



## Research article

# Dechlorination of PCBs in oil transformers by microwaves catalysed by Fe<sup>0</sup>, glycerol, and NaOH

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## ABSTRACT

Dechlorination of Polychlorinated Biphenyls (PCBs), carcinogenic substances used as oil in electrical transformers, remains an environmental challenge. This study aims to investigate the dechlorination of PCBs-contaminated transformer oils using microwave catalytic (Fe<sup>0</sup>, glycerol) pyrolysis and to study the kinetics of PCBs dechlorination. After determining the composition of PCBs-contaminated oil using Gas Chromatography with an Electron Capture Detector (GC-ECD), response surface methodology through a central composite design was used to optimize dechlorination factors (irradiation time, microwave power, NaOH). Finally, a kinetic study of PCBs dechlorination under optimal conditions was conducted. The results showed that the concentration of PCBs in the studied transformer oil was  $526 \pm 0.01$  mg/kg, composed of 14 congeners, with a dominance of hexa-PCBs (70 %) and penta-PCBs (18 %). The optimal PCBs dechlorination yield of 98.87 % was obtained under the following conditions: microwave power of 700 W, irradiation time of 8 min, and 0.3 g of NaOH. The kinetic study showed that PCBs degradation under optimal conditions follows a first-order reaction. These findings suggest that microwave-assisted catalytic pyrolysis is an effective and promising method for PCBs dechlorination in transformer oil, offering a potential solution for environmental remediation.

## 1. Introduction

Polychlorinated biphenyls (PCBs) are organic compounds consisting of carbon, hydrogen, and chlorine atoms. The physical and chemical properties of a PCBs molecules are determined by the number and position of its chlorine atoms, which can range from 1 to 10. Due to their properties, they have many industrial applications, such as dielectric fluids in capacitors, transformer oils, plasticisers, lubricant inks, paint additives, and adhesives [1]. However, PCBs exhibit carcinogenic properties, environmental persistence, bioaccumulation and biomagnification potential, reproductive effects, endocrine disruption, and impacts on the immune system [2–4]. Due to these characteristics, they are increasingly banned internationally.

The Persistent Organic Pollutants Convention of Stockholm states that PCBs are regulated or prohibited worldwide [5]. The Stockholm Convention, signed by 125 countries globally, brought several responsibilities, such as establishing inventories on the levels of POPs and decreasing and eliminating their environmental presence. Cameroon signed the Stockholm Convention on Persistent

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Organic Pollutants (POPs), ratified it in 2005, and became a country party in 2009. UNEP inventoried 1600 tonnes of PCBs-contaminated mineral oil [6] as part of the PCBs reduction in the Cameroon project, which utilised local expertise and developed national skills. The PCBs Elimination Network has ordered the parties to the Convention to ensure ecologically sound waste management of PCBs by 2028 and to stop using PCBs in any currently in-use equipment by 2025. The first steps in the worldwide management of PCBs are transformer sampling, dielectric oil analysis, transformer characterisation, and transformer disposal [7]. However, a significant amount of transformer oils containing PCBs are still in use or storage due to their benefits and adaptability to many environments [8]. Therefore, it is necessary to decontaminate PCBs transformer oil.

Incineration technology is quite effective for remediating PCBs-contaminated materials. However, highly toxic byproducts such as dioxins and furans, as well as disposal costs and the exclusion of reusable materials, are obstacles [9,10]. Another approach involves pyrolysis, chemical methods, supercritical water oxidation, and microbial degradation [11,12]. Among these methods, microwave catalytic dechlorination has been recognized as a promising technology due to its easy and simple procedures [13]. New techniques, including the use of an electron beam accelerator [14], plasma [15], lasers [16], adsorption, and absorption [17], have recently emerged. Base-catalysed decomposition is a valuable and reasonable method for decontaminating PCBs-contaminated transformer oils and soils [18,19]. In this method, PCBs are dechlorinated using a metal catalyst, a base, and a high-boiling-point hydrocarbon. Some hydrocarbons give off hydrogen, bases that act as nucleophilic activating groups, and metal catalysts that move electrons around in the PCBs dechlorination process [20].

Among metal catalysts, zero-valent iron ( $\text{Fe}^0$ ) has garnered significant attention for PCBs degradation due to its unique properties, including a large surface area, high reactivity, and cost-effectiveness [21–25]. Additionally,  $\text{Fe}^0$ , abundantly available as a recycled material and capable of completely degrading certain pollutants, is increasingly recognized as a sustainable solution [26,27]. Concerning the hydrocarbon component, trihydric alcohol glycerol is compatible with most substances, has no harmful effects on the environment, is less expensive, has a high melting point, and can act as a hydrogen donor and solvent in the base-catalysed decomposition process [20,28].

