retention time has no influence on emissions due to pyrolysis at 400°C, when the concentration of the PCBs in the released gases remains almost of the same value if the soil sample is treated for 30 or 60 min. The effect of the process time is important for high pyrolysis temperatures (600 and 800°C), when from 30 to 60 min. retention time conducted to two times concentration levels of PCBs in the pyrogases. This has happened for every single experiment concerning the PCB28, PCB77, PCB126 and PCBs behaviors. For instance, the concentration of PCB28 in the pyrogas formed during the pyrolysis process decreased with almost 6% from 30 to 60 min. retention time. Thus, the increase of the retention time decreases the PCB28 concentration level. The same behaviour was registered if the pyrolysis temperature is raised from 600 to 800°C (a decrease of 63% was registered for 30 min. and 53% for 60 min.). According to the results presented in the figures above, it seems that PCBs volatilization is the main mechanism for PCBs removal from contaminated soil under operational conditions applied in our experiments, phenomenon revealed in studies carried out by other authors [8] for a similar soil matrix.

Incineration of the artificially contaminated samples

Results obtained during the experiments demonstrate that during incineration, PCBs are almost entirely removed from the contaminated soil. Removal efficiency of PCBs from the soil is presented in figure 7.a and 7.b for different process temperatures (600, 800 and 1000°C) at different retention times of the solid material inside the reactor. As expected, for all studied cases, the removal efficiency was over 99%, except the PCB126 at 800°C and 30 min., for which, the removal efficiency was of approximately 97%. This could be explained by the temporary increase in concentration of PCB126 due to cracking mechanism of the heavier species of PCBs into PCB126.

As figure 8 shows, the volatilisation and removal rates were higher after 800°C; in case of PCB28; the removal efficiency increases from 99% (at 600°C) to over 99.9% (at 800 and 1000°C). In this case, the time factor had very sight influence on PCBs removal from contaminated soil. From the environmental point of view, as already mentioned, the Romanian regulations limit PCBs concentrations within soils based on its utilisation: agriculture or construction. Even if incineration seems to be one of the best technologies to remove and destroy PCBs from soils, it is very important to consider also PCBs emissions level within the process gas. Figure 9 shows the concentration of PCBs in flue gas in case of 30 min. retention time. As it can be observed, all studied congeners have the same trend.

Higher the temperatures, higher are the differences between devolatilization and the destruction rates. In order to completely destroy PCBs from the gas stream, the real incineration plants are built with a secondary combustion

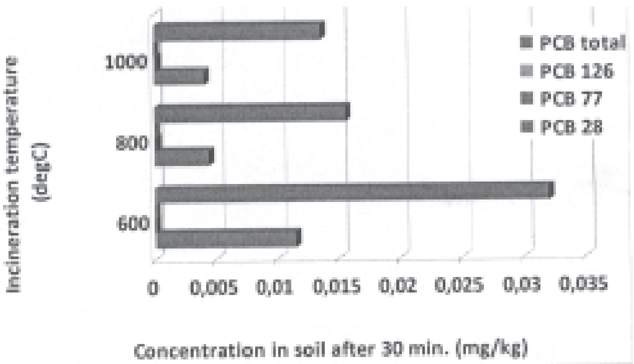

Fig. 8. PCBs concentration level in soil after the incineration process

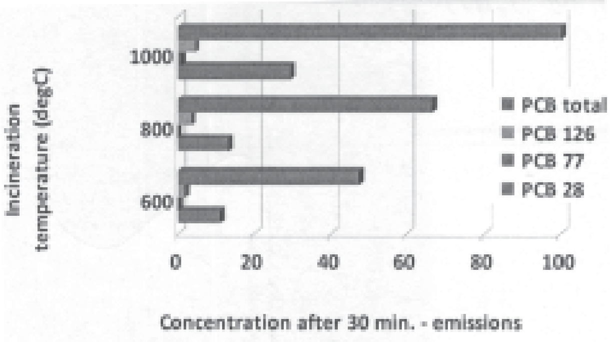

Fig. 9. Emission concentration level of PCBs from soil incineration (30 min. retention time)

