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RESEARCH ARTICLE

Assessment of Triclosan in Sewage, Sludge and Contaminated Soils

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ABSTRACT

Triclosan (TCS) is an antibacterial and antimicrobial compound, which is considered as "Emerging Organic Contaminants" and highly toxic to living organisms even at very low concentrations. It is an endocrine disrupter with anti-androgenic and oestrogenic properties and reduces serum level of thyroid hormone. TCS has highly persistence in sludge and sludge applied soils through sewage due to anthropogenic activities. The present study was carried out to assess the concentration of TCS in sewage, sewage sludge and contaminated soils from sewage treatment plants (STPs) of Ukkadam, Coimbatore. The observed TCS concentration in raw and treated sewage was 9.2 mg L^{-1} and 5.79 mg L^{-1}. The higher concentration of TCS was found in sludge sample (28.24 mg kg^{-1}) and the concentration of TCS in sewage contaminated soils of Coimbatore was 1.65 mg kg^{-1}. The plant samples collected from sewage irrigated lands of Coimbatore (Napier grass) also showed 0.019 mg kg^{-1} including samples of leaf, stem and root. The study showed the presence of TCS in sewage, sewage sludge, contaminated soils and plant samples that can be harmful to the environment.

Keywords: *PCPs, TCS, Sewage, Sludge, Contaminated soil*

Triclosan (5-chloro-2-(2,4-dichlorophenoxy) phenol, $C_{12}H_7Cl_3O_2$) was introduced more than 50 years ago and used as a preservative or as an antimicrobial agent in pharmaceutical and personal care products (PPCPs), such as soaps, cosmetics, shampoos, dental products, many household products, etc. The consequent discharge of TCS from domestic waste to wastewater treatment plants as effluent and to streams or rivers was identified as a primary source of TCS contamination, as TCS was removed incompletely from wastewater treatment plants. It has been reported that the raw, treated and sludge present in sewage treatment plant contained 2.01- 17.6 µg L^{-1}, 0.99- 13.0 µg L^{-1}, 3.65-15.0 µg L^{-1} of TCS (Lehutso *et al.*, 2017). This leads to environmental concerns, as TCS is toxic to organisms by blocking the fatty acid synthesis through inhibiting the enzyme enoyl–acyl carrier protein reductase. Also, TCS can cause endocrine disruption and also interfere with thyroid hormone metabolism (Rodricks *et al.,* 2010). In soil, sludge and sediments, TCS is readily degraded in the environment through photodegradation or sunlight irradiance with a half-life of a week in aerobic and a month to years in anerobic conditions, forming other compounds like, chlorophenols, chloroforms and also dioxins (Latch *et al.*, 2005). It is bacteriostatic at low doses and at higher doses; it acts as a bactericidal agent. TCS also inhibits fatty acid synthesis in plants and other living organisms. The aim of this study is to find out the concentration of TCS in sewage, sludge and contaminated soils of sewage treatment plant of Ukkadam, Coimbatore.

MATERIAL AND METHODS

Experiment was initiated by collecting sewage, sewage sludge and sewage contaminated soils samples with latitude of 10°59' N and longitude of 76°58' E from the sewage treatment plant (STP) in Ukkadam, Coimbatore. The sewage treatment plant has capacity of 70 MLD (Million Litres per Day) in Coimbatore. The treated waste water is used for irrigating gardens established with fodder crops.

Sample collection and preparation

Raw and treated sewage, sludge and soil samples were collected from sewage treatment plant of Ukkadam, Coimbatore. The liquid (sewage) and solid (sludge and soil) samples were stored at 4°C for analysis. Twenty soil samples were collected by random sampling method. The collected sludge (MLSS) and soil samples were air dried and sieved through 2 mm and used for further physic-chemical analyses. The initial characterization of sewage, sludge and soil samples was analyzed based on standard methods.

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Chemicals

TCS (2, 4, 4-trichloro-2-hydroxydiphenyl ether) (>95% of purity) were purchased from Sigma-Aldrich Bangalore, India to prepare working standards. Acetone, ethyl acetate and hexane used for analyses were of HPLC grade. Stock solutions were prepared by dissolving 10 mg of technical pure TCS in 100 mL of ethyl acetate: Acetone (1:1 v/v) and stored at 4°C. Working standards were prepared using stock solution.

