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Removal of α – and β – Endosulfan from Soils by **Using Natural and Synthetic Surfactants**

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Authors' contributions

This work was carried out in collaboration between all authors. Author IO carried out the endosulfan analysis and wrote the first manuscript version. Author MAAC developed the experimental study. Author LGT coordinated the whole work and participated in the correction of the paper. All authors read and approved the final manuscript.

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Original Research Article

ABSTRACT

Aims: The aim of this work was studying a process of surfactant-assisted soil washing for the remediation of a soil spiked with α - and β -endosulfan (6,7,8,9,10,10-Hexachloro-1,5,5a,6,9,9ahexahydro- 6,9-methano-2,4,3-benzodioxathiepine-3-oxide).

Place and Duration of Study: The work was carried out at UPIBI-IPN during 2016.

Methodology: An agricultural soil was collected and spiked with a commercial pesticide. Ten surfactants (nonionic, ionic, cationic, zwitterionic and natural) were selected for washing the soil using concentrations ranging between 0.001 to 0.2% w/w for each surfactant. Residual endosulfan concentrations were evaluated before and after washing soil process. Moreover, the extraction efficiency was related to surface tension and critical micellar concentration (CMC) of every surfactant.

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Results: Better extraction efficiency was observed for α -endosulfan, its average extraction efficiency was around 73% and 55% for β-endosulfan. These values were significantly higher (up to 40%) than those observed in control experiments (water without surfactant). The maximum extraction of α-endosulfan was 90% and it was obtained using canarcel 20 at 10 times CMC. On the other hand, the maximum extraction of β-endosulfan was around 80% when surfactant polafix LO was used at a concentration of 700 times CMC, indicating that the surfactant-assisted soil washing process was suitable for extraction of pesticides from polluted soil. However, the wastewater produced should be treated in a subsequent process.

Conclusion: Natural surfactants did not show CMC in the range of concentration tested. Guar gum did not show a reduction of surface tension, even when concentration increases up to 1200 mg/L. In the case of mesquite seed gum, the surface tension slightly decreases from 73 to around 65 dyn/cm. Tween 80, canarcel 20 and emulgin W-400 showed a CMC of 65, 60 and 10 mg/L. CMC of dehyquart A, surfacpol A and texapon KD was of 160, 250 and 900 mg/L, respectively. Considering all surfactants, the extraction obtained of α-endosulfan is in the range of 65 to 94% with a mean of 79%, while extraction of β-endosulfan was in the range of 41 to 80%, with a mean of 61%. Accordingly to the nature of surfactants, best extraction efficiencies were obtained as follows nonionic > nonionic natural > ionic. Best extraction efficiency of α-endosulfan was 94% obtained with canarcel 20 (C* of 11.6) while for β-endosulfanthebest extraction efficiency was 80% obtained using canarcel 20 (C^* of 11.6) and guar gum (383 mg/L). This information is highly valuable for designing a soil washing process for treatment of pesticide-polluted soil using natural compoundsas surfactants.

Keywords: Endosulfan; soils; surfactants; soil washing; critical micellar concentration.

1. INTRODUCTION

Pesticides have been used from early last century for controlling pests. The major source of pesticides in the environment is aconsequence of agricultural activities. Particularly, higher levels of pesticides in soils can be the result of spills and accidents involving pesticide handling that take place on farms, pesticide formulating and manufacturing plants [1]. Furthermore, thepresence of high concentration of could be also associated withtheindiscriminate use of pesticides, overtaking the natural capacity of soil microorganisms to eliminate them.

Among the organochlorine pesticides, endosulfan was one of the most widely used until its inclusion in the Annex A of the Stockholm Convention in 2011. This indicates that parties

must take measures to eliminate its production and use with specific exemptions [2]. Commercial endosulfan contains two stereoisomers, α and β, known also as endosulfan I and II in a 7:3 ratio (Fig. 1). Endosulfan and its transformation product, endosulfan sulfate are between the main pesticides detected in air and soil in Mexico [3-5].

Surfactants can be added to pesticidecontaminated soils to enhance the treatment efficiency of soil washing. The ability of surfactants to enhance the water solubility of hydrophobic organic compounds (HOCs) hydrophobic organic compounds provides a potential mean of improving the treatment efficiency of *ex-situ* soil washing systems for remediating pesticide-contaminated soils [6,7]. This treatment has been used for cleaning-up polyaromatic compounds, diesel and

Fig. 1. The molecular structure of a)endosulfan and b) β-endosulfan

crude oilcontaminated soils [8-10]. The removal of DDT (an organochlorine pesticide) also has been studied,in a washing process with a spiked soil, in a bioremediation system with biosurfactant-producing microorganism and in a remediation process using foam flushing [10-13]; while Wang and Keller [6,11] reported the treatment of atrazine and diuron contaminated soils using an anionic surfactant.Mixed systems of bacterial population with biosurfactant have been also studied for degradation of organophosphate pesticides [14].

