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ORIGINAL PAPER

Remediation of organophosphate pesticide-contaminated soil using soil washing and advanced oxidation processes

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Abstract
Large numbers of contaminants such as polycyclic aromatic hydrocarbons, pesticides and chlorophenols pass through sediments and soil, causing a giant danger to human health and ecosystem. To remediate the soil contaminated with these pollutants, various methods have been proposed including coupled soil washing with Fenton or Sono-Fenton process. In this study, non-ionic surfactants [Tween 85 AND linear alkylbenzene sulfonates (LASs)] were used for the removal of chlorpyrifos (organophosphate pesticide). The optimal conditions for LAS surfactant were found to be a concentration of 2.5 g/L with 20/1.5 ratio (liquid/solid), 360 min operation time and 120 rpm washing speed in room temperature; while 1 g/L Tween 85 concentration was more effective at 20:1 ratio (liquid solid), 360 min operation time and 60 rpm washing speed in room temperature, respectively. The results imply that combining both Tween 85 and LAS can be an effective way to remove large amounts of contaminants from soils quickly without damaging them further or harming humans who might come into contact with it afterward. The results of the experimental study on soil washing and Fenton/Sono-Fenton suggest that these two processes combined can be an effective way to remediate soils contaminated with chlorpyrifos. This combination was shown to provide superior results for both remediation and recovery of surfactants used in the cleaning process.

Keywords Fenton · Pesticides · Soil contamination · Sono-Fenton · Surfactant

Introduction

Soil quality is significant for the safety of produced foods, community health and a sustainable environment (Li et al. 2020). The formation of various substances that can change the quality and function of soil and damage its basic structure is defined as soil contamination (Sun et al. 2019), which is one of the most common problems around the world (Ramón and Lull 2019). Polycyclic aromatic hydrocarbons (PAHs), petroleum and related products, pesticides, chlorophenols and heavy metals are among the primary soil pollutants (Singh and Haritash 2019; Zeb et al. 2020).

Tons of pesticides are released into the biosphere both intentionally and unintentionally. Variable concentrations (ppt-ppm range) of pesticides were found in agricultural soils, surfaces and groundwater (Vryzas 2018) that are belong to very large amount of tons of pesticides were used worldwide (Neuwirthová et al. 2019). Pesticides are designed to disrupt the nervous and muscular systems and normal functions of microorganisms (Rajagopalan et al. 2023; Rajak et al. 2023). Unfortunately, pesticides applied to target organisms can quickly accumulate in many organisms, including humans (Zhen et al. 2018).

Organophosphates (OPs), a type of pesticide, were determined as the most used insecticides in the last decade (Aswathi et al. 2019). Chlorpyrifos, which is the most common organophosphate insecticide, has been used in agriculture (Alruhaimi 2023; Cheng et al. 2023).

Conventional treatment methods such as sorption (Liu et al. 2018), biological treatment system (Streptomyces consortium) (Fuentes et al. 2017), phytoremediation (Prabakaran et al. 2019), advanced chemical oxidation (Malakootian et al. 2020) and photochemical oxidation (Bae et al. 2023) and enzymatic conversion (Varga et al. 2019)

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or combinations of these methods (Sánchez et al. 2019) are used to remove pesticide residues from soil (Chen et al. 2019). Among these methods, the soil washing method is the most commonly used due to its simplicity and high efficiency in the treatment of different pollutants in soil (Gu et al. 2022; Tran et al. 2022). The basic principle of this method is solution extraction, which is used to efficiently transfer pollutants that are present in the soil to a liquid phase. The most used washing solution is surfactant solutions. Anionic surfactants are the most preferred surfactants due to their high extraction efficiency and low adsorption properties. One anticipated concept was that soil washing was the used media, which often contains chemical additives, may need specialized treatment which is generally difficult to do and expensive. Moreover, this issue could be attributed to the solution formed after washing creates an extremely dirty wastewater (Rajendran et al. 2022). Hence, it could be hypothesized that the usage hybrid treatment processes are more logical to overcome this problem. The combination of soil washing and advanced oxidation process such as Fenton and Sono-Fenton detemplation leads to more hydroxylated surfaces, with possible advantages for adsorption and catalyst preparation strategies was beneficial for utilization a sustainable solution on behalf of the green technology. The hydroxyl radicals have shown considerable selectivity in the elimination of different types of organic pollutants. It can be hypothesized that the cost-effectiveness of the whole soil washing process would be improved when the target pollutant (Chlorpyrifos, for example) is selectively removed by Fenton and Sono-Fenton processes.

