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ORIGINAL ARTICLE**Decomposition of pcbs(transformer oil) by Microwave Radiation in presence of solvent and H₂O₂/Tio₂ Agents****¹Reza Tajik, ¹Hasan Asilian, ¹Ali Khavanin, ¹Ahmad Joneidi, ²Babak Eshreti, ¹Ardalan solimanian**¹*Department of occupational and environment health, Faculty of medical sciences, Tarbiat modares University, Tehran, Iran.*²*Department of statistics and Epidemiology, Faculty of Health, Arak university of medical sciences, Arak, Iran.*Reza Tajik, Hasan Asilian, Ali Khavanin, Ahmad Joneidi, Babak Eshreti, Ardalan solimanian:
Decomposition of pcbs(transformer oil) by Microwave Radiation in presence of solvent and H₂O₂/Tio₂ Agents**ABSTRACT**

Poly chlorinated biphenyls (PCB_s) are the groups of organic chemical material and toxic, persistent, bio accumulate and pose a risk of causing adverse effects to human health and the environment. PCB compounds are caused the different health effects in human depending of age, sex, route of entry, intensity and frequency exposure. This study was conducted to determine the effect of microwave rays, hydrogen peroxide, Tio₂ catalyst and ethanol on the Decomposition of PCB_s. Temperature and pH was continuously monitored. In this experiment used a Microwave oven, Pyrex vessel reactor (250ml volume), Pyrex tube connector and condensing system. A 900w domestic MW oven with a fixed frequency of 2450 MHZ was used to provide MW irradiation. Radiation was discontinuous. Ray powers used in 540, 720 and 900w. The PCB_s were analyzed by GC-ECD. The degradation of total PCB_s in terms of 540, 720 and 900W was 85.12%, 90.15% and 97.33% respectively. The degradation of total PCB_s in terms of ratio to solvent with transformer oil in 1:1, 2:1 and 3:1 was 54.01, 79.07 and 95.01 respectively. The degradation of total PCB_s in terms of not using of H₂O₂ and using 10% and 20% of H₂O₂ was 72.93, 84.43 and 88.52 respectively. The degradation of total PCB_s in terms of not using of H₂O₂/ Tio₂ and using 10% of H₂O₂ and 0.05, 0.1, 0.15, and 0.2 g Tio₂ was 72.93%, 88.92%, 90.10%, 91.68% and 92.95% respectively. The degradation of total PCB_s in terms of not using of H₂O₂/ Tio₂ and using 20% of H₂O₂ and 0.05, 0.1, 0.15 and 0.2 g Tio₂ was 72.93%, 93.79%, 95.01, 96.06% and 97.33 % respectively. The Microwave thermal reaction was applied for the dechlorination of PCBs and high efficiencies were obtained. Microwave power and amounts of reactants added are important factors influencing dechlorination efficiency.

Key words: Askarel, degradation, H₂O₂, PCB_s, Tio₂**Introduction**

Polychlorinated biphenyls (PCBs) are one group of persistent organic pollutants (POPs) that are of international concern because of global distribution, persistence, and toxicity. Removal of these compounds from the environment remains a very different challenge because the compounds are highly hydrophobic and have very low solubility in water [1]. Although the production of PCBs has been banned all over the world, PCBs in some instances still remain in use in a variety of applications when circumstances permit [2]. Its were used in a wide range of industrial applications because of their

excellent physical and chemical properties like: oil in transformers, hydraulic fluids in hydraulic tools, dielectrics in capacitors and equipment and heat exchange liquids which are bio accumulate and pose great risk to the environment and human health. PCB compounds are caused the different health effects in human depending of sex, age, intensity and frequency exposure and route of entry [3]. Incineration is a reliable method for destruction of PCBs in transformer oil. It can destroy over 99.9999% of PCBs and is widely used [3,4]. However, there is widespread public opposition to this approach due to the potential for release of dioxin via flue gas, as stream [5]. Now people are more apt to accept the non-

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combustion technologies for the treatment of PCBs containing wastes. Alternative treatments under development for this type of residue are: chemical reaction with an alkaline hydroxide in polyethylene glycol; supercritical and wet oxidation; adsorption onto activated carbon-regeneration; and Fenton oxidation [3,6-13]. In recent years, microwave irradiation has been applied for the decomposition or dechlorination of chlorinated organic pollutants. In this field, a series of pioneer studies were carried out by Abramovitch et al who adopted microwave irradiation for the remediation of PCBs polluted soil and the results were attractive [14,15]. Subsequently Raquel et al investigated microwave decomposition of lindane supported on modified sepiolites, and found that trichlorobenzene and pentachlorocyclohexene formed with NaOH-modified sepiolite in a short time of Microwave irradiation [16]. Most of the dechlorinating processes for wastes containing PCBs were carried out in various organic solvents [3,6-11,17]. We believe that microwave heating can be employed as energy input to take the place of conventional heating to accelerate the thermal reaction [18].

