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Qualitative and quantitative characterization of hazardous triclosan traces in household personal care soaps marketed in UAE

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Abstract

Background: Many household personal care products such as soaps, cosmetics, shampoos, toothpastes and deodorants are profusely sold in most of pharmacies and grocery stores. Most of the customers excessively use them without knowing their main contents or what could be behind the label of each product. One of the hidden ingredients is triclosan (TCS), which is a broad-spectrum antimicrobial agent widely used in many personal care and household cleaning products. Some household personal care product manufacturers may use TCS as an ingredient without indicating it in the list of ingredients. This may lead to several toxic effects of TCS when is used excessively whether orally (as toothpastes) or topically (as soaps and cosmetics).

Purpose: Our study aims to investigate the presence of TCS in five soap bars from different brands marketed in the UAE.

Methods: Five soap bar products of commonly used brands marketed in UAE were purchased from local pharmacies and subjected to qualitative and quantitative characterization using Fourier transform infrared spectroscopy (FTIR) and high performance liquid chromatography (HPLC). The FTIR spectra of isolated TCS in each soap was compared to the spectrum of standard TCS which was previously obtained using the FTIR. The HPLC chromatographic conditions such as mobile and stationary phases were optimized using standard TCS to determine the optimum retention time at which TCS is retained. The chromatograms of the soaps were qualitatively compared to the standard TCS and quantitatively calculated according to the calibration curve of standard TCS.

Results: The FTIR spectra of the studied five soaps showed characteristic peaks that indicate the presence of TCS traces. Under the chromatographic conditions, TCS appeared in all chromatograms of the examined soaps at the same retention time that matches with the standard TCS chromatogram. However, the exact quantity of TCS varied from one soap to another.

Conclusion: More efforts should be addressed to improve the safety and efficacy to ensure they are TCS-free. This can be achieved by restricted inspection of all OTC products that were or will be entered to the market.

Key words: Triclosan, Toxicity, Soap, FTIR, HPLC, FDA

INTRODUCTION:

Triclosan (TCS) is a nonionic and relatively small molecule (289.54 g/mol) that is used in many personal care and household cleaning products. Since it is a broad spectrum antibacterial agent, it is involved extensively in many products such as toothpastes, cosmetics, deodorant sticks, mouth rinses, dentifrice gels, and antibacterial soaps (1). The physical state of TCS at standard temperature and pressure is white solid characteristic with a melting point of 54-57 ˚C and boiling point of 280-290 ˚C (2). TCS acts by the reaction with the enoyl-acyl carrier protein reductase (ENR), which is an essential enzyme for fatty acid biosynthesis in many bacterial species (3).

It was clearly demonstrated that TCS at its bactericidal concentration can cause cell wall disruption of the bacteria with nonspecific targets. However, its action is limited on specific targets when used at sub-lethal concentrations (4). Although TCS has beneficial disinfection activity, many toxic effects can result from extensive use of TCS-containing products. According to diverse animal studies, TCS contributes to endocrinal system disruption, especially thyroid gland and male reproductive-endocrinal system (5–7). A study published in 2012 reported that geno-toxicity was induced by TCS (8). Additionally, TCS has a potential effect in suppressing the immune system by reducing the concentration of T4 cells in juvenile male rats (9). Also, TCS has the ability to enhance the allergic response of certain allergens and increase the exacerbation of asthma (10,11). Moreover, another study revealed that TCS could interfere with the maintenance and development of normal placenta during the pregnancy by stimulating the estradiol and progesterone hormones and decreasing the human chorionic gonadotropin (hCG) hormone (12). Furthermore, TCS can be bio-accumulated in biological tissues and is associated with cross-resistance to many antibiotics in many microorganisms (13), yet, the significance of these effects on human's health has not yet identified. Other studies reported that TCS was detected in water sewage, soil and wastewater treatment influents, which could be attributed to the extensive use and physicochemical properties of TCS that influence its persistence in the environment (14,15). In 2016, the US Food and Drug Administration (FDA) has issued a warning and ordered to withdraw any over-the-counter (OTC) TCS-containing products and prohibiting their manufacture for personal care uses (16). Therefore, in recent years concerns have been raised and escalated about the presence of TCS in many daily use products, and its potential harmful impacts on human health and environment.

The present study aimed to screen some soaps available in the UAE market by using FTIR and HPLC. FTIR was used as qualitative process to identify the presence of TCS, while HPLC was used for qualitative and quantitative assessment of the presence of TCS in the tested soaps.

