



A Combination of Waste Biomass Activated Carbon and Nylon Nanofiber for Removal of Triclosan from Aqueous Solutions

Nor Khorih Eliysa Mohd Khor¹, Salmiati^{1,2*}, Tony Hadibarata³, Zulkifli Yusop^{1,2}

¹Department of Water and Environmental Engineering, School of Civil Engineering, Faculty of Engineering, Universiti Teknologi Malaysia, 81310 Skudai, Johor, Malaysia.

²Centre for Environmental Sustainability and Water Security (IPASA), Research Institute for Sustainable Environment (RISE), Universiti Teknologi Malaysia, 81310 Skudai, Johor, Malaysia.

³Department of Environmental Engineering, Faculty of Engineering and Science, Curtin University, Malaysia, CDT 250, 98009 Miri Sarawak, Malaysia

Received: 25/03/2020

Accepted: 26/06/2020

Published: 20/09/2020

Abstract

Triclosan (TCS) is one of the biocide used as antibacterial and antifungal agent to kill and hinder the growth of bacteria and also it is used in many personal care and health care products. However, TCS can cause health and environmental problems such as environmental pollutions, acute toxicity, etc. The aim of this study is to investigate the removal of TCS from aqueous solution by combining the coconut pulp waste (*Cocos nucifera*) activated carbon (AC) with nylon 6,6 membrane. To this end, first, the effects of physico-chemical characteristics of the membrane were studied. The nylon 6,6 membrane (14 wt.%) was prepared using electrospinning machine with injection rate at 0.4 mL/h, tip-to-collector distance at 15 cm, rotation speed at 1000 rpm, and applied voltage at 26 kV. The parameters studied for the membrane during the adsorption test were contact time, adsorbent dosage, agitation speed, initial TCS concentration, pH, and temperature of the TCS solution. The filtration test was done using flat sheet membrane test machine at pressure 1.0 bar. The characteristics of the membrane were analysed using the FESEM and FTIR tests. Based on the obtained results, the nylon 6,6 membrane can remove 90.2% of TCS within 5 minutes; the removal rate increased to 100% in less than 5 minutes after the membrane was combined with AC. This study proved that the combination of AC and nylon 6,6 membrane is able to maximize the TCS removal from water.

Keywords: Triclosan; Activated carbon; Coconut pulp waste; Nylon 6,6 membrane

1 Introduction

Triclosan (TCS) (5-chloro-2-(2,4-dichlorophenoxy) phenol) is a chlorinated aromatic compound with the molecular formula $C_{12}H_7Cl_3O_2$ and molecular weight of 289.54 g/mol. This organic compound, in the form of white powders, has functional groups of both ethers and phenols. Triclosan is one of the antibacterial and antifungal agents that are normally used in medical and consumer products, such as surgical scrubs, toothpastes, hand wash soaps, mouthwash, shampoos, plastics, toys, textiles, and deodorants [1]. It has the ability to hinder the growth of microorganisms, and due to its presence in many consumer products, it has been detected in most of the sediments, biosolids, surface water, soil, and aquatic species [2].

Though TCS is an antibacterial agent, it also poses a potential risk to the human health and the environment. Zhu *et al.* [3] reported that TCS was detected in 97% of urine samples of 471

men with concentrations from 0.41 to 2.95 ng (mg creatinine)⁻¹ and it caused some adverse effects to the semen quality such as low sperm production and poor forward mobility. Moreover, TCS has a high bioaccumulation potential and it can enter the food web system [4]. TCS can also cause toxicity to some aquatic life species such as algae, planktons, fishes, and frogs [5-10].

Therefore, several treatment methods have been implemented to remove TCS from the water, including those using cellulose acetate (CA) membrane [11], ammonia amendment and bioaugmentation in nitrifying activated sludge [12], dielectric barrier discharge plasma combined with activated carbon fibers [13], structure-directing agent modified mesoporous MIL-53 (Al) [14], dissolved organic matter on soybean peroxidase-mediated [15], ozonation [16], and microalgal species [17]. However, these treatments involved complex procedures, high costs of treatments and maintenance, large volumes of chemicals, and long

Corresponding author: Salmiati, (a) Department of Water and Environmental Engineering, School of Civil Engineering, Faculty of Engineering, Universiti Teknologi Malaysia, 81310 Skudai, Johor, Malaysia. (b) Centre for Environmental Sustainability and Water Security (IPASA), Research Institute for Sustainable Environment (RISE), Universiti Teknologi Malaysia, 81310 Skudai, Johor, Malaysia.

³Department of Environmental Engineering, Faculty of Engineering and Science, Curtin University, Malaysia, CDT 250, 98009 Miri Sarawak, Malaysia. Email: salmiati@utm.my.

processing times [18].

In recent years, the adsorption process has been one of the popular methods applied to remove chemicals and dyes in water and wastewater treatments due to its advantages of having less processing procedures with less sludge being produced. Several adsorption studies to remove TCS were done using rice straw-derived activated carbon [19], charcoal-based activated carbon [20], conventional activated carbon [21], civilian protective gas mask activated carbon [22], magnetic carbon composites from hydrochar [23], and wastewater biosolids-derived biochar [24]. High surface areas, micro-porous structures, and high degrees of surface reactivity cause activated carbons to become versatile adsorbents, particularly effective for the adsorption of organic and inorganic pollutants from aqueous solutions [25].