Material And Methods

Chemicals:

Oil transformers were obtained from regional Electric company in Tehran. Ethanol (C_2H_5OH), sodium hydroxide (NaOH) (all p.a. quality, Merck) and hydrogen peroxide (H_2O_2 , Merck) and used without any purification prior to use and the grain TiO_2 was GT01 (HSD co china) the particle diameter of GT01 is 180-250 μm which is small enough for suspension but large enough for settlement by gravity. The BET surface area of GT01 is 194.4 m^2/g and density is 900 kg/m^3 .

Sample preparation and reactor:

In the MW oven (380mm×380mm×300mm, 900w maximal MW output, made in LG company), there is a flat bottom flask (250ml), with which a 600mm long water reflux condenser is connected through a communication pipe. An aperture is made at the top of the oven for the communication pipe to go through. Note that an aluminum tube fixed in the aperture is used to eliminate MW leaking. The limit on the safe stray leakage of MW power density is kept below 0.5 mw/cm^2 at 2450 MHz measured at 200mm distance from the aperture. Ray powers used in 540, 720 and 900w. Temperature in the flask was measured by quickly stopping the MW oven and was found to be less than 80°C.

Transformer oil was dissolved in ethanol of hydroxyl group. Volumes of ethanol and transformer oil (1:1, 2:1 and 3:1) were in various samples. With due

the rapid increase the temperature of the contents of the reactor as the result of microwave radiation aimed at controlling the reactor during the reaction conditions, radiation was discontinuous. Thus after several trials, radiation was done 10 seconds with intervals 30, 40 and 50 seconds between both the stages of radiation for the selected exposure times were 3, 6 and 9 minutes respectively. The reaction mixture inside the reactor was continuously stirred the samples contained the appropriate volume of oil contaminated by PCBs, ethanol, sodium hydroxide solution (0.2 N), hydrogen peroxide (10% and 20%) and dioxide titanium (0.05, 0.1, 0.15 and 0.2g) for each assay the adjustment of the pH of the solution was done using NaOH solution. For the effect of initial PCB concentrations, H_2O_2 concentrations and TiO_2 quantities used were based on the optimum amount obtained, maintaining the PCB/hydrogen peroxide and dioxide titanium ratio in the solution in all the experimental runs. For the same exposure, the reactor was on the Turn section of microwave for the uniform of solution as the result of the microwave irradiation under the influence of electric field radiation, PCB and solvent molecules vibrated. In order to determine the effectiveness of the solution parameters on the degradation of oil, samples were used to control. The controls were treated similarly but not irradiated, in order to determine the losses not due to the reaction.

Analysis:

The PCBs were analyzed by GC/ECD (Trace Gc ultra USA) with decachlorobiphenyl as internal standard then data were analyzed using statistical software, spss16.0 for statistical analyses, repeated measurement ANOVA and t test and $p < 0.05$ was significant. The identification of the resulting by products was performed at the laboratory services of Kimia Shengerf Pars company of Iran by Gc/MAS. The pH of the samples was periodically controlled with a Methrom-Herisau pHmeter (E512). Samples were taken at varying time (3, 6 and 9 min) intervals and analyzed immediately or stored at 4°C up to 3-5 h before analysis. For quality control, the experiments were conducted in three replicates. To prevent from exposure to UV irradiation, samples covered with aluminium foil. Samples without the H_2O_2/TiO_2 and solvent were also used to investigate the effect of direct degradation of PCBs. The capillary column used was a DB-5 and the carrier gas was helium. The capillary column (narrow-bore) 30m×0.32mm ID fused silica capillary column chemically bonded with 5 percent phenyl methyl and 0.25 μm film thickness. Helium was used as carrier gas with a flow rate of 1.3 ml/min and N_2 was used as make-up gas with a flow rate of 60 ml/min . The injector and detector temperatures were set at 280 and 300°C respectively. A split-splitless injector in the splitless mode was used. The

column temperature was programmed as follows: 50°C(30Sc), 12°Cmin⁻¹ to 130°C and 5°Cmin⁻¹ to 280°C and 5min hold.

