

# Destruction and Dechlorination of Aroclor1254 in Real Waste Transformer Oil Using Microwave Irradiation, Microwave Absorbent and Reactive Materials

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## ABSTRACT

**Background:** In this research, the decomposition of Aroclor 1254, including 20 kinds of polychlorinated biphenyls (PCBs), in real waste transformer oil in the presence of PEG1000, NaOH, zero valent iron and H<sub>2</sub>O by microwave (MW) irradiation was studied.

**Methods:** Central composites design (CCD) method was carried out for experimental design. The effects of microwave power levels (200- 1000 W), reaction time (30-600 s), polyethylene glycol (PEG) (1.5-7.5 g), zero valent iron powder (0.3-1.5 g), NaOH (0.3-1.5 g) and H<sub>2</sub>O (0.4-2 ml) were surveyed on the decomposition efficiency of PCBs.

**Results:** The results specified that polyethylene glycol and NaOH, as reactants, greatly influenced the decomposition of PCBs. Nevertheless, zero valent iron had no effect, and H<sub>2</sub>O decreased the decomposition efficiency of PCBs. Furthermore, experimental results showed that at the optimum amounts of variables (PEG = 5.34g, NaOH= 1.17g, Fe= 0.6g, H<sub>2</sub>O= 0.8 ml, microwave power 800 w), 93% of PCBs were decomposed over the reaction duration of 6 min. Therefore, PCBs degradation efficiency without water rose up to 99.99% under the MW at 6 min.

**Conclusion:** The results indicated that MW, PEG and NaOH were important variables on PCBs degradation from real waste transformer oil. It is therefore suggested that microwave radiation with the assistance of PEG and alkali can be introduced as a potential technology for PCBs removal from real waste PCB-contaminated oil.

**Keywords:** Aroclors, Microwaves, Polychlorinated Biphenyls, Polyethylene Glycols.

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## INTRODUCTION

The wide use of polychlorinated biphenyls (PCBs) in various industrial applications, for their specific properties such as chemical and thermal stability and resistance to both microbial and photochemical degradation, have made them great risks to the environment [1,2].

For several decades (1920s– 1970s), PCBs were extensively employed in a wide range of industrial applications such as: capacitors' dielectrics, transformers' oils, hydrolytic liquids in hydraulic systems and machines, and heat exchange fluids [3, 4].

Due to the environmental problems attributed to the use of PCBs, almost all producers decreased or suppressed their productions of PCBs in the 1970s [5].

Stockholm convention in May 2004, prohibited using PCBs containing products by 2025 and also made it mandatory to eliminate all PCBs containing wastes by 2028 (Kim, Hirai et al. 2004; Kim, Son et al. 2010) [6,7]. However, because of their advantages and utility in various conditions, high quantities of PCB-containing transformer oils are still being employed or stored [1,8]. Therefore, it is necessary to decompose PCBs from transformer oils. Commercially usable destruction technologies for the liquid form of PCB wastes (usually as waste transformer oil) are as follows: incinerations, chemical decomposition (CD), advanced oxidation process (AOP), hydrothermal oxidation degradation, reduction and thermo-chemistry degradation, photo-disintegration, plasma degradation, etc [1, 9-11].

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Incineration is the conventional method for PCB destruction and removal from waste transformer oil, but with the possibility to produce dioxin and non-recycled waste oil [1, 12]

Some chemical destruction processes have been presented as simple, safe and economical methods. For example, in KPOG method, KOH as an activator and PEG as catalyst, produce complexes with potent nucleophilic qualities under conventional heating at mild temperatures (60-120 °C). But long reaction time and higher consumption of reactants are their [10, 13].

AOP produces highly reactive chemical oxidizers which in turn increase the decomposition rate of persistent organic pollutants (POPs). The AOP technology is merely usable for aqueous environments and cannot be employed for organic solids and liquids decomposition. Furthermore, AOP technology is complex needs specialized personnel and poses high-potential risks [9, 12]. All the above technologies and their alternatives have disadvantages that limit their application when compared to incinerations (Duffy, Anderson et al. 2000; Liu, Zhao et al. 2006) [14].

It was in the previous decade that microwave (MV) electro-magnetic radiation was introduced for decomposition of halogenated organic contaminants. Xitao et al, Raquel et al, Lee et al, Calinescu et al and other researchers have reported that MW decomposed chlorinated organic materials [15-17]. The main theme of the present study is also based on the application of MW instead of conventional heating as a reaction energy source.