However, the preparation of commercial activated carbons is a costly activity, which has encouraged researchers to search for low-cost materials as alternatives [26]. There are many types of natural wastes used as low-cost adsorbents such as human hair, sheep wool, cane bagasse, and many more [27, 28]. Among all, agricultural wastes are one of the promising sources as they are inexpensive, easy to collect, and environmentally friendly [29]. Furthermore, they have a high efficiency in trapping and removing chemicals and dyes from water due to possessing many functional groups such as alcohols, phenolic, amido, amino, carboxyl, carbonyl, and ester [30]. The agricultural wastes from coconut trees have become one of the promising materials to be used as adsorbents due to their abundance in nature, cheaper price, high porous structures, and high absorption capability. The coconut tree parts commonly used as adsorbents are the bunch [29], frond [31], pulp waste [32], husk [33], coir [34], leaves [35], and shell [36].

In addition, membrane is one of the technologies used for removal of various chemicals and pollutants from water. This advanced, well-known treatment technology has become one of the most preferred options for water and wastewater treatments in food industries, chemical industries, and pharmaceutical industries [37-39]. Such popularity of membrane treatment is because of many advantages such as no addition of chemicals required, no secondary pollutants produced, low energy consumption, easy to handle, low operating and maintenance costs, easy to scale-up, high porous structure, and high recovery and reusability [40, 41]. Most of the membranes are made from polymeric materials. Nylon 6,6 is one of the polyamide group that is excellent in mechanical strength, toughness, rigidity, and stability with self-lubricating properties and cost effectiveness in nature [42, 43]. It is also hydrophilic, thin enough, highly porous, highly permeant, acceptable in fouling resistant, and low complicated in structures [44]. These advantages have promoted nylon as a functional polymer for many biomedical and environmental applications [43].

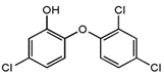
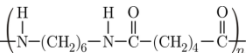
However, finding the best and the most affordable treatments for TCS (because of its long-term negative effects on the aquatic life, wild life, and human health) has remained a concern for researchers working in this field. Therefore, the aim of this research is to study the efficiency of combining both adsorption and filtration methods to remove TCS from water. The objectives of this study are to investigate the physico-chemical characteristics of nylon 6,6 membrane and to examine their effect on the TCS removal from water.

2 Materials and Methods

2.1 Chemical

In this study, the following chemicals were used: TCS and acetic acid that were supplied by Merck KGaA (Darmstadt, Germany), Tween 80 supplied by Sigma-Aldrich, ethanol 96% obtained from Qrec™ (Malaysia), Nylon 6,6 (polyamide 6,6) pellets supplied by DSM Co. (Netherlands), and formic acid supplied by HmbG Chemicals (Barcelona, Spain). The coconut pulp waste activated carbon was prepared during the preliminary study [45]. The physical and chemical characteristics of TCS and nylon 6,6 are shown in Table 1.

Table 1: Physico-chemical characteristics of TCS and nylon 6,6

	TCS	Nylon 6,6
Chemical structure		
IUPAC name	5-chloro-2-(2,4-dichlorophenoxy)phenol	Poly[imino(1,6-dioxohexamethylene) iminohexamethylene]
Appearance	White solid	White pellets
Physical state	Solid	Solid
Molecular weight	289.54 g·mol ⁻¹	262.3458 g mol ⁻¹
Chemical formula	C ₁₂ H ₇ C ₁₃ O ₂	C ₁₂ H ₂₂ N ₂ O ₂
Solubility	ethanol, methanol, diethyl ether, strongly basic solutions, and slightly soluble in water (10mg/L at 20°C)	Soluble in acid, slightly soluble in boiling water
Melting point	55-57 °C	255-265 °C

A 500 mg/L of TCS stock solution was prepared in 500 mL volumetric flask by dissolving 250 mg TCS powder into 500 mL ethanol with 0.1% Tween 80. Tween 80 is a surfactant that can solubilize hydrophobic organic compound and increase the treatment process efficiency to remove hydrophobic particles in water and soil. As stated by Cheng *et al.* [46], Tween 80 has many advantages such as cheap, low toxicity to environment, low polarity, and also has high solubilization capacity. As followed by Behera *et al.* [20], the stock solution was stored in refrigerator at temperature ± 4 °C and was used within one month from its preparation date. The standard solutions for adsorption process were prepared by diluting stock solution with distilled water.

2.2 Preparation of the nylon 6,6 membrane

Nylon 6,6 membrane was fabricated in an electrospinning machine (FNM Ltd., Iran). The preparation of one membrane sheet was done within three days. Firstly, 5 ml of acetic acid and 5 ml of formic acid were poured in a 30 ml glass bottle. Then, 1.40 g of Nylon 6,6 pellets were weighed using analytical weighing scale. The Nylon 6,6 pellets were dissolved with acetic acid-formic acid solution using magnetic stirrer for 12 hours. This procedure was done in order to get a homogenous solution.

For the electrospinning process, several parameters were set up for fabrication of Nylon 6,6 membrane. 5 ml syringe and 0.6 x 32 mm needles supplied by Terumo® (Canada) were used in this process. Firstly, the collector drum was covered with aluminum net as a membrane base support. This step was carried out so that the membrane sheet can be removed easily. The Nylon 6,6 solution prepared was sucked into the syringe and placed at the

syringe pump holder. A high voltage was supplied by clipping a small crocodile clipper at the middle of the needle used. As performed by Jasni *et al.* [43], the jetting flow rate, supplied voltage, drum collector speed, and tip-to-collector distance were set to 0.4 mL/h, 26 kV, 1000 rpm, and 15 cm, respectively. Then, the membrane sheet produced was removed from the drum collector and was dried in a clean cupboard at room temperature for 24 hours. Then, it was stored in a clean container for further research.