Extraction of triclosan from sewage

Five hundred millilitres of sample and five gram of NaOH was mixed and extracted through separating funnel and shaken well the content. The collected solutions were then washed with 50ml of n-Hexane and allowed it for 10 mins to separate. The aqueous solution was then transferred to separating funnel and pH was adjusted to 2. Then, aqueous solution was extracted twice with 50ml of n-Hexane (Nishi *et al.*, 2008).

Extraction of triclosan from sludge and soil

Ten gram of collected samples was homogenized with 50ml of acetone for 12 hours using orbital shaker. After shaking, supernatant will be filtered through Whatman.No.42 filter paper. Then the residues on filter paper were extracted twice with 25 ml of acetone. Combined extract were separated with 500 ml milli-Q water and five grams of NaOH in separating funnel. Fifty millilitres of n-Hexane was used for washing (Okumura and Nishikawa, 1996).

Silica gel cleanup

Three grams of silica gel stirred with 10ml of n-Hexane and acetone mixture (1:1, v/v) to make slurry and packed in glass column. Then Na_2SO_4 was layered on top and conditioned with 15 ml of n-Hexane and acetone mixture. Condensed extract then eluted through the glass column. The collected eluate then condensed to 1 ml using a rotary evaporator at 35°C and stored it in a vial at 4°C until GC-MS analysis (Okumura and Nishikawa, 1996).

Analysis of triclosan

The Gas Chromatography - Mass Spectrometer from Thermo fisher, Trace-1300 series, was used for the analysis. The instrument was set as follows, Injector port temperature set to 220°C, Interface temperature set as 250°C, source kept at 220°C. The oven temperature programmed as available, 75°C for 2 min, 150°C at 10°C min^{-1}, up to 250°C at 10°C min^{-1}. Split ratio set as 1:12 and the injector used was splitless mode. The DB-5 MS capillary standard non - polar column was used whose dimensions were 0.25mm OD x 0.25μm ID x 30 meters length procured from Agilent Co., USA. Helium was used as the carrier gas at 1.5 mL min^{-1}. The MS was set to scan from 50 to 550 of ion source. The source was maintained at 220°C and 4.5e-6 m torr vacuum pressure. The ionization energy was -70eV. The MS was also having in built pre filter which reduced the neutral particles. The data system has inbuilt libraries for searching and matching the spectrum.

RESULTS AND DISCUSSION

Effect of triclosan on pH in raw and treated sewage

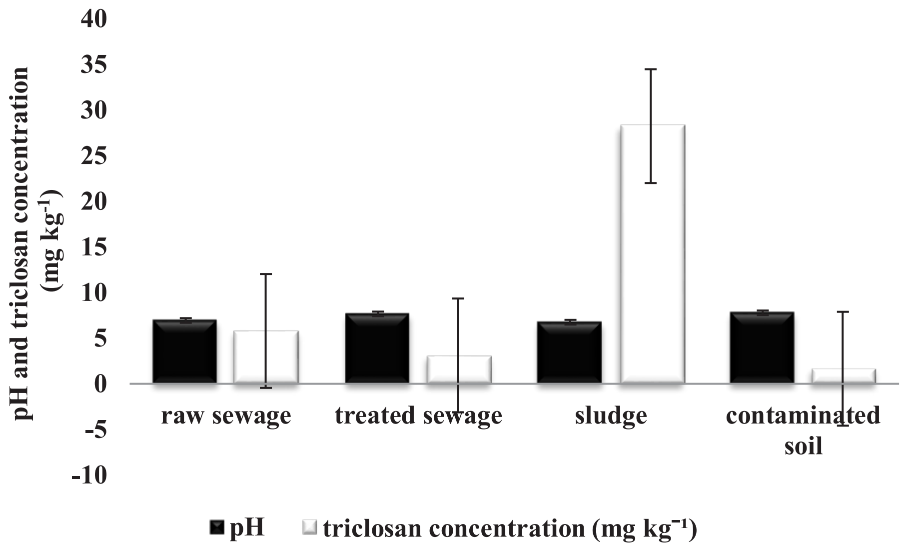
The concentration of TCS in raw sewage was 5.79 mg kg^{-1} with pH of 6.94 (Fig. 1) (Table 1). This is in line with the findings of Lehutso *et al.,* 2017 who reported that the concentration of TCS in raw sewage ranged between 2.01 to 17.6 μg L^{-1} with neutral pH. Dhillon *et al.,* 2015 have reported that the higher concentration of TCS in raw sewage with neutral to alkaline pH may be due to the presence of phenolate TCS. In treated sewage, the TCS concentration was 3.11 mg kg^{-1} with neutral pH.