MATERIALS AND METHODS:

Materials

Five soap bars from different commercial companies were purchased from local pharmacies in the UAE. Their band names are not revealed in this study, and S-I, S-II, S-III, S-IV and S-V names were assigned. All soaps were subjected to qualitative and quantitative analysis by using FTIR and HPLC techniques from Shimadzu (Japan). TCS standard was purchased from Sigma Aldrich (USA). Acetonitrile and ultrapure water were HPLC grade and purchased from Merck (Germany) were used as mobile phase.

Methods

FTIR

Fourier transform infrared (FTIR), Make: Shimadzu, Japan; Model: IRAffinity-1S, was used in this study as a qualitative tool to confirm the presence of TCS. This method measures the wavelength absorbed by the material and represents it as a characteristic spectrum. The TCS spectra (standard and soap samples) were scanned at a range from 400 to 4000 cm^{-1} and resolution of 2 cm^{-1}. A small portion of the standard TCS was placed in a sample holder where the IR beam passes through it. The frequency at which the sample absorbs the beam was scanned 20 times and represented as a spectrum by the LabSolution

software. The soap samples were finely powdered and subjected to the same procedures of the standard TCS.

HPLC

The HPLC analysis was performed using an i-Series Plus HPLC system from Shimadzu (Japan) which is facilitated with ACTO (Analytical Condition Transfer and Optimization) technology for easier migration of the materials. LabSolution software system was used for data acquisition and analysis. This is a new HPLC method which was carried out in accordance to the optimized conditions for analysis during the experimental study.

Chromatographic conditions

HPLC analysis was carried out using C8 column with particle size 5 µm (150 mm × 4.6 mm) as a stationary phase purchased from Waters, USA. The mobile phase consisted of a mixture of HPLC grade acetonitrile and water in a ratio of 70:30 v/v and separated by isocratic mode at flow rate of 0.7 mL/min. The column temperature was set at 35 °C and the UV detector was set at wavelength of 280 nm.

Standard solution, calibration curve and linearity

Standard stock solution was prepared by dissolving 0.03 g of TCS by the mobile phase in a 50 mL volumetric flask and sonicated for 10 minutes at 25 °C using the ultrasound. The standard TCS was injected at different volumes of 10, 20 and 40 µL. The calibration curve was constructed by plotting the amount of the standard injected in grams versus the area under the curve (AUC) and the linear equation was obtained.

Samples preparation

Aliquots of the studied samples were prepared at the same concentration by dissolving 0.02 g of each by the mobile phase in 50 mL volumetric flask and sonicating it for 10 minutes. From the resulting solutions, 20 µL was injected and detected at the same mentioned chromatographic conditions. The detected peaks at the same retention time of the investigated standard TCS peak were compared and calculations of the concentrations were derived from the linear equation based on the AUC values.

RESULTS AND DISCUSSION:

FTIR

The spectral FTIR scan of TCS presented a characteristic peak at 1473 cm^{-1} as shown in Figure 1 and this can be due to the presence of chlorobenzene in the structure of TCS (17). Similarly, a previous study reported the presence of TCS at the same range (18). The studied soaps were found to contain TCS in their formulations which was clearly appeared in their different FTIR spectra. The represented peaks of all five soaps were matched with the standard TCS characteristic peak; where all peaks of the five soaps ranged between 1450 and 1500 cm-1 as shown in Figures 2-6. Therefore, FTIR method is considered as a strong evidence for detecting the presence of TCS in the studies samples compared to the reference TCS. The quantitative assessment of TCS in the soaps can be determined by other methods for confirmation.

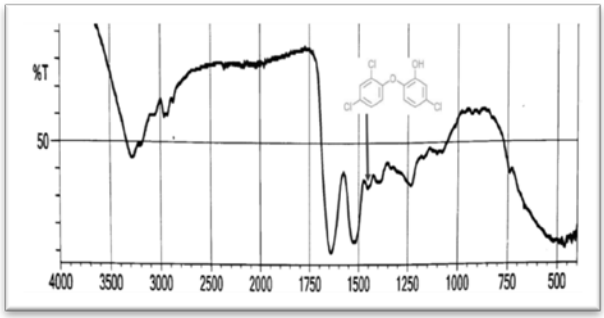

Figure 1. FTIR spectrum of standard TCS.