Compared with conventional heating, microwave heating has emerged as a novel method for PCBs degradation because of its high removal rate, high efficiency, quick reaction rate, and economic viability [29–36]. Many studies on the microwave removal of PCBs from transformer oil have focused on microwave-hydrothermal degradation [33,37,38], where they have demonstrated that factors such as microwave power, irradiation time, and the amount of reactants significantly influence dechlorination yield. This work aimed to investigate the dechlorination of PCBs-contaminated transformer oils using microwave catalytic pyrolysis with  $\text{Fe}^0$ , glycerol, and NaOH, and to study the kinetics of PCBs dechlorination under optimal conditions.

## 2. Materials and methods

### 2.1. Chemicals

The Ministry of Environment, Protection of Nature, and Sustainable Development of Cameroon provided mineral transformer oil. All reagents were purchased from Sigma-Aldrich Company, n-hexane (99 %, GC grade), high-purity silica gel (60–100 mesh), and anhydrous sodium sulphate were used. Standard solutions of PCB-Mix 14 components (PCB-28, PCB-52, PCB-31, PCB-77, PCB-101, PCB-105, PCB-118, PCB-126, PCB-138, PCB-153, PCB-156, PCB-169, and PCB-170) at 10  $\mu\text{g}/\text{mL}$  each in iso-octane were purchased from CPAchem. PCB-30 and PCB-209 at 10  $\mu\text{g}/\text{mL}$ , used as internal standards were purchased from Sigma Aldrich. Sodium hydroxide (NaOH) was purchased from Befar Group (China), and zero-valent iron ( $\text{Fe}^0$ ) was 0.74  $\mu\text{m}$  in diameter.

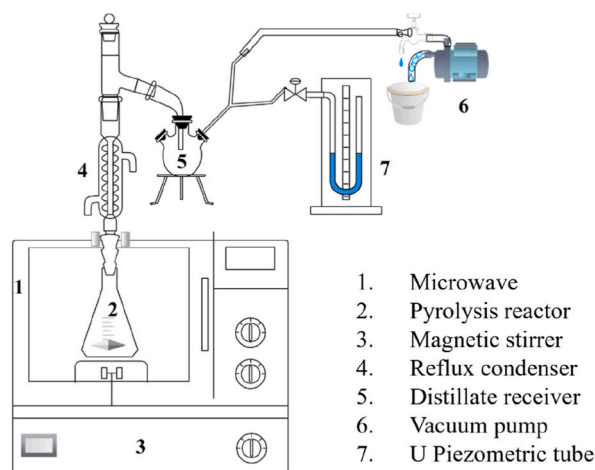


Fig. 1. Microwave pyrolysis system.

## 2.2. PCBs microwave dechlorination

The microwave dechlorination experiments were conducted in a modified domestic multimode microwave (Moulinex) oven with a maximum power of 800 W operated at 2450 MHz. The top of the oven was an opening, 40 mm in diameter, with a Teflon sealing ring. A glass condenser that allows outgoing vapour to condense protrudes through the opening. The condenser is connected to a 50 mL boiling flask, which resides inside the oven and serves as the reactor. A condensation system at the reactor outlet leads to a gas purification system with hexane. An inert internal environment in the reactor was created using a vacuum pump before and during microwave irradiation to reduce the risk of chlorinated dioxin formation further. After irradiation, the reactor was cooled quickly to 6 °C, after which the oily phase was separated by simple decantation. Fig. 1 shows the microwave pyrolysis system used in this study.

## 2.3. Experimental design

Design-Expert software 12.0 was used for the modelling and optimisation of microwave PCBs dechlorination. Response surface methodology (RSM) via central composite was used. Three (03) factors were considered: NaOH, microwave power, and irradiation time. These factors and their experimental domain are presented in Table 1. The value of each factor was chosen according to Kamarehie et al. [38,39] and to Asilian et al. [40], power, irradiation time, and NaOH. The factor Fe<sup>0</sup> (0.6 g) [38] and the oil PCBs/glycerol ratio (1/1) were kept constant during the optimisation [19].

