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## Human health risk assessment of pesticide residues in snappers (*Lutjanus*) fish from the Navachiste Lagoon complex, Mexico



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### ABSTRACT

Organochlorine pesticides (OCPs) residues were determined in fillets of *Lutjanus colorado*, *L. argentiventris*, and *L. novemfasciatus*. Fillet samples were collected bimonthly from February 2012 to February 2013. OCPs average concentrations do not differ significantly according to size, weight, or season, nor do they relate with the physico-chemical parameters of the sea water. The highest concentration and most frequently encountered OCPs were endosulfan sulfate, δ-HCH, and heptachlor epoxide, which indicates their use in the recent past and confirms their persistence. Average concentrations of  $\sum$ HCHs,  $\sum$ chlordane, and  $\sum$ heptachlor in samples were above cancer MRLs according to data from monthly consumed portions. HCHs and heptachlor are listed in Appendix III of the Rotterdam Convention of chemicals placed on a prior informed consent procedure for import and export purposes; they are considered illegal in Mexico. The OCPs concentrations above cancer MRLs in *Lutjanus* spp. turn its frequent consumption into a human health risk.

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### 1. Introduction

With the Industrial Revolution, the large-scale use of pesticides increased, requiring a greater capacity for their production, storage, as well as a better regulation for food protection. A clear example of this is the use of DDT (dichlorodiphenyltrichloroethane) patented by the chemist, Paul Herman Müller (Müller, 1945). Some organochlorine pesticides (OCPs) are persistent organic pollutants (POPs), and are prohibited or restricted in some countries like Mexico. Among them, dieldrin, endrin, aldrin, lindane, HCHs, and heptachlor are listed in Annex III subject to the Prior Informed Consent (PIC) procedure by the Rotterdam Convention (PIC, 2011). These OCP are usually introduced into the environment after their pest control use in agriculture activities (Cantu-Soto et al., 2011; Chen et al., 2011; Dhananjayan et al., 2012; González-Farias et al., 2002; Mugni et al., 2011; Panuwet et al., 2012; Yu et al., 2014). The continental region of the Gulf of California is surrounded by large areas of agricultural fields and one of the most important is the Guasave Valley, which discharges untreated residues into the adjacent Macapule-San Ignacio-Navachiste Lagoon complex (Gonzalez Farias et al., 2006;

Orduña-Rojas and Longoria-Espinoza, 2006). This complex receives large amounts of OCPs (Frías-Espericueta et al., 2011; Hernandez-Cornejo et al., 2005) through leaching, atmospheric transport, and/or erosion of agricultural soils. In addition, the lipophilic nature of OCPs (Arrebola et al., 2012) enhances their biomagnification and bioavailability (Leblanc, 1995; Mdegela et al., 2009) facilitating their incorporation into the food web of marine ecosystems (Carro et al., 2012; Kalyoncu et al., 2009; Kim et al., 2002; Muralidharan et al., 2009; Ramu et al., 2007; Ueno et al., 2003).

The consumption of OCP-contaminated fish could affect human health (Arrebola et al., 2012). The bioaccumulation of OCPs in sea food products is highly species-specific, due to ecological characteristics such as feeding habits (Qiu et al., 2012; Sundar et al., 2010) and habitats (Botello et al., 2000; Fung et al., 2005). Therefore, the presence of OCPs in the fish edible tissue is an effective environmental pollution-monitoring tool, and serves to evaluate the potential effects on human health. In this sense, snappers (*Lutjanus*) are especially suitable for this purpose as their carnivorous feeding habit makes them particularly vulnerable for the biomagnification of OCPs. It should be noted that this species is an important element in the diets of both the fishing populations around this lagoon complex and of high-income sectors in nearby localities along the adjacent coast. The presence of OCPs in muscle tissue of fish has been determined for other species from this

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region (Aksoy et al., 2011; Marburger et al., 2002; Mdegela et al., 2009; Shi et al., 2011). Therefore, the presence of OCPs in fish fillets constitutes a potential risk for human health (Dhananjayan et al., 2012; Hu et al., 2010; Moon et al., 2009; Perugini et al., 2004; Yu et al., 2010). Based on the afore mentioned, constant consumption of OCPs-contaminated *Lutjanus* spp. fillets can pose a health risk to the people who consume them. Thus, the main objectives of this study were to determine the levels of OCP residues in the fillets of snappers (*Lutjanus* spp.) collected bimonthly from February 2012 to February 2013 from the Navachiste region to evaluate the health risk posed by their consumption.