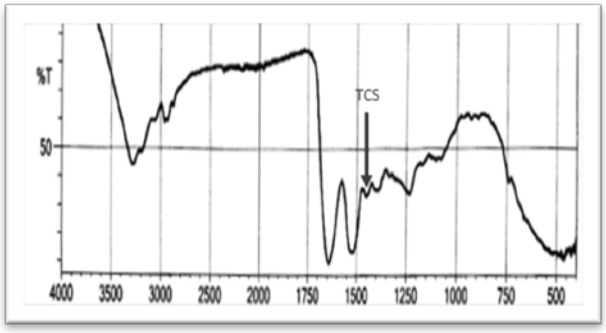

Figure 2: FTIR spectrum of S-I soap.

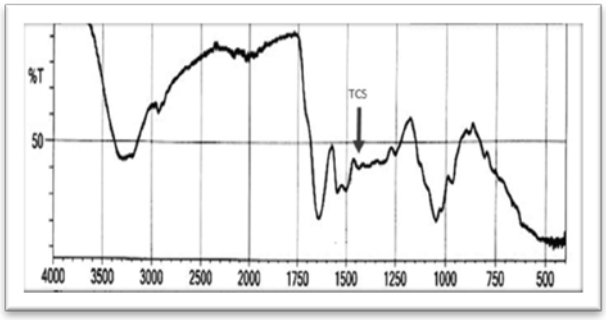

Figure 3: FTIR spectrum of S-II soap.

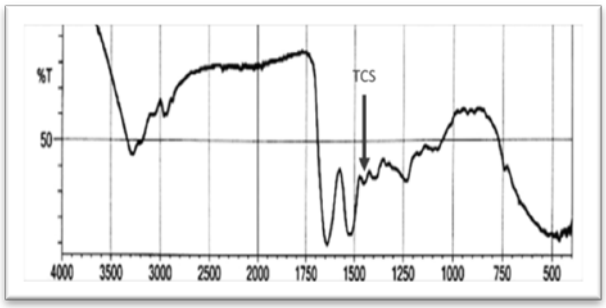

Figure 4: FTIR spectrum of S-III soap.

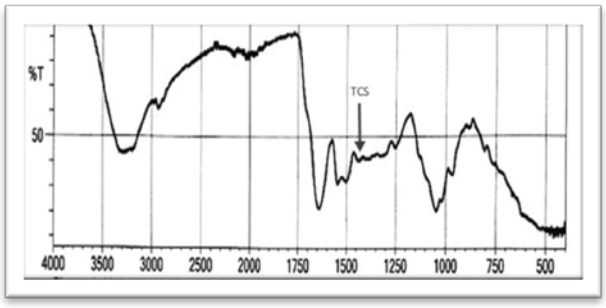

Figure 5: FTIR spectrum of S-IV soap.

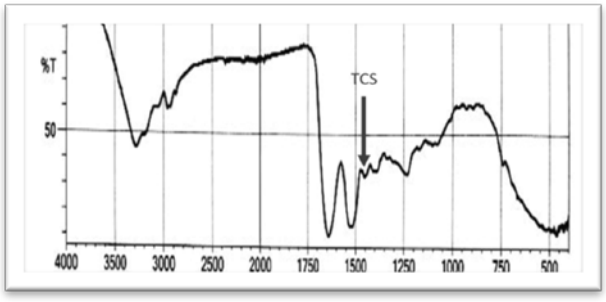

Figure 6: FTIR spectrum of S-V soap.

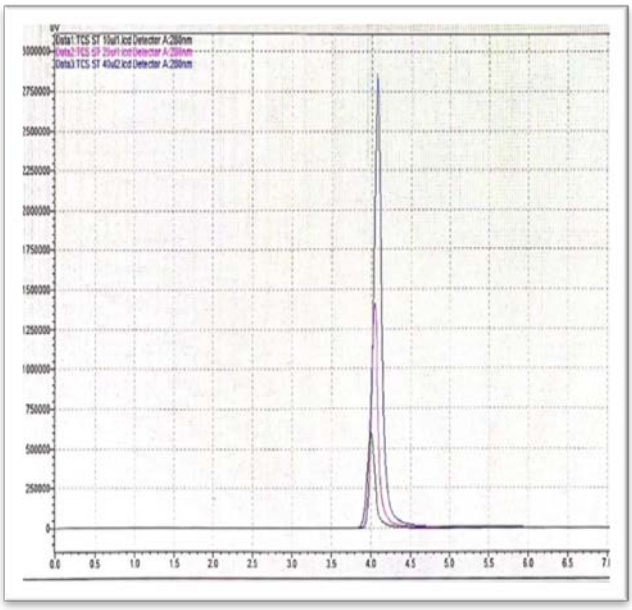

Figure 7: Overlaid HPLC chromatograms of standard TCS at different concentrations.