2.3 Adsorption studies

The adsorption studies of TCS adsorption using Nylon 6,6 membrane were conducted by means of 100 mL conical flask. Based on Jasni *et al.* [43], the batch studies were conducted in order to analyse the effect of various parameters on the uptake of TCS onto Nylon 6,6 membrane. As suggested by Muhamad *et al.* [47], for adsorption performance experiment, the membrane sheet was cut to smaller pieces (5 mm x 5 mm) before being weighed using analytical weighing scale. The adsorption tests were conducted using a conical flask and a shaker [43, 48]. The effects parameters analysed in this experiment were contact time, adsorbent dosage, agitation speed, initial TCS concentration, initial pH of TCS solution, and temperature.

The effects of contact time were studied from 10 minutes until 6 hours. Next, the effects of membrane dosage were investigated using 0.01 g, 0.05 g, 0.10 g, 0.15 g, and 0.20 g of nylon 6,6 membrane. For agitation speed, the speeds of orbital shaker were tested from 50 rpm to 250 rpm with 50 rpm interval. Then, the effects of TCS initial concentration were analysed at 5 mg/L, 10 mg/L, 30 mg/L, 50 mg/L, 70 mg/L, and 90 mg/L, and the effect of pH was investigated by varying the value from 3.0 to 9.0. Lastly, the effect of temperature was studied using incubator shaker at temperature 25 °C to 60 °C. A summary of design parameters for TCS adsorption using nylon 6,6 membrane is tabulated in Table 2. The remaining of the TCS concentration in water after adsorption treatment was determined using Ultraviolet-Visible (UV-Vis) Spectrophotometer (NANOCOLOR® UV/Vis Macherey-Nagel) at maximum wavelength of 279 nm.

Table 2: The design parameters for TCS adsorption using nylon 6,6 membrane

Parameters	Contact time (hr)	Membrane mass (g)	speed (rpm)	TCS conc. (mg/L)	pH	Temperature (°C)
Contact time	0.17-6.00	0.01	150	5.0	5.6	25
Adsorbent dosage	4.00	0.01-0.15	150	5.0	5.6	25
Agitation	4.00	0.01	50-250	5.0	5.6	25
TCS conc.	4.00	0.01	150	5.0-90.0	5.6	25
pH	4.00	0.01	150	5.0	3.0-9.0	25
Temperature	4.00	0.01	150	5.0	5.6	25-60

2.4 Filtration

Filtration experiments were done using the flat sheet membrane test. The filtration test was done to analyse the water flux, TCS flux, and permeate concentration of TCS solution after filtering with nylon 6,6 membrane. The membrane sheet was cut to an oval shape with 57 mm diameter to fit in the permeation cell. Then, the cell was tightened with screws. Afterward, the distilled water was poured in the feed tank for further tests. Next, after setting the permeation cell and tightening all screws, the membrane compaction test was conducted to allow all the

membrane pores to be filled and be in contact with water. The compaction test was done using 4000 mL distilled water with 1.5 bar pressure for one hour or until a stable permeation rate was achieved. The volume of permeate water from the membrane test cell outlet was recorded every 5 minutes to check the permeation rate pattern. After the permeation volume became stable, the pressure was reduced to 1.0 bar.

Then, the water flux test proceeded using distilled water. The volume of permeate water were recorded every 5 minutes. After that, the distilled water was removed and replaced with 4000 mL of 5 mg/L TCS solution for filtration process and TCS flux analysis. For TCS flux experiment, the volume of TCS solution permeated from the permeation cell was recorded every 5 minutes. Besides that, the TCS samples were taken from permeation cell and feed tank for TCS removal analysis. The entire membrane filtration tests were done at room temperature. The flux value was calculated using Equation (1) as follows:

$$\text{Flux, } J \text{ (L/m}^2\text{h)} = (V/t) / A \quad (1)$$

where V/t (L/h) is volume permeation rate and A (m^2) is membrane area ($A=0.002124 \text{ m}^2$).

2.5 Combination of activated carbon and nylon 6,6 membrane

The coconut pulp waste activated carbon and nylon 6,6 membrane were combined in order to maximize the TCS removal in water. All the optimum parameters conditions were obtained from the batch adsorption and membrane experiments. The combination test was done using flat sheet membrane test and a stand stirrer. A piece of fabric was installed at the inlet pipe in order to prevent the activated carbon from entrance to the membrane machine. The nylon 6,6 membrane was put in the permeation cell. After the compaction test, 4000 mL of the TCS solution with initial concentration at 5 mg/L was poured into the feed tank together with coconut pulp activated carbon. A stand stirrer was setup beside the feed tank with its stirrer pointed in the tank. Then, the TCS solution was stirred with activated carbon for 20 minutes. After that, the inlet valve was opened and TCS solution was filtered with nylon 6,6 membrane. The concentration of TCS permeated from the membrane was collected and analysed.

2.6 Characterizations of nylon 6,6 membrane

In this research, the surface structure and morphology of the nylon 6,6 membrane were analysed using Field Emission Scanning Electron Microscopy (FESEM) (FESEM, JEOL 6335f-SEM, Japan) test. The test was done for the membrane before and after treatments of TCS solution. As recommended by Jasni *et al.* [43], before conducting the FESEM test, the samples were coated using a gold sputter of a Bio Rad Polaron Division SEM coating system machine at 10^{-1} Mbar in order to reduce charging. Then, they were inserted in FESEM instrument to analyse their surface structures and morphologies. The magnifications scales were used from 5000x to 10000x.