Table 1. Concentration of triclosan in sewage, sludge and soil samples

Characters		Triclosan concentration (mg kg^{-1})
Sewage	Raw	5.79
	Treated	3.11
Sludge		28.24
Contaminated soil*		1.65

*Mean of twenty samples

This increase in TCS concentration might be due to discharge of untreated waste water from the sewage treatment plant and also non availability of technologies that remove contaminants of Pharmaceutical and Personal care products (PPCPs) (Lehutso *et al.,* 2017).



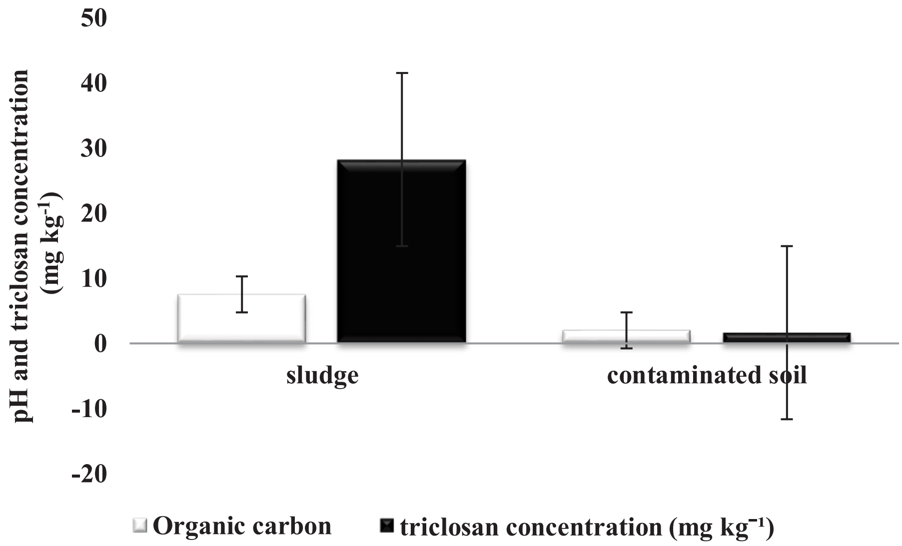


Figure 1. Effect of pH and Organic carbon on triclosan in sludge and contaminated soil

Effect of triclosan on organic carbon content in sludge and contaminated soils

The observed organic carbon content in sludge and soil samples in Coimbatore were 7.53% and 2.0% (Fig. 1), which is related to the results observed by Ibrahim *et al.* (2014) at 9.6% and the studies on sewage irrigated soils by Lal *et al.*, (2015) showed similar results of 0.86% and 0.65% organic carbon content in the soils. Long term irrigation with sewage tends to increase the soil organic carbon (SOC) content, which is most important indicator of soil health and also acts as a store house of plant nutrients and plays a major role in nutrient cycle. The concentration of TCS in sludge and soil samples were 28.24 mg kg^{-1} and 1.65 mg kg^{-1} (Table 1). Higher concentration of TCS drained into sewage systems, favours TCS partitioning into sewage sludge and subsequently their transfer to soil when applied to land surface. The increase in organic matter content leads to increase in concentration of TCS and the content of TCS can be degraded easily by soil microorganisms under aerobic conditions and highly persistent in anaerobic conditions (Mendez *et al.*, 2016).

PPCPs are used in many consumer products which are then released into sewage treatment plant through regular anthropogenic usage and leads to potential toxicity. The present study revealed the presence and their concentration of TCS in sewage, sludge and contaminated soils in sewage treatment plant of Ukkadam, Coimbatore.

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