In the present study, the soil washing studies were carried out using linear alkylbenzene sulfonates (LASs) and Tween 85 (TW 85) surfactant for agricultural soil contaminated with chlorpyrifos. The effects of the liquid/solid ratio, surfactant concentration, operation time, temperature and rotational speed parameters on the removal efficiency were investigated. Recovery of the washing solution obtained at the optimal conditions was carried out using advanced oxidation methods such as Fenton and Sono-Fenton. These processes are perhaps one of the most effectual methods for the removal of refractory pollutants.

Materials and methods

Sample preparation and analysis

The agricultural soil used in the study was obtained from 20 cm below the surface where the 38°12′13.5″N and 34°09′25.7″E points were specified. The soil samples were dried at 70 °C for 5 days after being passed through 50 mesh sieve analysis. Some of the dried samples were reserved for soil characterization studies, while the others were stored in airtight plastic boxes. The organic matter was determined by the loss on ignition method with a pH of 6.3. The main parameters were measured deal with methods such as: pH (ASTM D4972), bulk density (ASTM D7263), moisture (ASTM D2216), electrical conductivity (ISO 11265:1994), nitrogen and phosphorus (SM 4500-P J). The pH, electrical conductivity and other measurements were made by WTW pH330i/SET with different probes.

Table 1 Main parameters of the soil sample

Parameter	Unit	Value
pH	–	6.3
Bulk Density	g/L	65
Organic Matter	%	95
Moisture	%	45
Electrical Conductivity	μs/cm	335
Nitrogen	g/L	140
Phosphorus	g/L	160
Potassium	g/L	180

The characterization of the soil is given in Table 1.

The experiments were briefly as follows: 100 ml of 20 mg/L chlorpyrifos solution was added to a 100 g sample of dry soil. The mixture was mixed and stirred for 3 h to obtain homogeneous mixture. In order to examine the homogeneous distribution of the chlorpyrifos in the soil samples (three soil samples) were taken from different points, and recovery studies were carried out using acetonitrile and propionic solvents (20 mL solvent were used in experiments). The mixture was mixed and stirred for 3 days to obtain homogeneous mixture and evaporation of the solvent. The mixing and centrifuging studies were carried out by BIOSAN Multi RS-24 and Beckman Allegra X12 Centrifuge. The liquid–liquid extraction (LLE) (the extraction was made by BIOSAN Multi RS-24) method was selected and used for GC analysis (Shimadzu GC-2010 -GCMS-QP2010plus). An average of 77.78 ± 2.1% recovery was obtained when the acetonitrile solution was used, while 86.86 ± 2.4% recovery was achieved when the propionic solution was used.

The chlorpyrifos (organic phosphate pesticide) was purchased from Sigma-Aldrich. The chlorpyrifos and (isopropyl/ultrapure water) mixture (30:70) was dissolved for 24 h using a magnetic stirrer followed by a 40 °C hot water bath. The prepared solutions were injected into a gas chromatography (GC) using a micro syringe. The equation of the chlorpyrifos solution was created using GC and was linear ($y = 0.0162x + 0.0088$ $R^2 = 0.09996$). Limit of detection

Table 2 Operating conditions of the GC device

Carrier gas rate	1.0 mL/min
Carrier gas	Ultrapure Helium
Sample volume	1 µL
Injector temperature	250 °C
Auxiliary heater Temperature	230 °C
Temperature program	60 °C for 2 min, programed from 60 to 180 °C at 20 °C /min, and then increased at a rate of 10 °C /min 280 °C

Table 3 Different experimental conditions

Surfactant concentration (g/L)	0.5,1.0, 2.5, 5.0, 10.0, 15.0 and 20.0
Liquid/solid ratio	20/0.5, 20/1, 20/1.5, 20/ 2, 20/2.5 and 20/3
Operation time (min.)	15, 30, 60, 180, 360, 720, 1440, 2160, 2880 and 4320
Rotational speed (rpm)	60, 80,120 and 150
Temperature (K)	293, 298, 308 and 313

(LOD) and limit of quantification (LOQ) values for Chlorpyrifos were 0.273 and 0.909 µg/L. The chlorpyrifos pesticide was analyzed in a Shimadzu QP2010 Plus GC–MS device equipped with a DB-5MS capillary column. The conditions of the method for analysis are given in Table 2.

