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Running head: adsorbed chlordecone on microplastics

Title: Detection of adsorbed chlordecone on microplastics in marine sediments in Guadeloupe: a preliminary study

Sandre Fidji ^{1,2}, Dromard R. Charlotte ¹, Le Menach Karyn ², Bouchon-Navaro Yolande ¹, Cordonnier Sébastien ¹, Tapie Nathalie ², Budzinski Hélène ², Bouchon Claude ¹

¹ Laboratoire de Biologie des Organismes et Ecosystèmes Aquatiques (BOREA), Université des Antilles, MNHN, Sorbonne Université, Université de Caen Normandie, CNRS, IRD, Laboratoire d'Excellence « CORAIL », PB 592, 97157 Pointe-à-Pitre, France

² UMR CNRS 5805 EPOC – OASU, Equipe LPTC, Université de Bordeaux, 351 Cours de la libération, 33405 Talence Cedex, France

Corresponding author: Charlotte Dromard - charlotte.dromard@univ-antilles.fr
- Phone: +33 (0) 590 483 011

Abstract

Plastic pollution in the oceans is nowadays recognized as a worldwide problem. Since the 1950s, the production of plastics has been increasing and the first reports of microplastics (particles < 500 µm) in the marine environment began to appear in the 1970s. These particles represent a growing environmental problem due to their dispersion in seawater and marine organisms. The first purpose of the present study was to assess the level of contamination of marine sediments by microplastics in Guadeloupe. To do so, marine sediments were collected in 12 sites, in reef environments, around the island. Microplastics from each sample were then counted under a binocular. The results showed that microplastics are found on all the studied sites and that their distribution could be linked to marine currents or proximity to areas of significant human activities (port activities, agglomeration, etc.).

In addition, these micro-particles can adsorb pollutants that will then become bioavailable to organisms by being desorbed during digestion and could be an important pathway for the contamination of organisms. In the Lesser Antilles (Guadeloupe and Martinique), an organochlorine pesticide called "chlordecone" was used from 1972 to 1993 in banana plantations. This very persistent pollutant contaminates soils, rivers, and coastal marine areas and accumulates in marine foodwebs. So, the second objective of the study was to determine the ability of microplastics to adsorb chlordecone, as it has been demonstrated for other organochlorine pollutants. The preliminary results of the present study showed that chlordecone could be adsorbed on microplastics.

Key words: plastic fibers, pesticide, Caribbean, coastal pollution, coral reef

INTRODUCTION

Since the 1950s, global plastic production has become exponential due to its properties and the many advantages it offers: inexpensive, durable and light weight material (Andrady 2011). However, only a portion of these plastics is recovered and recycled at the end of their life. In 2016, out of 27.1 million tons of plastics collected in Europe, only 31.1% was recycled (Plastic Europe 2018). Plastics then accumulate in landfills and gradually spread into the environment. Because they are not digested, plastic particles can obstruct the respiratory or digestive tracts and may lead to the death of many organisms, such as marine mammals (Laist 1997), birds (Mallory 2008) or turtles (Bugoni et al. 2001). The first reports of microplastics in the marine environment appeared in the 1970s (Carpenter and Smith 1972, Carpenter et al. 1972, Harper and Fowler 1987). These publications invited the scientific community to debate on the origin of these microplastics and its environmental and health consequences. Produced directly as microplastics or resulting from physical, chemical or biological degradations, various polymers are found in seawater, of which the most common are PolyEthylene (PE), PolyPropylene (PP), PolyStyrene (PS), PolyEthylene Terephthalate (PET) and PolyVinylChloride (PVC) (Brien 2007). In 2003, Gregory and Andrady defined microplastics as plastic particles with a size of less than 500 μm . Below 1 nm, they are called « nano-plastics ».

In addition, plastics can adsorb several organic contaminants such as PolyChlorinated Biphenyls (PCBs), Polycyclic Aromatic Hydrocarbons (PAHs) or PolyBrominated Diphenyl Ethers (PBDEs) and heavy metals due to its hydrophobicity and its generally high partition coefficient with organochlorine compounds. Plastics may also carry endocrine disrupting substances (Teuten et al. 2007, 2009).