## 2. Materials and methods

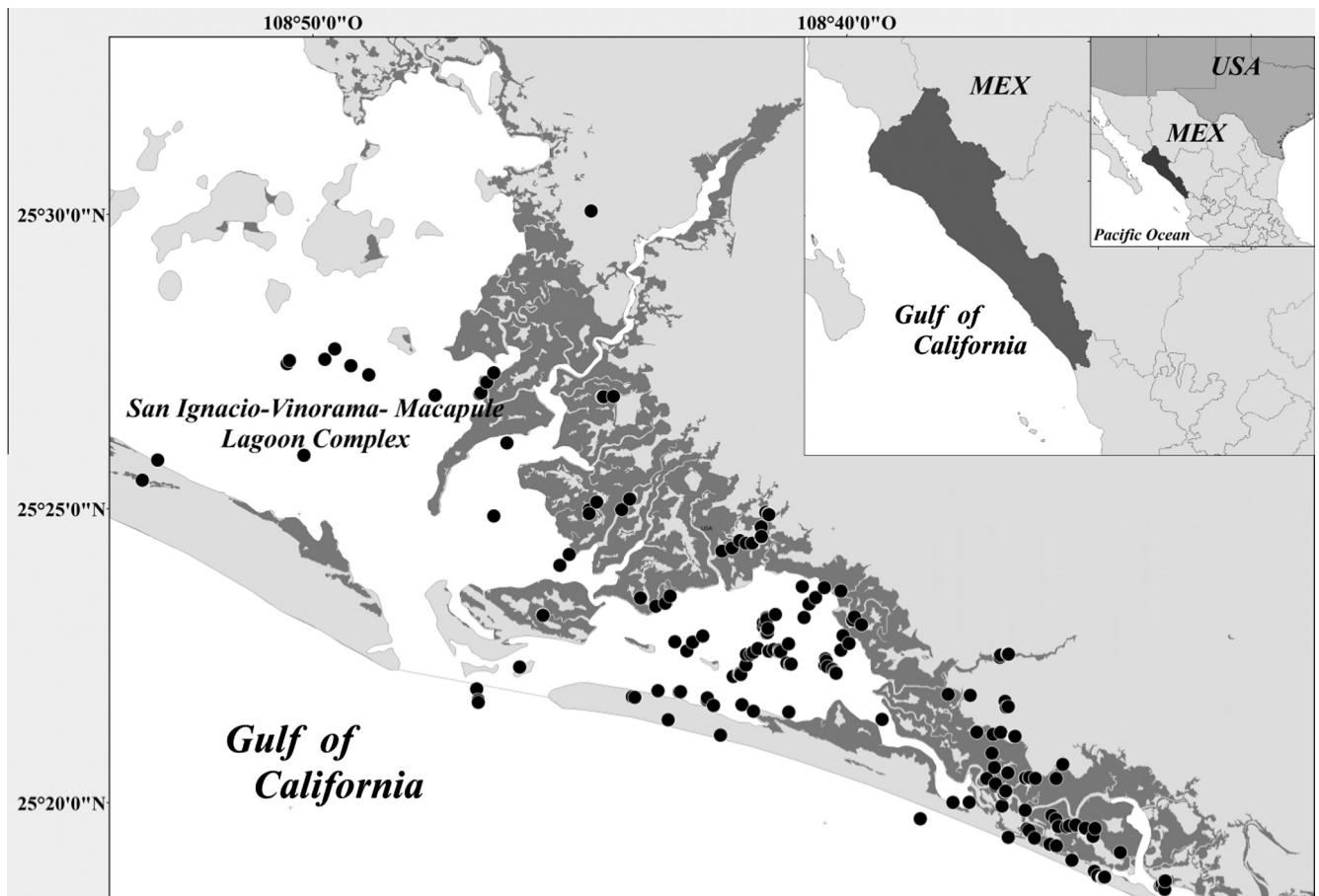
Snappers were collected from the San Ignacio-Macapule-Navachiste Lagoon complex (SIMN) covering a surface area of approximately 24,000 ha in the southwestern area of the Gulf of California, between 25.4° and 25.7° North latitude and 109.02° to 108.55° West longitude (Orduña-Rojas and Longoria-Espinoza, 2006). During the period of study, the following parameters were recorded: water pH, salinity, temperature, and dissolved oxygen, using a Hanna potentiometer®, a Vital Sine® refractometer, and a YSI-55® oximeter, respectively. A total of 110 organisms of the three snapper species, *Lutjanus colorado*, *L. argentiventralis*, and *L. novemfasciatus* were captured bimonthly between February 2012 and February 2013 in the SIMN (Fig. 1). Fishes were dissected to obtain samples of fillets that were then wrapped in aluminum foil, placed into plastic bags, and frozen at -20 °C (-4 °F) until processing.

### 2.1. Extracting OCPs and cleaning the tissue samples

For the extraction and separation of the OCP from the tissue, three 7.5-g portions were taken and ground individually from each sample, and placed in a porcelain pestle with 5 g of sodium sulfate anhydride (FAGALAB®, Cat. 2349-600) and 25 ml of chromatographic grade hexane (EMD®, Cat. MMEMHX02901). After the extraction process, 20 ml of extract (hexane with dissolved OCP) from each portion of tissue was recovered and extracts were then cleaned by passing them through glass columns packed with aluminum (FAGALAB®, Cat. 2269-100), MgO<sub>3</sub>Si (JT BAKER®, Cat. M368-07), SiO<sub>2</sub>, and Na<sub>2</sub>SO<sub>4</sub> (JT BAKER®, Cat. 3405-01) at 4, 4, 4, and 1 g, respectively, activated in an oven at 130 °C (266 °F) for 12 h and deactivated with deionized water (5% by weight). The extracts were then recovered in a 20-ml glass beaker and placed in sand at 50 °C (122 °F) to evaporate the hexane. The samples were re-dissolved in 2 ml of trimethyl pentane (GOLDEN BELL®, Cat. 37740-4000) and transferred to 5-ml vials (Galindo-Reyes et al., 1999).