In addition, the functional groups existed on the membrane were analyzed using Fourier Transform Infrared Spectroscopy (FTIR) (Perkin-Elmer spectrum ONE). The FTIR test was recorded in the spectral range of 4000 to 400 cm^{-1} at resolution 4 cm^{-1} . For the nylon 6,6 membrane, the FTIR analysis was conducted using the Attenuated Total Reflectance (ATR) technique. ATR mode was used because the membrane was

already in solid form and not in powder form. The ATR technique allows the solid or liquid samples to be examined directly without any preparation in advance.

3 Results and Discussion

3.1 Characteristics of the nylon 6,6 membrane

3.1.1 Surface morphologies

The FESEM test was done to analyze the surface morphologies of the nylon 6,6 membrane. Figures 1 (a) and (b) show the FESEM images of this membrane before and after the TCS adsorption, respectively. As can be seen in Figure 1 (a), the morphology of nylon 6,6 fiber threads appear to be thin, smooth, free from beads, and continuous. These results show that, the optimum electrospinning parameters used during the production of nylon 6,6 membrane can produce a quality nanofiber. Based on Figure 1 (b), on the other hand, can be used to analyse the activated carbon after adsorption of TCS. The image shows that the nylon 6,6 nanofiber threads were filled up by a lot of particles until most of the nanofiber threads were covered. This showed that the nylon 6,6 membrane can adsorb and trap TCS particles in aqueous solutions.

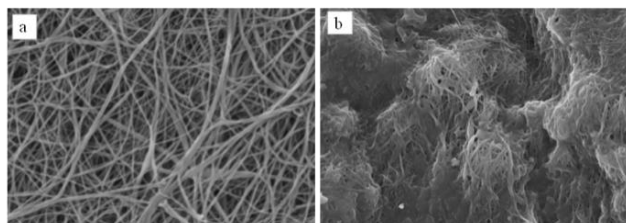


Figure 1: FESEM image of the nylon 6,6 membrane (a) before adsorption (magnification x10000) and (b) after TCS adsorption (magnification x5000)

3.1.2 Functional groups

FTIR is one of the important methods to identify and determine the functional groups of adsorbent samples and it influences the occurrence of the adsorption process. Figure 2 shows the FTIR spectra of the nylon 6,6 membrane. According to the obtained results, the nylon 6,6 has a medium band at peak 3298cm^{-1} that was attributed to the N-H stretch from amino groups [41]. Following this, a C-H stretching vibration due to alkanes group was observed with a medium peak at 2934cm^{-1} . At peaks 1636cm^{-1} and 1535cm^{-1} , two strong peaks were detected, and after 1500cm^{-1} , all the peaks weakened in intensity. The strong peaks formed at peaks range from 1500cm^{-1} to 1700cm^{-1} , due to amide I and II bands [42]. At peak 1636cm^{-1} , C=O stretching from the carbonyl group can bind with the amino group to form intra molecular hydrogen bonding, causing the C=O stretching that normally forms at peak 1760cm^{-1} to 1665cm^{-1} to be shifted to 1636cm^{-1} [43]. Meanwhile, the amide II band at peak 1535cm^{-1} appeared due to C-N stretching and N-H bonding.

Xu *et al.* [40] stated that “the hydrogen bonding interactions might play an important role in the sorption processes, because hydrogen bonds could be formed between phenolic hydroxyl group of TCS acting as hydrogen-bonding donors and carbonyl groups of electrospun fibrous membranes”. This FTIR test showed that the nylon 6,6 membrane has a carbonyl group and it can produce hydrogen bonding with the hydroxyl group of TCS molecules [43]. Therefore, it can be proven that the chemisorption process happened during the TCS removal using nylon 6,6

membrane.

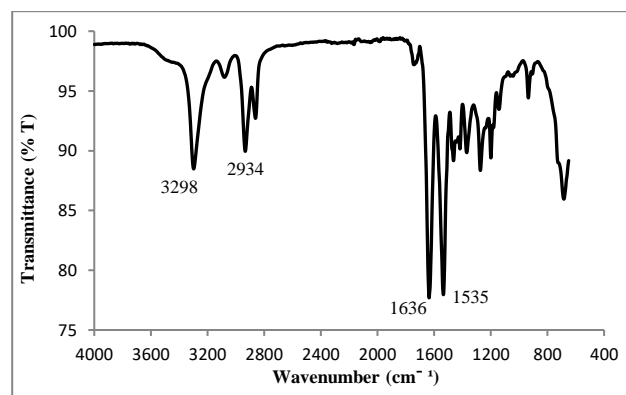


Figure 2: The FTIR spectra of the nylon 6,6 membrane

3.2 Adsorption studies

3.2.1 Contact time

The contact time was measured to determine the maximum time taken for adsorbate removal and adsorption capacity of the adsorbent until it reaches the equilibrium condition. The effect of contact time was examined through applying 0.1 g nylon 6,6 membrane to treating 50 ml of 5 mg/L TCS solution.

In Figure 3, it shows the effect of contact time on TCS removal by the nylon 6,6 membrane. The graph shows that the TCS removal and adsorption capacity increased with an increase of contact time. The TCS removal increased from 40.2% to 86.3% by the 4th hour before reaching its equilibrium conditions. Then, the TCS removal started to slightly decrease during the 5th and 6th hours from 85.3% to 83.3%. Meanwhile, the adsorption capacity also increased from 1.01 mg/g to 2.16 mg/g for 10 minutes during 4 hours of contact time before it decreased from 2.13 mg/g to 2.08 mg/g during the 5th to 6th hours of contact time. Therefore, the optimum time taken for TCS removal using nylon 6,6 membrane to reach equilibrium was achieved with 4 hours of contact time.