Soil washing test

Preliminary studies of the Tween 85, Span 80 and LAS surfactants were carried out in order to select the most suitable surfactant for the soil washing studies (Bolan et al. 2023; Buckley et al. 2022; Nagtode et al. 2023). The three surfactants were washed for 72 h under the same conditions, and after being mixed for 10 min at 3750 rpm, the upper liquid was filtered through 0.45 µm filters and then measured using the GC–MS. As a result of the preliminary studies, the most suitable surfactants were determined as LAS and Tween 85. Batch experiments were carried out to investigate the effect of different parameters on increasing the contaminated soil with LAS and Tween 85. The experimental studies were carried out in 50 mL glass bottles. In order to determine the optimal conditions for the washing process, the different experimental conditions are shown at Table 3.

Recovery of the washing solution with the advanced oxidation process

Recovery efforts were carried out for the solutions obtained at the end of the soil washing process, which was carried out under the optimal conditions for both surfactants. Fenton and Fenton + ultrasound advanced oxidation methods were used. The experiments were carried out to optimize pesticide removal under different Fe (II), frequency and H_2O_2

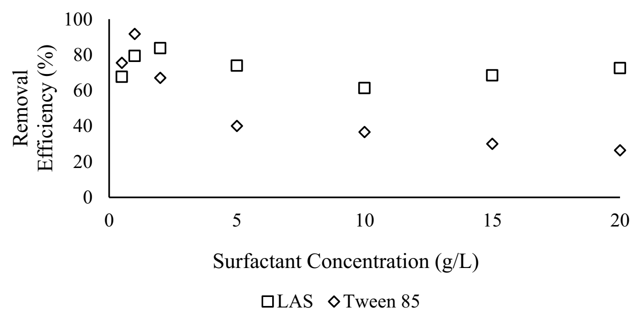

Fig. 1 Determination of the surfactant concentration for LAS and Tween 85

concentrations both with and without indirect ultrasound. In all of the experiments, the working time was kept constant for 10 min and the operating temperature at 293 K. The ultrasonic system used in this study was a power consumption of 100 W and was equipped with a heating power of 75% (Kudos, LHC Heating). All of the chemicals purchased from Merck were used as received.

Results and discussion

Effect of surfactant concentration

Due to their low cost, hypotoxic properties and high solubility, surfactants are widely used in soil washing of organic impurities (Tao et al. 2020). Studies were carried out with previously determined surfactant concentrations at room temperature, at a mixing speed of 120 rpm, a rate of 1/20