Previous studies confirmed that nucleophile agents such as polyethylene glycol (PEG), in alkaline environment, greatly influence the decomposition of PCBs [10, 13, 18]. It is therefore believed that the reaction rate of PCB decomposition can be accelerated using nucleophile PEG and alkali under MW heating, and this can take the place of conventional heating. On the other hand, hydrogen production is proven by iron oxidation in Fe+NaOH+H<sub>2</sub>O system at temperatures above 423K [19] which could be used for the decomposition of PCBs, particularly if the process was to be carried out by MW irradiation [1]. In order to overcome the shortcomings of the previous studies, in the

present study a modified domestic microwave system was applied for heating, decreasing reaction duration, increasing molecular vibration and increasing reaction rate. Also, in order to decompose PCBs in real waste transformer oil, we used a potent nucleophile material, PEG1000 as an activator of chlorinated compounds, NaOH, and also of Fe+NaOH+H<sub>2</sub>O system for PCB direct decomposition under MW, simultaneously. In the experiment, PEG, NaOH and H<sub>2</sub>O were MW absorbents. We waited, produced complex of PEG in the present NaOH, as a nucleophile material attack to C-CL bond of PCBs and decompose them, and also the produced hydrogen of the Fe+NaOH+H<sub>2</sub>O system substitutes chlorine ions in biphenyl rings and decomposes PCBs simultaneously (Ryoo, Byun et al. 2007; Ukisu 2008; Kastanek, Kastanek et al. 2011) [13, 20, 21].

From a practical, economic and ease of recycling point of view, PCB direct decomposition and removal from waste transformer oil is a suitable disposal method (Asilian, Gholamnia et al. 2010) [22, 23].

The main purpose of this study was to investigate the efficiency of PCB decomposition in the real waste transformer oil by microwave radiation, and its recycling and reuse after the process at a laboratory scale. Researchers of current work believe that this method is simple, safe, economic, available and an appropriate alternative to other PCB destruction methods. It can also be appropriate for PCB decomposition and dechlorination from real waste oil on storage sites in developing countries as Iran.

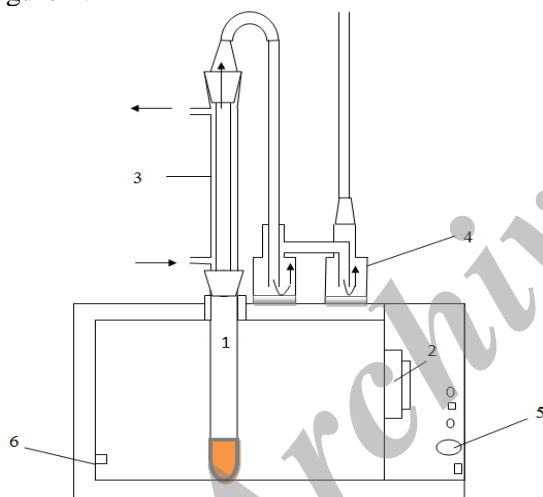
## MATERIALS AND METHODS

This experimental study was carried out in laboratory scale. PCBs containing waste transformer oil were supplied from a waste oil storage site in Hormozgan province in Iran. The waste oil contained 20 kinds of PCBs (Aroclor 1254) that included four, five and six chlorinated PCBs (No's 44, 49, 52, 66, 70, 74, 77, 82, 87, 99, 105, 114, 118, 126, 128, 138, 153, 156, 158 and 169). The detected concentration of aroclor1254 prior to the process was 4448 mg /L. All required materials were purchased from Merck with analytical grade.

In order to decompose PCBs in real waste transformer oil, we used a potent nucleophilic material (PEG1000) as an activator of

chlorinated compounds, NaOH, and Fe+NaOH+H<sub>2</sub>O system for direct decomposition of PCB under MW, simultaneously. In the experiment, PEG, NaOH and H<sub>2</sub>O were MW absorbents. After complexes of PEG in the presence of NaOH, as a nucleophilic material attack C-CL bond of PCBs and decompose them, the hydrogen produced by the Fe+NaOH+H<sub>2</sub>O system substitutes chlorine ions in biphenyl rings and decomposes PCBs simultaneously [13, 20, 21].

In this investigation, the experimental apparatus was a 1000w conventional microwave stove which was used to produce microwave irradiation; it had also been renovated by opening a 35 mm hole on the overhead of stove. The quartz reactor (30 mm i.d) was located into the microwave stove. The reactor outside was connected to a condenser system for collecting distillation and evolved gases passed via two series of vessels containing hexane as shown in Figure 1.



