

# Dechlorination and decomposition of Aroclor 1242 in real waste transformer oil using a nucleophilic material with a modified domestic microwave oven

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**Abstract** This research was done to assess the dechlorination and decomposition of polychlorinated biphenyls (PCBs) in real waste transformer oil through a modified domestic microwave oven (MDMW). The influence of microwave power (200–1000 W), reaction time (30–600 s), polyethylene glycol (PEG) (1.5–7.5 g), iron powder (0.3–1.5 g), NaOH (0.3–1.5 g), and H<sub>2</sub>O (0.4–2 ml) were investigated on the decomposition efficiency of PCBs existing in real waste transformer oil with MDMW. Obtained data indicate that PEG and NaOH have the greatest influence on decomposition of PCBs; while, iron did not influence, and H<sub>2</sub>O decreased, the decomposition efficiency of PCBs. Experimental data also indicated that with the optimum amount of variables through a central composites design method (PEG = 5.34 g, NaOH = 1.17 g, Fe = 0.6 g, H<sub>2</sub>O = 0.8 ml and microwave power 800 W), 78 % of PCBs was degraded at a reaction time of about 6 min. In addition, the PCBs decomposition without using water increased up to 100 % in the reactor with the MDMW at 6 min. Accordingly, results showed that MDMW was a very efficient factor for PCBs decomposition from waste transformer oil. Also, using microwave irradiation, availability and inexpensive materials (PEG, NaOH), and iron suggest this method as a fast, effective, and cheap method for PCB decomposition of waste oils.

**Keywords** Dechlorination · Decomposition · Polychlorinated biphenyls · Polyethylene glycol

## Introduction

Polychlorinated biphenyls (PCB) are used as dielectric fluids and fire retardants; because they are thermally stable, have good dielectric properties and are found in capacitors, sealants, plastics, etc. [1]. PCBs are derivatives of the biphenyl compounds in which chlorine atoms have been substituted with one to ten hydrogen atoms [2].

These synthetic materials were produced in high quantities from 1929 to 1976 because of their excellent chemical properties (thermal stabilities and electrical insulation); they have been used for a wide diversity of usages, as transformers dielectric oil and as capacitors, sealants, plastics, etc. [3, 4].

PCBs have hazardous properties and wide-spread environmental contamination, therefore, their production and use have been banned in various countries [5, 6].

High commercial value and large quantity production of PCBs between 1929 and 1976 are the causes for them to be still in use in a variety of applications; this also has caused them to be collected and stored in waste storage sites [7, 8]. Therefore, contamination of operation sites is one of the increasingly critical environmental challenges. Traditionally, transformer oils mainly consisted of commercial PCB mixtures, with the “Aroclor” registered trade-mark which contained 60–70 % PCBs [9].

In section 3 of the Basel convention on March 17, 2004 by the United Nations Environment Program (UNEP), the low-contamination limit to PCBs was determined to be 50 ppm for transformer oils [10].

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Various techniques have been investigated for the destruction of PCBs from waste transformer oil, such as incineration, advanced oxidation process (AOP), base catalyzed decomposition (BCD), cement kiln co-incineration, gas phase chemical reduction (GPCR), molten salt oxidation (MSO), plasma arc decomposition, sodium reduction, enhanced photocatalysis, etc. [10].

These methods have several technical and economical disadvantages; for example, the incineration technology with controlled high temperature is a conventional approach and quite effective for decomposition of PCBs. However, there is widespread public objection to this technology due to the disposal costs and the exclusion of reusable materials, as well as highly toxic by-products such as dioxin via flue gas stream. Thus, now people prefer a non-combustion method for the treatment of PCBs-containing transformer oils [3, 7]. AOPs act as professional decomposers for recalcitrant pollutants; however, this technology is very complex and expensive to be designed and operated [11]. Due to these disadvantages, researchers have been motivated to investigate other techniques for the treatment of PCBs-containing transformer oils.

In recent years, it has been proven that chlorinated organic wastes can be destructed and dehalogenated by microwave irradiation [12]. Microwave (MW) radiations are electromagnetic waves with wavelengths shorter than one meter and larger than a millimeter, and a frequency of 300 MHz–300 GHz [13]. Dipolar polarization and space charge polarization in materials which are an absorber of MW will cause the polarization to rotate quickly, and this leads to apparent thermal energy, then the activation energy of chemical reactions will reduce and their chemical bonds will weaken [14, 15].

Important investigations have been done in this field by Liu and Yu who studied the combined effects of microwave and activated carbon on the remediation of polychlorinated biphenyl-contaminated soil [16]. They found that microwave irradiation, with the assistance of activated carbon, can accelerate the decomposition process of PCB29 in soil [16]; subsequently, Loan et al., found that with NaOH, 2-propanol as solvent, and a few types of transition metals as catalyst (Pd, Pt, Ni, Mo, Co, Fe) dechlorinated PCBs used microwave radiation in the optimum temperature of 130 °C. So, their results show that microwaves are a clean and effective technique for destruction of PCBs [17, 18]. Kastanek et al. studied dechlorination of PCBs by nucleophilic reactants with the presence of ionic liquids and using microwaves [19]. They found that in the presence of polyethylene glycol 300, KOH, and 1-butyl-3-methylimidazolium hexafluorophosphate as an ionic liquid, the destruction rates of PCBs were accelerated about ten to twenty times by microwave heating, as compared to conventional heating destruction [19]. An alkaline

environment was used as a reactant for PCBs decomposition in the above-reported investigations, in the chemical dechlorination process of PCBs from waste transformer oil [7], in the base-catalyzed destruction process developed by US EPA [10], as well as in the results of the studies which were summed up by Kastanek [20], etc. The alkaline environment has activating and neutralizing roles for the produced HCL in the dechlorination process [21].

