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Research Article

INNOVATIVE ASSESSMENT OF ORGANIC MICROPOLLUTANTS IN SEWAGE DUMPING AREA OF TUTICORIN, SOUTHEAST COAST OF INDIA

Narmatha Sathish*and Jamila Patterson

Suganthi Devadason Marine Research Institute 44, Beach Road Tuticorin 628001, India

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ABSTRACT

Organic micropollutants such as phthalate, triclosan, octylphenol, polycyclic aromatic hydrocarbon and polychlorinated biphenyl were assessed in sewage dumping coastal area of Tuticorin, southeast coast of India. Nine phthalates were observed in the samples of coastal water and sediment and it ranged from 0.03 to 23.21µg/l and 0.056 to 46.52µg/kg respectively. Triclosan was detected both in water (31.52µg/l) and sediment (85.6µg/kg). Risk Quotient (RQ) values of diethyl phthalate, dibutyl phthalate and triclosan in the water were so high as to cause adverse effects on the aquatic system. But in the sediment, the phthalate concentration was below the USEPA sediment guideline. Traces of octylphenol were observed only in the sediment samples; and the concentrations of polycyclic aromatic hydrocarbon and poly chlorinated biphenyl were below the detectable level both in water and sediment samples. The screening and risk assessment of these pollutants are proven to be useful for better control and management of organic micropollutant discharges into the coastal waters.

INTRODUCTION

Water is a universal solvent because of its high dielectric constant and it has the property of dissolving most of the substances but the assessment of the substance leads to water pollution (Utkarsh *et al*. 2017). The *marine* ecosystem is the largest aquatic ecosystem of the earth. The increasing magnitude of pollution and industrial pressure along the coasts pose a threat to the coastal ecosystem. Raw sewage is allowed to mix with the sea water and this causes water pollution, which engenders serious repercussions for the marine environment. Sewage is a type of waste water consisting of domestic, industrial and agricultural wastes, and the surface runoff. Sewage may contain nutrients like nitrates, silicates, phosphorus, and solids including organic matter, pathogens such as bacteria, viruses and protozoa, oil and grease, and heavy metals (Monica 2003). Raw sewage needs to be treated properly before being discharged so that it will not harm the environment. Wastewater discharge into the sea is the major source of organic micro pollutants in the sea. A wide variety of organic micro pollutants are present in the marine ecosystem. Some of them are: polycyclic aromatic hydrocarbon (PAH) (Valavanidisetal.2008); plasticizers such as phthalate and bisphenol (Fromme *et al*. 2002); organochlorine pesticide, alkyl phenol (Sanchez-Avila *et al*. 2010); polychlorinated biphenyls and polybromodiphenyl ethers (Perez-Carrea *et al*. 2007). These pollutants generally have a significant effect on water quality and marine life. The coastal areas of Tuticorin have great ecological importance due to the presence of biological resources and industrial development. Thousands of coastal people in and around Tuticor in depend on the fishery resources for their livelihood (Diraviya Raj *et al*. 2017). Hence the protection of this region from continuous pollution is not only indispensable but also urgent. A large number of researchers have worked on the physicochemical properties of coastal water and sediment of Tuticorin coastal area (Puthiyasekar 2009; Rajchander *et al.* 2012; James *et al*. 2015; Clara *et al*. 2015 and Sekar *et al*. 2017). However organic pollutants have not been studied so far. This is the first report on organicmicro pollutants in the water and sediment of Therespuram coast of Tuticorin caused by the dumping of untreated sewage into the Therespuram coast via waste water channel. The objective of this study is to evaluate the occurrence and toxicity of phthalate (PAE), triclosan (TCS), polycyclic aromatic hydrocarbon (PAH), poly chlorinated biphenyl (PCB) and octylphenol (OP) in the water and sediment of Therespuram coast of Tuticorin. The Risk Quotient (RQ) was also calculated to determine the possible adverse effect of these compounds on the aquatic environment.