**Figure 1.** schematic of experimental set up; 1. Quartz reactor ; 2. lamp the microwave; 3. Condenser; 4. Bottles containing hexane; 5. canterol switch; 6. IR-temperature censor.

At the end of the reaction, the materials in the reactor were rapidly cooled to 6 °C. The oil phase was separated, and then the oil, the distilled vessels and the condenser washing solution (n-hexane) were mixed and analyzed in combination. Iron powder and PEG were precipitated and separated at 4 °C.

Central composites design (CCD) method was carried out for experimental design (Design-Expert 7 software and response surface

methods (RSM)). 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), with irradiation time of 360 s and microwave power levels of 200- 1000 w were used in Central composites design (CCD) method. These settings selected based on former studies and our initial tests. In all tests, the amount of waste oil used was 10 ml. The optimal mount of variables were obtained by the CCD method in 6 min. The PCB destruction rate was achieved by its decomposition in optimal condition and microwave power of 800w at durations of 0 to 600 s. Also the individual and combined influences of reactants on PCBs destruction were investigated.

Hexane was employed for dilution of organic phases (1/40), and the standard testing method (D 4059) used for analysis of aroclor1254 was gas chromatograph model 6990N AGILENT equipped with an electron capture detector (GC/ECD). The type of column was DB-5, diameter 0.32mm, capillary, length 30m and film thickness 0.25µm. The injector and detector temperatures were programmed at 270 and 300 °C, respectively.

Nitrogen was infused with a flow rate of 0.9 ml/min. The column temperatures were programmed as follows: initial temperature 120 °C, first increase rate of temperatures: 15°C/ min to 210 °C in isotherm 1 min, second increase rate of temperatures: 20°C/min to 240 °C in isotherm 2 min, and third: 30°C/ min to 295 °C in isotherm 2 min. The isotherms of PCBs were achieved via injection of their determined concentrations to GC/ECD and regarding to the same responses.

A VARIAN 4000 GC/MS was used for specifying byproducts. DB-5 MS was capillary column and helium was the carrier gas in GC/MS. N-hexan and deionized water were applied for washing separated solid particles, and then they were dried in ambient environment and were portrayed by X-ray diffractometer (XRD) with Cu K $\alpha$  radiation.

## RESULTS

### *Influence of Microwave Power*

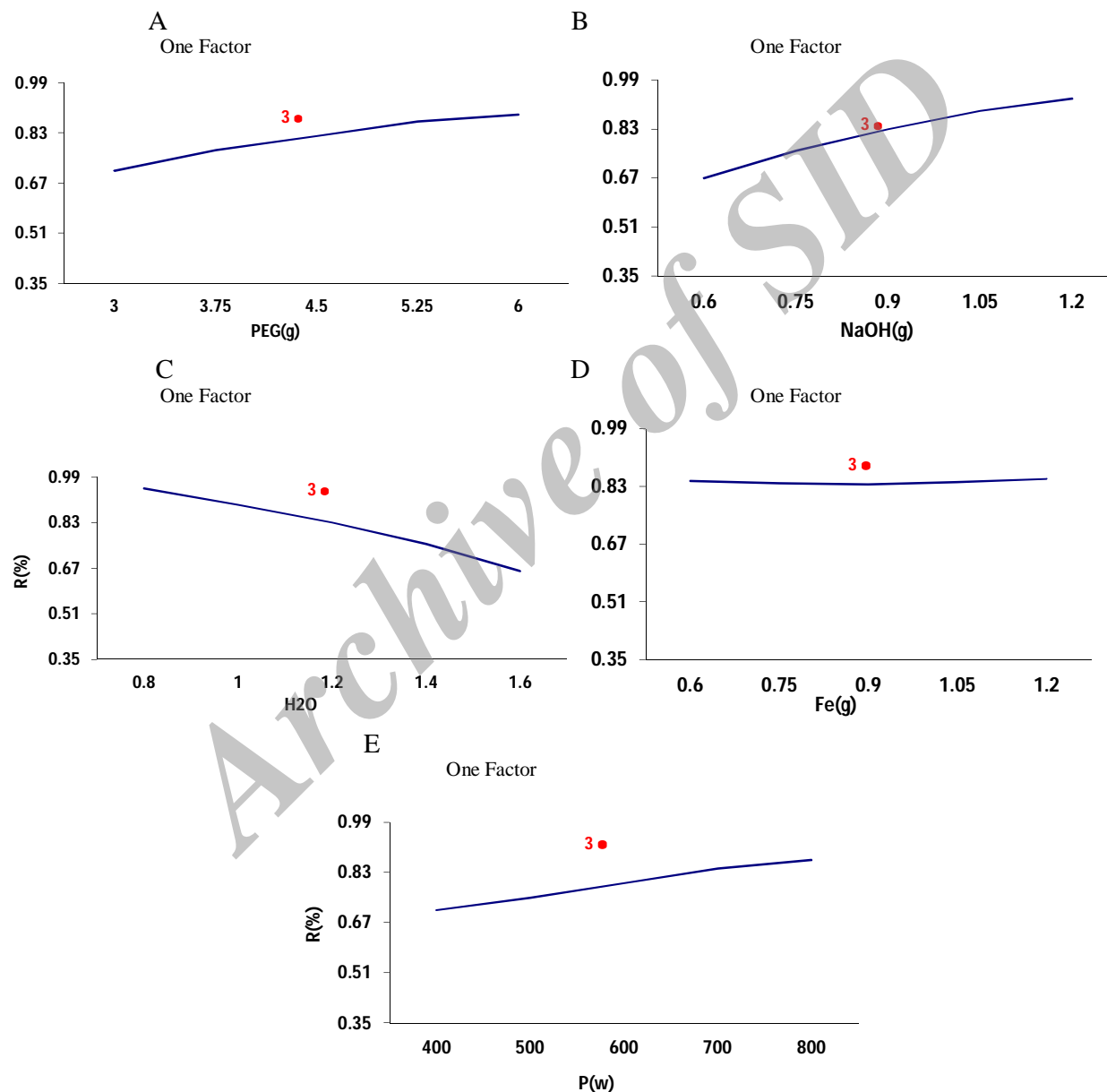
In this investigation, the four reactants PEG1000, NaOH, zero-valent iron and H<sub>2</sub>O affected decomposition efficiency of PCBs. The MW power levels were 200, 400, 600, 800, 1000 w, and other reactants range were as follows:

1.5-7.5 g PEG1000, 0.3-1.5 g Fe, 0.3-1.5 g NaOH, 0.4-2 ml H<sub>2</sub>O. The amount of waste transformer oil was 10 ml in all experiments. In all courses, the reaction temperature of reactor's outer wall in the end of the process was about 90 to 110°C.

Through RSM, the optimum quantities of factors were determined by primary stage of experiments which were as follows: 800 w power, 5.34g PEG, 1.17g NaOH, 0.6 Fe, and 0.8ml H<sub>2</sub>O.

The results attained from the tests showed that in all conditions, the decomposition efficiency of PCBs was increased by enhancing microwave power levels (from 200 to 1000w), as shown in Figure 2. In lower powers, the boiling times of materials were higher, and the boiling rate was lower.

The evaporation and condensation of water, and then its return to reactor caused the temperature of the reactor to be kept in the range of 90 to 110 °C.



**Figure 2.** The influence of microwave power levels and reactants amounts on the PCBs destruction efficiency at time 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, P=(200-800)w).

### Influence of Reactants Dosages

In this study, five levels of reactants, PEG, NaOH, zero valent iron and H<sub>2</sub>O, were selected and added to 10 ml of waste oil; their influences were determined via response surface method simultaneously (Figure 2). It was clearly seen that adding PEG (1.5 to 7.5g) and NaOH (0.3 to 1.5g) effectively raised the decomposition efficiency of PCBs, but adding H<sub>2</sub>O (0.4 to 2ml) obviously decreased PCBs decomposition efficiency. Contrary to this, adding zero-valent iron (0.3 to 1.5g) almost did not affect the PCBs decomposition efficiency, as shown in Figure 2.

When the optimum quantities of factors were used, 93.1% of aroclor1254 decomposed in 2 min; while after 10 min more than 97.5% was decomposed (Figure 3)

Additionally, their influences were determined with the optimum amounts of reactants and microwave power of 800 w in 6 min (Figure 4). The results displayed in Figure 4 shows the influence of variables on PCB decomposition efficiency in the optimum runs after 6 min and confirms the above findings. Furthermore, in table 1, the process temperature and degradation efficiencies with the optimum amounts of reactants and 800w power after 6 min are illustrated.

The PCBs decomposition rate was investigated using the optimum amounts of factors in time intervals of 20, 40, 60, 90, 120, 240, 360 and 600 s (Figure 5). The results showed that more than 70% of total PCBs were decomposed after 40 s, and also the decomposition rate decreased in longer durations of 60 s.

