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DEGRADATION OF DIISOPROPYL METHYLPHOSPHONATE IN AQUEOUS SOLUTIONS BY ULTRASONIC IRRADIATION

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Abstract. The degradation of diisopropyl methylphosphonate (DIMP) in aqueous solutions was studied using ultrasound irradiation with a fixed frequency of 26.6 kHz, following the first-order kinetic model. The study's primary goal was to determine the influence of the following experimental parameters: the pH (at different levels of 2, 7 and 10), the initial concentration of DIMP (at different levels: 7, 14, 30, 50, 80 mg/L), the processing time (at different values: 15, 30, 45, 60, 80, 90 min), and the concentration of the additive CCl₄ (at different concentrations: 0.002, 0.004, 0.006, 0.008 mg/L). A DIMP removal rate of 98% from aqueous solution was obtained at pH 10 and 0.008 mg/L CCl₄, after an ultrasound irradiation time of 45 min, pointing out the influence of the above-mentioned experimental parameters on the DIMP degradation process.

Keywords: ultrasound, diisopropyl methylphosphonate, sonochemical kinetic, aqueous solution, chemical oxidant.

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Introduction

Organophosphorus compounds (OPCs) are organic molecules formed from phosphoric acid and its derivatives, which include at least one carbon-phosphorus bond. Pentavalent phosphorus-containing compounds are largely used for socioeconomic and ecological purposes. Organophosphorus refers to any toxic phosphorus-containing chemical compound [1]. Some examples of organophosphorus compounds are thiols, amides, or esters of phosphonic, phosphinic, phosphoric, or thiophosphoric acids with two extra phenoxy, cyanide, or thiocyanate side chains [2]. Arylated organophosphorus species offer a wide range of uses, such as pesticides, materials, and analytical and medicinal chemistry [3]. Many of these types of OPCs are inactive in their native form and must undergo biotransformation to become active. This biotransformation involves the oxidation of different functional groups, such as the sulfur group (e.g., parathion and malathion),

the thioether group (e.g., disulfoton), the amide group (e.g., schradan and dichrotophos), and the alkyl group (e.g., triorthocresylphosphate) [4–11].

There are several ways of OPCs degradation, such as thermal decomposition, combustion, photolysis, and ultrasonic (US) irradiation [11–14]. An efficient way to study OPCs decomposition under laboratory conditions is by using diisopropyl methylphosphonate (DIMP) as a model system. Ultrasonic irradiation is a promising technique for pollutants elimination, along with chemical oxidation and biological degradation [15].

To the author's knowledge, no research has been previously done on the effectiveness of ultrasonic irradiation on DIMP degradation. Thus, the primary goal of the study was to determine the effect of the main process parameters on DIMP decomposition, such as pH value, processing time, initial concentration, and carbon tetrachloride (CCl₄) concentration.

Experimental

Materials

The reagents used in this study, namely: diisopropyl methylphosphonate (DIMP) (Hubei Bluesky New Material INC, China), CCl_4 (Merck Co., Germany), NaOH (Loba chemie, Mumbai, India), and HCL (Thomas Baker, India), were of analytical grade. In all experimental tests, fresh distilled water was used.

The procedure of ultrasonic irradiation of DIMP samples

The primary equipment used for this study was a high-intensity ultrasonic probe system, consisting of an ultrasonic generator (controller), a titanium horn micro-type, and an ultrasonic transducer (Hielscher, UP200HT, Germany). The experimental setup for the DIMP ultrasonic-assisted decomposition tests is depicted in Figure 1. DIMP solutions of increasing concentration (7, 14, 30, 50, 80 mg/L) at three pH levels (2, 7, and 10) were prepared, using a stirrer at a rotation of 300 rpm. Each concentration was prepared in a beaker of 1000 mL with distilled water and then transferred to a beaker of 250 mL. The solution pH was converted to acidic or basic, using HCl or NaOH respectively, and kept constant during the US-assisted treatment. Irradiation processes were then initiated at a fixed frequency (26.2 kHz) for six different processing times (15, 30, 45, 60, 80, and 90 min.). The temperature of the samples increased from initial value of 20°C up to 60°C during the irradiation process, while a temperature probe connected to a temperature controller stopped the excessive heating over 60°C by cooling with a water-jacketed vessel and switching off the USW Generator until the temperature decreased below the prescribed value.