Corresponding author:* **Narmatha Sathish
Suganthi Devadason Marine Research Institute 44, Beach Road Tuticorin628001, India

MATERIALS AND METHODS

Tuticorinis an industrial town located between latitude 8◦ 15′ to 9◦0′N and longitudes 77◦50′ to 78◦15′E. Water and sediment samples were collected during September 2017 from Therespuram (8° 43' - 8° 51' N latitude and 78° 5' - 78° 10' E longitude), a fishing village along the Tuticorin coast, where untreated sewage is dumped via Buckle channel into the sea. The collected samples of water and sediment were transferred to the laboratory in iceboxes. The sediment samples were stored at -20^{0}Cuntil extraction and the water samples were stored at 4◦ C. The physicochemical properties of the samples were analysed as per standard method (APHA, 2005). The dissolved oxygen was estimated by the modified Winkler's method, described by Strickland and Parsons (1972). The pH was measured soon after collection by pre-calibrated Eco pH tester and salinity was measured by Strickland and Parson method (1968).

Micropollutants were detected using Gas chromatograph mass spectrometerQP-2010 (Shimadzu GC MS). For phthalate, the sample was extracted by liquid extraction using dichloro methane. The analysis was performed using a gas chromatograph (Shimadzu GC- 2010) coupled with mass spectrometer (Shimadzu QP 2010) and an autosampler (AOC 20i, Shimadzu Corporation).Compounds were separated on a capillary column of 5MS 5% diphenyl-95% dimethyl polysiloxane (30m length, 0.25mm i.d., 0.25mm film thickness) from Thermo Scientific. The compounds were separated using the following oven program: the column temperature was initially set at 80^{0}Cfor 2 min, then increased at a rate of 17^{0}C/min up to 320^{0}C which was maintained for 5 min. Helium carrier gas (of 99.9999% purity) was maintained at a constant rate of 1.2 mL/min. Mass spectra were obtained. For the analysis of triclosan, the sample was passed through the C18 cartridge and eluted with methanol. The optimal condition was as follows: an Agilent SB-C8 analytical column (250 × 4.6 mm, 5µm)was utilized and mixed buffer solution of methanol and 0.01 mol /lphosphate (pH 3.0) (72: 28, V/V) was used for isocratic elution at a total flow rate of 1.0 mL/min. It was found that the calibration curves had a good linear regression with UV detection.

For PAH analysis, the sample was subject to liquid extraction using DCM: Hexane (1:1). The residue was reconstituted to 2ml with methylene chloride and injected to GC-MS. Foroctylphenol and PCB, the sample was extracted with methylene chloride and passed through SPE cartridge. A small amount of the residual water from the SPE cartridge formed an immiscible layer with methylene chloride. The obtained methylene chloride was evaporated and eluted to dryness under nitrogen, and the dried extracts were reconstituted into 1 ml of Acetone and filtered through 0.2 µm membrane and injected into the liquid chromatography.

Sea water risk assessment

The risk of organic pollutant in seawater was estimated on the basis of risk quotient (RQ) value (Sanderson *et al*. 2004; Vryz as *et al*. 2009; Ginebreda *et al*.2010). The risk quotient was an important line of evidence in characterizing the potential of substances that cause harm to ecosystems. A risk quotient was the ratio of predicted environmental concentration (PEC) to predicted no-effect concentration (PNEC). For the risk assessment, PNEC value based on the EC50 values (concentration that induces half the maximum effect) or LC50 (lethal concentration for 50% of the population) for acute toxicity tests for different organisms and NOEC values (No Observed Effect Concentration) used for chronic toxicity tests (Blair *et al*. 2013 and Tewari *et al*. 2013). For the determination of acute PNEC and chronic PNEC, EC50 values (or LC50) and NOEC were divided by an assessment factor (Yamamoto *et al*. 2011). In this study, literature value of LC50 and NOEC values were used to find risk quotient of phthalate and triclosan.

RQ<1, indicates no significant risk
1≤RQ≤10 indicates small potential for adverse effects
10≤RQ≤100 indicates significant potential for adverse effects
RQ≥100 indicates that adverse effects should be expected.