Since their discovery, organochlorine compounds have been widely used for many purposes: to produce materials such as PVC and Teflon, as pesticides or insulators such as PCBs (Lange 2001). This is why they have been so massively produced and have been responsible for many environmental issues. Indeed, most of them are toxic and have been distributed worldwide (Tanabe et al. 1996).

In the aquatic environment, organochlorine pesticides are generally present in low concentrations ($\mu\text{g.kg}^{-1}$) or even in trace concentrations (ng.kg^{-1}) but can nevertheless cause toxic effects in organisms due to bioconcentration and biomagnification phenomena (Sonnenschein and Soto 1998). The adsorption of these molecules on microplastics makes them much more bioavailable to aquatic fauna. The adsorbed concentrations can be several orders of magnitude higher than the concentration in seawater (Moore 2008). These pollutants can thus be desorbed when ingested by aquatic organisms (Devriese et al. 2017). Microplastics can also constitute a new pathway for contamination of marine food webs (Teuten et al. 2007). Guadeloupe Island is particularly affected by an organochlorine pesticide called « chlordecone ». About 300 tons of this molecule were used in banana plantations in Guadeloupe and Martinique between 1972 and 1993 (Devault et al. 2016), which causes severe damages to the terrestrial and aquatic environment (Merlin 2015). Classified as POPs (Persistent Organic Pollutants) in the Annex A of the Stockholm

Convention (*i.e.* « to be eliminated »), this pesticide is highly toxic to many organisms (MacPhee and Ruelle 1969, Roberts and Bendl 1982). Due to its structure, this molecule is very persistent. Its half-life time is estimated at several decades (Cabidoche et al. 2009). Chlordecone is also biomagnified in the marine food-webs (Bouchon and Lemoine 2007, Dromard et al. 2018).

In recent years, many studies have focused on contaminant adsorption to microplastics (Daugherty 2016, Devriese et al. 2017, Hüffer & Hofmann 2016, Koelmans et al. 2016, Teuten et al. 2007, 2009), but none have been conducted on tropical marine ecosystems and none have been focused on chlordecone. The present study aims to quantify the microplastics present in the marine sediments around Guadeloupe Island and their capacity to adsorb chlordecone.

MATERIALS AND METHODS

Sampling protocol

Marine sediments were collected in 12 sampling sites, located around Guadeloupe Island (Figure 1). These sites were chosen in function of their geographic location around the island (North, South, East and West coasts), and of their different potential levels of exposure to chlordecone pollution. Samples of sediment were collected in reefs between 3 and 5 m deep using glass containers to avoid contact with plastic material. For the same reason, the use of plastic gloves was avoided during sampling and laboratory experiments. Three replicates were collected per site. Sediments were transferred to aluminum trays and dried in an oven (60°C) until obtaining constant dry weights.