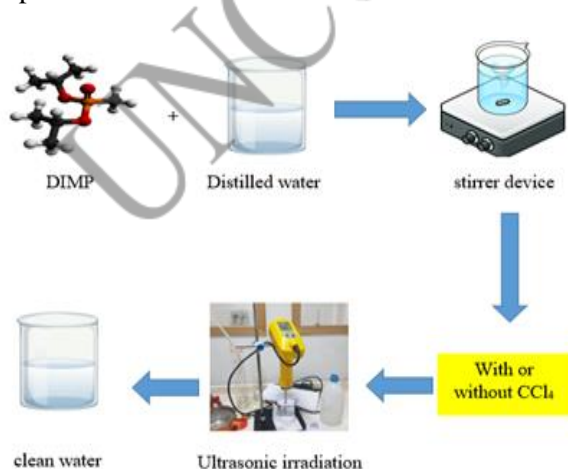


Figure 1. Brief graphic illustration of the experimental procedure of DIMP ultrasonic irradiation.

The vessel was periodically opened, and all samples were evaluated by Liquid Chromatography. During the irradiation treatment, samples of the liquid mixture were withdrawn at pre-established times, in order to be analyzed by HPLC. Measurements were carefully done in triplicate, therefore the accuracy for the determined DIMP concentration is an average of measurements with standard deviation less than 10%.

First-order kinetics model evaluation

In order to obtain the best removal rate in the frames of this study, the test was carried out at pH 10 and DIMP initial concentration of 80 mg/L was selected. The kinetic degradation of DIMP is governed by the first-order kinetic model, taking into consideration that the correlation coefficient (R^2) was closer to one (0.967) than that obtained when a second-order kinetic model was applied (0.762).

The first-order kinetic model in the linear form is given in Eq.(1) [15].

$$\ln \frac{C_0}{C} = kt \quad (1)$$

where, C_0 - the initial concentration (80 mg/L) of DIMP;

C - the concentration of DIMP after ultrasonic irradiation at different treatment times;

k - the first-order degradation rate constant, which can be derived from the line slope by plotting $\ln \frac{C_0}{C}$ vs t , as illustrated in Figure 2.

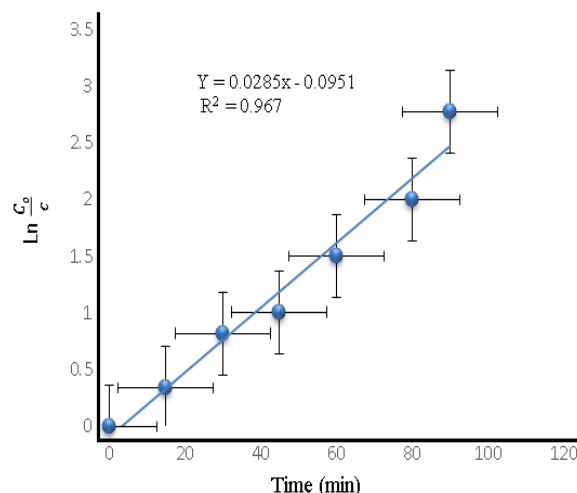


Figure 2. First order kinetic model for DIMP concentration of 80 mg/L at pH 10.

The pseudo-first and second-order kinetic models have been evaluated as well, but these are not presented, due to the lack of good fitting between the experimental data and those predicted by these models.

There are some previous studies on organophosphorus compounds (as pollutant models), which demonstrated that many ultrasonic degradation processes have specific kinetics and can be described by kinetic models that include pseudo, first, and second-order models [17–19].

The concentrations of diisopropyl methylphosphonate and intermediate compounds produced by ultrasonic (US) treatment were determined by *high-performance liquid chromatography* (HPLC) (SAYKAM Germany). The stationary phase of 5 mm particle size was contained in a C8 column with the following dimensions: 250 mm in length, and 4.6 mm internal diameter. The mobile phase for diisopropyl methylphosphonate is a 70:29:1 mixture of acetonitrile, water and H_3PO_4 . HPLC was conducted at a flow rate of 0.7 mL/min. The UV detectors pointed out peaks at a wavelength of 210 nm. All samples were filtered using 0.2 μ m syringe filter.

Results and discussion

In order to reach the aim of this work related to determining the effect of process parameters (pH, processing time, initial concentration, and CCl_4 concentration) on the degradation of DIMP, the main results are further graphically presented.

The results of HPLC analysis for DIMP degradation are shown in Figure 3(a,b). Figure 3(a) shows a decrease in DIMP concentration in the solution of 30 mg/L initial concentration after 30 min of treatment, due to ultrasonic irradiation and generation of hydroxyl radicals-rich species. Figure 3(b) shows a decrease in the concentration of DIMP in the solution of 80 mg/L initial concentration after 30 min, due to irradiation and the addition of the chemical oxidant CCl_4 , which enhanced the decomposition process based on the generation of chlorine radicals.