RESULTS AND DISCUSSION

Physicochemical property

The values of pH, salinity and dissolved oxygen concentration of the samples were 7.5, 35.3ppt and 2 mg/l respectively. James *et al.* 2015 reported that the salinity of Tuticorin coastal water ranged from 32 to 35.6ppt, and that it was low during the monsoon season and high during the summer season. A low dissolved oxygen value (2 mg/l) was observed in the present study and a similar pH (7.4) and lower value of 2 mg/l dissolved oxygen were reported in Therespuram coastal water by Puthiyasekar *et al.* 2009.

Phthalate

Phthalates are esters of phthalic acid (1, 2-benzene dicarboxylic acid) and endocrine distributing compounds (EDC).

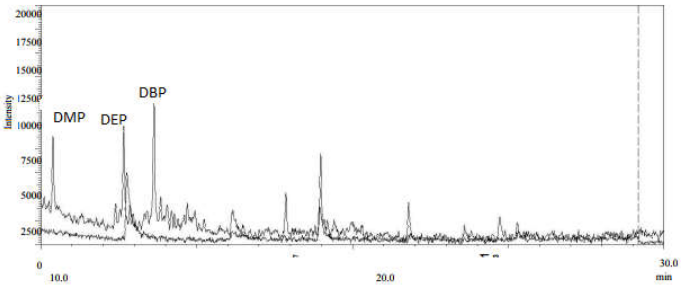

Fig 1 Phthalate in water (µg/L)

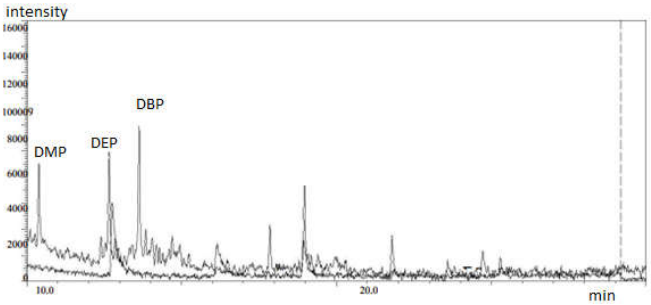

Fig 2 Phthalate in sediment(µg/kg)

They are used as plasticizers in plastic (PCV) for flexibility (Jaeger Rubin *et al*. 1970), in cosmetics, personal care products (Ron and Lisa 2012) and in insect repellants, and solid rocket propellant (CCREM 1987). In the present study, phthalates

were observed in the water and sediment samples. The data are presented in Fig 1 and 2. The values of phthalate concentration in water are DMP (23.2 µg/l), DEP (11.325µg/l), DBP (16.806µg/l), DHP (0.085µg/l), BBP (0.032µg/l), DBEP (0.125µg/l) and DEHP (0.098µg/l). The concentration of phthalate in sediment are DMP (46.52 µg/kg), DEP (30.06 µg/kg), DBP (26.93 µg/ kg), DHP (0.125 µg/kg), BBP (0.056 µg/kg), DBEP (0.865 µg/kg) and DEHP (0.155 µg/kg). In this study the concentration of phthalates were found to be higher in sediment than in water.

Eric Lichtfouse and Jan Sachwarzbauer (2012) reported that phthalates are generally less soluble in salt water than in fresh water. Even phthalates of lower molecular weights are only slightly soluble in salt water. So this may be the reason for the higher concentration of phthalates in sediment than in water. Of the nine phthalates, dimethyl phthalate (DMP), diethyl phthalate (DEP) and dibutyl phthalate(DBP)were detected in higher concentrations in water and sediment samples, whereas dihexyl phthalate (DHP), benzyl butyl phthalate(BBP), bis(2-n-butoxy ethyl) phthalate (DBEP) andbis (2-n-ethylhexyl) phthalate (DEHP) were observed in lower concentrations. The other two phthalates Di-n-octylphthalate (DnOP) and diisononyl phthalate (DiNP) were observed in concentrations below the detectable level. Fatoki and Noma (2002) reported similar results [DMP (0.03-31.7 µg/l), DEP (0.03-33.1µg/l), DBP (2.8 - 12.19µg/l) and DEHP (0.06 - 19.74µg/l)] in East London Port of South Africa. Marti *et al*. 2011 reported a similar range of phthalates in Valencia sea water (0.025 to 20 µg/l). Juans *et al*. reported lower values of phthalates (0.001to5.9µg/l) in the Mediterranean Sea.