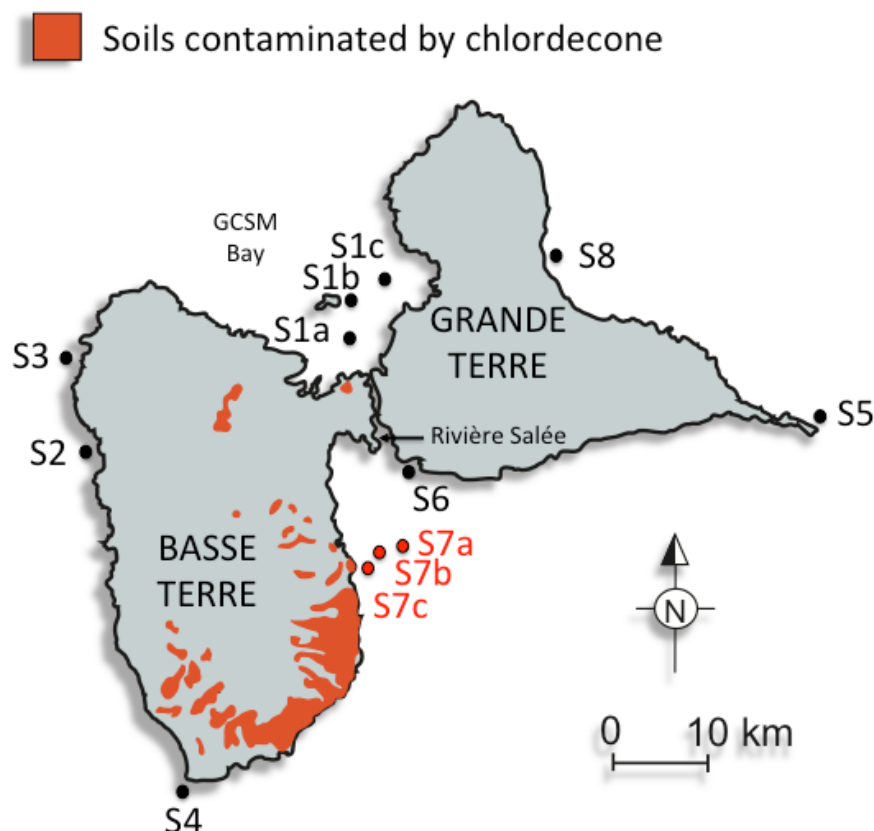


Figure 1 Location of the different sampling sites around Guadeloupe Island. Sites with red label were used to study adsorbed chlordecone on microplastics. S1a: Lagoon of Grand Cul-de-sac Marin Bay (GCSM), S1b: Passe-à-Colas, S1c: External reef slope of GCSM, S2: Pointe Noire, S3: Deshaies, S4: Anse Dupuy, S5: Pointe des Châteaux, S6: Îlet Cochon, S7a: Caye à Dupont, S7b: Îlet Fortune, S7c: Lagoon of Petit-Bourg, S8: Moule.

Microplastics counting

Two methods were used to extract microplastics from sediment.

The first method consisted in a direct observation of sediment under a binocular microscope and was used to assess the level of contamination of sediment around Guadeloupe. For each replicate (three per site), two subsamples of 5 g of sediment were observed under a binocular microscope in a Petri dish. Microplastics (fibers and fragments) were then sorted using fine pliers, and counted. The identification of microplastics was done according to the size, the color and the shape of the particle, and following recommendation from Hidalgo-Ruz et al. (2012). The amount of microplastic particles per site was expressed as the mean number of microplastics per 5 g of sediment. Numbers of microplastics per site were compared using a test of Kruskal-Wallis, followed by a post-hoc comparison test. Statistical analyses were performed with the software R, using 0.05 as critical significance level.

The second method was used to extract microplastics that were used to study the adsorption of chlordecone on plastic particles. To do so, three sites were chosen due to their proximity to chlordecone-contaminated watersheds (sites S7a, S7b and S7c,

Figure 1). To extract the microplastics from these samples, two 200 g sub-samples of sediment were added to 200 ml of a NaCl solution with a salinity of 140, following the protocol of Martins and Sobral (2011). The supernatant was recovered and rinsed three times in 50 ml of the solution of NaCl. This method was chosen to obtain a high amount of microplastics for the analysis. The supernatant, containing microplastics and recovered from sediments, was treated with a sodium hypochlorite solution (8°) to remove the organic suspended matter present. The supernatant was then filtered through glass microfiber filters (Whatman GF/F), rinsed with distilled water, which remained in an oven (60°C) until obtaining constant dry weights.