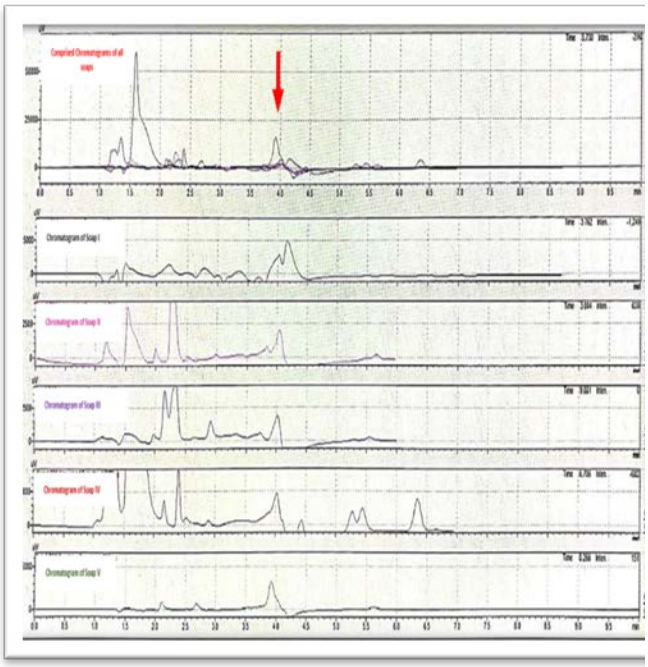

Figure 8: HPLC chromatograms of the five studied soaps. The arrow indicates the retention times correspond to TCS.

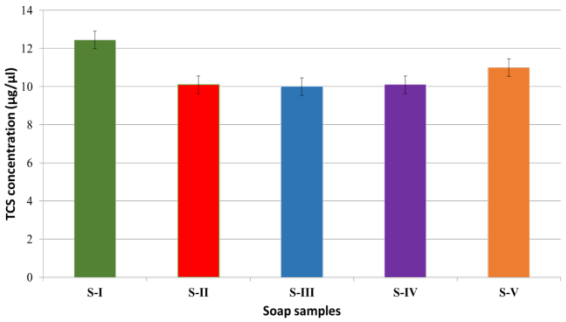

Figure 9: TCS concentration (µg/µl) in all the studied soaps.

HPLC

The HPLC chromatograms of the studied soaps confirmed the FTIR results of the presence of TCS in studied soap products. The injected volumes of standard TCS at a flow rate of 0.7 mL/min prepared at concentration of 0.03 g TCS with 50 mL mobile phase represented with characteristic peaks at a retention time ranging from 3.7 – 4.3 min (Figure 7). The chromatograms of the soap samples were detected by comparing the retention times of samples to the retention time of standard reference TCS. The optimum mobile phase composition was acetonitrile:water, 70:30, while the optimum flow rate was 0.7 mL/min. The oven temperature of 35 °C and wavelength of 280 nm were used. HPLC is a good technique to confirm the FTIR results of the presence of TCS in the studied samples since all the soaps revealed a peak at a retention time from 3.7 – 4.3 min. Furthermore, the HPLC was used to quantify the TCS in each sample. A serial of TCS standard concentrations gave a linear calibration with area under the curve (AUC) with a correlation coefficient of 0.9999. In the present study, five commercially available soaps in the pharmacies were analyzed for the presence of TCS under the proposed HPLC method. Figure 8 shows the chromatograms of the five soap with a characteristic peak with retention times within the range of 3.7 – 4.3 min, which is the same range for the retention time of standard TCS. However, each of the five soaps showed different AUCs, which indicates different TCS concentrations. Figure 9 showed that some of the commercial soaps contain more TCS than others although all of the manufacturers did not mention that on the label, which is considered as illegal according to the FDA recommendations in 2016 (16). This was consistent with a previous study where all tested products contained TCS at different concentrations (19). According to our results of analysis, it can be demonstrated that the proposed method of HPLC analysis is good for identification and quantification of different soap bars for the presence of TCS.

CONCLUSION:

Both FTIR and HPLC methods offer good qualitative and quantitative analysis of different personal care products. Our proposed HPLC method was valid in representing the exact content of certain substances such as TCS in the studied household soap products. The results of analysis demonstrated that TCS is present in many personal care products although the manufacturer labels did not claim that. Therefore, more efforts and inspections are recommended to test the commercial personal care products to ensure they are TCS-free.

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