Higher concentration of DMP, DEP, DBP were observed in water samples, and this is due to the direct discharge of sewage water into the sea. Phthalates like dimethylphthalate (DMP), dibutylphthalate (DBP), diethylhexylphthalate (DEHP), and benzylbutylphthalate (BBP) are widely used in plastics (Oehlmann *et al*. 2009). Similarly Meeker *et al*. (2010) reported phthalates of low molecular weights such as DMP, DEP, and DBP in seawater brought through the wastewater from washes of personal care products such as cosmetics, facial cream, lotion and shampoo. Even though DMP has a moderate toxicity, it degrades through the environmental factors into intermediate mono methyl phthalate which is an endocrine disruptor so it may affect the development and reproductive system of animals (Brar *et al.* 2009). Lower concentration of DEHP (0.098 µg/l) and BBP (0.03 µg/l) were observed in water.

used as plasticizers in plastic and there is no covalent bond between plastics and the plasticizer (PAEs) and they are merely entangled within the plastic, so it can be released into the environment by exposure to heat (Wilkes and Summer, 2005). In the present both these micropollutants were observed in minimal concentrations and their presence in water may be due to the dumping of plastic wastes into sea. The USEPA (2012) has classified DEHP (class B2), BBP (Class C) as possible human carcinogens. Phthalate concentration data obtained in the site under study are compared with those reported from other locations in the world (Table 1) for the evaluation of the extent of contamination.

Petrovic *et al.* 2001 reported that sediment may act as the repository of these pollutants which are insoluble in water. Yuan *et al*.2002 and Chang *et al.* 2004 & 2005 informed that the half-life periods of phthalates in sediment with an anaerobic condition were 3-10 times higher than those in aerobic condition.

In the present study, the sea water was found to have low DO (2mg/l). This anaerobic condition of sea water of the site under study is due the discharge of raw sewage into sea. In the sediment, concentration of phthalates were in the order DMP>DEP>DBP>DBEP>DEHP>DHP>BBP.

Somaye Mohammadian *et al.* (2016) declared that adsorption of all phthalates are slightly influenced by the increase in salinity from 0 to 40ppt and in the present study the salinity observed in the coastal water was 35.3ppt. Xue-Kun *et al*. (2004) reported that salinity increases the partition coefficient and so bioaccumulation takes place. So the higher concentration of phthalate in sediment may be due to higher salinity and lower dissolved oxygen of the water. Mackintosh *et al*. (2004) reported that the sediment not only was the final sink of phthalates but also may play an important role in phthalate conversion from the media to the biological systems in an aquatic environment. Thus the affected organisms may be an unexpected source of exposure to humans. The site of the present study has a low oxygen level due to sewage dumping and hence it is vulnerable to micropollutant accumulation. Similarly Srinivasan *et al.* (2010) reported the concentration of phthalate in the sediment of river Gomathi: BDL-490 µg/kg (DMP), BDL -350 µg/kg (DEP), BDL-340 µg/kg (DBP), BDL -3240 µg/kg (DEHP) and BDL-530 µg/kg (DOP). Here too the higher concentration of phthalates in the sediment are due to the high level of waste water dumping into the river water.

Table 1 Comparison of phthalate reported in coast of India and other countries(µg/l)

Location	DMP	DEP	DBP	BBP	a	DOP	Reference
UK(Bay)	<1	0.025-0.5	0.47-0.55	-	0.98-2.2	-	Law *et al*. 1991
Spain(fishing port)	1.6	1.4	1.3	0.5	2.1	-	Penalver*et al*. 2001
Japan(Bay)	0.01-0.09	<loq-0.31	0.01-0.54	<loq-0.06	-	-	Suzuki *et al.* 2001
Taiwan(Bay)	-	<loq-2.5	1-13.5	-	<loq-18.5	-	Yuen *et al*. 2002
Spain(sea)	0.01	0.03	0.08	0.01	0.06	-	Prieto*etal.* 2007
Iran(sea)	0.49	0.52	-	-	-	-	Hadjmohammadi *et al.* 2011
Canada(sea)	-	-	0.18-3	-	0.01-0.95		Keil *et al*. 2011
USA(sea)	-	-	-	-	0.06-0.64	-	Keil *et al*. 2011
India(sea)	23.2	11.3	16.8	0.032	0.098	-	Present study 2017