The study of reactants optimum quantities influences (PEG, NaOH, zero valent iron and

H<sub>2</sub>O) on PCBs degradation showed the maximum decomposition efficiencies of PCBs were related to both runs (PEG, NaOH) and (PEG, NaOH, Fe), 99.95% and 99.99%, respectively. Water had a negative effect on the reactor temperature and also had a negative effect on activation of PEG. By comparing the data of the two courses of reactions in the presence and absence of water, negative influence of water was affirmed. Kastanek and Liu attained similar results in their researches [1, 13, 17].

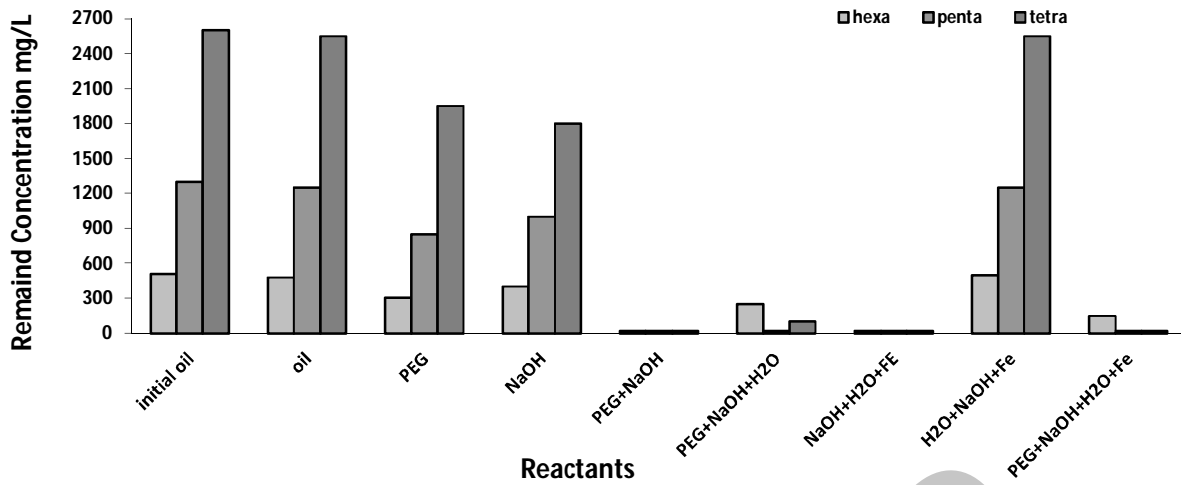
The absence of water and also the increase in temperature of reaction in the two above courses to 160°C can be important reasons for efficiency enhancement; while in other courses, the maximum temperature of reactions was 110°C. The influence of high temperature on decomposition efficiency of PCBs have already been confirmed in previous studies [1].

The analysis of aroclor 1254 decomposition during the process indicated that the degradation kinetic was fitted with the apparent one-order kinetics,  $k = 0.0192$  (Figure 6). Moreover, the results illustrated that higher chlorinated biphenyls were faster decomposed than the lower chlorinated biphenyls in all courses.

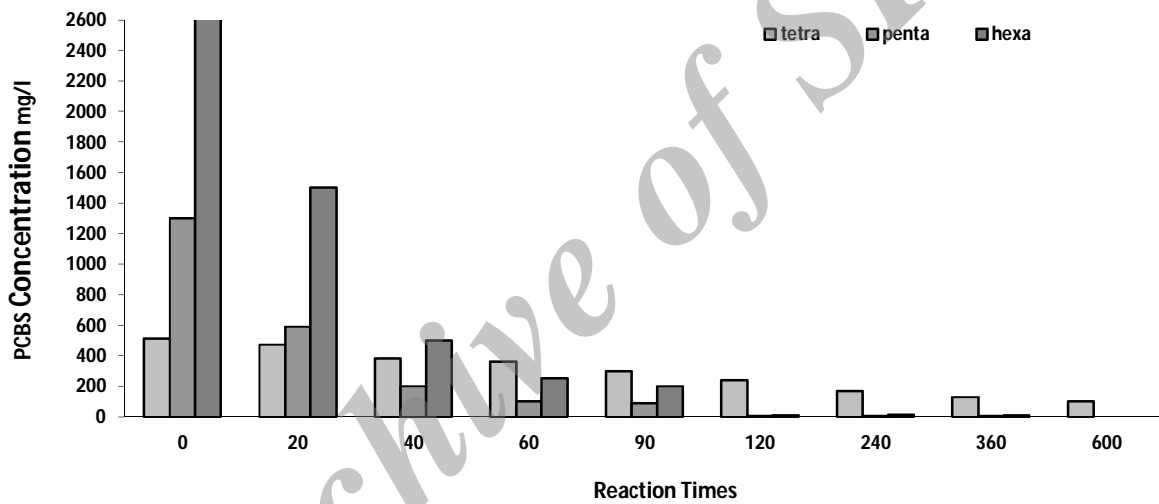
The decomposition rates were as follows hexa > hepta > tetra, and also the decomposition efficiencies of tetra-, penta- and hexa-chlorinated biphenyls after 120 s were 74% , 96% and 97% and after 600 s were 80% , 99.9%, and 99.9% respectively (Figure 5). Also, to confirm PCBs degradation, chloride concentration was measured in residual wastes of the reactions and the chlorine balance mass was determined (Figure 7).