Also transition metals, alkaline-earth metals, and precious metal catalysts can increase the hydrothermal dechlorination of chlorinated compounds [17, 22]. Other alternatives such as zero-valent iron would be useful and can replace the above-said catalysts, due to their potential toxicity and high cost. Ye et al. found that zero-valent iron, as an effective reactive material, increased dechlorination efficiencies of HCB [23]. Liu et al., also showed that in the microwave-hydrothermal process, zero-valent iron, as reactant for dechlorination of PCBs in the simulative transformer oil, increased decomposition efficiencies of PCBs [24]. Uchida et al. [25] have suggested that iron hydrothermal oxidation in an alkaline environment and temperatures above 423 K produce hydrogen gas; this might be used for dechlorination of PCBs.

The polyethylene glycol (PEG) in an alkaline environment, as a potent nucleophilic agent, can affect the removal of PCBs from transformer oil. For example, the potassium polyethylene glycol, which was produced via reaction between PEG and KOH, can attack the C–Cl bond at relatively low temperatures of about 60–120 °C [19, 20].

Our literature review indicated that the degradation of PCBs from real waste transformer oil by MDMW has not been reported using a combination of reactants (PEG1000, NaOH, zero-valent iron, and H<sub>2</sub>O).

In this investigation, microwave irradiation was used instead of conventional heating, due to its advantages such as lower energy consumption, high efficiency, shorter destruction time, smaller equipment, etc. [26]. We believe that, on the one hand, combining NaOH and PEG as nucleophilic reactant and microwave radiation absorbent can act as an effective factor in the PCBs destruction of transformers oil in a short time and at a temperature of 160 °C. On the other hand, production of hydrogen gas via hydrothermal reaction of Fe, NaOH, and H<sub>2</sub>O in the reactor, can be used for destructing PCBs. Zero-valent iron was used as a catalyst in the hydrothermal reaction, and NaOH and water were used as microwave absorbent.

The goal of this research was dechlorination and/or decomposition of PCBs from real waste transformer oil by using a modified domestic microwave oven (MDMW) at a laboratory scale, and then recycling and using them again. This method is simple, economical, accessible, and a desirable alternative in comparison to other PCBs destruction methods.

## Materials and methods

### Material

Waste transformer oil samples, contaminated with PCBs, were obtained from a waste oil storage site in Bandar Abbas, a town in the south of Iran. The waste oil contained 11 kinds of PCBs (Aroclor 1242) that included two, three, and four chlorinated PCBs (No's, 8, 28, 37, 44, 49, 50, 52, 66, 70, 74, and 77).

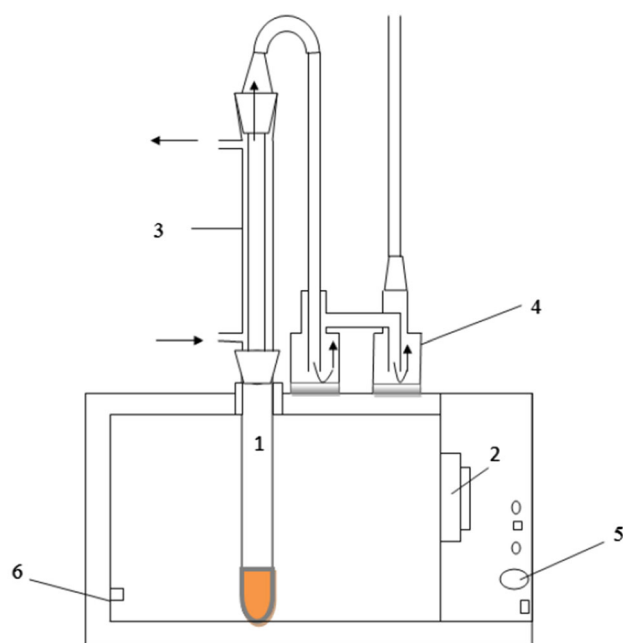
The concentration of Aroclor 1242, which was detected prior to the process, was 710 mg/l (containing 1.34 mg/l di-, 171.5 mg/l tri- and 537.5 mg/l tetra-chlorinated biphenyls).

Other required chemicals for the experiments including *n*-hexane, Polyethylene glycol (PEG1000), NaOH (with 97 % purity), and zero-valent iron powder were in analytical grade which were obtained from Merck.

### Method

Experiments with MW irradiation were performed in a modified domestic microwave stove with a cooling system (2450 MHz, 1000 W, Delongi Co).