Similar concentrations had been reported by Prieto *et al*. (2007) from the coastal waters of Spain: 0.06 µg/L of DEHP and0.01 µg/l of BBP. The most toxic pollutants DEHP and BBP are

Although at present the concentrations of all phthalates are low, the accumulation of these compounds in the medium is bound to increase over the years and will lead to negative impacts on the aquatic organism. Phthalates are identified as

environmental contaminants because of their presence in sewage, rain water, soil, natural water, sediments and in aquatic organisms (Berge *et al*. 2013; Hongjum *et al*.2013; Teil *et al*. 2013). Phthalates are good indicators of environmental risk due to human activities. Soour landfill and sewage should be removed and treated appropriately.

Octylphenol

Octylphenol is an endocrine disrupting compound. Traces of octylphenol (0.011µg/kg) were observed in sediments but in water it was found in the below-detectable level only.

Hansen and Lassen (2008) observed similar lower concentration (<0.2) in the coast of Baltic Sea. Khimetal. (1999) observed higher concentration (91.5ng/g) of octylphenol in Masan bay in South Korea. The octylphenol observed in sediment samples of the present study may be due to discharge of sewage water into sea. ECHA (2011) reported that many consumer products such as detergent, cosmetics, lotion liquid, ink, and paint containoctylphenol compound. These products washed down into the household drainage release octylphenol into the coastal environment. It is toxic to the aquatic organisms, it is not easily degradable, and so it has a potential to cause endocrine disrupting effect (OSPAR 2006). Even though octylphenol was found to be in smaller concentration in this study prolonged exposure to it may cause adverse effect on the aquatic environment.

Triclosan

Triclosan (5-Chloro-2-(2,4-Dichlorophenoxy) phenol) is an antibacterial agent. It possesses both phenol and ether functional group.TCS has been mentioned as an endocrine disruptor (Forenetal.2000). Because of the wide use of triclosan in personal care products (Kirk-Othmer 1993, Perenvich *et al*. 2001), household items, pesticide, health care products such as first aid, antiseptic products and preservatives, it is easily discharged into the environment (Reiss Mackay, 2002). In the present study, triclosan was observed both in water and sediment samples at 85.6µg/kg and 31.5 µg/l respectively, and it is shown in Fig 3 and 4. Similarly Tatarazako and Ishibashi (2004) studied coastal water quality of Rhode Island near the outfall of wastewater treatment plant and reported that, triclosan observed in effluent samples in the range of 10-20 µg/l and of 80-100 µg/kg in sediment samples.

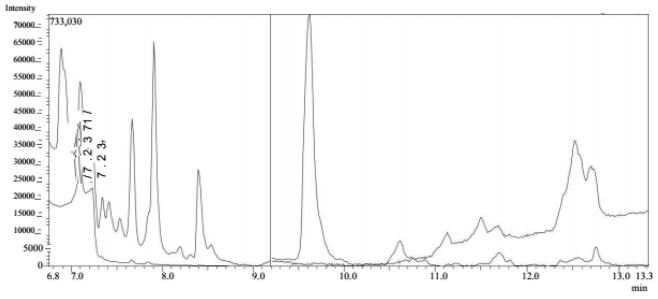

Fig 3 Triclosan in water(µg/l)

Name	R.Time	Targetion	Type	Area	Height	Conc
Triclosan	7.237	TC	Target	12440	52178	31.52

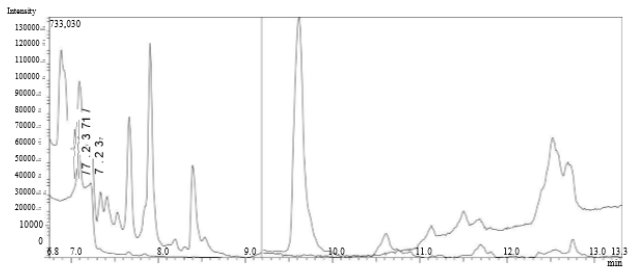

Fig 4 Triclosan in sediment (µg/l)

Type	Name	R.Time	Target ion	Area	Height	Conc
Target	Triclosan	7.237	TC	36440	96178	85.6

They declared that incomplete removal of TCS from WWTPs and application of TCS laden bio-solids into agricultural soils lead to TCS contamination of the environment. These findings agree and coincide with the results of the present study. This indicates that the concentration of triclosan in the marine environment studied may be due to the discharge of untreated sewage into sea.