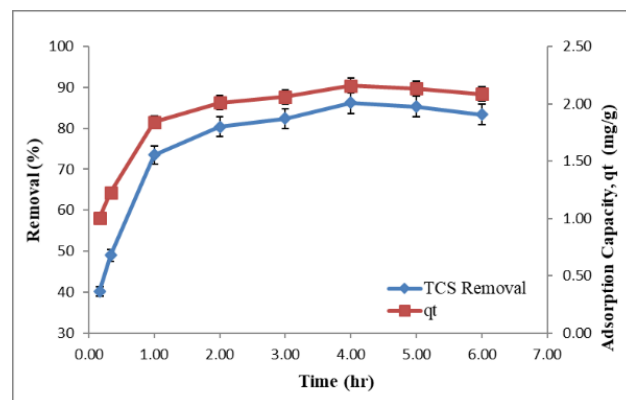


Figure 3: The effect of contact time on the TCS removal using nylon 6,6 membrane

According to the results, the higher and rapid adsorption rates at the initial period were due to the number of vacant sites available at the initial stage on the external surface of nanofibrous adsorbents [49]. On the other hand, the slow adsorption rate from 2 hours to 4 hours of contact time was due to the availability of

decreased membrane pores and the fact that TCS had to move into deeper sites [26,49]. The decrease in removal percentage after equilibrium might also be due to some adsorbate particles which started being released from the adsorbent surface into the solution [30]. The same pattern for adsorption capacity was also achieved in [50] for chitosan/PVA nanofibers to remove nickel and cobalt where it reached equilibrium at 120 min. Razzaz *et al.* [49] also reported the same trends for adsorption capacity of chitosan/TiO₂ nanofibrous for Pb(II) and Cu(II) ions removal, reaching equilibrium at 30 min of contact time.

3.2.2 Adsorbent dosage

Adsorbent dosage is a parameter that affects the availability of the adsorption sites. The effect of adsorbent dosage was examined by varying the amount of nylon 6,6 membrane from 0.01 g to 0.20 g. The experiments were conducted at room temperature to treat 50 ml of 5 mg/L TCS solution, at 150 rpm. Figure 4 shows the effect of adsorbent dosage on TCS removal using the nylon 6,6 membrane. As depicted by the graph, the removal of TCS increased from 56.9% to 86.3% with an increase of membrane mass from 0.01 g to 0.10 g. However, when the mass of the nylon 6,6 was added in the range of 0.15 g to 0.20 g, the TCS removal also decreased from 82.4% to 70.6%. On the other hand, the adsorption capacity of nylon 6,6 membrane was found to decrease by increasing the membrane mass. The adsorption capacity decreased from 14.22 mg/g to 0.88 mg/g when the membrane was increased from 0.01 g to 0.20 g. Thus, the optimum membrane dosage to remove 5 mg/L TCS solution was achieved with 0.10 g of the adsorbent.

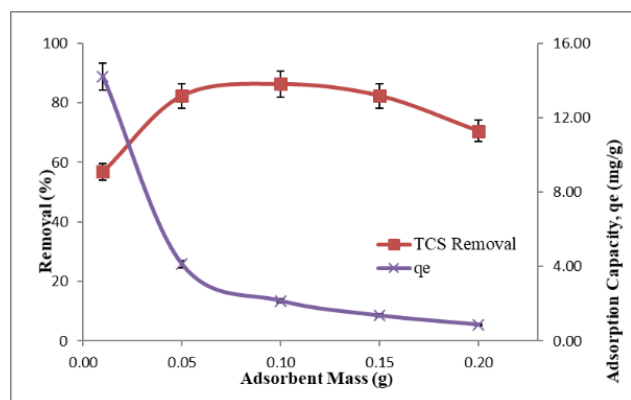


Figure 4: The effect of adsorbent dosage on the TCS removal using nylon 6,6 membrane

The increase in removal percentage from 0.01 g to 0.10 g was due to the high surface area and the availability of binding sites for adsorption [51]. As for the decrease in the removal after the optimum dosage was given, it was attributed to the overlapping of adsorption sites due to crowded membranes that would reduce the active sites for adsorption [15]. Moreover, the decrease of adsorption capacity with an increase in the adsorbent dosage was due to the fact that the adsorbent sites available were not fully utilized at a higher adsorbent dosage. When the adsorbents increased, there were more sites available in number which finally reduced the amount of TCS adsorbed for each unit weight of membranes [52]. Additionally, at a high adsorbent dosage, the interfacial tension between two phases increased, which reduced the driving force for the mass transfer, hence reducing the

adsorption capacity [53]. A similar trend for effect of membrane dosage was also reported by Li *et al.* [15] in their study into methylene blue dye removal using calcium alginate membrane. The study achieved the optimum dye removal of 96.0% using 20 mg of membrane.

3.2.3 Agitation

Agitation was performed to ensure the maximum contact of fiber surface with the TCS in the solution [48]. The effect of agitation was investigated by varying the agitation speed of the shaker from 50 rpm to 250 rpm. Figure 5 shows the effect of agitation speed on TCS removal using the nylon 6,6 membrane. According to the graph, the optimum TCS removal and adsorption capacity of 86.3% and 2.16 mg/g, respectively, were achieved at 150 rpm. A slower speed will reduce mobility and transfer force between TCS and the adsorbent, while a higher speed will lead to weakening of the bonding strength between the TCS and the membrane surface area. It results in low removal percentage and adsorption capacity using activated carbon. Therefore, the best agitation speed for TCS adsorption using the nylon 6,6 membrane was achieved at 150 rpm, and it was used for all the other parameters during this study.