**Table.1.** The influence of the temperature and variables kinds on PCBs decomposition efficiency in the optimal amounts of reactants(PEG=5.34g, NaOH= 1.17g, Fe= 0.6g, H<sub>2</sub>O=0.8ml, P=800w, T=6min).

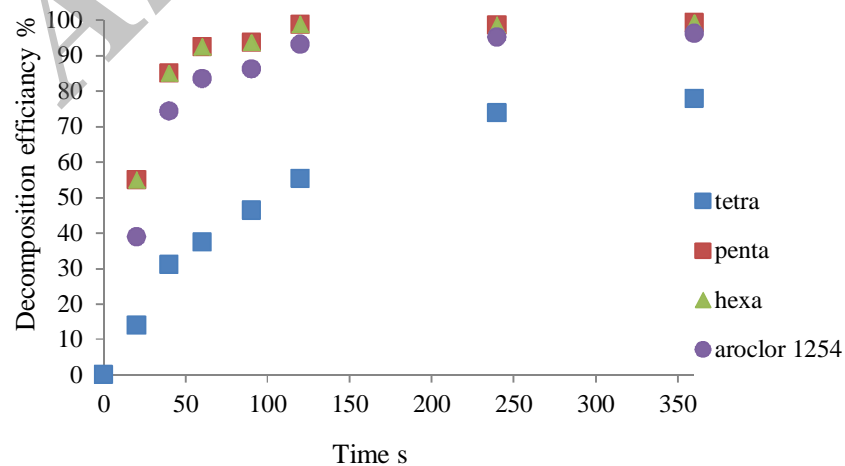
| runs | Kinds of reactants in various runs | destruction efficiency % |       |       |              | T °C    |
|------|------------------------------------|--------------------------|-------|-------|--------------|---------|
|      |                                    | Total                    |       |       |              |         |
|      |                                    | tetra                    | penta | hexa  | aroclor 1254 |         |
| 1    | oil                                | 6.5                      | 5.1   | 3.69  | 4.3          | 60-70   |
| 2    | PEG                                | 45                       | 33    | 24.5  | 28.29        | 120-160 |
| 3    | NaOH                               | 24                       | 21    | 31    | 27.56        | 120-160 |
| 4    | PEG+ NaOH                          | 99.5                     | 99.98 | 99.97 | 99.95        | 120-160 |
| 5    | PEG+NaOH+H2O                       | 51                       | 97.68 | 95.8  | 93.87        | 90-110  |
| 6    | PEG+NaOH+FE                        | 99.9                     | 100   | 99.99 | 99.99        | 120-160 |
| 7    | H <sub>2</sub> O+NaOH+Fe           | 9.1                      | 5.7   | 2.15  | 8.22         | 90-110  |
| 8    | PEG+NaOH+H <sub>2</sub> O+Fe       | 54.178                   | 99.94 | 99.96 | 97.39        | 90-110  |



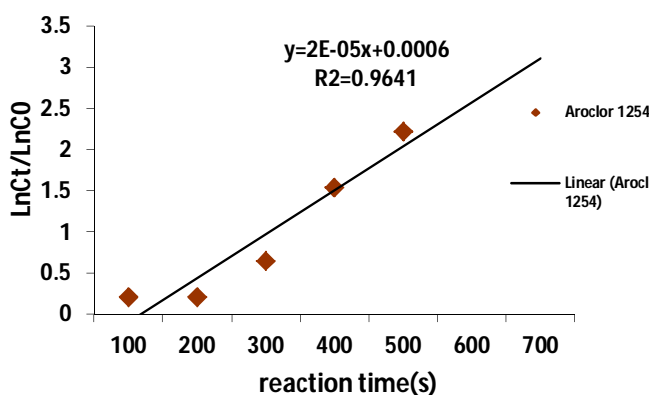
**Figure 3.** Changes the homologic groups decomposition amount of PCBs by MW in the optimum amounts of variables at time 6 min (PEG=5.34g, NaOH= 1.17g, Fe= 0.6g, H<sub>2</sub>O=0.8ml, P=800w).