It is well known that surfactants form aggregated structures called micelles above a certain aqueous surfactant concentration, called Critical Micellar Concentration (CMC) which is defined as the concentration where the surface tension of surfactant solution reaches a limiting constant value [15]. In aqueous solutions, surface tension decrease with theaddition of surfactant and CMC is the point where reaches constant surface tension [16]. CMC is a variable that is used as areference. This value is determined by diluting surfactant in distilled water, but this variable can change in presence of solids such as soil particles [17].

Natural surfactants are surface-active compounds synthesized by a variety of microorganisms or contained in seeds, which has been studied as an alternative to removing organic and inorganic contaminants [18-20]. Additionally, removal of pollutants by washing surfactant solution has been demonstrated by pilot-scale [21,22]. More recently, in our previous works, we reported the soil washing of a soil contaminated with 2,4-D using different surfactants in a turbine-agitated acrylic tank and the elimination between 63 and 98% of the initial concentrations of methyl parathion by surfactantenhanced soil washing and treatment of the produced wastewaters in an aerobic-submerged biofilter [23,24].

The aim of this work was to evaluate the surfactant-enhanced soil washing of soils spiked with α and β -endosulfan as a remediation process.

2. MATERIALS AND METHODS

2.1 Soil Characterization and Contamination

Soil employed along this work was collected from a farm, which produced chili. The site is localized in the Estate of Hidalgo, México at 20°10'14.19"N and 99°18'40.77"W, where endosulfan has been used. The soil was collected following NOM-021- SEMARNAT-2000, [25] it was collected superficial soil (0-5 cm), the sample was placed in a plastic bag and transported and stored at 4°C. The soil was screened through mesh 10 (<2 mm). Some characteristics of the soil sample were carried out, such as texture, pH, water capacity, bulk density and total heterotroph's count, all determinations in accord with Torres et al. [24].

The soil was spiked with a commercial pesticide (Thiodan, BAYER, Mexico containing 33% w/w of α – and B–endosulfan) and allowed to rest at 4°C in a dark environment. The soil was mixed one time by week during one month. At the end of the impregnation process, soils were sampled and the amount of pesticide was measured accordingly to the method explained later in this section. Real concentration was obtained by theanalytical method. Pesticide extraction percent was calculated by thedifference between background concentration, spiked concentration and residual concentration.

2.2 Surfactants

Different surfactants were employed for enhanced soil washing including, nonionic, ionic and zwitterionic surfactants, their characteristics are summarized in Table 1. Nonionic surfactants included tween 80 (DrogueríaCosmopolita, Mexico); canarcel 20 (Canamex, Mexico); emulgin W-400 (Conjunto Lar, Mexico) and two natural surfactants, mesquite seed and guar gums. Two ionic and one cationic surfactants were also tested, surfacpol A and texapon KD and dehyquart A, respectively, all obtained from Conjunto Lar, Mexico. The two zwitterionic surfactants, polafix LO and dehyton KB were obtained from Polaquimia, Mexico. Mesquite seed gum is obtained from a native tree (*Prosopissp*) that grows in arid zones in Mexico [28]. Mesquite seed gum is extensively used in a variety of industrial applications due to their emulsifying, microencapsulation, thickening and stabilizing properties, among others [29]. Guar gum is obtained from the seed of the legume Cyamopsis tetragonolobusis legume *Cyamopsis tetragonolobusis* (Leguminosae), this polysaccharide that has high molecular weight and high viscosity [30,31].

2.3 Surface Tension Measurements

The surface tension of all solutions including the stock was determinate using a tensiometer