Results and Discussion

Identified PCBs in PCB transformer oil is included PCB101, PCB138, PCB153, PCB180. The initial concentration of PCBs is showed in table 1. Also, the table 1 is showed percent of degradation of PCBs without solvent and H₂O₂ and TiO₂.

Table 1: initial concentration of PCBs in PCB transformer oil

PCB	PCB101	PCB138	PCB153	PCB180
Value (µg/L)	421	2492	1997	3333
% degradation of PCBs (blank sample-without ethanol and H ₂ O ₂ and TiO ₂)	2.6	1.6	0.4	2.2

Experimental conditions: pH= 7±0.5, Temperature= 76 ± 1.5°C, exposure time= 9 min , power=900w

Table 2 and figure 1 shows the effect of power of microwave in similar times in samples. The degradation of total PCBs in terms of 540, 720 and 900w was 85.12%, 90.15% and 97.33% respectively.

Table 2: Effect of MW power on PCBs degradation efficiency (%)

PCBs Power	Exposure Time (min)	PCB101	PCB138	PCB153	PCB180	Mean
540w	3	81.94	83.62	84.37	82.47	83.10
	6	83.84	84.10	84.77	83.55	84.06
	9	84.79	85.39	85.57	84.75	85.12
720w	3	87.64	86.83	87.98	86.43	87.22
	6	89.54	88.60	88.18	87.27	88.39
	9	90.49	90.20	89.78	90.15	90.15
900w	3	94.29	95.50	93.39	93.51	94.17
	6	96.19	96.30	94.39	95.67	95.63
	9	97.14	97.27	97.59	97.35	97.33

Experimental conditions: Volumes of ethanol and transformer oil (3:1), pH= 7±0.5, Temperature in 540w= 37 ± 1.5°C (3min) , 41 ± 1.5°C (6min) and 46 ± 1.5°C (9min) , In 720w =51 ± 1.5°C (3min) , 55 ± 1.5°C (6min) and 60 ± 1.5°C (9min) , In 900w =65 ± 1.5°C (3min) , 69 ± 1.5°C (6min) and 74 ± 1.5°C (9min)

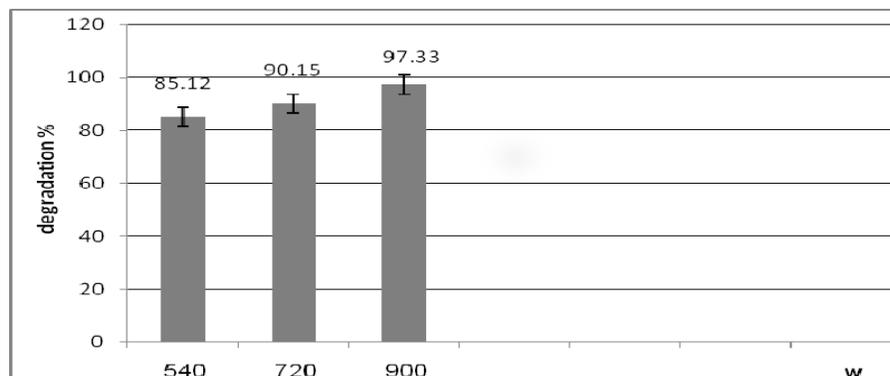


Fig. 1: Effect of MW intensity on PCBs degradation (%)

Table 3 and figure 2 shows the effect of volume of solvent , i.e.ethanol on PCBs degradation. The degradation of total PCBs in terms of ratio solvent with oil transformer in 1:1 , 2:1 and 3:1 was 54.01 , 79.07 and 95.01

Table 3: Effect of volume of solvent on PCBs degradation efficiency (%)

PCBs Volume of solvent to oil	PCB101	PCB138	PCB153	PCB180	Mean
1:1	53.91	53.85	54.03	54.27	54.01
2:1	79.33	78.69	79.26	79.02	79.07
3:1	95.24	94.86	94.99	94.95	95.01

Experimental conditions: pH= 7±0.5, Temperature= 74 ± 1.5°C, exposure time= 9 min , power=900w