Effect of pH

The influence and efficacy of pH on the degradation of DIMP were investigated, and the experimental findings revealed that the optimal degree of degradation proceeded in alkaline solution (pH 10) followed by neutral (pH 7) and acidic (pH 2) ones, as shown in Figure 4. Because the hydroxyl radicals generation rate during sonochemical degradation is controlled by the solution's pH, higher pH solutions result in more

radicals being recombined to create H_2O_2 , which reduces the amount of radicals that may be used in the intended degradation reaction [21,22]. In addition, a high pH value may generate more free radical scavengers, which reduces the HO^\bullet radical concentration [23,24].

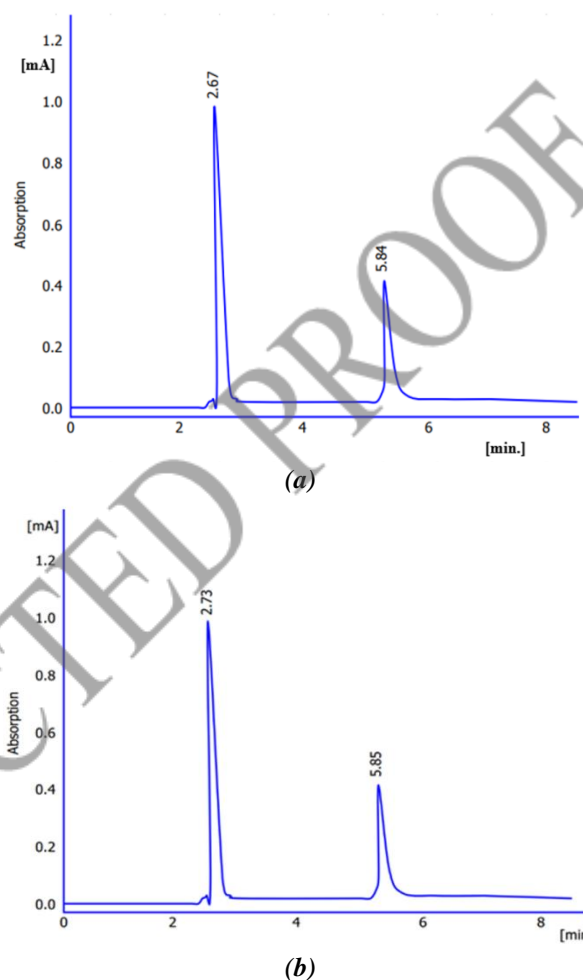


Figure 3. HPLC analysis for DIMP degradation: 30 mg/L at pH 2 and 30 min with ultrasonic irradiation (a); 80 mg/L at pH 10 and 30 min with 0.006 mg/L of CCl_4 (b).

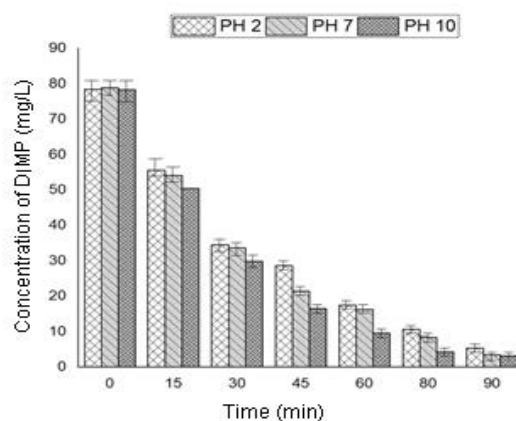


Figure 4. Time evolution of DIMP concentration in a solution of 80 mg/L initial concentration, at different pH values

Degradation kinetics using sonochemistry at various pH levels is affected by the pollutant molecule's ionic or molecular state. The influence of pH on the degradation rate is thus highly dependent on the pKa value of the ionizable organic pollutants. Since a pKa of 7.9 indicates a strong alkaline medium, the optimal environment for DIMP degradation is found in basic solutions, where the ionic form predominates, and bubbles of the compound cannot form. This ionic species is limited to the film area of the interface and reacts with the OH[•] radicals [25].

Effect of initial concentration

The initial concentration can affect the efficiency of US-assisted degradation, as the degradation efficiency increases as the concentration lowers. The degradation rate was affected by rising or decreasing concentration, therefore the decomposition was constant. This is because in bulk solution hydroxyl radicals were produced, which reacted with molecules during acoustic decomposition to form H₂O₂ [26].