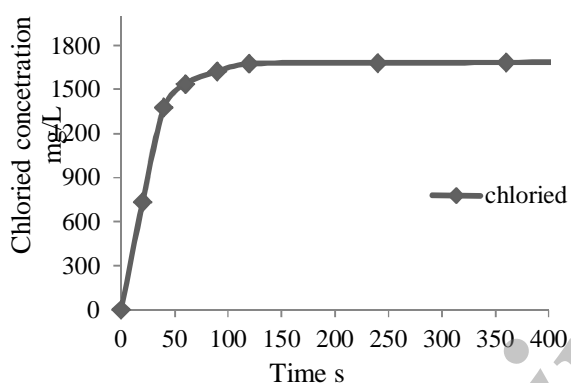
**Figure 4.** The remained concentration of special PCBs in the end of reaction in optimum amounts of variables at different times (PEG=5.34g, NaOH= 1.17g, Fe= 0.6g, H<sub>2</sub>O=0.8ml, P=800w).



**Figure 5.** The decomposition efficiency of special PCBs and total aroclor 1254 in the optimal amount of variables by MW (PEG=5.34g, NaOH= 1.17g, Fe= 0.6g, H<sub>2</sub>O=0.8ml, P=800w).



**Figure 6.** The decomposition kinetic of aroclor 1254 in the optimal amount of variables by MW (PEG=5.34g, NaOH= 1.17g, Fe= 0.6g, H<sub>2</sub>O=0.8ml, P=800w).



**Figure 7.** Chloride ions produced during the decomposition of Aroclor1254 by MW after of process (PEG=5.34g, NaOH= 1.17g, Fe= 0.6g, H<sub>2</sub>O=0.8ml, P=800w, T=6min).

## DISCUSSION

Microwave irradiation motivates migrant ions by dipole exchange and causes vibration of sidelong molecules, that generates heat [18, 24, 25].

MW radiation weakens strong chemical bonds, and decreases the activation energy of the refractory organic materials [26]. Microwave absorbent materials increase these thermal effects. The radiation intensity and heating of MW directly depend on consumed power levels [6].

In this process, two mechanisms might occur for PCB decomposition in the reactor. The first mechanism is the hydrothermal reaction in Fe+NaOH+H<sub>2</sub>O system under MW radiation, in

which hydrogen might be produced and participates in the PCBs dechlorination. In order to determine the dominant mechanism, different runs were carried out with the optimum amounts of reactants (Table. 1). The results are specified when a mixture of reactants Fe, NaOH and H<sub>2</sub>O used 8% of degraded PCBs. Whilst, merely in alkaline condition, 28% of PCBs were degraded which is an indication of the fact that hydrothermal reaction did not happen. Furthermore, x-ray powder diffraction pattern of remained iron powder in the bottom of reactor showed that Fe<sub>3</sub>O<sub>4</sub> and Na<sub>2</sub>FeO<sub>2</sub> were not produced in the process.

Hydrothermal oxidation of zero valent iron in alkaline condition and also hydrogen gas production from this reaction in temperatures above 300°C is proven in other reports; nonetheless, the possibility of low temperature (110°C) and reaction condition to cause this mechanism is impossible in the reactor [1, 19; 27].

The second mechanism constituted an attack to C-CL band of the PCB rings by producing a complex of PEG and alkali, as a potent nucleophilic agent, under MW radiation. Both PEG and NaOH are MW absorbent; meanwhile, NaOH acted as an activator and neutralizes separated chloride ions of PCBs (13, 20, 21). However, the second mechanism (the mixture of NaOH and PEG) distinctly had the greatest influence on PCB decomposition in real waste transformer oil by MW radiation.

The results exhibited in Figure 5 and Table 1 show that more than 99% of PCBs degraded when a mixture of reactants, PEG1000, NaOH and Fe, were used. The main factor was sodium polyethylene glycol alkoxide, produced from the mixture of PEG and NaOH by MW. Sodium PEG alkoxide can phase transfer itself into the organic phase, through self-complexation, and since PEG alkoxide is a potent nucleophile, it can attack C-CL bands of the PCBs and separate chlorine ions from their rings. The aryl polyglycol products are end products of the process and are 300 times less reactive than the initial PCBs [28].