The second-order polynomial model was used to determine the correlation between the dechlorination factors and the microwave PCBs dechlorination yield. This model is given by equation (1).

$$Y = a_0 + a_1X_1 + a_2X_2 + a_3X_3 + a_{12}X_1X_2 + a_{13}X_1X_3 + a_{23}X_2X_3 + a_{11}X_1^2 + a_{22}X_2^2 + a_{33}X_3^2 + e \quad (1)$$

where  $Y$  is PCBs dechlorination yield,  $a_0$  is the intercept,  $a_1, a_2, a_3$  are the linear coefficients of the independent factors,  $a_{11}, a_{22}, a_{33}$  are the quadratic coefficients of factors,  $a_{12}, a_{13}, a_{23}$  are the different interaction coefficients between the input factors  $X_1, X_2$ , and  $X_3$ , and  $e$  is the error of the model.

## 2.4. Sample clean-up and analysis

A clean-up step is required before analysis of the microwave PCBs dechlorination sample in GC-ECD. This method involves mixing 1 g of microwave-treated oil sample with 1 mL of internal standard PCBs and adjusting the volume to 10 mL with n-hexane in a flask. This mixture was then allowed to clean-up using a column containing 0.5 g of deactivated silica gel (with 0.5 % distilled water). The column was first washed with 1 mL of n-hexane, then a microwave-treated oil sample (500 µL) was added, and the column was eluted with 1.5 mL of n-hexane. The eluted sample was collected in a 5 mL gauge flask, and the volume was adjusted with n-hexane.

Quantitative analysis of microwave-treated oil was performed by Gas Chromatography with an Electron Capture Detector (GC/ECD 2030) (Shimadzu). A specified SH-Rxi-XLB column (60 m\*2.5 µm\*2.5 mm) was used to separate the PCBs. The injector and detector were set at 210 and 300 °C, respectively. Helium (99 % purity) was used as the carrier gas at a flow rate of 1 mL/min, and the column temperature program was 80 °C, 4 min, and 4 °C/min to 300 °C. Hold for 5 min at 0 °C. One microliter of the purified sample was analysed.

## 2.5. Kinetic study of microwave PCBs dechlorination

The dechlorination kinetics were determined over a time range from 2 to 10 min. The other factors (microwave power and NaOH amount) are set to their optimal values. Kinetic modelling of PCBs dechlorination congeners according to De Filippis et al. [41] was performed via equation (2).

$$C_t = C_0 * e^{(-kt)} \quad (2)$$

where  $C_t$  and  $C_0$  are the residual and initial PCBs concentrations (mg/kg), respectively;  $k$  is the rate constant ( $\text{min}^{-1}$ ); and  $t$  is the irradiation time (min).

**Table 1**  
Optimisation factors studied and experimental domain.

Factors	Experimental domain				
	$-\alpha$	Low	Medium	High	$+\alpha$
	-1,68179	-1	0	1	1,68179
NaOH (g)	0.1	0.3	0.9	1.5	1.9
Power (W)	164	300	500	700	836
Time (min)	1.3	3	5.5	10	12

### 3. Results and discussion

#### 3.1. PCBs profile in transformer oil

PCBs reduction in Cameroon using local expertise and the development of national capacities inventoried 1600 tonnes of PCB-contaminated mineral oil. In this study, GC-ECD was used to analyse the profile of PCBs-contaminated oil. Fig. 2 shows the distribution of 14 PCBs weights in transformer oil. Based on the Balschmitter method, considering only PCB-28, PCB-52, PCB-101, PCB-138, PCB-153, and PCB-170, the concentration of PCBs is  $526 \pm 0.01$  mg/kg of oil. This concentration is 10.5 times higher than the maximum allowed (50 mg/kg) by the Stockholm Convention.

A grouping of the PCB congeners identified previously into homologous groups shows a high proportion of hexa-PCBs (70 %) and Penta-PCBs (18 %), as shown in Fig. 3.

#### 3.2. Optimisation of transformer oil PCBs dechlorination

Table 2 presents the result of a central composite design aimed at optimizing the dechlorination of PCBs based on three main factors: NaOH, microwave power, and irradiation time.

The PCBs dechlorination yield varies from 82.43 % to 87.21 %, with most values clustering around 84 %–86 %, indicating substantial changes in dechlorination efficiency based on different factor combinations. The average dechlorination yield was around 84.95 %, suggesting good overall performance for the conditions tested. According to the literature, PCBs dechlorination yield varied between 83 % and 97 %. Akhondi et al. [19] obtained 99.8 % PCBs removal via base-catalysed decomposition (BCD) using sodium hydroxide, zero-valent iron, and glycerol to dechlorinate transformer oils with low polychlorinated biphenyl concentrations. Repeated conditions (0.9 g NaOH, 500 W, 6.5 min) show good reproducibility, indicating stability under moderate conditions. Extreme levels of power (700 W) and time (10 min) tend to yield higher dechlorination rates, suggesting that more intense conditions may be beneficial.