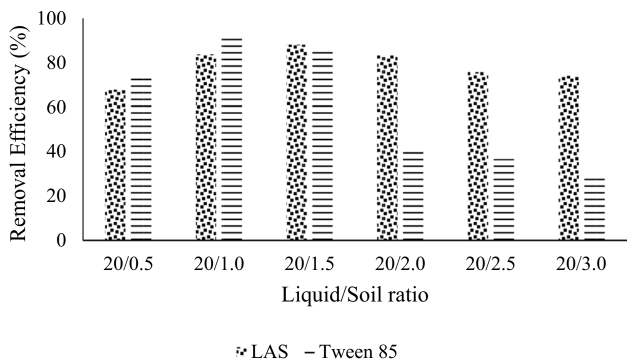

Fig. 2 Determination of the liquid/solid ratio for **a** LAS and **b** Tween 85

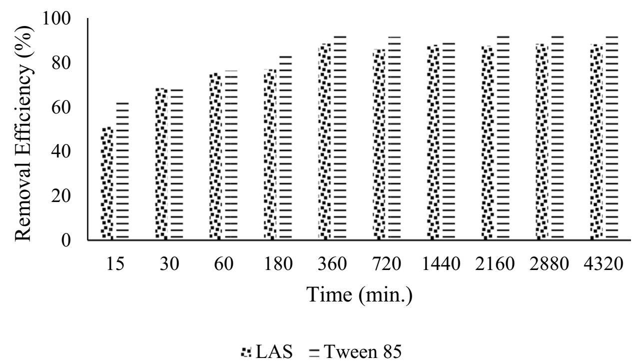

Fig. 3 Operation time for **a** LAS **b** Tween 85

(solid/liquid), for 4320 min. The optimal concentrations for LAS and Tween 85 are given in Fig. 1.

The removal efficiency of 2.5 g/L LAS surfactant was found to be 83.73%, and this was the highest efficiency obtained among the other investigated concentrations. It efficiently removed pesticide contamination in the soil of low concentrations. Some researchers obtained similar results in their study for the removal of the atrazine pesticide. High surfactant concentrations caused a decrease in removal efficiency (Dos Santos et al. 2015).

Higher removal efficiencies were obtained at low Tween 85 concentrations. 91.71% removal efficiency was observed for the concentration of 1 g/L Tween 85. Increasing concentrations led to a gradual decrease in removal efficiency. It was determined that the removal efficiency decreases up to 26.42% at the concentration of 20 g/L surfactant.

When both surfactants were compared, it was determined that higher removal efficiencies were obtained at lower concentrations of Tween 85 surfactant. The main reasons for this are the higher molecular weight of Tween 85 and its ability to form more bonds with chlorpyrifos. As mentioned in the review of literature, a study mentioned the using a large amount of surfactant may causes a back-adsorption effect on pollutant, resulting in unsuccessful removal efficiency (Ren et al. 2023). The selection the surfactants and determining the surfactant concentrations were critical issue to help the providing an advantages for sustainable removal of target pollutant (Silva et al. 2021).

Effect of liquid/solid ratio

Liquid/solid ratio is another parameter that affects removal efficiency. The rate to be determined varies according to the pollutant status and soil structure, but it is often applied without sufficient consideration. A wide range of liquid/solid ratios (1/1–100/1) have been reported in the literature (Gautam et al. 2020). The effect of liquid/solid ratio on removal efficiency at optimal surfactant concentrations is given in Fig. 2 for both washing solutions.

88.22% removal efficiency was obtained in the washing processes with 1.5 g of agricultural soil. The increase in the amount of soil in the washing solution caused the removal efficiency to decrease to 74.13%. The ratio of 20/1.5 (liquid/solid) was found to be optimal. Some researchers observed similar results in the removal of heavy metals found in agricultural soils and mine soils contaminated by soil washing method (Wang et al. 2020).

91.71% and 86.60% removal efficiencies were obtained in 20/1–20/1.5 (liquid/solid) ratio for Tween 85. The increase in the amount of agricultural soil has led to a decrease in the efficiency of the washing process. As a result of this, washing process could not be carried out effectively and chlorpyrifos was attached to the agricultural soil and could not pass into the surfactant. These results and findings also matched those mentioned in some studies, the number of micelles generated in the surfactant solution with optimum liquid/solid ratio that was lower than the effective critical micelle concentration (CMC) required for target pollutant removal from the soil media. The occurrence of a adequate amount of micelles in the surfactant would also solubilize higher amounts of chlorpyrifos and increase removal efficiency (Mirzaee and Sartaj 2022).

Effect of operation time

One of the most important parameters to be optimized in soil washing is the operation time. Washing time affects the degree of adsorption and washing efficiency (Cheng et al. 2020). It is necessary to examine the operation time with extensive studies. For both washing solutions, removal efficiencies from 15 to 4320 min are given in Fig. 3.