Xie *et al*. (2008) and Fair *et al*. (2009) reported that the worldwide data of triclosan in sea water ranged from<0.001-100 ng/ l in water and from 0.02 - 35 µg/kgin sediment. But the area under the present study had higher concentration of triclosan in water and sediment which is due to the disposal of untreated sewage into the sea. It may cause adverse effect to the environment because triclosan is converted into dioxin when it is exposed to sunlight in an aqueous medium. Triclosan combines with chlorine in seawater to form chloroform (Rule *et al*. 2005 and Fiss *et al*. 2007) which is listed as a probable human carcinogen. Triclosan was observed in higher concentrations in sediment due to the persistent degradation processes under anaerobic condition (Mc-Avoy *et al.* 2002). Ying *et al.* (2007) conducted a laboratory experiment and proved that TCS degraded in the aerobic soil with the half-life period of 18 days, but persisted in the anaerobic soil during the 70 days of the experiment.

Even though triclosanis represented as an important health care and sanitary tool, constant exposure to triclos an becomes a health and environmental hazard. Triclosan develops the antibacterial resistance in pathogenic bacteria (Kola, *et al*. 2015). Many research works have reported that triclosanbio-accumulates in algae (Dury *et al*. 2013), zebra fish (Tatarazako *et al*. 2004), snail and marine mammals (Crofton *et al*. 2007) Gibeliioncatla Fish (Govindaraj and Shanmugam *et al*. 2014) and human breast milk (Adolfsson-Erici *et al.* 2000). Due to the dynamic state of TCS, it is difficult to find the accurate risk posed to environment; and even at small quantity it affects organisms. So it is imperative to minimize the amount of triclosan reach to the environment.

PAH and PCB

PAHs are compounds consisting only of carbon and hydrogen atoms. Chemically, the PAHs are composed of two or more benzene rings bonded in linear or cluster arrangement. PAH compounds are produced during combustion and pyrolysis processes of anthropogenic and natural origin (Bertilsson *et al*. 2002). A high amount of PAH is emitted from coal processing, during incomplete combustion of organic matter like wood and fossil fuel and from motorcycle exhaust and cigarettes. In

India, Dhananjeyan *et al*. (2012) reported the concentration values of PAH (8.66 ng/l to 46.74 ng/l in water and 2608 ng/g to 134134 ng/g dry weight in sediment) for Harbourline in Mumbai. They declared that intense shipping, oil refinery activities, sewage discharges, and discharging of black smoke from ship and boat transport were the reason for the high concentration of PAH. In the site of the present study, PAHs concentrations were below the detectable limit because fuel contamination was limited in the sewage of this study area.

Polychlorinated biphenyl is an organic chlorine compound with the formula $C_{12}H_{10-x}Cl_x$. PCBs are widely deployed as dielectric and coolant fluids in electrical apparatus, in the manufacture of carbonless copy paper and in heat transfer fluids. PCBs are produced by biomass burning, leakage from transformers, shipwreck, and municipal waste burning (McElroy *et al.* 1989). PCB concentration was below the detectable limit in both water and sediment of the sample of this study. Babu *et al*. (2004) reported that in Tamil Nadu, the highest concentration of total PCB was found in sediment samples from Chennai harbour (6570 pg/g) followed by sediments samples of Cooum River mouth in Chennai (505 pg/g), Cuddalore fishing harbour (335 pg/g) and Mandapam (251 pg/g). They opined that PCB may come from industrial and domestic waste water discharges, and that the nature of sewage varied from place to place. In the present study even though sewage is directly discharged into the sea, the study area had concentrations below the detectable level of PCB for the coastal samples had not been as yet contaminated with the detectable level of PCB.