### 2.2. Determination and quantification of OCPs

From each vial, 2 µl were extracted and injected into a SHIMADZU GC-17A gas chromatograph (GC) equipped with an electron capture detector (ECD), following this program: the initial oven temperature of 99 °C was maintained for 2 min, then it was increased by 8 °C per minute to 290 °C, and held there for 3 min before being reduced back to 99 °C. The temperature of the injector and detector were 260 °C and 310 °C, respectively. A Restek® column (Rtx-5) with chromatographic grade nitrogen (INFRA®) was



**Fig. 1.** Study area and sampling points in the San Ignacio-Navachiste-Macapule Lagoon complex.

used as the carrier gas at a pressure of 4.22 kg cm<sup>-2</sup> and a flow of 31 ml min<sup>-1</sup>. For the GC quality control, an internal standard (2 ng µl<sup>-1</sup> of SUPELCO® standards: EPA 8080 Pesticides Mix, Cat. 4-7913) was prepared and analyzed in the GC equipment before and after sample analyses to ensure OCPs quantity standardization and their identification. Standards included the analytes, α-BHC, β-BHC, lindane, δ-BHC, aldrin, 6-hydroxy-2-naphthyl disulfide, 1,1-dichloro-2,2-bis (4-chlorophenyl) ethane, 4,4'-DDT, dieldrin, α-endosulfan, β-endosulfan, endosulfan sulfate, endrin, endrin aldehyde, heptachlor, heptachlor epoxide and methoxychlor. All samples were analyzed on a wet mass basis.

### 2.3. Statistical analysis

Data were analyzed with SAS®(Ver. 9) and Statistica®(Ver. 7) software and ( $\text{Log}_{10}$ )-transformed for an ANOVA test ( $p < 0.05$ ,  $\alpha = 0.05$ ), and since these were not normally distributed (considering as normal distribution values of  $p > 0.05$ ,  $\alpha = 0.05$  through the Kolmogorov-Smirnov test) and lacked homoscedasticity, data were subjected to a Kruskall-Wallis test ( $p < 0.05$ ,  $\alpha = 0.05$ ). Correlation of OCP concentration levels with weight, size, and physico-chemical parameters (salinity, temperature, oxygen) was determined with the Pearson correlation test ( $p < 0.05$ ).

## 3. Results

OCP concentrations among species, *L. argentiventralis*, *L. novemfasciatus*, and *L. colorado*, (0.002–0.057, 0.001–0.035, and 0.001–0.095 mg kg<sup>-1</sup>, respectively) were not significantly different ( $p < 0.05$ ); hence, the concentrations among these species were considered as samples of one population on a wet mass basis (Table 1).

**Table 1**  
Average concentration of OCPs in three snapper species of the San Ignacio-Navachiste-Macapule Lagoon complex (mg kg<sup>-1</sup>). <sup>a</sup>Mean, <sup>b</sup>(Min-Max), ND: Value below detection limits.

Analyte	Snapper species			Total
	<i>L. argentiventralis</i>	<i>L. novemfasciatus</i>	<i>L. colorado</i>	
α-HCH	0.002 (0.002–0.002)	ND	ND	0.002 (0.002–0.002)
β-HCH	0.003 (0.002–0.004)	0.003 (0.002–0.003)	0.003 (0.003–0.005)	0.003 (0.002–0.005)
δ-HCH	0.017 <sup>a</sup> (0.002–0.057) <sup>b</sup>	0.035 (0.035–0.035)	0.02 (0.002–0.087)	0.028 (0.002–0.18)
γ-HCH	0.002 (0.002)	ND	ND	0.002 (0.002–0.002)
Heptachlor epoxide	0.003 (0.002–0.057)	0.003 (0.001–0.005)	0.004 (0.002–0.026)	0.004 (0.002–0.026)
Heptachlor	0.005 (0.002–0.018)	0.003 (0.003)	0.004 (0.002–0.01)	0.004 (0.002–0.018)
α-chlordane	0.004 (0.002–0.005)	0.003 (0.002–0.003)	0.014 (0.002–0.095)	0.010 (0.002–0.095)
γ-chlordane	0.014 (0.002–0.003)	0.011 (0.011)	0.011 (0.003–0.027)	0.013 (0.002–0.003)
Endosulfan sulfate	0.008 (0.002–0.017)	0.009 (0.001–0.009)	0.007 (0.001–0.025)	0.007 (0.001–0.025)
Endosulfan I	0.002 (0.002)	ND	0.002 (0.002–0.003)	0.002 (0.002–0.003)
Endosulfan II	ND	ND	0.006 (0.003–0.008)	0.006 (0.003–0.008)
4,4'-DDT	0.003 (0.002–0.007)	ND	0.004 (0.002–0.001)	0.004 (0.002–0.001)
4,4'-DDD	0.003 (0.003)	ND	0.002 (0.002)	0.003 (0.003–0.003)
4,4'-DDE	0.003 (0.003)	ND	0.004 (0.003–0.005)	0.004 (0.003–0.005)
Aldrin	0.003 (0.003–0.004)	0.003 (0.003–0.003)	0.004 (0.002–0.006)	0.003 (0.002–0.006)

During February 2012, the OCPs with the highest concentrations were α-chlordane (0.034 mg kg<sup>-1</sup>) and δ-HCH (0.031 mg kg<sup>-1</sup>), whereas in May, these were α-chlordane (0.006 mg kg<sup>-1</sup>) and endosulfan sulfate (0.006 mg kg<sup>-1</sup>). For June, October, and December 2012 these were δ-HCH with 0.022, 0.015 and 0.060 mg kg<sup>-1</sup>, respectively, and γ-chlordane with 0.011, 0.011 and 0.022 mg kg<sup>-1</sup>, respectively. In August 2012 and February, the highest was endosulfan sulfate with 0.016 and 0.005 mg kg<sup>-1</sup>, respectively.