The second-degree polynomial equation used to relate the PCBs dechlorination yield to the studied factors is provided by equation (3).

$$Y(\%) = 84.34 + 0.15NaOH + 1.04Power + 0.25Time - 0.75NaOH * Time - 0.08Power * Time - 0.27NaOH * Power + 0.03NaOH^2 + 0.10Power^2 + 0.70Time^2 \quad (3)$$

Each coefficient in the equation represents the main effect, interaction, or quadratic term of the factors studied on the PCBs dechlorination yield. Positive coefficients indicate an increase in the dechlorination yield as the factor increases, while negative coefficients indicate a decrease.

- Statistical analysis of the models

The analysis of variance of the central composite design of the mathematical model of PCBs dechlorination yield is shown in Table 3, where the p-values of power irradiation are less than 0.05, which highlights the great influence that microwave power has on the dechlorination PCBs yield.

The ANOVA table for the quadratic model of transformer oil PCBs dechlorination reveals several significant findings. The model demonstrates a high degree of statistical significance ( $p < 0.05$ ) and substantial explanatory power, as evidenced by an  $R^2$  value of 0.92, indicating that the model can explain 92 % of the variance in PCBs dechlorination. Only microwave power shows a significant main effect, independently influencing dechlorination yields. Interaction effects between NaOH and time are also significant, suggesting that these combinations amplify the dechlorination process beyond their impacts. Moreover, the quadratic term for time demonstrates a pronounced nonlinear relationship with dechlorination, underscoring the importance of considering both linear and

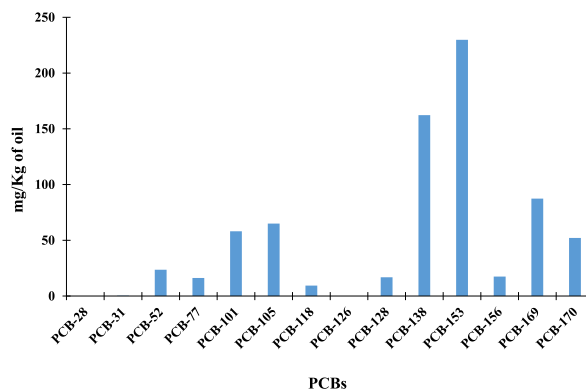


Fig. 2. Distribution of PCBs-contaminated oil.

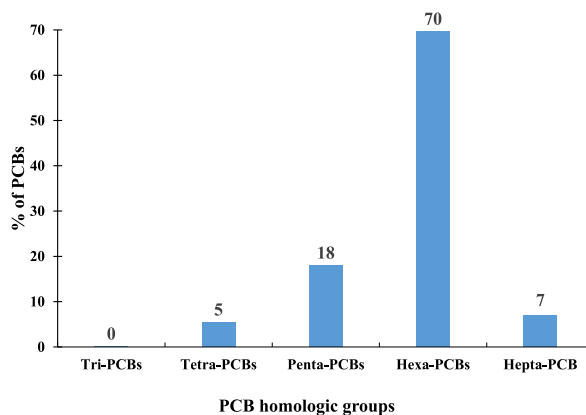


Fig. 3. PCBs homologue in transformer oil.

Table 2

Matrix of central composite design.

Run	Factors			Dechlorination (%)
	NaOH (g)	Power (W)	Time (min)	
1	0.3	700	3	85.04
2	0.9	164	6.5	82.58
3	0.9	500	0.6	86.56
4	0.9	500	6.5	84.68
5	1.5	700	3	86.09
6	2	500	6.5	84.88
7	0.9	500	6.5	83.81
8	1.5	700	10	85.54
9	0.3	700	10	87.21
10	1.5	300	3	84.88
11	0.3	500	6.5	84.21
12	0.9	500	6.5	84.49
13	0.3	300	10	85.25
14	0.9	836	6.5	86.89
15	0.3	300	3	82.43
16	0.9	500	12	86.31
17	1.5	300	10	84.36

Table 3

ANOVA of the quadratic model of transformer oil PCBs dechlorination.