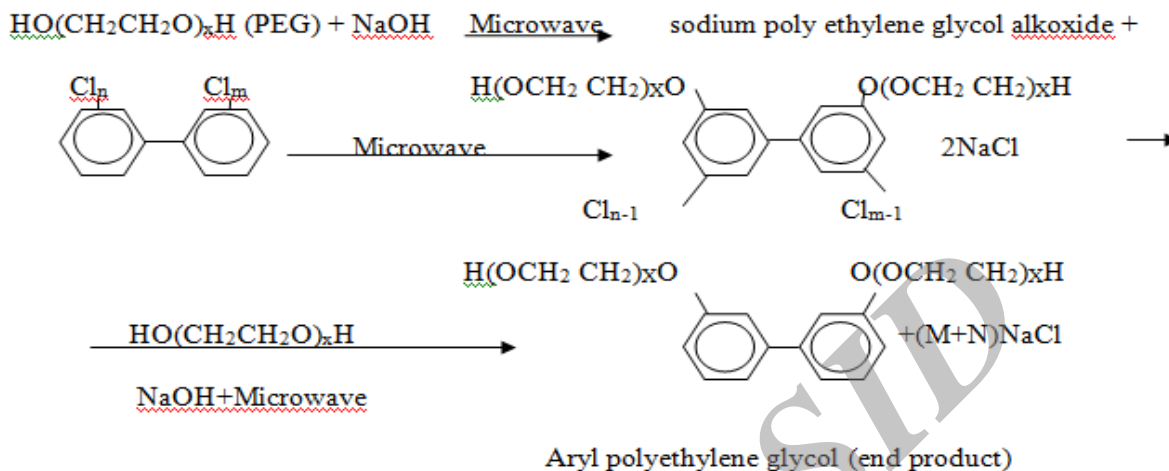
In this process, zero valent iron had little influence (<1%) on PCB decomposition. Fe acted as a MW absorbent and catalyzed and increased the degradation efficiency of PCBs, especially in high chlorinated biphenyls.



Also, observation of reactions behavior and MW radiation of waste oil containing either PEG or NaOH, specified that both of them acted as MW absorbent while waste oil was not MW absorbent. PEG and NaOH, hence, accelerated

the degradation rate of PCBs. The dechlorination pathways suggested in the process are as follows:

When PEG and NaOH are used for dechlorination of PCBs:



In this process, it is possible that very small amounts of active by-products (i.e., the polychlorobiphenyl alcohol) or very toxic intermediate products (i.e., polychlorodibenzodioxins (PCDD)) and polychlorodibenzofurans (PCDF)) are typically formed, but it is proven that all of them react with PEG/NaOH and are converted to its aryl polyglycol during the reaction [28]. The GC-MS chromatograms showed that hazardous intermediates such as dioxin and other toxic chlorinated organic compounds were not generated in the process. Previous studies did not confirm the production of toxic intermediates by this method [17, 29]. But research is still open for investigating the production of dangerous intermediates in the process.

The chlorine balance mass in the reactions provides very important informations about PCB decomposition. The chlorine balance mass chart illustrates that chloride ions amount were almost equivalent with the degraded chlorinated organic materials. It is confirmed that PCB decomposition by the reaction was through dechlorination, not via other paths.

It is proven that the perpendicular or symmetrical structures of PCB molecules decompose later than coplanar or asymmetric structures [20, 27]. This study indicates that PCB52 with the perpendicular and symmetrical

structures have the greatest resistant to degradation in all conditions.

High efficiency, short duration of reaction, easy recycling of waste oil and the low cost are considerable advantages of this method that can be used for the removal of PCBs in waste oil on site.

## CONCLUSION

From a practical, economic and ease of recycling point of view, PCB direct decomposition and removal from waste transformer oil seems to be a suitable disposal method [22, 23].

The main purpose of this study was to investigate the efficiency of PCB decomposition in the real waste transformer oil by microwave radiation, and its recycling and reuse after the process at a laboratory scale. Researchers of current work believe that this method is simple, safe, economic, available and an appropriate alternative to other PCB destruction methods. It can also be appropriate for PCB decomposition and dechlorination from real waste oil on storage sites in developing countries such as Iran.

In this study, the PCB decomposition in real waste transformer oil was investigated using various substances (PEG, NaOH, iron powder, H<sub>2</sub>O) and MW radiation. The MW radiation, PEG and NaOH were important factors in increasing the degradation efficiency. The amounts of reactants, zero valent iron powder,



and water were not influential in this process. These reactants are inexpensive and widely available, and the complete decomposition of PCBs by MW can be carried out in a very short time. In further works, this approach might be valuable for recycling real waste oil and other POPs-containing wastes.

## ACKNOWLEDGEMENT

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