Figure 5 illustrates the time evolution of DIMP concentration at different initial concentrations at pH 10 and reveals that a percent degradation rate of up to 98% occurred at a concentration of 7 mg/L in 45 min, whereas at a concentration of 80 mg/L, the degradation rate was of only 78%, which demonstrates the effect of low concentrations on the degradation processes: at lower DIMP concentration, the degradation rate was higher than at high concentration. The same results were obtained for pH 2 and 7, as shown in Figures 6 and 7.

Effect of ultrasonic processing time

In a nutshell, the chemical breakdown of DIMP depends on the elapsed treatment/irradiation time. Longer exposure time results in more hydroxyl radicals being produced, as seen in Figures 5-7. Degradation of organophosphorus compounds has been studied extensively, and the obtained results were in concordance with the aspects pointed out in literature. In a previous study [26] was reported the sonolysis of azinphos-methyl over a period of 60 min of treatment, and showed that the effectiveness of azinphos-methyl elimination improved rapidly in the first 20 min, but dropped considerably between 20 and 60 min. Sonolysis's ability to remove pesticides did not change in terms of yield [26].

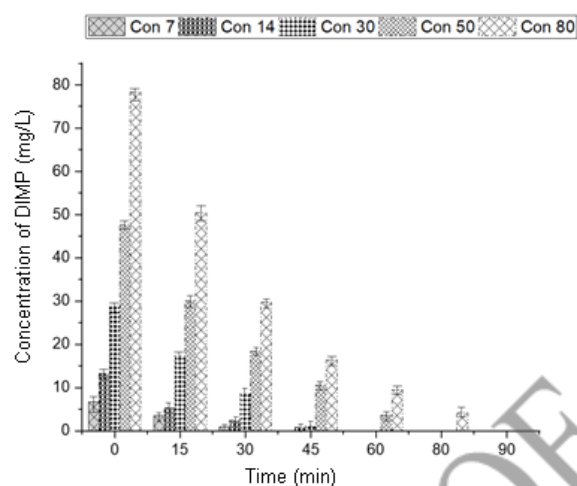


Figure 5. Time evolution of DIMP concentration at different initial concentrations, at pH 10.

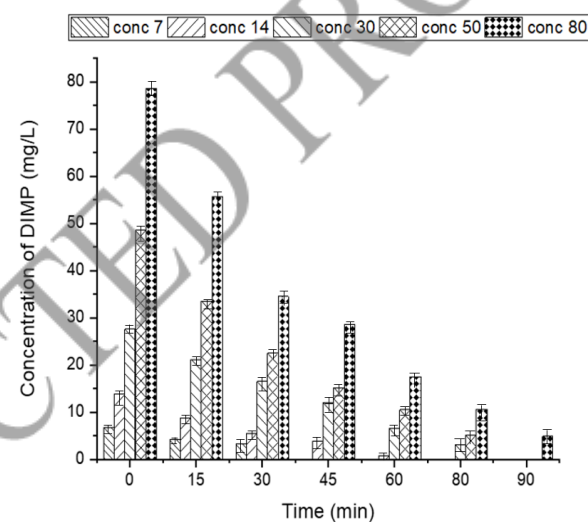


Figure 6. Time evolution of DIMP concentration at different initial concentrations, at pH 2.

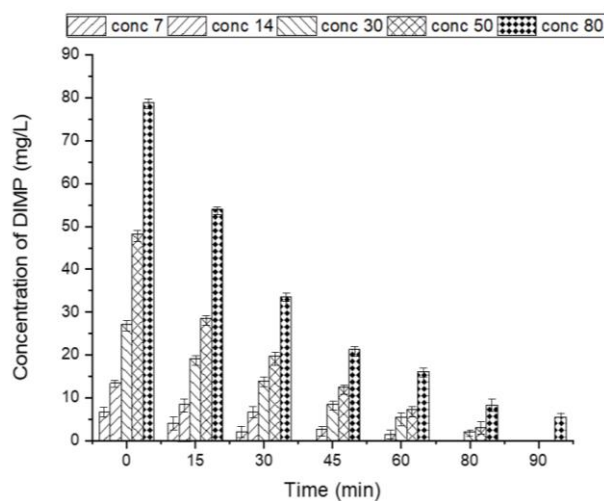


Figure 7. Time evolution of DIMP concentration at different initial concentrations, at pH 7.

Effect of CCl₄ addition

The influence of CCl₄ concentration (0.002, 0.004, 0.006, 0.008 mg/L) on the degradation of DIMP in solution of 80 mg/L initial concentration was examined throughout time periods ranging from (15, 30, 45, 60, 80 min). This demonstrates that a complete elimination of 0.008 mg/L was achieved in a short period of time. Figure 8 and Table 1 illustrate the percentage degradation rate of DIMP over time, at different CCl₄ concentrations.