Chlordecone extraction and analysis

Glass microfiber filters, containing microplastics, were placed in microwave fields in pressure-resistant vessel containing 15 ml of a 50/50 mixture of dichloromethane and methanol, to extract the amount of chlordecone potentially adsorbed on microplastics. Due to the small amount of microplastics collected on filters, replicates of each site were pooled for the analysis. The extracts were recovered on glass-fiber filters. However, as a deposit of material was visible after filtration, they were thus filtered a second time on Whatman filters. The extracts were concentrated under nitrogen flow and transferred into injection vials (three rinses). A blank was used to control that there was no external contamination during the experiment. Then, chlordecone analysis was performed by liquid chromatography coupled with a tandem mass spectrometer (GC-MS-MS analysis, Agilent LC 1290 Infinity™) with internal calibration. Internal standard and measurement solution were prepared from a chlordecone solution marked with the carbon isotope ^{13}C at $5.549 \mu\text{g}\cdot\text{g}^{-1}$ and a native chlordecone solution at $5.668 \mu\text{g}\cdot\text{g}^{-1}$. Acetonitrile and water were used as solvents for chlordecone analysis and two ionic transitions were detected at m/z 506,9 to 426,7 and m/z 508,9 to 428,7. Acetonitrile blanks (spike) were introduced between each sample during injection.

RESULTS

Contamination of marine sediments by microplastics

Microplastics, principally fibers, were found in the marine sediments of all the studied sites (Figure 2). Considering all replicates, the number of microplastics varied from 0 to 52 particles per 5 g of sediment. Mean number of microplastics was significantly different between sites (Kruskal-Wallis, $X^2=29.8$, $df=11$, $p=0.0017$).

A post-hoc multiple comparison test showed that the differences in the mean number of microplastics according to site were observed between sites S7c and S4 and between sites S7c and S1b, *i.e.* sites with the lowest number of microplastics and those with the highest number of microplastics.

The maximum number of microplastics counted was observed along the drop-off of Passe-à-Colas channel (site S1b) and high values were also observed in the lagoon of the Grand Cul-de-Sac Marin (GCSM, site S1a), Anse Dupuy (site S4) and Îlet

Cochon (S6). The sites containing the lowest amount of microplastics were Caye à Dupont (site S7a) and the lagoon of Petit-Bourg (site S7c).