Commercial brand	Chemical nature	HLB	CMC	
[CAS number]			mg/L	$mM^{\overline{a}}$
Nonionic:				
Tween 80	Polyoxyethylenesorbitanmonooleate	15	25° ;	0.02^b -
$[9005 - 65 - 6]$	(POE 20)		$(13-65.4)$	0.05
			[16,21,24,26,27]	
Canarcel 20	Sorbitanmonolaureate (POE 20)	8.6	60 ^b	NR.
$[9005-64-5]$				
Eumulgin W-400	Ethoxylatednonylphenol	NR.	10 ^b	0.03 ^b
$[9016 - 45 - 9]$	(POE 6)			
Mesquite	Galactomannan	NR.	NR.	NR.
Guar gum	Galactomannan	NR.	NR.	NR.
lonic:				
Surfacpol A	NR.	NR.	250 ^b	NR.
Texapon KD	Sodium lauryl ether sulphates	NR.	160° ; 1,458 [27]	0.42^{b}
[68585-34-2]				
Cationic Dehyguart A	Cetyltrimetyl ammonium chloride	21.4	900° ; 5500 [27]	0.281^{b}
$[112-02-07]$				
Zuitterionic:				
Polafix LO	Myristyl dimethylamine oxide	NR.	180 ^b	0.53^{b}
$[3332-27-2]$				
Dehyton KB	Propyl Cocoamidopropyl betaine	NR.	180 ^b	0.70^{b}
[4292-10-8]	(CAPB)			

Table 1. Surfactants employed in this work and some of its physicochemical properties

HLB- hydrophile-lipophile balance. CMC- critical micelle concentration. ^a This value depends of the molar mass. ^b This work. POE- Nonionic polyoxyethylene. NR- Not reported

(Model 14814, Fisher Scientific). Calibration was made using distilled water and hexane. A stock solution was prepared to provide a concentration of 0.5% (w/w) of all surfactants except for natural surfactants, which was prepared at 0.1% (w/w). From stock solution, seven solutions were obtained in the range of 0.001 to 0.2% w/w. This procedure has been previously described by Hagenhoff et al. [32]. The values were calculated from the average of four calculated from the average of four replications. CMC was calculated by the interception of two lines drawing in a chart of concentration vs surface tension of surfactants solutions.

2.4 Soil Washing

The soil washing procedure was as follows, 10 g of contaminated soil were put inside 80 mL flask with 40 mL of surfactant solution prepared with distilledwater and shaken during 23 hours at 28ºC. Flasks were closed with plastic caps and covered with aluminum foil to prevent light exposure. Soils were dried at environmental
temperature. Pesticide concentration was Pesticide concentration was assessed in the spiked soil and the washed samples, following the methodology described below.

2.5 Pesticide Extraction and Quantification

Endosulfan was extracted by sonication (US-EPA Method 3550C) [33] using 10 g of soil, the pesticides extraction recovery from soil is above 96% with this methodology. The quantification was performed by gas chromatography (US-EPA Method 8081B) [34] with an electron capture detector (Varian 3400, USA) equipped with a J&W Scientific DB-5 column (Agilent, USA). The temperatures of the detector and injector were 300°C and 250°C, respectively, while oven initial temperature was 160°C and increased up to the final temperature of 240°C, at a rate of 5°C/min. Nitrogen was used as carrier gas.

3. RESULTS AND DISCUSSION

3.1 Soil Characterization

Some characteristics evaluated for this soil are presented in Table 2. As observed, it is mainly a clayed-loamy soil with a pH slightly lower than 7, bulk density is about 2 g/cm^3 , water capacity is above 8% and the total heterotrophs count is in the rage of reported values for a bioremediation process (1x10 6 FCU/g).

Table 2. Soil characterization

3.2 Surfactants Critical Micellar Concentration

3.2.1 Nonionic surfactants

In the case of natural surfactants, they did not show CMC in the range of the concentrations tested (Fig. 2a) this has been also reported by other authors [19]. Guar gum did not show a reduction of surface tension, even when concentration increases up to 1200 mg/L.The natural molecules maybe do not lower the surface tension of water, but they form stable emulsions and helps in the contaminated soil washing process [27]. In the case of mesquite seed gum, the surface tension slightly decreases from 73 to around 65 dyn/cm. These values are comparable to those reported by Torres et al. [27] where thesurface tension of natural surfactants, *i.e*., locust bean gum (LBG), guar gum and a rhamnolipid produced by *Pseudomonas sp* were compared*.* They observed that while the rhamnolipid reduced the water surface tension to values up to 29 dyn/cm (at concentrations around 0.04%), LBG and guar gum reached values of 58 and 59 dyn/cm, respectively (at concentrations of 0.5 and 0.03%). This lack of true micellar formation ofbiosurfactants has been identified as one of their advantages over synthetic surfactants in enhancing biodegradation processes [35].

At Fig. 2b is compared the CMC of tween 80, canarcel 20 and emulgin W-400. These three surfactants showed a CMC of 65, 60 and 10 mg/L. These values can be related to the molecular weight of these compounds since tween 80 is a heavy molecule (1309.68 g/mol) while emulgin W-400 has a mass molar of 308.46 g/mol.