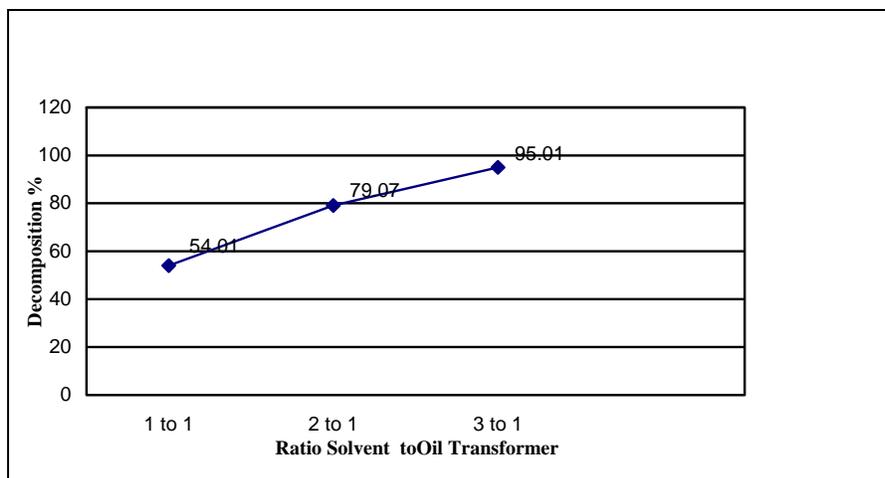


Fig. 2: Effect of volume of solvent on PCBs degradation (%)

Experimental conditions: volume of ethanol and transformer oil (1:1, 2:1 and 3:1), pH=7±0.5, Temperature= 74 ± 1.5°C, exposure time= 9 min , power=900w

the use of MW/H₂O₂ process. The degradation of PCBs in terms of use of 10% and 20% of total volume in samples of H₂O₂ were 84.43% and 88.52% respectively.

Table 4 and figure 3 shows the effect of initial H₂O₂ concentration on the degradation of PCBs with

Table 4: Effect of Hydrogen Peroxide on PCBs degradation (%)

PCBs	Time (min)	PCB101	PCB138	PCB153	PCB180	Mean
10 % H ₂ O ₂	3	82.89	83.14	82.57	83.31	82.51
	6	83.84	83.46	84.17	83.91	83.67
	9	84.79	84.10	84.57	84.27	84.61
20 % H ₂ O ₂	3	86.69	86.51	86.17	85.95	86.91
	6	87.64	87.31	87.78	86.79	87.95
	9	88.59	88.44	89.18	87.87	88.85

Experimental conditions: Volumes of ethanol and transformer oil (3:1), pH= 7±0.5, Temperature = 65 ± 1.5°C (3min) , 69 ± 1.5°C (6min) and 74 ± 1.5°C (9min) , power = 900w

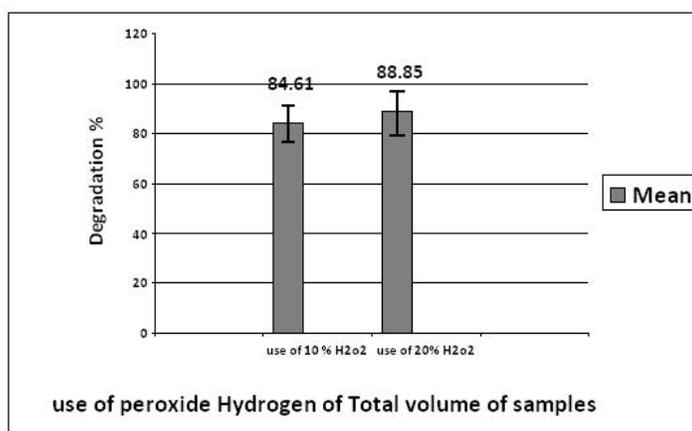


Fig. 3: Effect of hydrogen peroxide on PCBs degradation(%)

Experimental conditions: volume of ethanol and transformer oil (1:1, 2:1 and 3:1), pH=7±0.5, Temperature= 74 ± 1.5°C, exposure time= 9 min, power=900w

Table 5 shows the effect of degradation of PCBs in terms of not using of H₂O₂/TiO₂ and using 10% and 20% of H₂O₂ and 0.05,0.1,0.15 and 0.2 g TiO₂. The degradation of total PCBs in terms of not using of H₂O₂/TiO₂ and using 10% of H₂O₂ and

0.05,0.1,0.15 and 0.2 g TiO_2 was 72.93 , 88.92, 90.10, 91.68and 92.95 respectively and The degradation of total PCBs in terms of not using of

H_2O_2/TiO_2 and using 20% of H_2O_2 and 0.05,0.1,0.15 and 0.2 g TiO_2 was 72.93, 93.79, 95.01, 96.06 and 97.33.