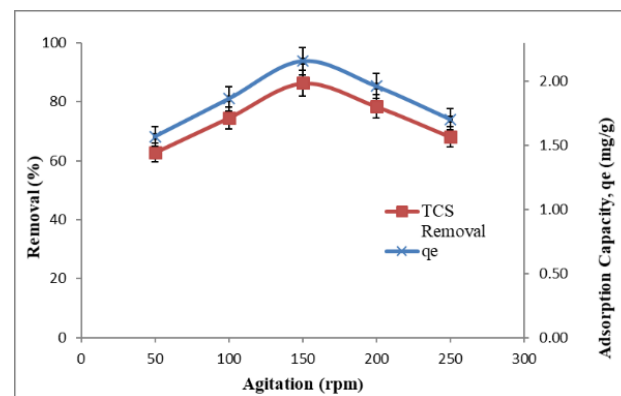


Figure 5: The effect of agitation speed on the TCS removal using nylon 6,6 membrane

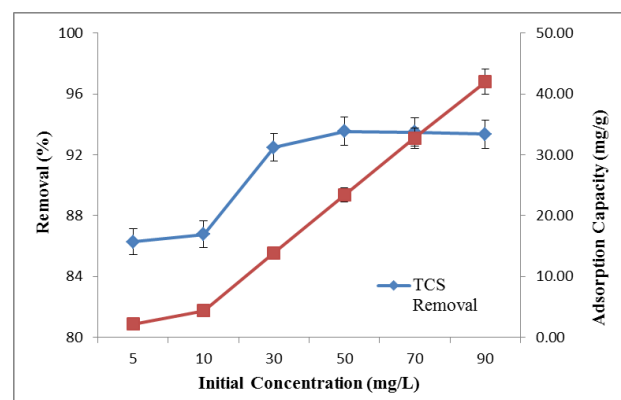


Figure 6: The effect of initial TCS concentration on the TCS removal by using nylon 6,6 membrane

3.2.4 Initial TCS concentration

The effects of initial TCS concentrations were tested from 5 mg/L to 90 mg/L, while the other parameters were kept constant. Figure 6 shows the effect of initial TCS concentration on its

removal using nylon 6,6 membrane. From the graph plotted, when the initial concentration of TCS was increased from 5 mg/L to 50 mg/L, the TCS removal also increased from 86.3% to 93.5%. This was due to the many active sites available for the adsorption process at low concentrations [54]. However, when the TCS concentration was increased to 70 mg/L and 90 mg/L, the removal percentage almost remained constant with a slight decrease to 93.5% and 93.4%, respectively. According to Feng *et al.* [55], this could be due to the decrease and the saturation of active sites on the adsorbent surfaces. While the membrane adsorption capacity increased with an increase in the TCS initial concentration. The nylon 6,6 membrane adsorption capacity increased from 2.16 mg/g to 42.01 mg/g in 5 mg/L to 90 mg/L TCS concentrations. Increase of the concentration will increase the electrostatic interactions between the adsorbate and adsorbent, hence improving the adsorption capacity of the adsorbent [56].

Literature consists of a number of studies reporting similar trends on the effect of the initial concentration using various nanofibers. Li *et al.* [15] reported that the adsorption capacity of calcium alginate membrane increased from 520 mg/g to 1680 mg/g when methylene blue initial concentration was increased from 30 mg/L to 170 mg/L.

3.2.5 The pH effect

The effect of pH value was tested through fixing it at 3.0-9.0. The pH values selected were in range of TCS pK_a value and isoelectric point of the nylon 6,6 membrane. Figure 7 shows the effect of pH on the TCS removal using the nylon 6,6 membrane.

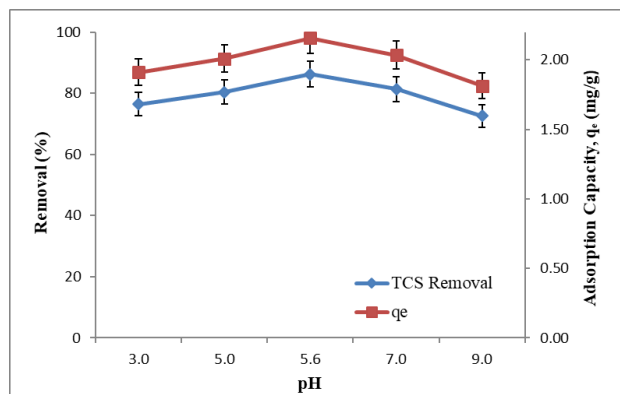


Figure 7: The effect of pH on the TCS removal using nylon 6,6 membrane

As shown by the graph, the highest removal percentage and adsorption capacity of 86.3% and 2.16 mg/g, respectively, were achieved at pH 5.6. The other pH values resulted in lower TCS removal percentages and adsorption capacities. At a pH lower than TCS $pK_a = 7.90$, the TCS molecules are available in a protonated form [40]. Based on Jasni *et al.* [43], the isoelectric point of the nylon 6,6 membrane is at pH 5, where the nylon 6,6 membrane is in positive charge at pH 5 and below, but it turns to negative charge with pH values higher than 5. Therefore, the TCS removal and adsorption capacity were low at pH 5 and below due to the electrostatic repulsions between the positive charge of TCS and the positive charge of the nylon 6,6 membrane. On the contrary, for pH higher than 5, the nylon 6,6 membrane was turned to the negative charge nanofiber. As a result, it increases both TCS adsorption and adsorption capacity due to attraction

forces between the positively charge TCS and the negatively charge membrane. At pH values higher than 7.90, TCS particles will be deprotonated and become negatively charged. Thus, the electrostatic repulsion between the deprotonated TCS and nanofibers caused a reduction for both TCS removal and adsorption capacity [40]. Xu *et al.* [40] also reported a similar trend for the adsorption of TCS using electrospun fibrous membranes and achieved the best TCS removal at pH 6 compared to those fixed at pH 4, 8, and 10.