In snapper fillet, the OCPs with the highest concentrations were δ-HCH (0.028 ± 0.041 mg kg<sup>-1</sup>), γ-chlordane (0.012 ± 0.009 mg kg<sup>-1</sup>), α-chlordane (0.001 ± 0.24 mg kg<sup>-1</sup>), and endosulfan sulfate (0.007 ± 0.005 mg kg<sup>-1</sup>). According to frequency, quantifiable endosulfan sulfate, heptachlor epoxide, and δ-HCH reached 72.8%, 34%, and 33%, respectively, whereas quantifiable aldrin, endosulfan I, endosulfan II, α-HCH, 4,4'-DDE, 4,4'-DDD, and γ-HCH were found in less than 10% of samples (Table 2). No significant differences ( $p > 0.05$ ) were observed according to sampling period (Fig. 2).

No significant correlations existed between the physico-chemical parameters of the seawater (temperature, dissolved oxygen, and salinity) and OCP concentrations ( $p > 0.05$ ), although there was a significant correlation of size and mass (0.95,  $p < 0.05$ ) with each species, *L. argentiventralis*, *L. colorado*, and *L. novemfasciatus* (0.57, 0.95, and 0.99, respectively) (Table 3).

The detected mean concentrations were all lower than the maximum residue limits (MRLs) established for these substances in animal edible products (DOF, 1994; EU, 2006; FAO-WHO, 2013) (Table 4).

For the 95th percentile, the averages of the highest exposure were for chlordanes, endosulfan I and II, and DDTs (0.079, 0.055 and 0.044 ng kg<sup>-1</sup>, respectively). The other analytes revealed an average exposure below 0.02 ng kg<sup>-1</sup> (Fig. 3).

**Table 2**Average concentration (AC) ( $\text{mg kg}^{-1}$ , wet weight) and frequency of OCP presence in the samples.

Analyte	February 12	May 12	June 12	August 12	October 12	December 12	February 13	AC ( $\text{ng g}^{-1}$ )	Freq. %
Endosulfan sulfate	0.008	<b>0.006</b>	<b>0.004</b>	0.006	0.007	<b>0.005</b>	<b>0.007</b>	7.71	<b>72.8</b>
Heptachlor epoxide	0.006	0.002	0.002	0.003	0.003	0.004	0.004	6.03	<b>34.0</b>
$\delta$ -HCH	<b>0.031</b>	0.002	0.022	<b>0.015</b>	<b>0.060</b>	<b>0.013</b>	<b>0.028</b>	31.29	<b>33.0</b>
$\beta$ -HCH	0.003	0.004	ND	0.003	0.003	0.003	0.003	2.79	26.2
Heptachlor	0.004	0.002	ND	0.007	0.003	0.004	0.004	4.36	26.2
$\gamma$ -Chlordane	0.010	ND	0.011	<b>0.011</b>	<b>0.022</b>	0.005	<b>0.012</b>	10.04	18.4
$\alpha$ -Chlordane	<b>0.034</b>	<b>0.006</b>	0.003	ND	0.004	0.002	<b>0.010</b>	34.09	14.6
Aldrin	0.006	ND	0.003	0.002	0.003	0.003	0.003	5.77	6.8
Endosulfan I	0.002	0.002	ND	0.003	0.002	ND	0.002	2.13	4.9
Endosulfan II	0.006	0.005	ND	ND	ND	ND	0.006	5.81	3.9
$\alpha$ -HCH	ND	ND	ND	ND	0.002	0.002	0.002	0.01	2.9
4,4'-DDT	0.007	0.004	ND	0.003	0.003	0.004	0.004	6.97	19.4
4,4'-DDD	0.003	ND	ND	ND	0.003	ND	0.003	2.71	1.9
4,4'-DDE	0.003	ND	ND	0.005	0.003	ND	0.004	3.25	2.9
$\gamma$ -HCH	ND	ND	ND	ND	ND	0.002	0.002	0.00	1.0
$\sum$ HCH	0.019	0.004	0.022	0.014	0.050	0.012	0.021	19.28	51.46
$\sum$ DDTs	0.007	0.004	0.002	0.004	0.004	0.004	0.004	6.64	21.61
$\sum$ CHLs	0.020	0.006	0.006	0.010	0.014	0.006	0.012	20.10	56.60
$\sum$ Endosulfan	0.008	0.006	0.004	0.006	0.007	0.005	0.007	7.74	82.33
$\sum$ Heptachlor	0.007	0.002	0.002	0.007	0.005	0.006	0.006	7.17	44.25

Numbers in bold indicate the highest values per period.

According to the Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories (EPA, 2000) quotients of non-carcinogenic risk were below 1 (Fig. 4a); in contrast, quotients of carcinogenic risk were higher than 1 (Fig. 4b).

#### 4. Discussion

The pesticides with the highest concentration in the muscle tissue of the studied population were:  $\delta$ -HCH ( $28 \pm 41 \text{ ng g}^{-1}$ ),  $\gamma$ -chlordane ( $13 \pm 9 \text{ ng g}^{-1}$ ),  $\alpha$ -chlordane ( $10 \pm 24 \text{ ng g}^{-1}$ ), endosulfan sulfate ( $7 \pm 5 \text{ ng g}^{-1}$ ). Previous studies show that HCHs, like lindane and its isomers, are the most frequent OCPs found in organisms and sediments of the Gulf of California (Carvalho et al., 2002). In this sense, the pesticide with the highest concentration in the muscle tissue of the studied snappers was  $\delta$ -HCH, ( $28 \pm 41 \text{ ng g}^{-1}$ ) higher than reported by Reyes-Montiel et al. (2013) for *Mugil cephalus* from the same ecosystem ( $0.003\text{--}0.017 \text{ mg kg}^{-1}$ ) and by Hernández and Hansen (2011) for water and sediments affected by agricultural activities (Table 5).