Source	Sum of squares	df	Mean	Coeff	F-value	p-value
Intercept	27.212	9	3.024	84.341	9.531	0.003 <sup>a</sup>
NaOH	0.318	1	0.318	0.153	0.970	0.350 <sup>b</sup>
Power	14.757	1	14.757	1.040	46.51	0.000 <sup>a</sup>
Time	0.892	1	0.892	0.256	2.81	0.138 <sup>b</sup>
NaOH*Power	0.595	1	0.595	-0.273	1.87	0.213 <sup>b</sup>
NaOH*Time	4.594	1	4.594	-0.758	14.48	0.007 <sup>a</sup>
Power*Time	0.060	1	0.060	-0.087	0.188	0.677 <sup>b</sup>
NaOH*NaOH	0.013	1	0.013	0.030	0.024	0.848 <sup>b</sup>
Power*Power	0.115	1	0.115	0.101	0.368	0.566 <sup>b</sup>
Time*Time	5.563	1	5.563	0.702	17.80	0.004 <sup>a</sup>
Residual	2.222	7	0.317	0.317		
Lack of Fit	1.807	5	0.361	0.361	1.743	0.403
Pure Error	0.415	2	0.207	0.207		
Cor Total	29.434	16				
Std.Dev	0.563		R <sup>2</sup>	0.92		
Mean	85.01		Adjusted R <sup>2</sup>	0.75		
C.V. %	0.66					

<sup>a</sup> Significant ( $p < 0,05$ ).

<sup>b</sup> Non-significant ( $p > 0,05$ ).

quadratic effects in optimizing the process. The lack of fit test, yielding a non-significant result ( $p = 0.403$ ), reinforces the model's adequacy in fitting the data.

#### • Influence of the factors on the PCBs dechlorination and optimal conditions

A detailed analysis of the interactions between factors is necessary to refine the optimisation process. Figs. 4–6 present the effects of the interactions Power\*Time, NaOH\*Power, and Time\*NaOH, on PCBs dechlorination yield, respectively. Both contours and a three-dimensional depiction of the effect of interactions are represented.

From Fig. 4, there is a close relationship between the increase in power and the increase in time. An increase in these two parameters is followed by an increase in PCBs dechlorination. The dechlorination reaches a maximum of approximately 87.21 % for a power of 700 W and a time of 10 min when the NaOH is at its lowest value (0.3 g). As the NaOH concentration increases, the optimal zone for dechlorination shifts. For instance, when the mass of NaOH increases from 0.3 to 1.5 g, the new optimal zone shifts to a lower irradiation time of approximately 3–4 min at similar power levels. The increase in NaOH enhances the reaction temperature, allowing the same dechlorination yield to be achieved in a shorter time.

There is a strong interaction between the microwave power and the mass of NaOH (Fig. 5). As previously described, the simultaneous increase in power and NaOH increases the dechlorination yield. However, the maximum dechlorination yield varies as a function of time. For low irradiation times (less than 4 min), the maximum degradation is obtained for high powers and high masses of NaOH. When the irradiation time increases from 3 to 10 min, the optimal zone shifts; at a power of 700 W, the highest dechlorination yields are achieved with lower masses of NaOH. This shift in the optimal zone is due to the combined effects of increased reaction time and the influence of NaOH on the reaction temperature, which enhance the dechlorination process.

Fig. 6 illustrates the significant influences of NaOH concentration and irradiation time on PCBs dechlorination across different microwave powers (300 W, 500 W, and 800 W). It demonstrates an increase in the dechlorination yield when simultaneously increasing the irradiation time and the mass of NaOH. The optimal zone shifts as the power increases. The dechlorination reaction is thought to be highly sensitive to microwave power because the power level directly affects the maximum temperature that the catalyst or reaction system may achieve. As a result, an increase in microwave power will provide more thermal energy and thus increase the degradation efficiency [42]. The degradation efficiency generally increases with increasing microwave power [32,43]. The reactants NaOH, Fe<sup>0</sup>, and glycerol are first exposed to microwave radiation by space charge polarisation and dipolar polarisation, and the incident microwave energy is immediately transformed into thermal energy [32]. Since the breakdown of glycerol is the primary mechanism for the formation of hydroxyl radicals, increasing the power also increases the rate at which hydroxyl radical hydrogen is generated, which in turn causes PCBs to oxidise [44]. Sodium hydroxide plays an important role in the dechlorination reaction; it activates PCBs and facilitates the transfer of hydrogen species to activated PCBs [45]. The HCl produced during the dechlorination of