3.2.6 Temperature

The surrounding temperature influences the bonding strength between TCS particles and the surface of the membrane. The temperature was varied from 25 °C to 60 °C to treat 50 mL of the 5 mg/L TCS solution. The experiments were conducted in an incubator shaker. Figure 8 shows the effect of temperature on the TCS removal using the nylon 6,6 membrane. The results show that the TCS removal and adsorption capacity decreased with an increase in temperature. The TCS removal and adsorption capacities both decreased from 86.3% to 55.9% and from 2.16 mg/g to 1.40 mg/g, respectively, when the temperature was increased from 25 °C to 60 °C. The melting point of TCS is 57 °C; therefore, TCS solubility in water increases when the temperature increases, hence causing the adsorption process to recede [57]. Furthermore, increasing the temperature weakens the electrostatic interactions between the adsorption surface and adsorbate particles and this will reduce the adsorption capacity and TCS removal [15]. The adsorption of TCS using the nylon 6,6 membrane was an exothermic process since its removal rate and adsorption capacity lowered with the increase of temperature [53].

Aluigi *et al.* [53] also reported a similar trend for the removal of methylene blue using keratin nanofibrous membrane where the adsorption capacity and the methylene blue removal both decreased from 175 mg/g to 128 mg/g and from 68% to 51%, respectively, when the temperature was increased from 20 °C to 50 °C.

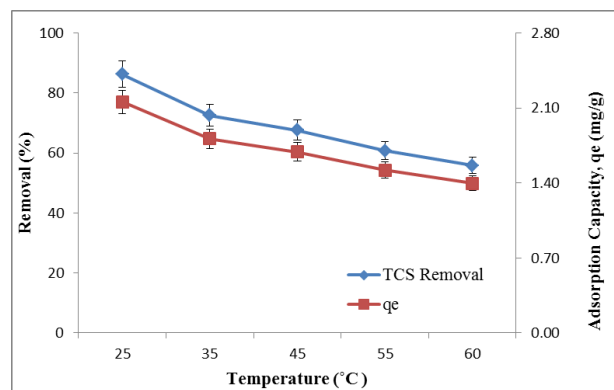


Figure 8: The effect of temperature on the TCS removal using nylon 6,6 membrane

3.3 Filtration studies

3.3.1 Compaction

For the filtration method, the pre-compaction test was necessary before the compound filtration process can be done. This procedure was done in order to reduce the interference of compaction with other factors such as fouling, and the test was

conducted until a steady flux was achieved [58]. The pre-compaction test was done in order to maximize the water contact in all membrane pores before the filtration test can be done. The time taken to achieve a steady flux can vary based on the types and material of membrane. Hussain and Al-Saleh [58] also stated that the steady flux for ultrafiltration membranes can be reached within minutes, but it will take more time for reverse osmosis membranes. For the compaction test, a volume of 4000 mL distilled water was used and poured into the feed tank. The test was conducted for 90 minutes while the pressure was set to 1.5 bar.

The water flux achieved during the pre-compaction test using the nylon 6,6 membrane is shown in Figure 9. Based on the results, the water flux decreased from 2028 L/m²h to 1570 L/m²h and reached a steady flux in 75 minutes. The compaction process caused the membrane structure porosity to be reduced and a flux reduction occurred [58]. After a steady flux was achieved, the compaction test was stopped and the pressure was reduced to 1.0 bar before continuing further experiments.

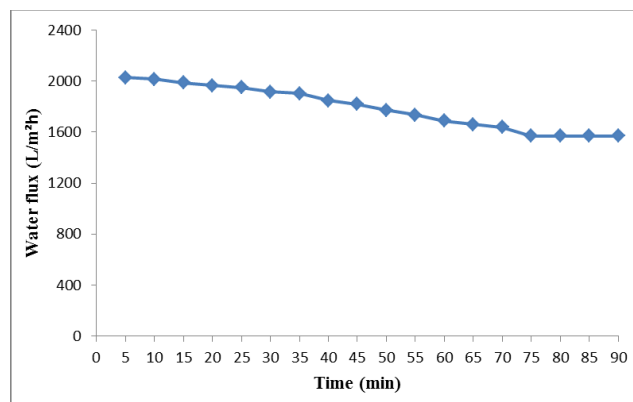


Figure 9: The water flux during pre-compaction test

3.3.2 Water flux and TCS solution flux analysis

After the compaction process, the pressure was decreased to 1.0 bar. Subsequently, the flux analysis using distilled water was conducted for 30 minutes at pressure 1.0 bar. Figure 10 shows the distilled water flux, TCS solution flux, and TCS removal within 30 minutes. As can be observed in the figure, the distilled water flux is higher than the TCS solution flux. The water flux was recorded with 1655 L/m²h during the first 5 minutes before it decreased to 1491 L/m²h at 30 minutes. As for the TCS solution, the flux also decreased from 1209 L/m²h to 1062 L/m²h, from 5 to 30 minutes. The reduction of flux when using TCS solution might be due to the presence of TCS particles trapped into the membrane pores, causing their porosity to be reduced for water permeation.

The removal of TCS by filtration method decreased with the increase in time. The TCS removal decreased from 90.2% to 17.7% from 5 minutes to 30 minutes. The same results were also reported by Muhamad *et al.* [47] in their experiment for the removal of BPA using PES-SiO₂ membrane, where the BPA removal decreased from 81% to 52% from 10 minutes to 170 minutes during the filtration test. This occurred due to the reduction of adsorption sites available for TCS after its saturation [47]. According to the obtained results, the TCS achieved its maximum removal within 5 minutes of the filtration process.