A condensing system was installed above the reactor for preventing liquid evaporation (Fig. 1). We opened a hole with a 35 mm diameter above the stove from which a quartz batch reactor (30 mm, i.d) was inserted into the



**Fig. 1** Experimental set up Scheme: 1 quartz reactor, 2 microwave generator, 3 condenser, 4 vessels containing hexane, 5 control switch, 6 IR-temperature sensor

microwave stove. The space between the hole and reactor was filled with a Teflon ring. This reactor was connected to a condenser for collecting distillation, and so the produced gas could pass through two containers which were connected in series and containing *n*-hexane. The materials in the reactor cooled in 6 °C after the reaction ends, then they are mixed with *n*-hexane in bottles and PCB can be detected. All these experiments were operated in a batch reactor; the main operational variables were tested in treating waste transformer oil.

First, experimental design was carried out through the central composites design (CCD) method (Design—Expert 7 software). The operational parameters were used in the CCD following the irradiation time (360 s), microwave power levels (200–1000 W), polyethylene glycol (1.5–7.5 g), iron powder (0.3–1.5 g), NaOH (0.3–1.5 g), and H<sub>2</sub>O (0.4–2 ml) that were selected based on previous studies and our initial tests. The amount of waste oil used in all experiments was 10 ml. Afterward, the optimum amounts of reactants and microwave power were achieved using the CCD method at 360 s, then the reaction rates were investigated using the optimum amounts of reactants ranging from 0 to 600 s. At the end of the experiments, the dechlorination and decomposition mechanisms were investigated. In every stage, duplicate or sometimes triplicate samples were carried out and analyzed.

PCBs were analyzed using an AGILENT gas chromatograph which was equipped with an electron capture detector (model 6990 N) based on standard testing method D 4059, as reported in ASTM. Hexane was used for the dilution of organic phases (1/40).

The type of column was DB-5, capillary, diameter 0.32 mm, length 30 m, and film thickness 0.25 μm. The injector and detector temperatures were at 270 and 300 °C, respectively. The carrier gas was N<sub>2</sub> with a flow rate of 0.9 ml/min. The column temperature was programmed as following: initial temperature 120 °C; first temperature increase rate was 15 °C/min to 210 °C in an isotherm of 1 min, second increase rate: was 20 °C/min to 240 °C in an isotherm of 2 min and third: 30 °C/min to 295 °C in an isotherm of 2 min. The PCBs isotherm was obtained by injecting pre-determined concentrations to GC/ECD.

A VARIAN 4000 GC/MS was used for detection of the PCBs decomposition byproducts. DB-5 MS and helium were used as a capillary column and the carrier gas, respectively.

To confirm the decomposition of PCB, deionized water was used for diluting the precipitated solution, then the chloride concentration was detected by ion chromatography according to ASTM-D4327-11 as a reference standard.

To confirm the possibility of the hydrothermal reaction, acetone and deionized water were used to wash precipitated solid particles, then they were dried in open air;

finally, they were portrayed by an X-ray diffractometer (XRD) with Cu K $\alpha$  radiation.