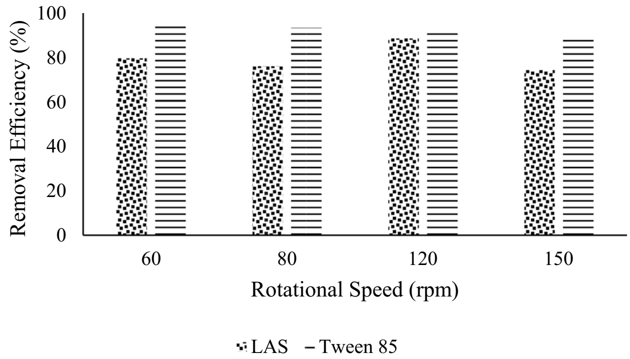

Fig. 4 Determination of rotational speed for LAS and Tween 85

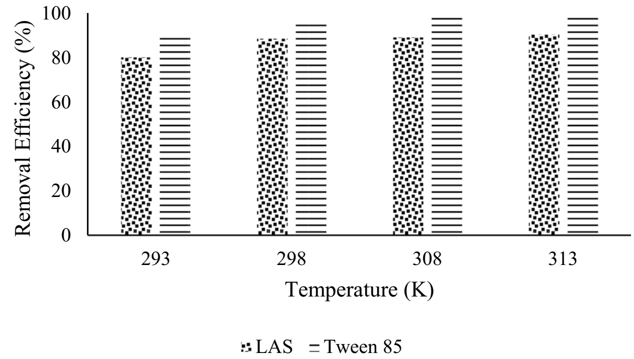

Fig. 5 Determination of operation temperature for LAS and Tween 85

It was determined that removal efficiencies are around 70% in low operation times. It was determined that the removal efficiency at the end of the 360 min of operation time was 88.51%, which was the optimum. Although the further increase of the operation time caused both an increase and decrease in removal efficiency, at the end of 4320 min of operation, 88.22% removal efficiency was achieved.

In the soil washing process where Tween 85 is used, the optimal operation time has been determined as 360 min, which results in 92.78% removal efficiency. The higher operation times on removal could not be effective.

It was observed that the percentage of chlorpyrifos removal in both surfactants increased rapidly for up to 60 min and then remained at more stable values. The 360-min washing period in which steady state effect was present was chosen as the optimum. Some scientists reached the approximately same washing times in their studies investigating the removal of target pollutants using soil washing methods (Ma et al. 2023; Offiong et al. 2023). The operation time values may be controlled up to a limited value, a higher values cannot be effective for removal and recovery (Silva et al. 2021).

Effect of rotational speed

In the washing processes, it was ensured that the mixing speed creates turbulence in the solution by means of causing random and changing speeds. Figure 4 shows the effect of mixing speed on removal efficiency.

Increasing mixing speeds generally leads to an increase in removal efficiency, but when it goes above a certain level, a decrease in removal rates was observed. High mixing speeds help to separate absorbed contaminants (Befkadu and Quanyuan 2018). Increasing the rotational speed to 120 rpm depending on the fluidity of the LAS washing solution caused it to be applied to the surface more effectively.

The viscous nature of the Tween 85 washing solution caused it to slide over the agricultural soil and form a mixture. For this reason, increasing the rotational speeds caused decreases on the removal efficiency. While the optimal rotational speed was determined as 60 rpm, the removal efficiency was 95.44% (Fig. 4).

Effect of operation temperature

The effect of temperature on removal efficiency in soil washing operations performed at 4 different operating temperatures is given in Fig. 5.

It was determined that the removal efficiency was 90.44% at the highest operating temperature level (313 K). This is mainly due to the decrease in tensile force between surfaces (Fanaei et al. 2020). When considered economically, the optimal operating temperature was found to be 298 K.

A similar situation was observed for the Tween 85 surfactant. The removal efficiency, which was 89.83% in the Tween 85 soil washing process carried out at room temperature, changed to 98.07% when the temperature was increased to 313 K. When examined in terms of applicability and economic evaluation, the optimal operating temperature was accepted as 298 K. These findings corroborate the ideas of some researchers (Ren et al. 2023).