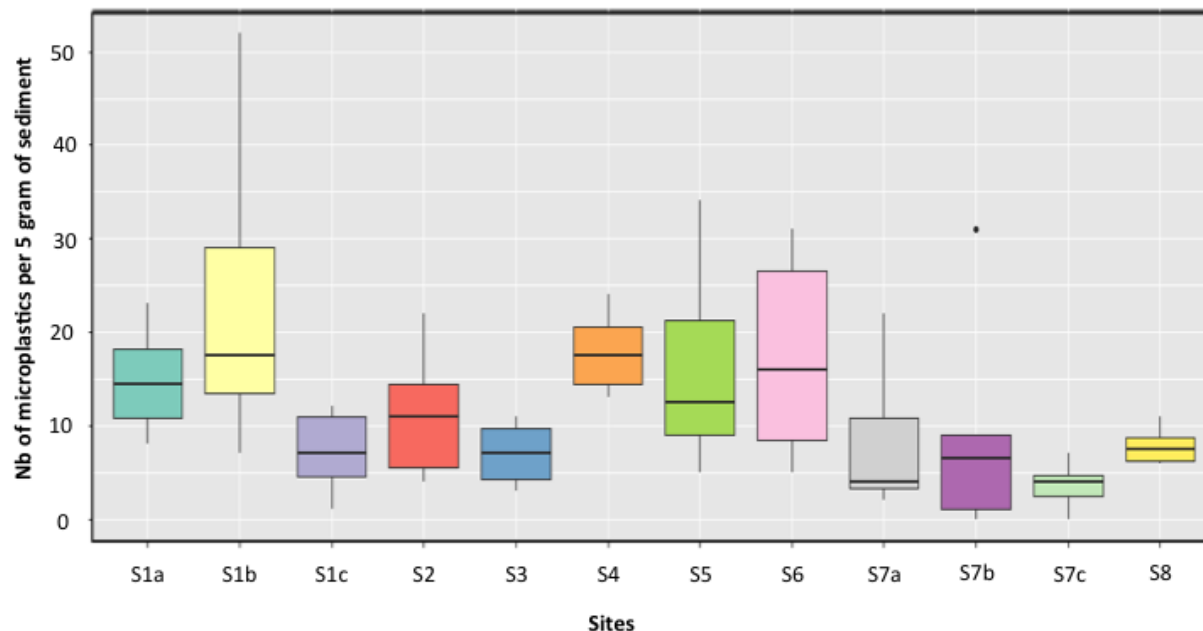


Figure 2: Mean number of microplastics per 5 grams of sediment in each site. Location of the study sites is given in Figure 1. S1a: Lagoon of Grand Cul-de-sac Marin Bay (GCSM), S1b: Passe-à-Colas, S1c: External reef slope of GCSM, S2: Pointe Noire, S3: Deshaies, S4: Anse Dupuy, S5: Pointe des Châteaux, S6: Îlet Cochon, S7a: Caye à Dupont, S7b: Îlet Fortune, S7c: Lagoon of Petit-Bourg, S8: Moule.

Concentration of adsorbed chlordecone

Chlordecone concentrations measured in each sample were determined by the internal standard quantification method.

The high values of the dry weights of filters are due to the presence of mineral residues on filters. The number of microplastics was estimated from the data obtained by the counting of particles carried out on these same samples (see previous section). The mean weight of microplastics per filter was estimated according to the nature of the polymers (Callister 2007). The area values obtained by LC-MS-MS were used to calculate the quantity of chlordecone adsorbed (in μg) per μg of microplastic collected on the filters (Table 1).

Table 1: mass of chlordecone (in μg) per μg of microplastic collected on the filters, calculated from LC-MS-MS data. CLD: chlordecone, MP: microplastics

Sample	m_{CLD} (μg) per filter	M_{MP} (μg) per filter	m_{CLD} ($\mu\text{g}\cdot\mu\text{g}^{-1}$ of MP)
Site S7a	0.00131	0.76000	0.00173
Site S7b	0.00030	0.84000	0.00036
Site S7c	0.00044	0.35000	0.00124

DISCUSSION

The results of the present study highlight the presence of microplastics in the sediments of all the studied sites. This result shows the omnipresence of microplastics along the coasts of Guadeloupe Island, as it has been demonstrated in other regions (Claessens et al. 2011, Graham and Thompson 2009).

The location of sites S1a, S1b, and S1c can partially explain the different amounts of microplastics found in marine sediment. Site S1a is located in the lagoon of the Grand Cul-de-Sac Marin Bay (GCSM Bay) and is protected from marine swell by a long reef barrier, except during climatic events like hurricanes or in the case of strong northern swell. On the other hand, its proximity to the Rivière Salée, an inlet that separate the two island of Guadeloupe (Grande Terre and Basse Terre) and that cross over the urbanized area of Pointe-à-Pitre could explain its high plastic content. Indeed, a study on the currentology of Guadeloupe indicated that the circulation of water in the Rivière Salée follows an alternating rhythm depending on the tide, but also mentioned that under usual conditions, waters from the Rivière Salée flow south to north (that is in direction to the GCSM Bay) (Egis Eau 2012). In addition to currents, the renewal rate of the lagoon waters is low and would then allow the accumulation of microplastics at the surface of the sediment, in this part of the bay.

The site S1b is the site where sediment exhibited the highest concentrations of microplastics. While it is further north of the land and urban discharges, this site is located along a channel and is therefore more exposed to waters movements. The high contamination of this site by microplastics could be due to inputs from outside the island, but also from an influence of the Riviere Salée. In this channel, waters flow in both sides (north to south and south to north depending on the season, the tide and the wind) (Castaing et al. 1984, Pujos et al. 1992).