According to the information on the order of HCH isomer persistence in the environment (Ameur et al., 2013; Kouras et al., 1998), the observed percentage frequency ( $\delta$ -HCH >  $\beta$ -HCH >  $\alpha$ -HCH >  $\gamma$ -HCH) and the concentration in the snappers' muscle tissue ( $\delta$ -HCH >  $\beta$ -HCH >  $\gamma$ -HCH >  $\alpha$ -HCH) suggest a historical contamination due to the use of a technical mixtures of HCH (Montes et al., 2011) at the following proportions: 55–80% of  $\alpha$ -HCH, 5–15%  $\beta$ -HCH, 8–15%  $\gamma$ -HCH, and 2–16% of  $\delta$ -HCH (Willett et al., 1998). In this regard, the presence of HCHs in the snappers of the lagoon complex suggests that insecticides containing HCH are being currently used. The  $\beta$ -HCH is considered as the most persistent and derived from the degradation of  $\alpha$ -HCH (Phillips et al., 2005) and of  $\gamma$ -HCH (lindane), and has a wide spectrum insecticide activity (Li et al., 1998). The presence of  $\alpha$ -HCH and  $\gamma$ -HCH in the snapper fillets indicates their recent application in agricultural activities (Hu et al., 2010; Kalyoncu et al., 2009), which, in this study, involves the Guasave Valley. Also, its persistence is not affected by the oxic and anoxic conditions of the aquatic systems (Wu et al., 1997), as they present low volatility and resistance to microbial degradation (Phillips et al., 2005; Rajendran et al., 2005).

Endosulfan sulfate was the most frequent OCP (72.8%) and the one with the highest concentration in the snapper fillets

( $7 \pm 5 \text{ ng g}^{-1}$ ), followed by endosulfan I and II (5% and 4%, respectively). This indicates the predominance of the degraded form, resulting from the oxidation and/or photolysis of endosulfan I (Montes et al., 2011). Finding the two isomers (I and II) in the snapper fillets indicates that they are still being used as insecticides in the agricultural activity of the state of Sinaloa (Garcia de la Parra et al., 2012), as a technical mixture in a 7:3 proportion (Westbom et al., 2008). Finding it in the snapper fillets is due to the degradation rate of isomer I, which reaches 22 days, whereas that of isomer II is achieved in 8.3 days (Carvalho et al., 2002). Based on the obtained experimental information and the data compiled by Montes et al. (2011) and Reyes-Montiel et al. (2013), we can infer their recent use in neighboring agricultural zones.

Snappers are highly sensitive to these OCPs as they generate in them severe metabolic disturbances by being endocrine disruptors and highly neurotoxic (Ballesteros et al., 2009; Da Cuna et al., 2011; Nowak, 1992; Pérez-Ruzafa et al., 2000; Tellez-Banuelos et al., 2011). However, the samples were obtained from February 2012 to February 2013, it was only in that last year that endosulfan has been subjected to import restriction, and starting in December 2014 its use has been banned by the Mexican government. Hence, future studies in these and other fishes frequently consumed will be necessary to discard or confirm the use of this chemical in the agricultural activity and its anticipated decline.

Heptachlor is one of the OCPs that most rapidly is degraded in aquatic systems; in two weeks it is transformed to heptachlor epoxide (more toxic) and in three weeks it is completely degraded. In this sense, the literature reports very low levels of heptachlor or none at all (Clark et al., 2001). Heptachlor was found at higher concentrations (25%) than heptachlor epoxide (34%) of the studied population, concentrations that are slightly higher than those reported in these species (Rosales and Escalona, 1983; Rueda et al., 1997), indicating its recent use despite its listing in Annex 1 by the Stockholm Convention.

Our results on chlordane differ from previous studies in the region in which this OCP was not detected (Osuna-López et al., 2009). It is a mixture of more than 140 related chemical substances, such as  $\gamma$ -transchlordane,  $\alpha$ -cis-chlordane,  $\beta$ -chlordane, heptachlor, trans-nonachloride (Cuozzo et al., 2012), chlordanes and nonachlorides (Bondy et al., 2003). In addition, its wide use from 1948 to 1988, added to its persistence, accounts for the detection of residues in coastal systems (ATSDR, 2011). In Mexico, this OCP is restricted to be used only as termiticide since 30 years