## Results and discussions

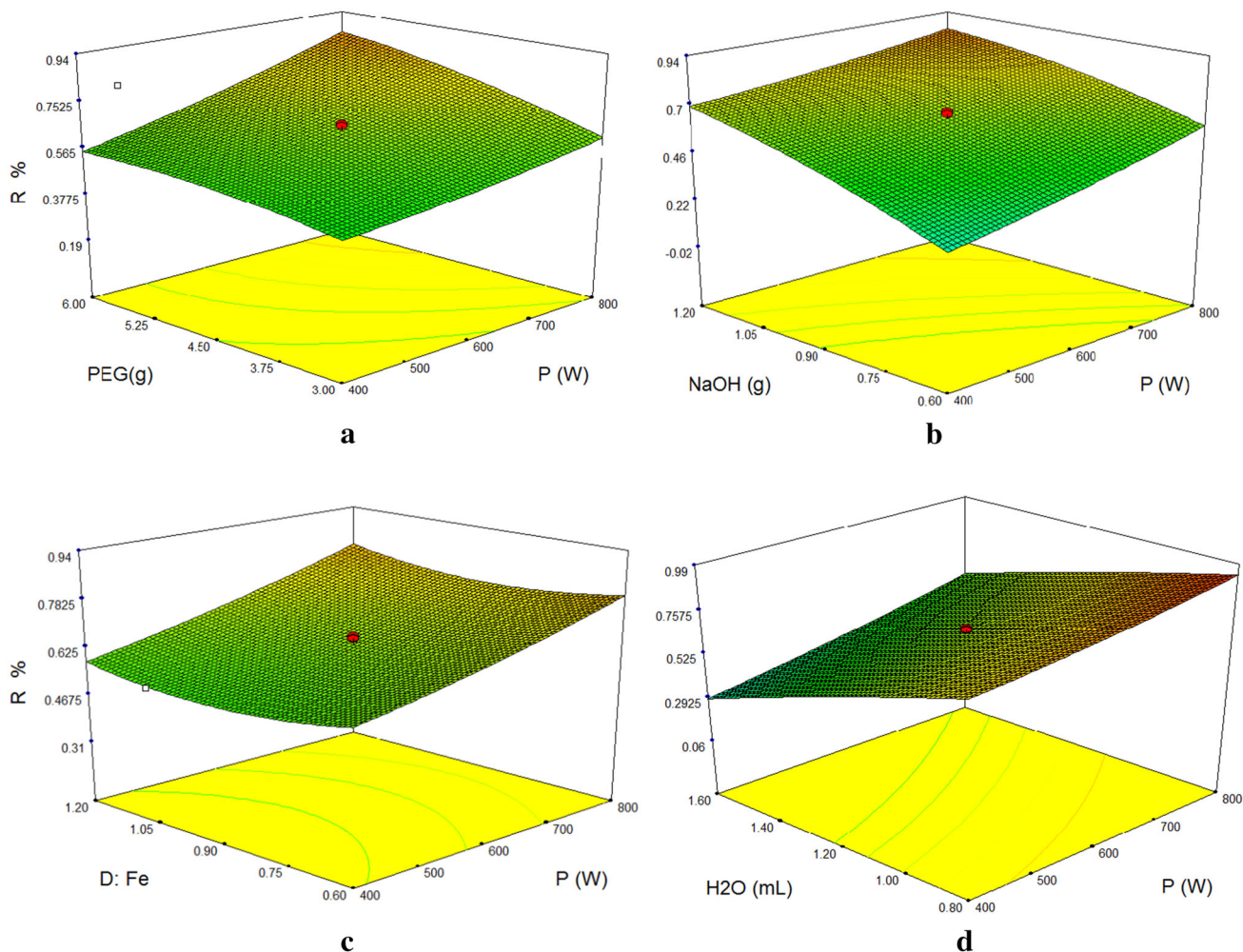
### Influence of microwave power

A very important variable in the dechlorination and decomposition of PCBs was microwave power; the temperature of the whole process and the radiation intensity of MW were directly related to the power levels [27, 28].

In the study, the MDMW power levels were 200, 400, 600, 800, and 1000 W and other parameters were as follows: 1.5–7.5 g polyethylene glycol, 0.3–1.5 g iron powder, 0.3–1.5 g NaOH, 0.4–2 ml H<sub>2</sub>O, 10 ml waste transformer oil and 360 s microwave irradiation.

The results obtained from experiments showed that the decomposition efficiency of PCBs in the whole amounts of

various reactants had increased by adding microwave power, as shown in Fig. 2. The recorded temperatures in the reactor (after the end of the microwave oven phase) specified that, in all runs, the reactor temperatures were almost about 90–110 °C. The boiling time of solution was shorter in higher powers in comparison to lower powers, and its boiling rate was more as well. The reactor temperature was kept between 90 and 110 °C due to evaporation and condensation of water, and then to return water to the reactor. The negative effect of water on the reactor temperature and PCB decomposition was confirmed by comparing two runs of reactions in the presence and absence of water, as shown in Table 1. By using the response surface method, the optimum amounts of variables were specified by an initial stage of experiments that were as follows: 800 W power, 5.34 g PEG, 1.17 g NaOH, 0.6 g Fe and 0.8 ml H<sub>2</sub>O. When the optimum amounts of variables were applied, almost 57 % of Aroclor 1242 degraded at a reaction time of 2 min; while in 10 min,

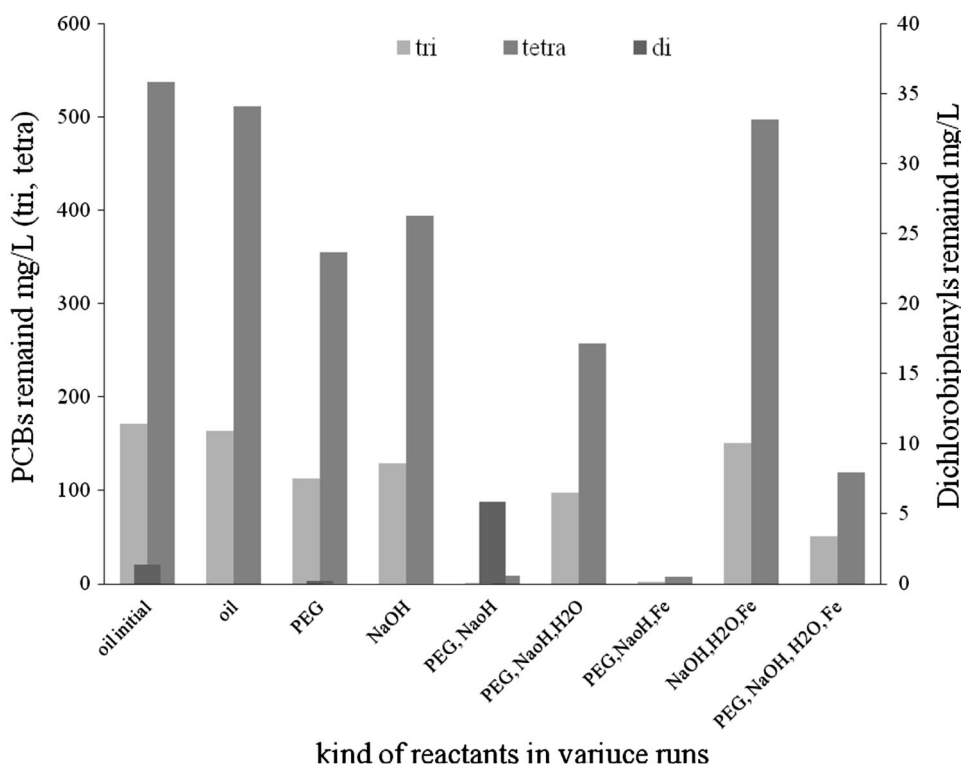


**Fig. 2** The effect of reactants and microwave power levels on decomposition efficiency of PCBs at 6 min [PEG (1.5–7.5) g, NaOH (0.3–1.5) g, Fe (0.3–1.5) g, H<sub>2</sub>O (0.4–2) ml, MW power (200–1000) W]