Table 1

Degradation efficiency/rate of DIMP at 80 mg/L initial concentration and pH 10, at various CCl₄ concentrations.

Time (min)	CCl ₄ Concentration (mg/L)			
	0.002	0.004	0.006	0.008
15	32%	39%	51%	52%
30	66%	77%	81%	85%
45	83%	90%	94%	98%

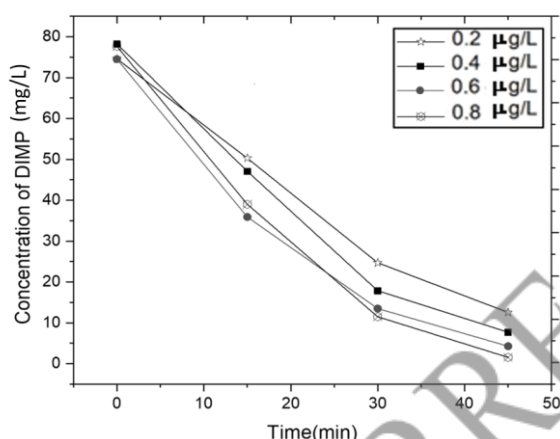
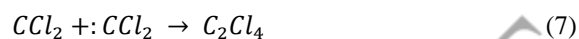
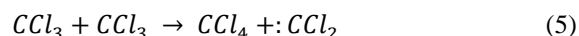
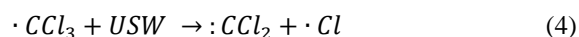
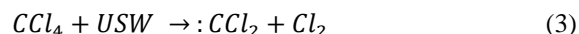
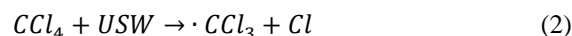


Figure 8. Degradation of DIMP solution 80 mg/L and 10 pH of DIMP at different CCl₄ concentrations (0.002–0.008 mg/L) over 45 min treatment.

As a result of the creation of additional oxidizing species in the system, during ultrasonic treatment, the addition of carbon tetrachloride can accelerate the rate of pollutant elimination. Due to its hydrophobic nature, CCl₄ is easily sucked into cavitation bubbles, where it undergoes a cleavage, leading to the dissociation of CCl₄ molecules and the formation of highly reactive chlorine radicals [27].

The formation of chlorine radicals will result in a sequence of recombination reactions that produce highly oxidizing species inside the system [28,29]. The formation of chlorine radicals leads to the recombination of strong

oxidizing types in the system, as shown in Eqs.(2-9) [16].



where, USW - ultrasonic waves.

Other oxidizing species attack the organic component in the bulk phase or at the gas-liquid interface, in addition to the hydroxyl radicals created by the sonolysis of water vapor in the cavitation bubble. This coordinated assault in the presence of CCl₄ dramatically speeds up the degradation of contaminants. It is also crucial to remember that the addition of CCl₄ can impact the system's final toxicity levels if the additive is not properly used during the treatment process. An excessive amount of CCl₄ in the reaction system produces the generation of vaporous cavitation bubbles, which restricts the net release of energy during the implosion of the bubbles and hence reduces the synthesis of oxidants due to the extremely volatile nature of the additive [24].

The obtained results of this study pointed out once again the enhancing treatment efficiency of the organic refractory pollutants from wastewater, using the combining ultrasonic irradiation with other wastewater treatment process [30], even when the ultrasonic irradiation could act independently with enough good treatment efficiency.

Conclusions

This study investigated and clarified the degradation of DIMP in aqueous solutions under ultrasonic irradiation, by means of the effect of different influence factors on DIMP decomposition, including initial concentration, pH, treatment time, CCl₄ concentrations, reaction rate, and intensity of the irradiator. The highest degradation was observed at pH 10, compared with pH 2 and respectively pH 7, which values changed during experiments. The pH of the

solution has a major role in the generation of hydroxyl radicals through the collision of solution molecules with each other. In particular, the pH of the solution (pH>7) played an essential impact on the effectiveness of degradation, particularly when different amounts of CCl₄ were utilized as a DIMP breakdown catalyst.

The most favourable experimental conditions for DIMP decomposition in the presence of CCl₄ were 0.008 mg/L CCl₄ and 45 min of ultrasonic treatment time when a quasi-complete degradation of 98% was recorded. Sonolysis of water vapour in the cavitation bubble results in the production of hydroxyl radicals, which then attack the organic substance that is present either in the bulk phase or on the gas-liquid interface. Additional oxidizing species participate in this attack. The first-order kinetics model can be used to describe the degradation kinetics of DIMP in aqueous solution.

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