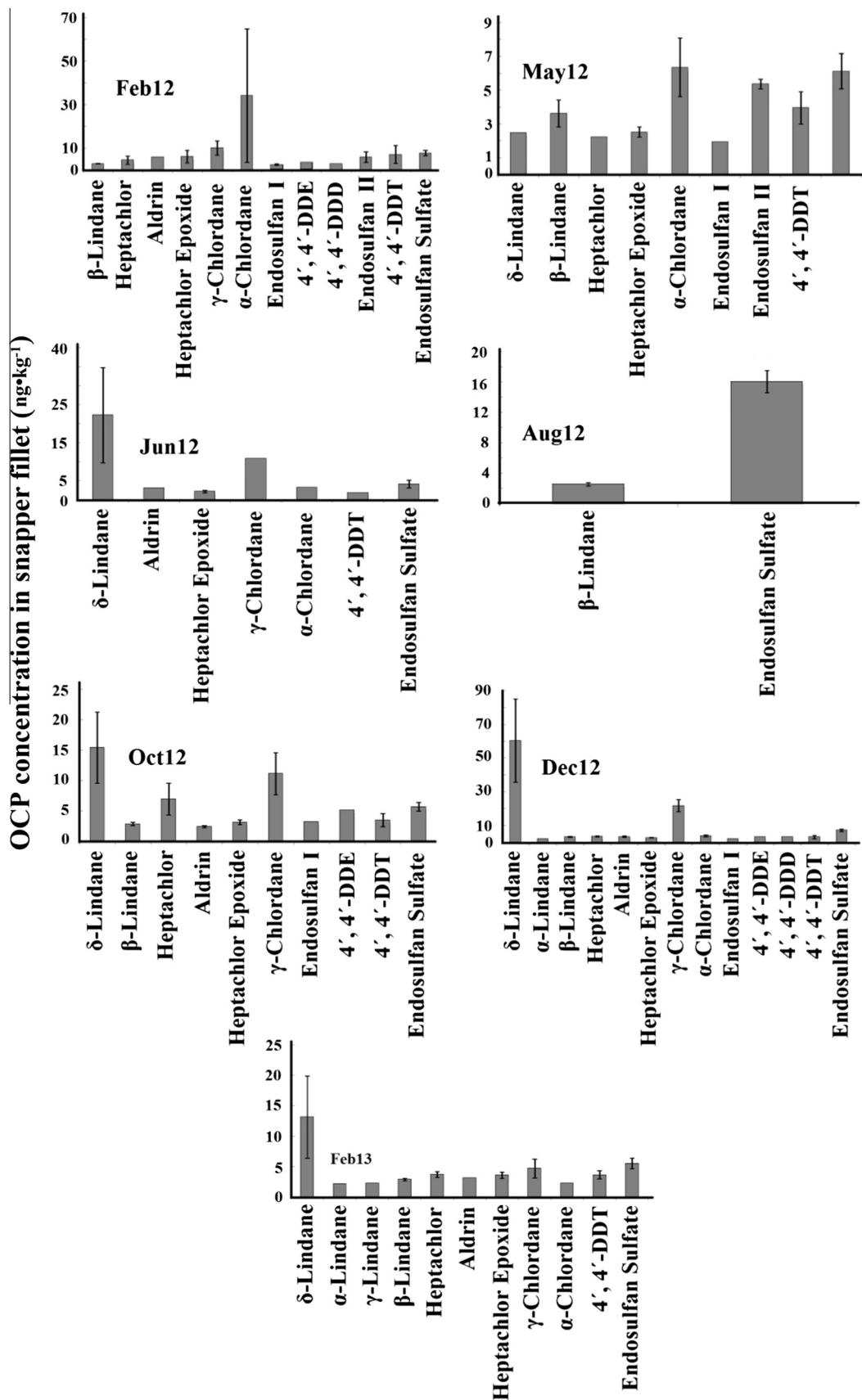


Fig. 2. Average concentration of OCP ( $\text{ng}\cdot\text{kg}^{-1}$ ) in snapper fillet per sampling period, from February 2012 to February 2013.

**Table 3**

Correlation coefficient values of OCP concentration in snapper's muscle tissue and the physico-chemical parameters of the seawater from the lagoon complex ( $p < 0.05$  is considered significant). <sup>a</sup>Mean, <sup>b</sup>(Min-Max), ND: Value below detection limits.

OCP detected	Temperature	Salinity	Dissolved oxygen (DO)	Weight	Size	Samples (n)	Freq (%)
$\alpha$ -HCH	-0.8	0.7	-0.4	-0.6	-1.0	3	2.9
$\beta$ -HCH	0.1	0.2	0.0	0.2	0.4	27	26.2
$\delta$ -HCH	-0.1	0.1	0.1	-0.1	-0.1	34	33.0
$\gamma$ -HCH					1		1.0
Heptachlor	0.2	0.3	-0.1	0.0	0.0	27	26.2
Heptachlor epoxide	0.0	0.0	-0.1	-0.1	-0.1	35	34.0
Aldrin	-0.3	-0.2	0.7	0.1	0.2	7	6.8
$\alpha$ -Chlordane	-0.2	0.4	0.0	0.2	0.2	15	14.6
$\gamma$ -Chlordane	-0.1	0.2	0.1	0.1	0.0	19	18.4
Endosulfan sulfate	0.2	-0.3	0.0	-0.1	-0.1	76	72.8
Endosulfan I	0.0	1.0	-0.5	0.4	0.4	5	4.9
Endosulfan II		-0.1		0.2	0.1	4	3.9
4,4'-DDE	1.0	1.0	-1.0	0.9	0.9	3	2.9
4,4'-DDD				-1.0	-1.0	2	1.9
4,4'-DDT	-0.2	-0.7	0.7	-0.2	-0.2	20	19.4

**Table 4**

OCP concentration with respect to the MLRs in edible animal products ( $\text{mg kg}^{-1}$ ). <sup>a</sup>Mean, <sup>b</sup>(Min-Max), ND: Value below detection limits.

OCP detected	This study	NOM-004-ZOO-1994 (DOF, 1994)	CE/178/2006 (DOCE, 2006)	CE/149/2008 (DOCE, 2008)	Codex Alimentarius (FAO & WHO, 2013)
$\alpha$ -HCH	0.00377	0.3	0.1	0.2	
$\beta$ -HCH	0.00292	0.3		0.1	
$\delta$ -HCH	0.02768	0.3			
$\gamma$ -HCH <sup>a</sup>	0.00213	4		0.1	0.1
Heptachlor epoxide	0.0033	0.2			
Heptachlor <sup>a</sup>	0.00445	0.2			0.2
Aldrin <sup>a</sup>	0.00445	0.3		0.2	0.02
$\alpha$ -Chlordane	0.00364				
$\gamma$ -Chlordane	0.01248				
Endosulfan sulfate	0.00732	0.2			
Endosulfan I	0.01012	0.2			
Endosulfan II	0.00286	0.2			
4,4'-DDT	0.00556	5	0.1		
4,4'-DDD	0.00386	5			
4,4'-DDE	0.00226	5			
$\sum$ Chlordane <sup>a</sup>	0.00225		0.1	0.5	0.05
$\sum$ Heptachlor	0.02259		0.1	0.2	
$\sum$ DDT <sup>a</sup>	0.00809		0.1	1	5
$\sum$ Endosulfan <sup>a</sup>	0.01514		0.1	0.05 <sup>(a)</sup>	0.2

ago, but its presence is still reported (Lewi et al., 2002; Zapata-Perez et al., 2007), just as in this study in fish.