**Table 1** Effect of temperature and the kind of reactants on decomposition efficiency of special PCBs in the optimum amounts of reactants ( $P = 800$  W, PEG = 5.34 g, NaOH = 1.17 g, Fe = 0.6 g,  $H_2O = 0.8$  ml,  $T = 6$  min)

Runs	Participated variables in process	Decomposition efficiency %				$T$ (°C)
		Di	Tri	Tetra	Total PCBs or Aroclor 1242	
1	Oil	100	3.9	4	4	60–70
2	PEG 1000	100	34	34	34	120–160
3	NaOH	100	36	27	29	120–160
4	PEG + NaOH	100	100	98	98	120–160
5	PEG + NaOH + $H_2O$	100	43	52	50	90–110
6	PEG + NaOH + Fe	100	98	99	99	120–160
7	NaOH + $H_2O$ + Fe	100	12	8	9	90–110
8	PEG + NaOH + Fe + $H_2O$ (optimum amounts)	100	70	78	76	90–110

**Fig. 3** Decomposition changes of homological groups of PCB by MDMW (800 W) with optimum amounts of variables ( $P = 800$  W, PEG = 5.34 g, NaOH = 1.17 g, Fe = 0.6 g,  $H_2O = 0.8$  ml,  $T = 6$  min)



more than 78 % of them became degraded. These results are similar to other reports published by researchers, such as Kastanek and Liu [16, 19, 29].

**Influence of reactants dosages**

As mentioned, four reactants participated in the decomposition process of PCBs from real waste oil by MDMW. Reactants were added at five levels, and their influences were investigated by using the RSM method simultaneously. Also, their influences were investigated using the optimum amounts of reactants. Dechlorination and decomposition efficiency of PCBs increased by adding from 1.5 to 7.5 g of PEG1000 and 0.3 to 1.5 g of NaOH, but decreased by adding 0.4–2 ml  $H_2O$ . PCBs

decomposition efficiency did not change by adding zero-valent iron (from 0.3 to 1.5 g), as shown in Fig. 2. To prove the above results, influences of different kinds of variables on PCBs decomposition efficiency was investigated using the optimum amounts of reactants and 800 w power in 6 min; these results are shown in Fig. 3. The results again proved that PEG and NaOH were important reactants influencing the decomposition of PCBs, while iron and  $H_2O$  did not increase the decomposition efficiency;  $H_2O$  also decreased decomposition efficiency.

Decomposition efficiency and the process temperature in optimum amounts of reactants and 800 W power at 6 min are illustrated in Table 1. Results show that when a mixture of PEG and NaOH reactants was used, the maximum decomposition efficiency of PCBs was attained. The



decomposition efficiency in either runs [(PEG, NaOH) and (PEG, NaOH, Fe)] were 98 and 99 %, respectively. One of the most important reasons for increasing efficiency of decomposition in the above runs could be raising the temperature to 160 °C, another important reason for increasing efficiency of decomposition in the above runs might be decreasing the negative effect of water on activation of nucleophile (PEG) [30]; while in other runs, H<sub>2</sub>O evaporation and condensation keep the reactor temperature between 90 and 110 °C. These results are similar to reports published by other researchers such as Kastanek et al. [18, 19, 31].

#### Investigation of specific PCBs decomposition rate

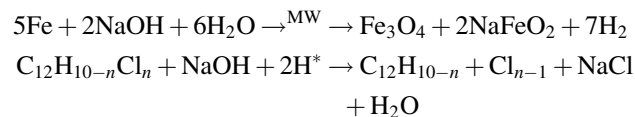
The decomposition rate of different kinds of PCBs was investigated in the optimum amounts of variables at 20, 40, 60, 90, 120, 240, 360 and 600 s. Figure 4 shows the remaining concentrations of PCBs at the end of the process in the optimum condition, at different intervals. The results showed that more than half of the total PCBs were destructed at the initial 2 min of reaction, and as the time passed, the decomposition rate decreased. Also, the results indicated that the tetra chlorinated biphenyls decomposition rate was faster than tri chlorinated biphenyls after 10 min, but due to a high concentration of PCBs and a high rate of decomposition, tetra- and tri-chlorinated biphenyls decomposition occurred non-selectively in the first 2 min; as the results show decomposition efficiencies of tetra- and

tri-chlorinated biphenyls in 90 s were 48 and 59 %, and in 600 s were 80 and 74 %, respectively, as shown in Fig. 5.