Recovery of the washing solution by Fenton

The Fenton process or Fenton-type process is based on the reaction between iron ions and hydrogen peroxide to produce hydroxyl radicals at ambient temperature. Among advanced oxidation processes (AOP), the Fenton-type process is an effective method for the removal of contaminants. The Fenton process has several advantages such as being safe and environmentally-benign nature of reagents, having relatively

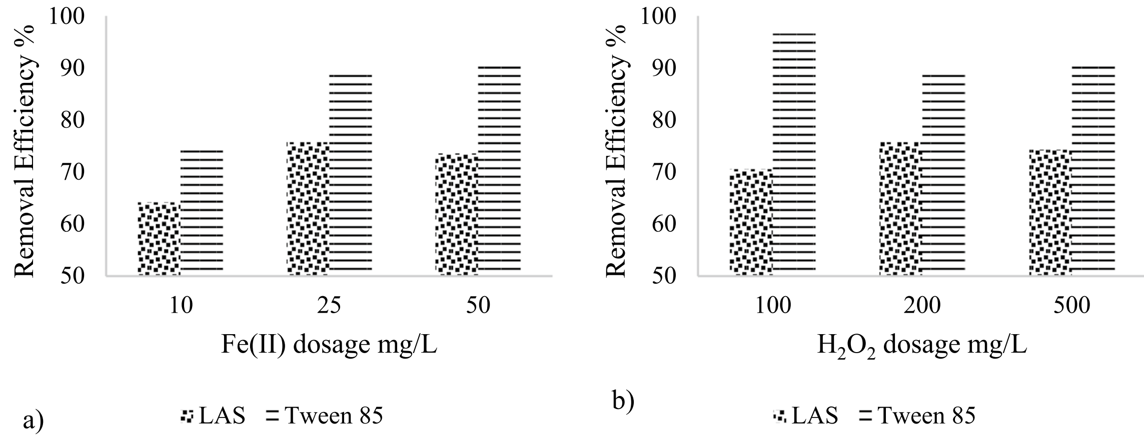

Fig. 6 Effect of **a** Fe (II) dosage **b** H_2O_2 dosage for LAS and Tween 85

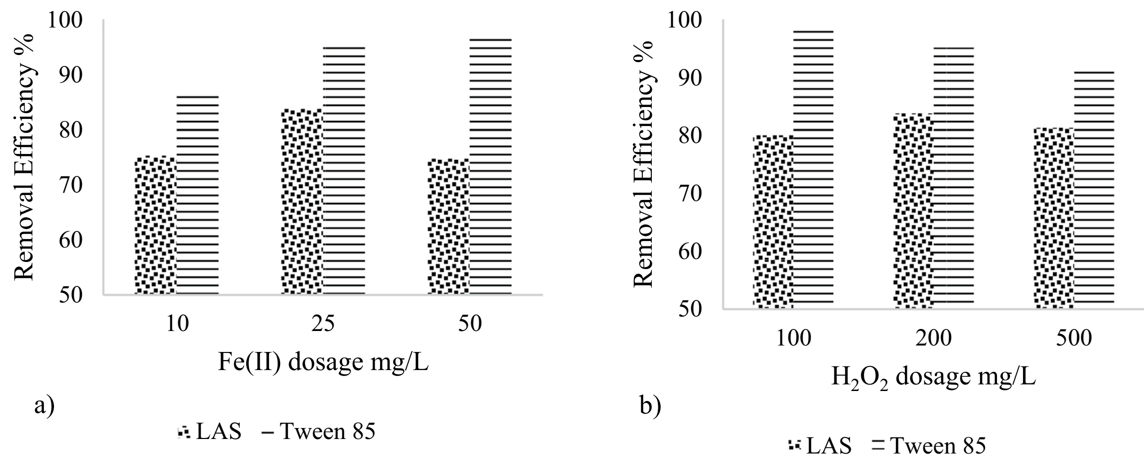

Fig. 7 Effect of **a** Fe dosage **b** H_2O_2 dosage for LAS and Tween 85 (53 kHz)