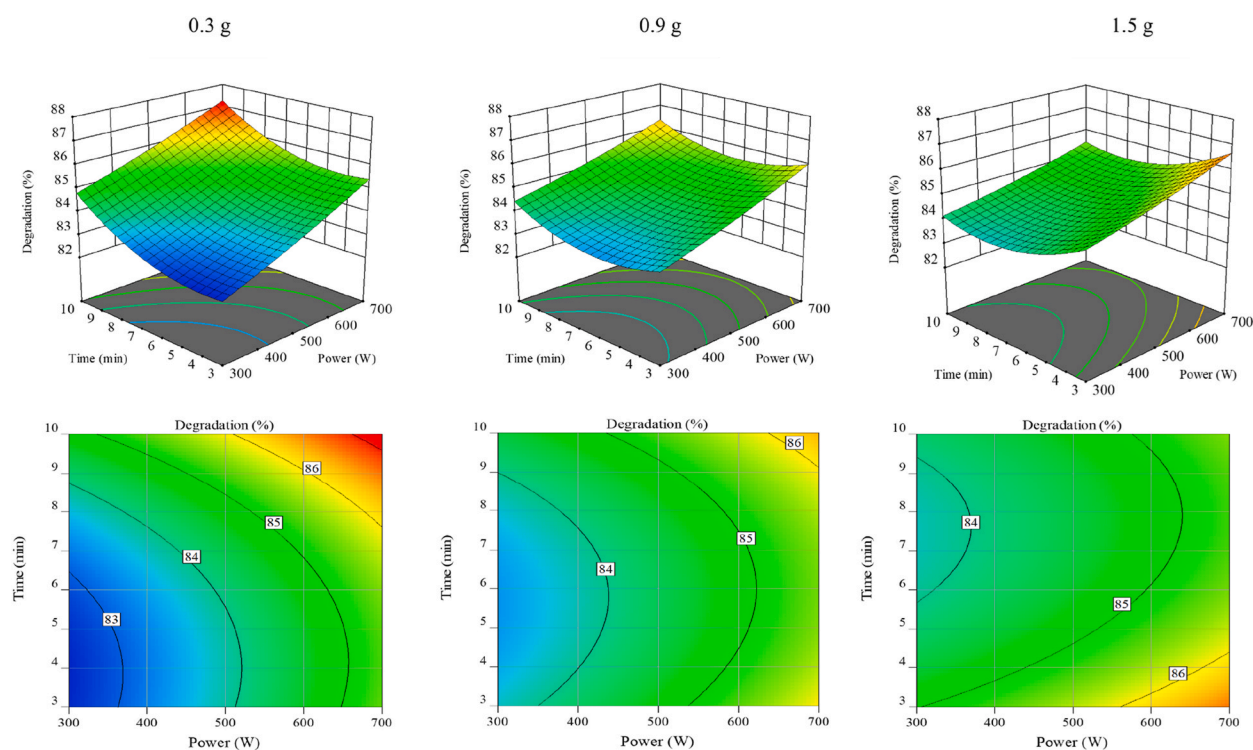


Fig. 4. Iso-response surfaces and contour diagrams of the interaction power\*time at different NaOH concentrations.