According to Waliszewski et al. (2014), DDT metabolites persist in the environment in cases of extreme contamination, as can

occur in the soils neighboring the Guasave agricultural valley. The proportion of metabolites allows determining their historical use (Zhu et al., 2005) and the proportion of *pp'*-DDT *pp'*-DDE<sup>-1</sup> found in this study (0.58) is similar to the findings by Montes et al. (2011), which could indicate a historical contamination, probably due to run-off processes and soil erosion as Muralidharan et al. (2009) stated. Notwithstanding, more detailed and specific studies have to be made to allow determining whether the abundance of DDT in the muscle tissue of the studied population of snappers is due to persistence or because of the recent use of this OCP in the neighboring areas.

OCP concentrations can vary as a function of the seasons of the year (Muralidharan et al., 2009; Pérez-Ruzaña et al., 2000), runoff, bioavailability, solubility, polarity and molecular size or the physico-chemical parameters, lipid content and rainy season (Carro et al., 2004; Marburger et al., 2002; Mdegela et al., 2009; Montes et al., 2011; Sundar et al., 2010). Nevertheless, no influence of the physico-chemical parameters of the seawater on OCP concentrations in tissues of snapper fillets was found in this study similarly to findings by Ueno et al. (2003).

The less volatile OCP depends on temperature (Felipe-Sotelo et al., 2008) that influences different toxicological responses (Capkin et al., 2006) and can increase the metabolism in adult snappers, as found in their juvenile (Wuenschel et al., 2005). Chemical elimination through fecal egestion, respiratory surfaces,

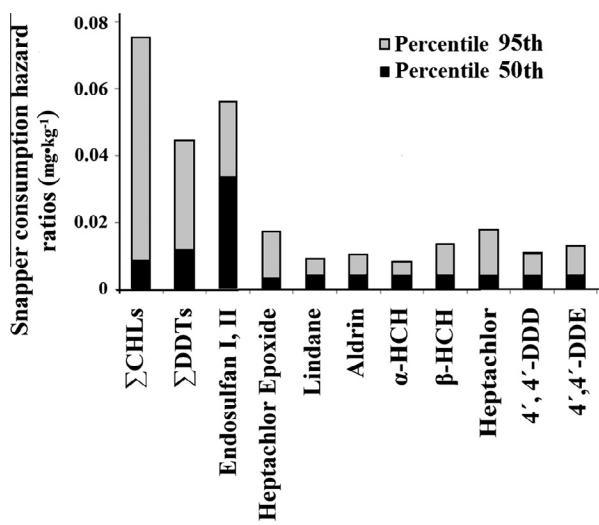
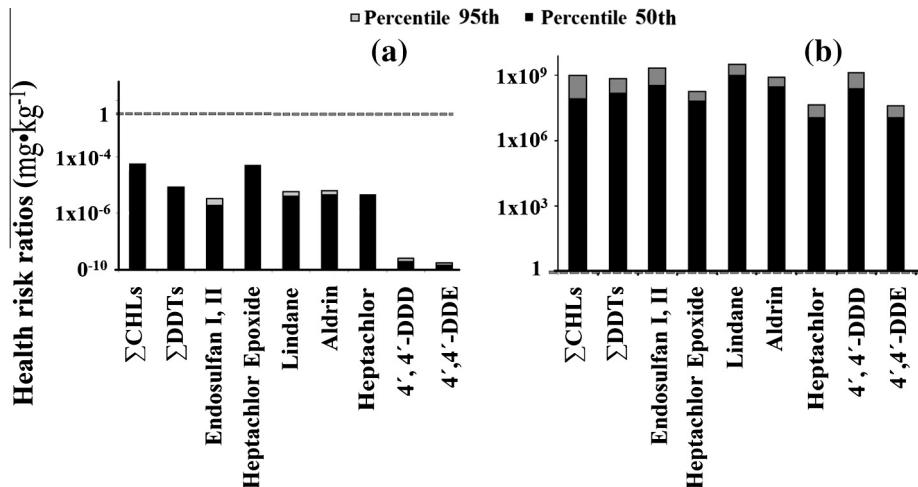


Fig. 3. Average of estimated exposure ( $\text{ng kg}^{-1}$ ) per consumption of snapper.



**Fig. 4.** Risk quotients for non-carcinogenic (a) and carcinogenic (b) effects due to consumption of snapper with concentrations in the 50th and 95th percentiles ( $1 \times 10^9$ ).

**Table 5**

Average OCP concentrations in studies reported in the San Ignacio-Macapule-Navachiste Lagoon system for sediments and seafood edible tissue.