simple operating principles, short reaction time and no mass transfer limitations (Malakootian et al. 2020). The dosage of the ferrous ion is one of the main operating parameters that determines the treatment efficiency and operating costs of the Fenton process, as ferrous ion produces OH● by catalytically decomposing hydrogen peroxide (Özdemir et al. 2011). The increasing trend is due to the fact that higher ferrous dosages can form more OH● through the Fenton reaction, thus leading to a higher removal rate. Therefore, the resultant reactive oxidants, such as hydroxyl radicals, eventually promote the chemical oxidation process to degrade biologically and chemically recalcitrant pesticide compounds in soil at circum-neutral pH (Reddy and Kim 2015). Hydrogen peroxide plays a vital role in the Fenton oxidation process, as it is the source of OH●. However, an excess amount of hydrogen peroxide not only reduces the treatment efficiency, but also raises the cost of treatment, as it is the main expense. H_2O_2 concentration has a significant effect because excess H_2O_2 can destroy the newly formed hydroxyl radicals causing a reduction in the performance of the process.

After determining the optimal conditions for both washing solutions, studies of recovering the washing solution water were carried out by Fenton advanced treatment method. In order to determine the Iron (II) concentration, 200 mg/L hydrogen peroxide (H_2O_2) was kept. In the recovery studies for Iron (II) concentrations, the effects caused by the solution resulting from the washing processes with LAS are given in Fig. 6.

The highest removal efficiency was obtained in 25 mg/L Fe (II) concentration. H_2O_2 concentration determination studies were made by keeping this value constant (Fig. 6).

In the concentration of H_2O_2, it was determined that increasing the ratio increases the removal efficiency. When the removal efficiencies were examined, it was determined that high concentrations were not feasible. The optimal H_2O_2 concentration was determined as 100 mg/L. In the Fenton study with 25 mg/L Fe (II) and 100 mg/L H_2O_2 made for the recovery of the washing solution with LAS, the removal efficiency was obtained as 70.54%. The studies were carried out under the same advanced oxidation conditions for washing solutions made with Tween 85. Iron (II) concentration studies are given in Fig. 6.

In the case of adding Fe (II) at concentrations of 10 mg/L, 25 mg/L, 50 mg/L, removal efficiencies were 84.96%, 89.81%, 91.32%, respectively. 25 mg/L was chosen as the optimal Fe (II) concentration. Figure 7 shows the studies for the determination of the H_2O_2 concentration.

After adding 100 mg/L H_2O_2 to the washing solution, the recovery efficiency was determined as 96.60%. For the washing solution formed as a result of soil washing process with Tween 85, it was determined that the recovery with Fenton is higher than the one with LAS. The amounts of radical producers reagents such as Fe and H_2O_2 dosages were critical. Additionally, a high concentration of surfactant at the advanced oxidation section is undesired so it can make the process insufficient owing to the removal efficacy. There are several possible explanations for such a result. One of them, surfactant micelles can be a layer toward radical oxidation of target pollutant and the other explanation was, the competition between the surfactant and contaminants (Checa-Fernández et al. 2023; Garcia-Cervilla et al. 2022).

Recovery of the washing solution by Sono-Fenton

The removal efficiency is significant with increasing acoustic power. The greatest overall removal of the pollutant was observed at 53 kHz. An almost 1.2-fold increase was observed in removal efficiency at 53 kHz, in comparison to 35 kHz. The increasing frequency of the ultrasonic equipment reduced the pollutant degradation (Eren 2012).

As hydrogen peroxide and iron doses increase, Fe^{+2} ions were reduced in the presence of hydrogen peroxide and OH● radicals were formed, thus optimal conditions are determined as seen in Fig. 7.

After the predetermined concentrations of Fe (II) and H_2O_2 were added to the LAS washing solution, an advanced ultrasound oxidation method was applied at a frequency of 53 kHz. In the ultrasound method, the internal temperature of the tank was determined as 25 °C and the operation time as 10 min, during the reaction. Recovery percentages

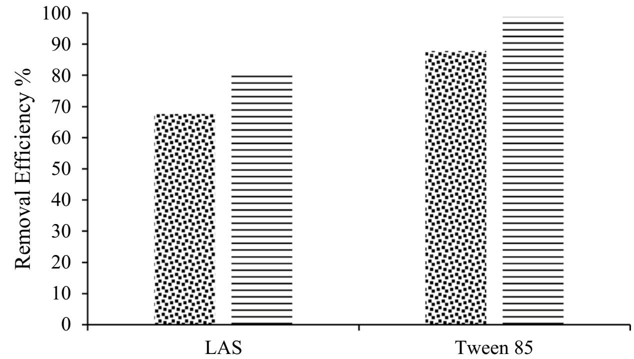

Fig. 8 Effect of ultrasound frequency for LAS and Tween 85 (optimum conditions)

obtained according to Fe (II) concentrations as a result of ultrasonic processes are given in Fig. 7.