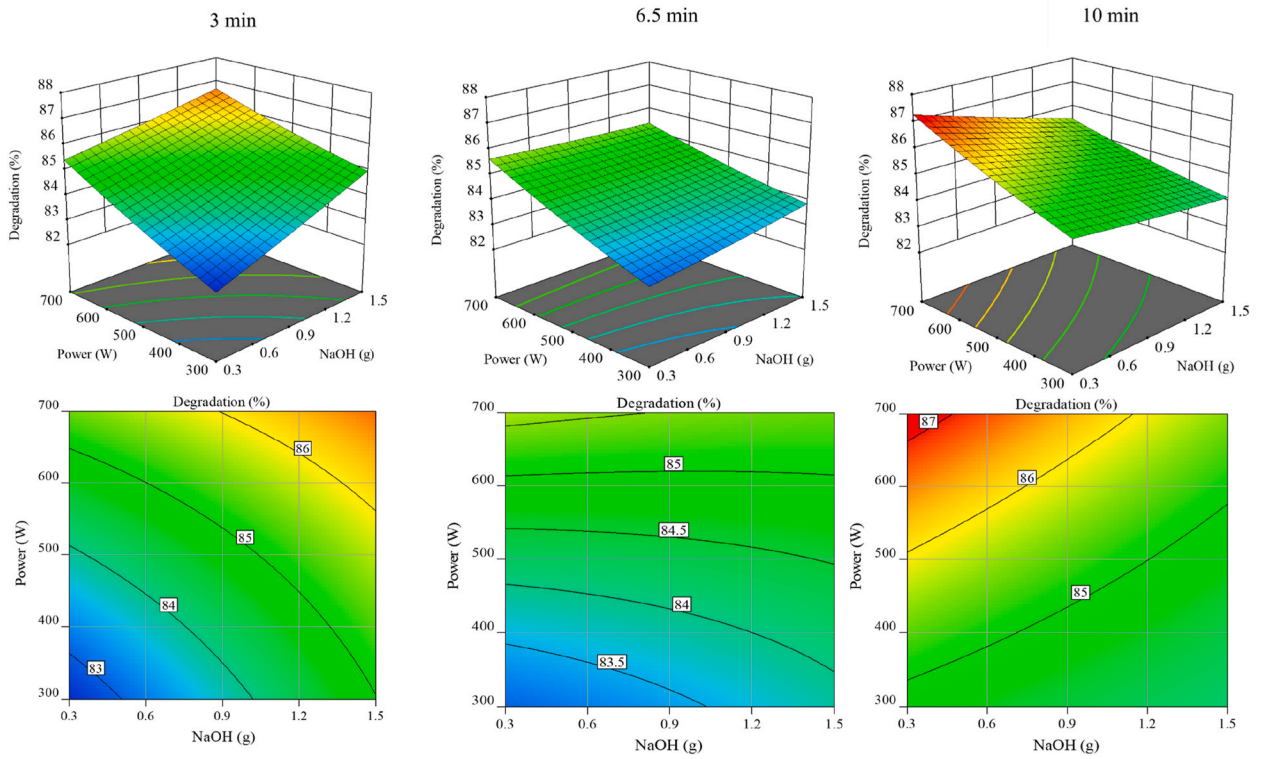


Fig. 5. Iso-response surfaces and contour diagrams of the interaction power\*NaOH at different times.

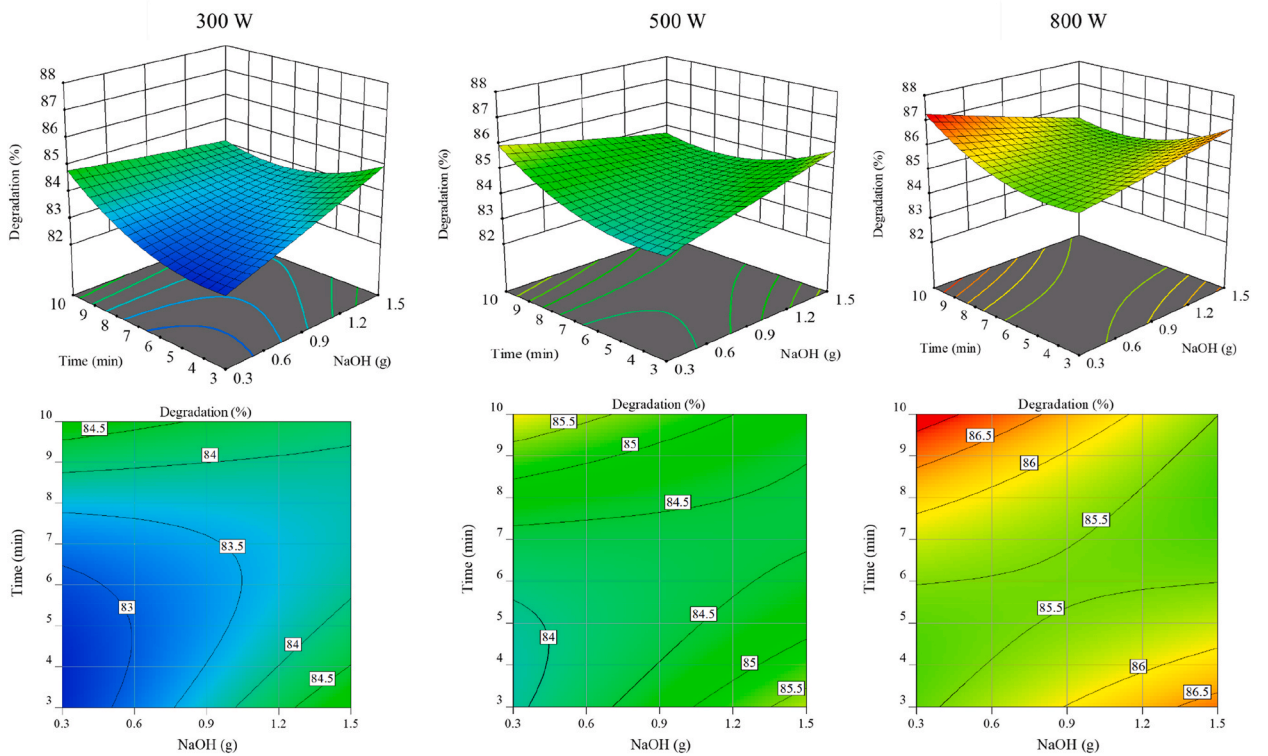


Fig. 6. Iso-response surfaces and contour diagrams of the NaOH\*time interaction at different powers.