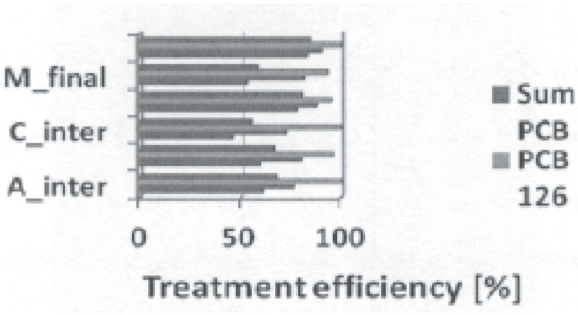

Fig. 10. The time influence on the removal efficiency of the electrochemical treatment (12 and 21 days)

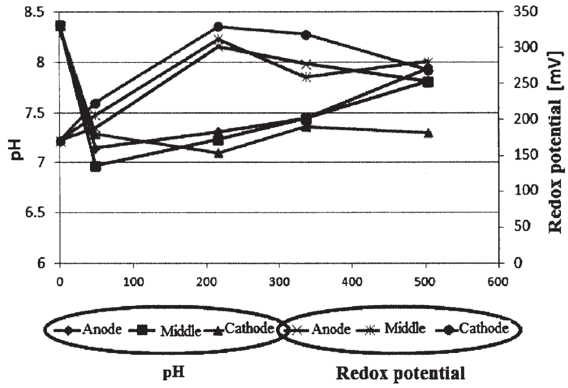

Fig. 11. Comparison between pH and ORP variation during the electrochemical experiment

Table 2
INITIAL AND FINAL CONCENTRATION FOR THE PCBs OF CONGENERS AFTER APPLYING ELECTROCHEMICAL TREATMENT [mg/kg$_{dw}$]

	PCB 28	PCB77	PCB126	PCBs
Initial	1.3593	0.0576	0.0026	4.4661
A_inter	0.5288	0.0137	n.a.	1.4263
M_inter	0.5459	0.0113	0.0001	1.4830
C_inter	0.7302	0.0157	n.a.	1.9900
A_final	0.2926	0.0069	0.0010	0.8714
M_final	0.6323	0.0105	0.0020	1.83
C_final	0.2318	0.0057	n.a.	0.67

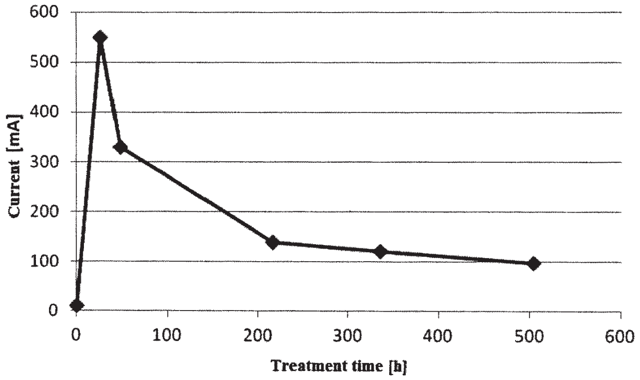

Fig. 12. Current trend in time during the electrochemical treatment

chamber [9] where the gas stream is completely oxidized for 2 s at temperatures of about 1200°C. In this manner, the production of future dioxins and furans is avoided.

Electrochemical treatment

As previously underlined, the electrochemical treatment has been applied on an artificially contaminated soil with PCB-containing transformer oil and anthropogenic with heavy metals. The electrochemical treatment was applied on almost 60 kg of PCB polluted soil, for a period of 21 days. The specific voltage was 1 V/cm; that means an applied voltage of 50 V (the distance between the electrodes was set at 50 cm).

As in case of thermal treatment methodologies illustrated before, the behaviour of PCB 28, PCB 77, PCB 126 and PCBs, prior and after the electrochemical treatment, were analyzed. The highest initial PCB28 concentration was of 1.3595 mg/kg$_{d.w.}$ (table 2). During the experiment, a continuous monitoring of the key parameters has been carried out (electric current, *p*H, redox potential and temperature). The soil *p*H represents a measure of acidity which plays an important elements and processes from soils [10-13]. Samples after 12 days, as well as at the final experiment (after 21 days) have been characterized in order to determine the concentration levels for the already mentioned PCBs congeners. The soil sampling has been done from three different areas: anode, middle and cathode. The treatment efficiencies have been assessed as presented in figure 10 and table 2.