3.2.2 Ionic surfactants

The commercial products, dehyton KB and polafix LO, has the same active substance according to the suppliers in consequence, the CMC was similar for this two surfactants, $~180$ mg/L (Fig. 3a). In Fig. 3b is shown the comparison of CMC of dehyquart A, surfacpol A and texapon KD (160, 250 and 900 mg/L, respectively). CMC values obtained in this work for texapon and dehyquart are one order of magnitude lower than that reported by Torres, et al. [27]. Nevertheless, reported CMC values depends on the specific product purity, the method employed for measurement, etc.

3.3 Extraction of Endosulfan

The initial concentration of α-endosulfan and βendosulfan determinate in theno-washing sample was 0.640 and 0.135 mg/kg, respectively (αendosulfan: β-endosulfan ratio ~5:1). Controls without surfactant using only distilledwater were performed obtaining an average reduction in theconcentration of 60.5% and 39.14% for α- and β-endosulfan. Kumar and Philip [36] found that distilledwater desorbed around 40% of αendosulfan, however in a sandy soil desorption was around 85%, indicating that adsorption of αendosulfan towards soil particles was influenced by the composition of the soil.

Considering all surfactants, the extraction obtained of α-endosulfan was in the range of 65 to 94% with a mean of 79%, while extraction of β-endosulfan was in the range of 41 to 80%, with a mean of 61%, each caseis discussed in the following sections. The lower extraction efficiency of β-endosulfan could be related to physical processes of adsorption into soil. However, Kumar and Philip [36] determined that αendosulfan had higher adsorption capacity than β-endosulfan in four types of Indian soils.

3.3.1 Nonionic surfactants

The extraction efficiency of α - and β -endosulfan with the two natural nonionic surfactants tested is shown in Fig. 4.

Extraction of β -endosulfan was lower than α -endosulfan in all cases but it was not observed a correlation between surfactant concentrations and extraction of both isomers. The highest extraction of α - and B-endosulfan was around 90% and 80%, respectively and they were obtained using 383 mg/L of mesquite seed gum. In the case of gum guar, the maximum extraction of α - and B-endosulfan was obtained using 683 mg/L reaching around 80% and 70%, respectively. These maximum extraction values of α - and β -endosulfan were significantly higher than those values found in controls without surfactant, 60 and 40%, respectively. On the contrary, in the two lowest concentrations tested the extraction efficiencies observed were similar of α - and β -endosulfan were significantly higher to those of the controls. Furthermore, at than those values found in controls without concentrations above of 683 and 383 mg/L of surfactant, 60 and 40%, respectively

concentrations above of 683 and 383 mg/L of mesquite seed and guar gums, it seems that the effect in the extraction was similar or lower to the maximum obtained. to those of the controls. Furthermore, at concentrations above of 683 and 383 mg/L of mesquite seed and guar gums, it seems that the effect in the extraction was similar or lower to the

Fig. 2. Surface tension as a function of nonionic surfactants concentration a) mesquite; guar gum. b) tween 80; canarcel 20; eumulgin W-400. Bars mean standard deviation ean with n=4

Fig. 3. Same as in Fig. 2. a) ◇dehyton KB; ▼polafix LO. b) \Box surfacpol A; ▲ dehyquart A; **texapon KD. Bars m mean standard deviation with n=4**

Fig. 5 shows the extraction efficiency of the other nonionic surfactants used, in this case, it was plotted the surfactant concentration calculated as surfactant concentration divided by CMC. Surfactants concentrations under and above CMC were studied (C*<1 and C*>1, respectively). Fig. 5 shows the extraction efficiency of the other
nonionic surfactants used, in this case, it was
plotted the surfactant concentration C*,

In the case of tween 80 extractions of 86% and 73% for α - and β - isomers, respectively were obtained at a C* of 40.5 and comparable extraction efficiencies were found using eumulgin W-400 at a C* of 15.4. On the other hand, the higher extraction efficiency of both isomers was obtained using canarcel 20 (94 and 80% for α - and β - isomers, respectively) at a C^{*} of 11.6. In the case of tween 80, the extraction efficiency was not related to the surfactant concentration.

It has been previously discussed that under the CMC value of surfactants, the predominant phenomenon is the lowering of the solution surface tension and hence, an increase in the

CMC value, the predominant mechanism is the micellization of pesticide, though the
phenomena of increment in the phenomena of increment in the solubilization.