Table 5: Effect of Hydrogen Peroxide and dioxide titanium on PCBs degradation (%)

H_2O_2/TiO_2	PCBs				Mean
	PCB101	PCB138	PCB153	PCB180	
Not using of H_2O_2/TiO_2	73.39	74.15	71.55	72.63	72.93
10% H_2O_2 + 0.05 g TiO_2	87.64	90.20	88.78	89.07	88.92
10% H_2O_2 + 0.1 g TiO_2	89.54	90.52	90.58	89.79	90.10
10% H_2O_2 + 0.15 g TiO_2	91.44	92.13	92.18	90.99	91.68
10% H_2O_2 + 0.2 g TiO_2	93.34	92.45	92.98	93.03	92.95
20% H_2O_2 + 0.05 g TiO_2	93.34	94.06	93.79	93.99	93.79
20% H_2O_2 + 0.1 g TiO_2	95.24	94.86	94.99	94.95	95.01
20% H_2O_2 + 0.15 g TiO_2	96.19	95.82	95.99	96.27	96.06
20% H_2O_2 + 0.2 g TiO_2	97.14	97.27	97.59	97.35	97.33

Experimental conditions: volume of ethanol and transformer oil (3:1) , pH= 7±0.5, Temperature= 74 ± 1.5°C, exposure time= 9 min , power=900w

Discussion:

Effect of MW power:

The result of Table 2 showed that the degradation rate was very high during the first three, four minutes, point which was then slow down and level off when the irradiation proceed. This indicated that the reactor was highly efficient in dechlorinating the congeners tested, and optimising the time for degradation reduced the reaction time and therefore the main cost of the treatment, i.e. energy cost of producing MW radiation can be minimised [7-9,17].

An important variable influencing dechlorination is Microwave power. Microwave power was regarded as a very important factor for the dechlorination reaction, because the temperature that the whole reaction system or catalyst could reach was directly related to the power level as a result, increasing the MW power will provide more thermal energy and so increase the degradation efficiency [19]. At first, NaOH, H_2O_2 , TiO_2 and C_2H_5OH received Microwave irradiation by space charge polarization and dipolar polarization, and the incident Microwave energy was instantly converted into thermal energy [20]. Microwave energy has been used more and more on synthetic organic chemistry because of its great ability to accelerate reactions and to improve yields and selectivity [21]. recently, some researchers have started to investigate catalytic reactions assisted with Microwave irradiation [22,23].

Figure 1 showed the effect of changing MW intensity on the degradation efficiency of

PCB congeners. In general the degradation efficiency increase with increasing MW intensity and decreasing the Microwave intensity decreasing the dechlorination rate, and also the degradation efficiency tended to plateau from 540w to 900w [7-9,17].

In general, the effect of the MW power is expected to be straightforward. Since the major mechanism for generation of hydroxyl radical is degradation of hydrogen peroxide, increasing the power of the MW should increase the generation rate of hydroxyl radical and thus the oxidation of the PCBs [7-9,17,24].

As the Microwave oven used in this study was a modified one, the power levels used here were discontinuous, just timed mark/space ratio control. three power levels (540w, 720w and 900w) were investigated, and other parameters were as follows: 10% and 20% of H_2O_2 (0.058 mole and 0.116 mole), 0.05, 0.1, 0.15 and 0.2 g TiO_2 , NaOH 0.2N (1^{cc}) , ratio ethanol solvent with transformer oil 1:1 (25 cc ethanol and 25 cc oil), 2:1 (33.33 cc ethanol and 16.67 cc oil) and 3:1 (37.5 ethanol and 12.5 oil) and 3, 6 and 9 min Microwave irradiation.

The six reactants participated in both the Microwave thermal reaction and the dechlorination process. So the amounts of reactants being added are crucial and their effects should be investigated by adding 0.116 mol of H_2O_2 , 0.2 g TiO_2 , 900w power, NaOH 1 cc, 9 min exposure time and 3:1 ratio to solvent with oil high efficiency of dechlorination was obtained. therefore, we designed experiments to investigate the effect of reduced amounts of

reactants on dechlorination and dechlorination efficiencies were calculated based on chlorine ions produced.