Treatment efficiencies for the test performed on PCBs contaminated soil, increased in time. Concerning the PCBs concentration level, after 21 days the concentrations in the electrodes areas are smaller than the intervention threshold for sensible use mentioned in O756/1997 (removal efficiency ranged from 59% to 85%). If the attention is focused on PCB 28, the electrochemical treatment should continue for at least other 2 weeks (over 21 days of the experimental study), considering that a lower efficiency was obtained: from 53% to 83% related to the different sampling areas (anode, middle and cathode). The two PCBs congeners that are toxic for human health (PCB77 and PCB126) showed good removal efficiencies. Even if higher removal efficiency was obtained for PCB126 (between 94% and 100%) the results concerning PCB77 could be considered more accurate because of the initial concentration of two congeners (about 0.0026 mg/kg$_{d.w.}$ for PCB126 thus, 12 times smaller than the one for PCB77).

Concerning the ORP that was checked across the experiments, this is a measure of the system ability to oxidize (accept electrons) or to reduce (donate electrons) in the system. Consequently, there are ensured conditions for oxidizing or to reducing other components in the system. If ORP is positive, less oxidant will be required to oxidize a component [14 - 16]; in this way, soil or sediment remediation is expected.

*p*H is another key parameter of the electrochemical remediation process with important influence on method efficiency because is playing a part in the range that oxidation and reduction can take place. The reason, for which ORP was monitored during the electrochemical tests, was both to observe where the oxidation/reduction reactions took place and to control the treatment efficiency along the soil sample. Results gained until now and published in the specialized literature showed that, if applying a constant voltage to an organic polluted soil and the changing ORP in a considerable way, a non uniformity in removing organic pollutant is observed at the end of the process [17]: higher removal at anode where the oxidation reaction occurred and smaller at the cathode. With the aim of avoiding these kinds of results (no uniformity in removing organic pollutants from soil), during the experimental tests, the oxidation reactions along the soil sample were encouraged: every single time when it was observed that ORP was below 0, the electric potential was changed and in this way anode became cathode and the cathode became anode.

The third key parameter monitored across the electrochemical tests was the current that has an influence on the overall costs due to the fact that it will increase the costs with energy expenditure. Usually when we have a high value for the current in an area, in the same area we have strong oxidation reactions.

Results gained throughout the experimental research concerning the key parameters of the processes are according to figures 11 and 12.

Conclusions

This paper presents the results of the research on three decontamination methods for PCBs removal from soil, namely: pyrolysis, incineration and electrochemical remediation. For every single decontamination method, the behaviour of PCB28, PCB77, PCB126, and PCBs has been studied, using different process parameters. The initial PCBs concentrations, according to the national regulation, were 1.3593 $mg/kg_{d.w.}$ for PCB28 and 4.4661 $mg/kg_{d.w}$ for PCBs. Envisaging the future use of the decontaminated soils for agriculture, the authors considered the national acceptable levels for intervention thresholds (sensitive use) as reference levels. In this case, the results showed that the initial concentration for PCBs exceeded by our times the legal limit, while the PCB28 concentration level was of about 135 times in excess. With respect to the process temperature of 600 and 800°C for the thermal treatment methodologies, our results showed that both pyrolysis and incineration are efficient methods for removing PCB28, PCB77, PCB126 and PCBs from contaminated soils, generally attaining an efficiency of over 99%, independently of the process parameters. Exception to the rule was PCB126 that seems to be more difficult to be removed from soil at 600 and 800°C for 30 min.: for pyrolysis the efficiencies were 98.077 and 98.462%, respectively, while for incineration, 98.367 and 95.769%, respectively. As expected, the lower pyrolysis process temperature at 400°C for 30 min. retention time ensured a lower efficiency of 97.349%, and the higher incineration process temperature of 1000°C for both 30 min. and 60 min. retention times, guaranteed higher efficiencies, in particular, over 99%. Even though, thermal treatment ensured high efficiencies, it must be said that, special attention should also be paid to emission levels and energy consumption. Also for this reason, research was additionally focused on electrochemical remediation. The temporary results (after 12 days), as well as the final ones (after 21 days), pointed out that PCBs concentration levels exceeded the nationally acceptable levels for intervention thresholds (sensitive use) and a high efficiency was gained just for PCB126 removal (over 99%). This is an important aspect if one would take into account that, in case of the thermal treatment, PCB126 removal was problematic. With regard to other congeners and PCBs, the results indicated that, in order to have higher efficiency, the electrochemical treatment needs more time. Future research will be focused on costs, environmental impact and energy consumption in relation with the tested remediation methods.

The subject has been also studied in [18].

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