Pesticides	Montes et al. (2011) sediments (ng g⁻¹ dw)	Galindo-Reyes et al. (1999) shrimps (µg l⁻¹)	Galindo-Reyes et al. (1999) shrimps (ng g⁻¹)	Reyes-Montiel et al. (2013) mullet (mg kg⁻¹)	Osuna-López et al. (2009) sediments (ng g⁻¹)
ΣHCH			101.7		
α-HCH	5.1			0.188	
β-HCH	18.2				
δ-HCH	7.5			0.0062	
γ-HCH	1.3			0.009	
Heptachlor epoxide	0.5			0.015	
Heptachlor	3.2	0.0353	93.244 112.51		
ΣChlordane					
α-Chlordane					
γ-Chlordane					
Endosulfan sulfate	1.9	0.0629			
Endosulfan I	1.0	0.0011	205.225	0.034	
Endosulfan II	0.7			0.0095	
4,4'-DDT	1.0			0.009	
4,4'-DDD	0.5			0.008	
4,4'-DDE	0.9		24.015	0.01433	76.8
Aldrin	0.6	0.0026		0.00975	205.7
Methoxychlor	11.9			0.1435	
Dieldrin	0.7	0.0156		0.11	
Endrin	4.5	0.0456		0.00525	
Endrin aldehyde	0.7		105.143		
Endrin ketone	2.1				
Methylparathion				98.6	
HCB					

and metabolic transformation could cause a decrease in OCP concentration in tissues (Fu and Wu, 2006). Elimination rates of organochlorine pollutants are increased with the high water temperatures of the summer and autumn (Blanes et al., 2009). In this study, we did not find a direct influence of the water temperature on the OCP concentration in the edible tissue of the analyzed snappers; the reduction of OCP levels in fish is not fully understood yet and more research is needed to focus on the enrichment of lipid content in fish.

Regarding the size, weight, and time of sampling, these influence the bioaccumulation of contaminants in aquatic organisms (Ferrante et al., 2010; Galindo-Reyes et al., 2014; Guo et al., 2010; Kucuksezgin et al., 2001; Larsson et al., 1991; Vives et al., 2004, 2005).

Nevertheless, we did not find, similarly to Ueno et al. (2003), a significant correlation between these parameters and OCP concentrations. As indicated above, the lipid content (Felipe-Sotelo et al., 2008), or the bioaccumulation among species (Szlinder-Richter

et al., 2008) can influence the OCP concentration. These pollutants do not have a normal distribution and show discrepancies among individuals of the same size, weight, and gender (Moriarty, 1972) as seen in our results.

#### 4.1. Maximum residue limits (MRLs)

To estimate the risk for human health and carcinogenic effects by the ingestion of OCPs in snappers from the Navachiste Lagoon complex region, the levels of exposure were compared with the MRL concentrations for each OCP as established by the "Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories" (EPA, 2000), which sets the portion of fillet equivalent to 0.229 kg month⁻¹. In this way, the studied OCPs detected in snappers of the Navachiste region can cause carcinogenic effects in humans: ΣHCHs concentrations reveal that the average concentrations of the samples (June 12, October 12, and February 13) were equivalent to carcinogenic concentrations if more than three

monthly portions are consumed ( $>0.018\text{--}0.024 \text{ mg kg}^{-1}$ ). In the case of  $\Sigma$  chlordane, carcinogenic concentrations were presented on February 12 with one portion month $^{-1}$  ( $>0.13\text{--}0.27 \text{ mg kg}^{-1}$ ) and in August 12 and February 13 with two portions month $^{-1}$  ( $>0.089\text{--}0.13 \text{ mg kg}^{-1}$ ). It is important to point out that  $\Sigma$  heptachlor presented carcinogenic concentrations in all months with a consumption above four portions month $^{-1}$  ( $>0.0013\text{--}0.0026 \text{ mg kg}^{-1}$ ). Finally,  $\Sigma$  DDT, which is a mixture of three DDT forms: 4,4'-DDT (71%), 2,4'-DDD (14%), 4,4'-DDE (4%) and other impurities (ATSDR, 2002), was not within the carcinogenic levels.

Considering the potential risks posed by the consumption of snapper that occurs regularly in gourmet restaurants, more integrated risk assessments must be performed to accurately determine the risks for human health posed by the exposure to the consumption of snappers from the Navachiste Lagoon complex.

## 5. Conclusions

Some OCP concentrations were above cancer MRLs, which suggest that, in the Guasave Valley, the application of banned OCPs is still happening, as has been reported before (Montes et al., 2011). Under these circumstances, the Navachiste Lagoon system, surrounded by one of the most important agricultural valleys in Mexico (Carrasquilla-Henao et al., 2013), can be influencing the deposition of OCPs in sediments and their bioavailability to the biota. Nevertheless, it is necessary to perform specific studies regarding the bioavailability of OCPs, health conditions, lipid content, and the biomagnification into the trophic web among the marine organisms that inhabit this lagoon complex.

The agricultural activity added to the carnivorous feeding habits of the snappers from the Navachiste Lagoon complex, which involves biomagnification, can be used to predict the trophic biomagnification of some OCPs in the aquatic food web (Wang and Wang, 2005). Both situations constitute a potential risk if snappers contaminated with OCP concentrations above carcinogenic concentrations are consumed.

The detected levels of total OCP ( $0.072 \text{ mg kg}^{-1}$ ) in this study were lower than the MRLs for Fish and Fishery Products Hazards and Controls Guidance set by the US Food and Drug Administration (FDA, 2011) under Compliance Policy Guide Sec. 575.100 (FDA, 2010). Overall, total OCP levels in the fish samples were less than the default MRL. Meal size is 8 oz (0.227 kg) (EPA, 2000) and the highest detected level (w.w basis), represents the worst case scenario for a human weighing 70 kg. The estimated daily intake (EDI) of these contaminants for a Human using a fish consumption rate for chronic health effects were  $0.02739 \text{ g day}^{-1}$ .

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