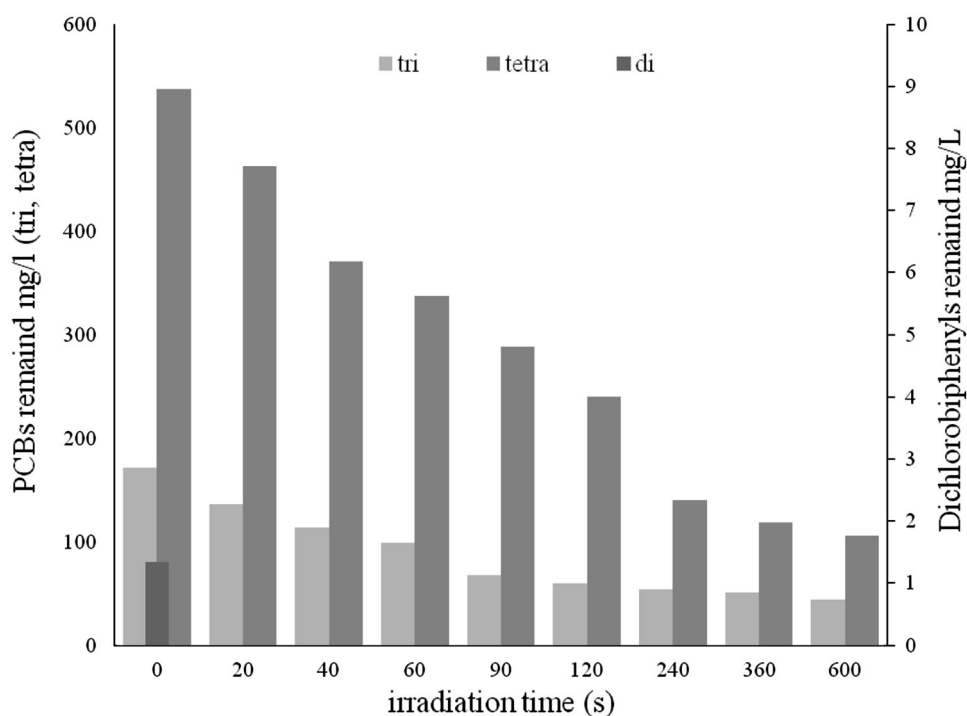
The investigation of Aroclor 1242 decomposition during the reactions indicated that the destruction kinetic was fitted with the apparent second -order kinetic,  $k = 2e^{-6}$  (Fig. 6). Nevertheless, other studies have confirmed that PCBs with low chlorine decompose harder in comparison to PCBs with high chlorine, by conventional heating systems [32].

#### Investigation of dechlorination and decomposition mechanisms

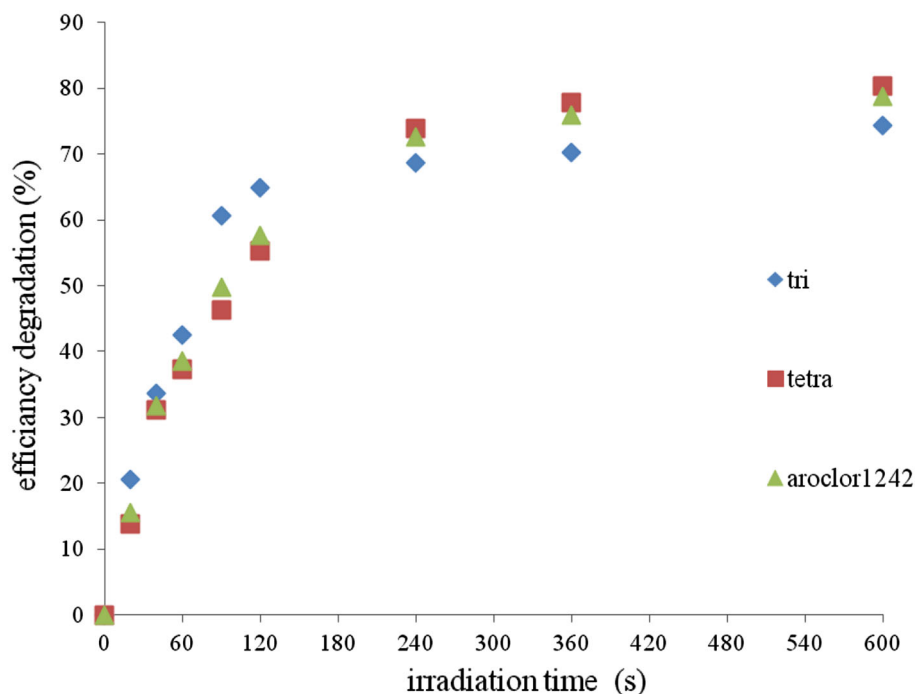
We expected that two effective mechanisms occur in reactor for PCBs decomposition. The first mechanism was the reaction between PEG and NaOH, in which PEG (as a potent nucleophilic agent and MW absorbent) and NaOH (as activator and MW absorbent) attack the C–Cl bond and/or the positive charge generated in the biphenyl ring under MDMW [7, 33]. The second mechanism was composed of the hydrothermal reaction of NaOH, iron powder, and H<sub>2</sub>O (under microwave irradiation); the possible resulting hydrogen of this process participated in PCB dechlorination of real waste transformer oil [21, 24]. The dechlorination hydrothermal reaction might be as in the following:



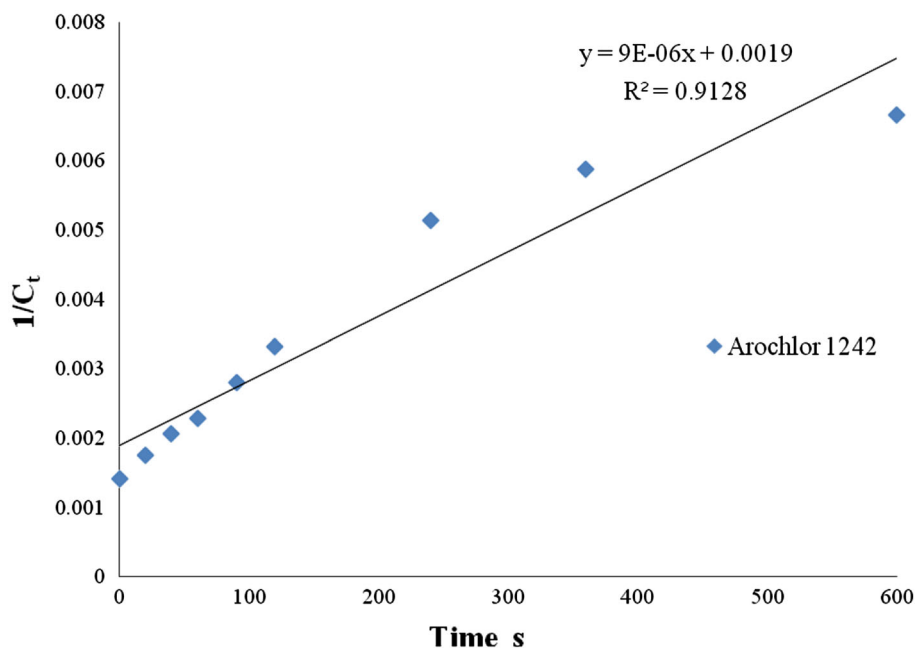
**Fig. 4** Special PCBs concentration remained at the end of the process with optimum amounts of variables at various times ( $P = 800$  W, PEG = 5.34 g, NaOH = 1.17 g, Fe = 0.6 g, H<sub>2</sub>O = 0.8 ml)



**Fig. 5** Decomposition efficiency of special PCBs and total Aroclor 1242 by MDMW ( $P = 800$  W, PEG = 5.34 g, NaOH = 1.17 g, Fe = 0.6 g,  $H_2O = 0.8$  ml,  $T = 6$  min)



**Fig. 6** The decomposition kinetic of Aroclor 1242 in the optimal amount of variables by MW (PEG = 5.34 g, NaOH = 1.17 g, Fe = 0.6 g,  $H_2O = 0.8$  ml)



The results specified that, with combining Fe, NaOH, and  $H_2O$ , at most 10 % of PCBs decomposed. Whereas, this was 29 % when only NaOH was used in the waste oil. Therefore, it is highly possible that a hydrothermal reaction did not occur at all.

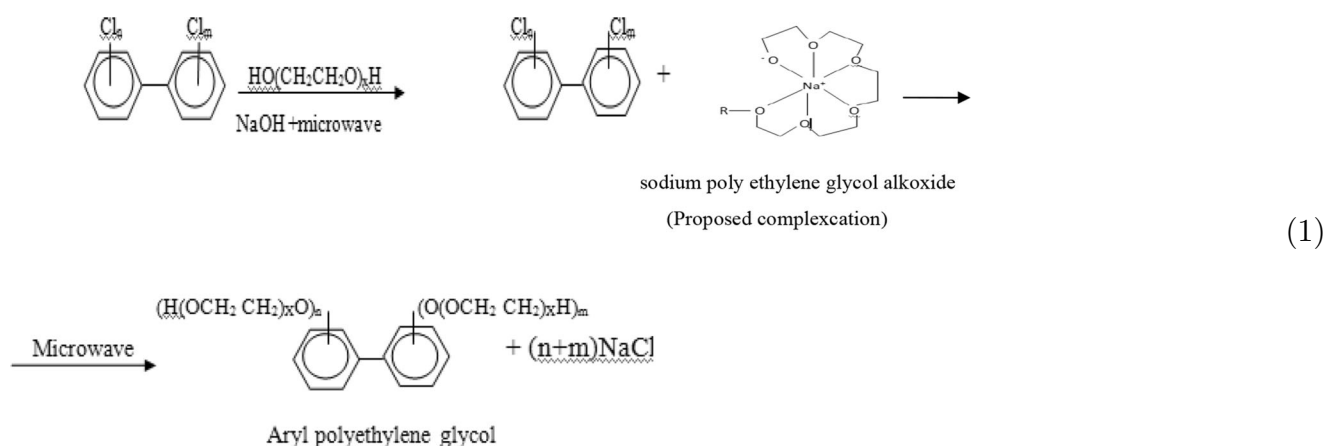
Hydrothermal oxidation of iron powder in an alkaline environment and producing hydrogen gas at temperatures above 300 °C was reported in other studies [34]; but, due to

low temperature in the reactor (110 °C), occurrence of this mechanism is impossible.