Fig. 5 shows the extraction efficiency of the other solubilization of the pesticide [1,17]. Under that producined as unfact
ant concentration coincilization of pesticide, though the periodical
collated as unfactant concen Jayashree et al. [37] reported the surfactant washing of soils contaminated with a mixture of α - and B-endosulfan. They employed a natural surfactant (surfactin) and two nonionic synthetic surfactants (tween 80 and triton X-100). The soil was spiked with 10-80 mg/kg of a mixture of α and β -endosulfan. Surfactant concentrations were in the range of 500-2,000 mg/L, for desorption times between 6 and 48 hours. Maximum endosulfan removals were about 91, 85 and 70% for surfactant tween80 and X100, respectively. However, values of removal with controls (using only water) were not reported. on of the pesticide [1,17]. Under that

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is soils contaminated with a mixture of

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On the other hand, Torres et al. [24] reported the washing of a garden soil intentionally

Fig. 4. Extraction efficiency of (███) α-endosulfan and (███) β-endosulfan with nonionic surfactants: a) guar gum; b) mesquite

Fig. 5. Extraction efficiency of (\Box **)** α -endosulfan \quad and (\Box) β-endosulfan with nonionic tion efficiency of (██)α-endosulfan and (██)β-endosulfan with non
surfactants a) tween 80; b)canarcel 20; c) eumulgin W-400. C*= Surfactant **concentration/CMC**

contaminated with methyl-parathion in concentrations of 0.413 and 12.9 mg/kg. Surfactant-enhanced washing was carried out using nonionic, anionic and natural surfactants yielding methyl-parathion removal between 75 and 97% (for the higher methyl parathion concentration). For the lower methyl-parathion soil concentration,
removals between 63 and 87% were removals between 63 and 87% were achieved. Best methyl-parathion removal was observed with locust bean, guar and contaminated with methyl-parathion in
concentrations of 0.413 and 12.9 mg/kg.
Surfactant-enhanced washing was carried
out using nonionic, anionic and natural
surfactants yielding methyl-parathion removal
between 75 and 97% contaminated with methyl-parathion in mesquite seed gums in a concentration of 100
concentrations of 0.413 and 12.9 mg/kg. mg/L.
Surfactant-enhanced washing was carried
out using nonionic, anionic and natural 3.3.2 lonic

mg/L.

3.3.2 Ionic surfactants

The extraction efficiency obtained with ionic surfactants is shown in fig. 6, values ranging from 72-85% and 54-79% for α -endosulfan and -endosulfan were observed. Worst results, in terms of thelower extraction efficiency and thehigher concentration of surfactant added raction efficiency obtained with ionic
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Fig. 6. Extraction efficiency of (███) α-endosulfan and (███) β-endosulfan with ionic **surfactants a) dehyton KB; b)polafix LO; polafix c) dehyquart; d) texapon KD; e) surfacpol A. C*= endosulfan d) Surfactant concentration Surfactant concentration/CMC**

were found with texapon kd while with dehyquart best results were observed. However, these values are lower compared with the extraction with natural nonionic surfactants. Torres et al. [24] reported that texapon 40 at a concentration of 100 mg/l was able to remove 85% and 69% of methyl-parathion form a soil spiked with initial concentrations of 0.413 and 12.9 mg/kg.

4. CONCLUSION

Natural surfactants did not show CMC in the range of concentration tested. Guar gum did not

were found with texapon kd while with dehyquart show a reduction of surface tension, even when
best results were observed. However, these concentration increases up to 1200 mg/L. In the
values are lower compared with the e concentration increases up to 1200 mg/L. In the case of mesquite seed gum, the surface tension slightly decreases from 73 to around 65 dyn/cm. Tween 80, canarcel 20 and emulgin W W-400 showed a CMC of 65, 60 and 10 mg/L. CMC of dehyquart A, surfacpol A and texapon KD was of 160, 250 and 900 mg/L, respectively. Considering all surfactants, the extraction obtained of α-endosulfan is in the range of 65 to showed a CMC of 65, 60 and 10 mg/L. CMC of dehyquart A, surfacpol A and texapon KD was of 160, 250 and 900 mg/L, respectively. Considering all surfactants, the extraction obtained of α-endosulfan is in the range of 65 to endosulfan was in the range of 41 to 80%, with a endosulfan was in the range of 41 to 80%, with a
mean of 61%. Accordingly to the nature of show a reduction of surface tension, even when
concentration increases up to 1200 mg/L. In the
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surfactants, thebest extraction efficiencies were obtained as follows nonionic > nonionic natural > ionic. Best extraction efficiency of α-endosulfan was 94% obtained with canarcel 20 (C* of 11.6) while for β-endosulfan best extraction efficiency was 80% obtained using canarcel 20 (C* of 11.6) and guar gum (383 mg/L). This information is highly valuable for designing a soil washing process for treatment of pesticide-polluted soil usingnatural compoundsas surfactants.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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