PCBs is neutralised by NaOH; it is also assumed that NaOH could also serve as a hydrogen donor. The work by Kawahara and Michalakos [45] showed that no reaction occurs at temperatures below 250 °C in the absence of a base.

By analysing these results, optimal conditions can be determined to maximize the PCBs dechlorination rate using specific combinations of NaOH, microwave power, and irradiation time. This approach facilitates the optimisation of the dechlorination process in transformer oil, providing valuable insights for future experimental design and process refinement. The optimal conditions for PCBs dechlorination are 0.3 g of NaOH, 700 W of microwave power, and an irradiation time of 8 min. Under these conditions, the predicted PCBs degradation rate is 98.87 %.

### 3.3. Kinetics of microwave PCBs dechlorination

Table 4 shows the results of the kinetic study of the dechlorination of the different PCBs congeners. The reaction rate varies depending on the type of PCBs congener. Previous research has shown that removing chlorine from the para position at each phenyl ring is easier than from the ortho and meta positions. Furthermore, releasing the ortho position is generally more rapid than the meta position [46]. The overall quantity of chlorine atoms had an impact on the degradation reactions. As shown in Table 4, the largest range of dechlorination rates was between 0.282 and 0.588. The reaction time of the chlorine atom increases as the dechlorination rate constant increases, and tetrachlorobiphenyls have the highest kinetic constants. The principal factors influencing PCBs resistance to decomposition include the quantity and orientation of chlorine ions on the biphenyl rings and the symmetry and asymmetry of the molecules [47].

## 4. Conclusion

This study aimed to investigate through central composite design the dechlorination of PCBs-contaminated transformer oils using microwave catalytic pyrolysis ( $\text{Fe}^0$  and Glycerol) with power, NaOH, and irradiation time as factors and to study the kinetics of PCBs dechlorination. The transformer oil used had a PCBs concentration of 526 mg/kg, comprising 14 congeners. For a long irradiation time, at higher microwave powers, the reagents in the medium absorb the radiation, and the rate at which the reactor's temperature rises accelerates the breakdown of PCBs in the oil. Zero-valent iron and glycerol have little influence on degradation. The optimal conditions allowing maximum dechlorination (98.87 %) are 0.3 g of NaOH, a microwave power of 700 W, and an irradiation time of 8 min. The kinetic study demonstrated that microwave PCBs dechlorination reaction follows a first-order reaction. This approach is easy to use, inexpensive, secure, and an acceptable solution for existing PCBs removal techniques.

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## Ethical approval statement

Not applicable.

## Data availability statement

Data will be made available on request.

## CRedit authorship contribution statement

**Kom Regonne Raïssa:** Writing – original draft, Methodology, Conceptualization. **Kodami Badza:** Writing – original draft, Methodology, Conceptualization. **Nkenmogne Kamdem Ines Estelle:** Methodology. **Ngassoum Martin Benoit:** Writing – original

**Table 4**  
The rate constant of dechlorination kinetics of PCBs congeners.

Congeners	IUPAC Name	K ( $\text{min}^{-1}$ )	R <sup>2</sup>
PCB-77	3,3',4,4'- Tetrachlorobiphenyl	0.588	0.919
PCB-126	3,3',4,4',5- Pentachlorobiphenyl	0.343	0.441
PCB-101	2,2',4,5,5'- Pentachlorobiphenyl	0.491	0.956
PCB-105	2,3,3',4,4'- Pentachlorobiphenyl	0.468	0.969
PCB-118	2,3',4,4',5- Pentachlorobiphenyl	0.508	0.996
PCB-128	2,2',3,3',4,4'- Hexachlorobiphenyl	0.282	0.370
PCB-138	2,2',3,4,4',5'- Hexachlorobiphenyl	0.480	0.942
PCB-169	3,3',4,4',5,5'-Hexachlorobiphenyl	0.422	0.964
PCB-156	2,3,3',4,4',5- Hexachlorobiphenyl	0.465	0.973
PCB-153	2,2',4,4',5,5'- Hexachlorobiphenyl	0.407	0.968
PCB-170	2,2',3,3',4,4',5- Heptachlorobiphenyl	0.312	0.987



draft, Supervision, Conceptualization.

### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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