Effect of Ethanol(solvent):

Figure 2 showed the effect of changing volume of solvent on the degradation efficiency of PCB congeners. The degradation of total PCBs in terms of ratio to solvent with transformer oil in 1:1, 2:1 and 3:1 was 54.01, 79.07 and 95.01 respectively. Because all chlorobiphenyls are poorly soluble in water, early work on PCB degradation was carried out in alkanes and alcohols. The many organic solvents are employed in previous studies. Solvents such as hexane, isooctane, 2-propanol and methanol are used by some researchers [7,9,17]. The overall photochemistry in both organic and aqueous media is predominantly dechlorination, but, depending on the solvent used, some solvent derivatives can also be found [8,13,24-26]. Photochemical decomposition using alkaline isopropyl alcohol would not be useful in aqueous media because water inhibits the reaction by protonating the reactive radical. However, there were a few reports found that employ ethanol. It was reasonable to predict that the performance of ethanol should be in between or similar to others solvent. The some of solvents, for example, hexane and isooctane is highly flammable, and the degradation may take long time for high PCB concentration, fire hazard is of concern. However, the reaction rate of PCB was found to be faster in hydroxylic solvents than non-polar solvents [24]. we used of ethanol solution as intermediate solvent for absorbing thermal energy of Microwave irradiation and spreading it.

In general, the effect of the ethanol volume is expected to be straightforward. Since the major mechanism for generation of hydroxyl radical is degradation of hydrogen peroxide, increasing the concentration of ethanol should increase the generation rate of hydroxyl radical and thus the oxidation of the PCBs.

Effect of hydrogen peroxide:

Fig. 3 shows the effect of initial H₂O₂ concentration on the degradation of PCBs with the use of MW/H₂O₂ process. The degradation of total PCBs in terms of use of %10 and %20 of volume of H₂O₂ were %84.61 and %88.85 respectively. As expected, the degradation rate of PCBs increased with increasing the concentration of H₂O₂ added. This can be explained by the effect of the additionally produced OH^{*} radicals [27].

Results showed that irradiation alone cannot be used as an effective procedure for the degradation of PCBs. Some studies indicated that the degradation efficiency of PCBs by MW radiation could be raised by the addition of H₂O₂. When H₂O₂ is added, the

dominant mechanism of PCBs destruction becomes hydroxyl radical mediated advanced oxidation, and the highly reactive OH radical species produced in the process are expected to quickly react with PCBs. The hydroxyl radical is an extremely reactive and nonselective oxidant and, thus, when produced in sufficient quantities, can lead to complete oxidation of organic compounds to carbon dioxide, water, and inorganic ions [25]. The OH^{*} radical may oxidize substrate, but because of its high reactivity it also may react with a variety of other substances commonly found in PCBs.

The hydrogen peroxide dose is very important parameter for the efficiency of the treatment process and the economic desirability. In the MW/H₂O₂ process, the degradation of hydrogen peroxide is the major mechanism generating hydroxyl radicals. Therefore it is logical to assume that increasing the dose of hydrogen peroxide would increase the transient-state concentration of hydrogen peroxide. This is not the case though, because hydrogen peroxide acts as a hydroxyl radical scavenger too [13,24-27]. It is obvious that at low initial hydrogen peroxide concentrations, increasing the dose significantly increase the degradation rate of the PCBs. At higher concentration, the effect of increasing the dose is negligible. This is a reasonable result since the hydroxyl radical production is limited from the degradation rate, increasing the hydrogen peroxide dose further from that point acts only to increase the hydroxyl radical scavenging from hydrogen peroxide.

Effect of Dioxide Titanium:

Titanium dioxide (TiO₂) power is generally used as one of the most popular photocatalysts in photocatalytic oxidation for water and wastewater treatment because of its nontoxicity, chemical inertness and low costs [28]. GT01 had a comparatively good performance in MW/TiO₂ degradation PCBs and was easily separated from the contents of reactor. The concentration of 4g/l (0.2g/50 cc) was the optimum dosage of GT01 under the experimental condition in terms of catalytic oxidation rate. Microwave enhances the reactants mobility and diffusion leading to increased exchange of reactants between catalyst surface and solution [28].

Conclusion:

The Microwave thermal reaction was applied for the dechlorination of PCBs and high efficiencies were obtained. Microwave power and amounts of reactants added are important factors influencing dechlorination efficiency.

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