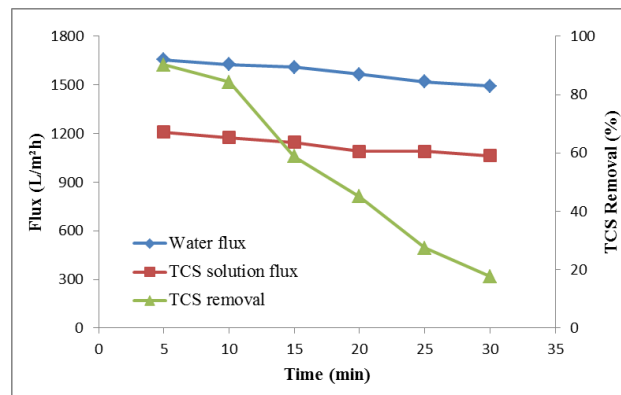


Figure 10: The results of distilled water flux, TCS solution flux, and percentage TCS removal obtained using nylon 6,6 membrane through filtration process

3.4 The effect of combined activated carbon and membrane on TCS removal

The combination method was done in order to find the best setting for TCS removal from aqueous solutions. The experiments on the combination method using coconut pulp waste activated carbon and nylon 6,6 membrane were conducted using a flat sheet membrane test machine. The optimal parameters values obtained from previous experiments were used in this process where 4 L of 5 mg/L TCS solution was poured into the feed tank with 8.0 g coconut pulp waste activated carbon and was stirred with a stand stirrer. After 20 minutes of the adsorption process, the TCS solution was filtered using the nylon 6,6 membrane by unfastening the inlet valve from the feed tank. The TCS solution pH at 5.6, room temperature, and inlet pressure 1.0 bar were set up for this test. Figure 11 shows the TCS removal after combining both the adsorption and filtration methods.

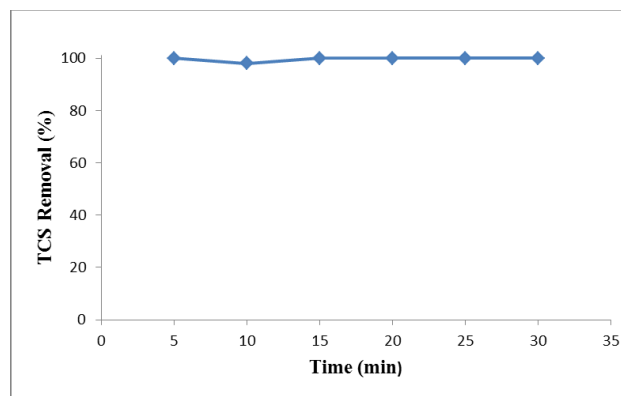


Figure 11: The TCS removal through combining the adsorption method using coconut pulp waste activated carbon and filtration method using nylon 6,6 membrane

According to the obtained results, TCS was removed 100% in 5 minutes of the filtration process where it already reached its equilibrium. During the adsorption process, most of the TCS particles in the solution were already removed. As such, the nylon 6,6 membrane only filtered the leftover of TCS particles that escaped during the adsorption process. According to Skouteris *et al.* [59] and Wang *et al.* [60], the activated carbon can be used as

a pre-treatment for membrane by removing the majority of dissolved organic matters and inorganic particles in the water, thereby reducing the membrane fouling. Therefore, it shows that this combination method can increase the TCS removal in aqueous solutions within a shorter time.

Similar results were reported by Wang *et al.* [60], using the combination of coconut shell activated carbon and polyamide NF membrane to remove dimethyl phthalate, di-(2-ethylhexyl) phthalate and dioctyl phthalate where more than 99% of removal rate was achieved for all the three chemicals.

4 Conclusion

Based on this study, 90.2% of the TCS removal was achieved using the nylon 6,6 membrane through filtration method within 5 minutes. Then, after combining both coconut pulp waste activated carbon and nylon 6,6 membrane, the TCS removal was increased up to 100% within less than 5 minutes. Therefore, this combination method can help to increase the TCS removal from the water and reduce the fouling probabilities. Based on the FESEM images, the results showed that the nylon 6,6 membrane had thin, smooth, beads-free, and continuous fibers. While the results of the FTIR test showed that the nylon 6,6 membrane had carbonyl group and it was able to produce hydrogen bonding with hydroxyl group of TCS molecules. Thus, it proved that the chemisorption process happened during the TCS removal using the nylon 6,6 membrane. As a conclusion, combining activated carbon and nylon 6,6 membrane is one of the promising methods that can help to increase the TCS removal from the aqueous solutions.

Acknowledgment

The authors would like to acknowledge the Ministry of High Education of Malaysia for providing LRGS Grant on Water Security entitled Protection of Drinking Water: Source Abstraction and Treatment (203/PKT/6720006) and Universiti Teknologi Malaysia (R.J130000.7809.4L810, Q.J130000.2522.19H06 and Q.J130000.2422.04G06) as financial support of this project.

Ethical issue

Authors are aware of, and comply with, best practice in publication ethics specifically with regard to authorship (avoidance of guest authorship), dual submission, manipulation of figures, competing interests and compliance with policies on research ethics. Authors adhere to publication requirements that submitted work is original and has not been published elsewhere in any language.

Competing interests

The authors declare that there is no conflict of interest that would prejudice the impartiality of this scientific work.

Authors' contribution

All authors of this study have a complete contribution for data collection, data analyses and manuscript writing

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