Aquatic risk assessment

Risk assessment study is imperative for assessing the water health status. In this study, literature value of LC50 and NOEC (no observed effect concentration) values were used to find risk quotient of phthalate. Depending on the phthalate ester, their toxicity to salt water organism varies. RQ value of fish *Rivulusmarmoratus* was calculated using NOEC value reported by Adams *et al*. (1995) with the Assessment factor 100. DBP exhibited the chronic toxicity to egg and larvae of *Rivulusmarmoratus* with 1.6 (RQ >1). RQ value of DEP calculated using LC50 of invertebrate had been reported by Adams *et al* (1995).RQ value(1.1) showed that it causes acute toxicity to invertebrate (mysid shrimp). In the present study area, only DEP and DBP exhibited small potential for adverse effect on the aquatic species.

Phthalate sediment concentration was compared with screening benchmark value given by USEPA 2006 (Table 2). None of the value exceeded the screening bench mark value. So there is no risk to the environment.

Table 2 Screening benchmark (SCB) for phthalate in coastal sediment (µg/kg)

Phthalate	Sediment	Coastal sediment SCB
DMP	30.06	200
DEP	26.93	1160
BBP	0.056	16800
DEHP	0.055	182

Risk assessment of triclosan to aquatic species was observed using PNEC value. ECETOC (2003) and NICNAS (2009) adopted PNEC (0.05ug/l) value for marine ecosystem. Compared with this finding, the study area had higher concentration of TCS in water. Water Frame Work Directive UK TAG (2009) proposed PNEC value of triclosan at 0.1, 0.28, and3.8ug/l for saltwater species like algae, taxa, and secondary poisoning predator respectively. Calculated RQ values for these species are shown in Fig 5.

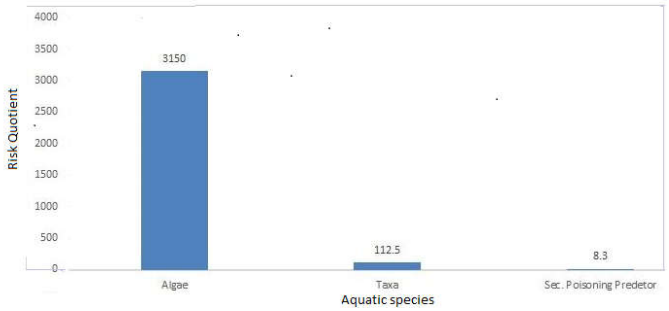

Fig 5 Aquatic risk assessment of triclosan

RQ values of algae, taxa, and secondary poisoning predator are above 1. This indicates that the pollutant poses risk to the aquatic species. The RQ value for algae and taxa is >100. This shows that they have significant potential for adverse effect. Algae cause possible destruction in ecological balance (Ricart *et al*. 2010). Small potential effect was caused for secondary poisoning predator (1<R<10). LogKow of triclosan is 4.76,which exceed TGD trigger value(>3).So it is important to assess the sediment quality. The PNEC value is not available, and data on direct toxicity to sediment-dwelling organism are limited (WFD UK TAG 2009). PNEC value of octylphenol is higher than PEC value of sediment. So it does not cause any risk to the environment.

CONCLUSION

This is the preliminary report on organic micropollutants in seawater and sediment samples of Therespuram. Phthalate, triclosan and octylphenol were detected in this area. Butpolycyclic aromatic hydrocarbon and poly chlorinated biphenyl were found to be in concentrations below the detectable limit in water and sediment samples. Diethyl phthalate, dibutyl phthalate and triclosan exhibit small potential adverse effect to aquatic species. The other seven phthalates and octylphenol are in concentrations below the risk level. The by-products of these pollutants are even more toxic and pose a potential risk for aquatic species. So a continuous monitoring of the coastal water and sediment is necessary for better management and control of pollutant discharges into the sea. Lots of wastes are dumped into the m*arine* ecosystem and their accumulation creates problem to the environment and also pose a bigger threat to the present and the future generation. The present study reports the occurrence of pollutants, and further studies are necessary to identify and quantify the chronic effects on fish and other aquatic species, because a large population in this area depends on fishery resources for their livelihood. The input of sewage is hazardous to the environment so it is urged that the sewage must be treated before the discharge point.

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