Also, iron powder did not change after the reaction, because the X-ray diffraction pattern of solid material which remained in the bottom of reactor confirms that  $Fe_3O_4$  and  $2NaFeO_2$  were not produced in the process as per the above equations.

But NaOH and PEG had distinctly influenced PCBs decomposition. In fact, the first mechanism was the main

operative decomposition of PCBs from real waste transformer oil. With adding PEG in the presence of NaOH, sodium poly ethylene glycol alkoxide was generated which can transfer itself into the organic phase, via self-complexation and, as a potent nucleophile, can attack the positive charge carbon in PCBs. Thereby, the chlorine ions were neutralized and, consequently, the PEG activation for attack to the positive charged carbons was raised and PCBs removal efficiency was increased (Eq. 1). The increase of dichlorinated biphenyls concentration in PEG + NaOH run confirmed that a potent nucleophilic agent attacked the C–Cl bond of PCBs in the process, as shown in Table 1. The positive effects of PEG and alkali on PCBs decomposition are confirmed in reports published by other researchers, such as Kastianke et al. [19, 35, 36]. The suggested dechlorination pathways of PCBs are as follows:



The chromatograms, attained from GC–MS of treated oil, did not show biphenyl carbon. Therefore, it is speculated that the PCB rings have been fractured, and, consequently, chlorine ions have been separated from rings. The chart in Fig. 7 illustrates the chlorine balance mass in the process. Results showed that the formation of chlorine ions during the reaction was almost equal to the decomposed chlorinated organic biphenyls. Therefore, it can be realized from the chlorine balance mass that the decomposition of PCBs in the process was via dechlorination and not through evaporation.

In the end of the reaction, aryl polyethylene glycol was likely produced and precipitated with PEG in the bottom of the reactor. The investigations carried out by EPA with conventional heating in PCBs dechlorination showed that the produced mixture of aryl polyethylene glycol and PEG at the end of the reaction was neither toxic nor mutagenic for various animals [35]. The dioxin produced from PCBs decomposition during reactions should probably be low, because the reactor had a reduction environment and, also, the reaction temperature was lower than 160 °C. The chromatographic analysis did not detect toxic chlorinated compounds; previous studies did not confirm dioxins and toxic compounds in the presence of PEG + NaOH [37]. But research is still open for the possible generating of dangerous intermediates from the reactions.

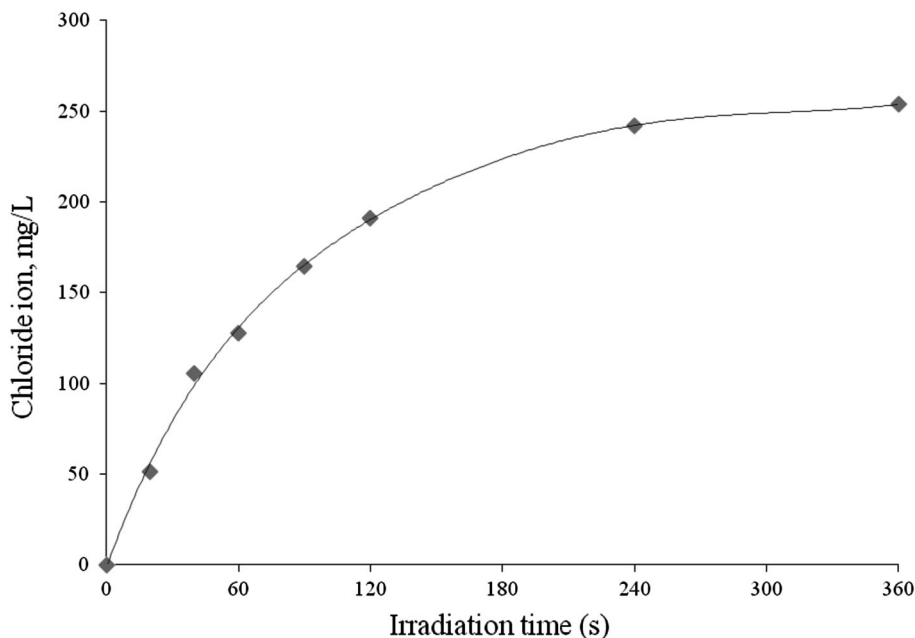
Finally, this method can be considered as a convenient and economical way for its low cost, accessibility of its material, high efficiency, and high speed in PCBs removal from waste oil in site.

## Conclusion

In this study, the MDMW radiation and various kinds of reagents, namely PEG, NaOH, iron powder, and H<sub>2</sub>O were used for PCBs dechlorination and decomposition from real



**Fig. 7** Produced chloride ions from decomposition of Aroclor 1242 by MDMW in the end of process at various times



waste transformer oil. The MDMW radiation and quantities of PEG and NaOH reactants were main factors influencing decomposition efficiency of PCBs.

Iron powder and H<sub>2</sub>O reactants did not increase the decomposition efficiency; while, H<sub>2</sub>O decreased decomposition efficiency.

The materials used are available and cheap, and, by MDMW decomposition, can be completed in very short time. In future work, this method might be noteworthy for the treatment of POPs-containing wastes.

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