The high values of the sites S4 and S6 could be linked to local anthropogenic activities. The site S4 (Anse Dupuy) is located near a fishing harbor and a nautical work site area. These activities involve the use of synthetic resins that could be found in the surrounding environment. The site S6 (Ilet Cochon) is the closest to the center of the island and the most important agglomerations, which can be a source of microplastics discharge (in particular through the massive use of water bottles (PET) combined with poor waste management on the island). For these two sites, the contamination therefore seems to come directly from the island. The low concentrations of microplastics found at Petit-Bourg (sites S7a and S7c) could be explained by their exposition to dominant east-west marine currents. These sites are particularly exposed to the swell and winds that push the water mass towards the coast, due to dominant winds ("Alizées") that blow from east to west for most of the year. These conditions would therefore not be favorable to the retention of microplastics by the sedimentary compartment. To sum up, the abundances of microplastic differ according to their geographical distribution. Exposure to marine swell or proximity to more urbanized or harbor areas seem to play a role in the number of microplastics present in marine sediments. However, further studies are needed to confirm this link between human activities and the number of

microplastics: a more detailed study of the current patterns or the identification of the chemical nature of plastics would make it possible to trace their origin.

The presence of chlordecone was detected in the three samples analyzed. While it has been suggested, the present study evidences for the first time the capacity of chlordecone to adsorb on microplastics. Significant adsorption of chlordecone on microplastics reflects its high affinity with plastic as many studies have already shown the strong affinity of organic pollutants to microplastics. This adsorption depends on different parameters. The polymer type could impact the adsorption affinity. Indeed, Cavani and Trifiro (1995) showed high adsorption rates of PAHs and PCBs on polystyrene. Daugherty (2016) showed that polypropylene sorbed more pollutants than polyethylene. But the sorption forces were weaker and thus the pollutants could be more easily bioavailable to organisms. Bakir et al. (2012) found that PVC and PolyEthylene Hight-Density (HDPE) could adsorb different concentration of Dichloro-Diphenyltrichloroethane (DDTs) or Phenanthrenes (Phe). The contaminant also plays a role in the adsorption rate. Frias et al. (2010) studied adsorbed Persistent Organic Pollutants (POPs) on plastic samples and found a high concentration of adsorbed PCB regarding to others categories of POPs. Pollutants also showed a competitive sorption. For example, Bakir et al. (2012) demonstrated that DDT competitively interfered with the sorption of Phe. Finally, the size and age of plastics are also important: older plastics would be more degraded and have a higher adsorption capacity (Endo et al. 2005, Ogata et al. 2009). In this study, it is not possible to conclude on the differential capacity of chlordecone to be adsorbed on different types of plastic because chlordecone was quantified on the entire samples (all categories of microplastics confounded). An FT-IR analysis should be necessary to determine the characteristics of the plastics found in these environmental samples.

Chlordecone adsorbed on microplastic particles suggest the possible existence of a new pathway of transfer of the molecule into marine trophic food-webs. However, while model of accumulation and biomagnification of chlordecone in the marine environment have been studied (Coat et al. 2011, Dromard et al. 2018), none of them have considered this potential pathway of contamination. The competitiveness of chlordecone compared to other pollutants, such as DDT or DDE, found in the waters around Guadeloupe is also not unknown. Adsorption tests on microplastics of pollutant mixtures in environmental concentrations would provide information on the behavior of chlordecone and allow a better estimation of its potential impact on marine organisms.

In conclusion, microplastics were found in all the sites studied around Guadeloupe. This pollution seems to come from both local sources, due to human activities on the island, but also from more distant sources (*via* oceanic swell). Similar patterns were found in corals colonies, collected in the 12 same study sites, during a previous study in Guadeloupe (Rochas 2018). This preliminary study assessed chlordecone contamination adsorbed on microplastics from marine sediments. Further studies would be needed to focus on pollutants adsorbed on microplastics distributed in the water column or those present in marine organisms to get a more global overview of the contamination. In addition, a qualitative study to determine the nature of all

pollutants adsorbed on microplastics would be necessary to estimate the potential synergistic effects of these molecules. A limited number of sites have been sampled to measure chlordecone adsorption, so the results will not be generalizable to the entire contaminated area. However, since the sites were chosen for their high level of chlordecone contamination, the study will allow for no doubt to reveal the maximum chlordecone values that can be found on microplastics of the sediments of Guadeloupe. Finally, a FT-IR analysis combined with tests on chlordecone adsorption could give important information about the nature of plastic and their adsorption rate and allow a better estimation of the potential impact on marine wildlife.

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