When 25 mg/L Fe (II) was added to the LAS washing solution and advanced ultrasound treatment method with 53 kHz was applied for 10 min at room temperature, a recovery efficiency was obtained as 83.75%. In advanced ultrasound treatment, the optimal Fe (II) concentration, with Fenton, was determined as 25 mg/L. The results of the study done for the determination of the concentration and recovery efficiency for H_2O_2 are given in Fig. 7.

In studies for Tween 85 washing solutions performed with 53 kHz ultrasound, recovery percentages obtained for Fe (II) and H_2O_2 concentrations are given in Fig. 7.

Although the removal efficiency increases with increasing iron concentrations, due to the problem of precipitation of excess iron doses, the optimal dose was chosen as 25 mg/L. However, the case with hydrogen peroxide is not the same. When given an excessive dose of hydrogen peroxide, it reacts with itself and disintegrates. Therefore, the removal efficiency was low at high doses (Fig. 7).

The effect of 35 kHz ultrasound treatment on the recovery efficiency was determined by using the optimal Fe (II) and H_2O_2 concentrations for 53 kHz advanced ultrasound treatment. The results of the advanced ultrasound treatment method with 35 kHz, which was applied by adding 25 mg/L Fe (II) and 100 mg/L H_2O_2 to both LAS and Tween 85 washing solutions and where the internal temperature of the tank was 25 °C and the operation time was 10 min. As a result of the advanced ultrasound treatment with low frequency (35 kHZ) performed after the Fenton process, it was determined that the recovery was 67.64% in the LAS washing solution and 87.76% in the Tween 85 washing solution (Fig. 8). It was observed that low ultrasonic frequencies decreased the recovery efficiency for both washing solutions.

As mentioned in the review of the literature, The incline in the removal efficiency with the oxidant dosage (Fe (II) and H_2O_2 dosage, etc.) was mainly depends on the rise in the number of produced radicals with the high amount of oxidant dosage, which assisted the removal of target pollutant. Even though, the removal efficiency went on to increase when the oxidant dosage was increased up to limited level, the target removal efficiency stayed stable. This situation may be attributed to the generation of excess iron amount that leads to quench radicals (Sun et al. 2023) and this hypothesis are also matched with our experimental results.

Conclusion

Remediation of chlorpyrifos was utilized via two surfactants (LAS and Tween 85) enhanced soil washing and Fenton/Sono-Fenton advanced oxidation processes. According to the results, it was found that the operational time, surfactant concentration, liquid/soil ratio, rotational speed and temperature have a positive impact on pollutant desorption from soil. Comparison with two surfactants Tween 85 and LAS, Tween 85 surfactant was found to be more effective washing solution than LAS surfactant.

The advanced oxidation was studied using different oxidation processes, between which the Fenton and Sono-Fenton systems showed noteworthy benefits with approximately 75.7% (for LAS) 96.6% (for Tween 85) and 80% (for LAS) and 98.7% (for Tween 85) chlorpyrifos removal. In these oxidation processes, the oxidant dosages (Fe (II) and H_2O_2 dosages) and ultrasound frequency (for Sono-Fenton process) were critical.

In this study, it was determined that the chlorpyrifos pesticide residue in the soil can be effectively purified by soil washing method. Meanwhile, the solutions obtained from soil washing method can be cleaned up and recovered by Fenton/Sono-Fenton oxidation processes.

All of the results show that full-scale remediation studies can be done based on this study that prove a promising future horizon for hybrid technologies. Further studies of combined processes that account for different variables (different type of pesticides, different surfactant types, different operational parameters and different advanced oxidation processes, etc.) need to be undertaken.

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Author contributions All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by EB and ŞT. The first draft of the manuscript was written by EB and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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Declarations

Conflict of interest The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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