Fabrication of electrospun ultrafine fibreous membrane and their application in paraben adsorption

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K E Y W O R D S	A B S T R A C T
Electrospinning Nano Fibres Fabrication Electrospun Ultra Fibreous Paraben Adsorption	Electrospinning provides a simple and versatile method for generating ultrafine fibers from a rich variety of materials that include polymers, composites, and ceramics. Find applications in various fields, including tissue engineering, filter media, reinforcement in nanocomposites, and micro/nano-electro-mechanical systems. In this study the polystyrene fibers in 10%, 20% and 30% (w/v) in THF: DMF solvents were characterized by ATR-FTIR, AFM and SEM. The obtained fibers exhibited a smooth surface with fine structures, emphasizing a ribbon-like morphology with bead-like features within the range of 3 µm to 5 µm in diameter. The aim of this study was to extract the parabens at optimized condition as pH, adsorbent dose, contact time and the elution of solvent. To examine the mechanism of adsorption kinetics, the two most commonly used kinetics models (pseudo-first order and pseudo-second order) were employed to test experimental data along with Morris-Weber diffusion model. The obtained data showed that the maximum sorption efficiency obtained at pH 3.0, adsorbent dose optimized as 10 mg of fiber with time of 24 hours, the methanol was sufficient to elute paraben from fibers met. The experimental data of the adsorption followed by first order reaction with mass transfer-controlled diffusion mechanism.

1. Introduction

Ultrafine fibers have been examined effectively as a modern functional material [1]. They offer a substantial surface area towards multiple groups which sensing enzymes and chromophores components [2]. Fibers can be produced from various polymeric solutions including natural, synthetic, degradable and non-biodegradable polymers [3]. Solid nano-rods, nanowires, hollow cylinders, fibrils, spheres and micelles, nano fibre webs, ultrafine fiber, nano-structured walls, pores, and nano-patterned materials have been prepared using a number of methods [4-5]. Electrspun ultrafine fibers have low basis weight, small pores, and high surface areas,

make them appropriate for a wide range of uses with high permeability [6]. Polystyrene is most common polymer used in various fields as biomedical, durability and disposal petri plates due to such advantages polystyrene is cost effective, inert in nature and nontoxic [7]. Polystyrene mainly belongs to the family of vinyl group that produces wide range of polymer through vinyl monomers contain the C=C bonds and it shows super-hydrophobic nature and can be electrospun by the electrospinning technique [8]. The electrospinning process uses polymer solution with net charge density, viscosity and surface tension [9]. The electrospinning process also effected by changing the parameters such as concentration,

pressure in capillary tube solution properties, potential at the tip, distance between collector and the tip, conductivities of the solvents etc.

Generally, Alkyl esters of p-hydroxybenzoic acid known as parabens that have been used as antimicrobial and anti-fungal activities in various products and they are extensively used as preservatives in multiple commodities (e.g, skin care products, cosmetics, beverages and toothpaste) and food processing [10]. The various commercial products consist of 75% to 95% parabens at level of 0.01% - 0.3% [11]. Parabens are esters derived from large groups of synthetics of p-hydroxybenzoic acid; these synthetics esters comprised of methyl paraben (MP), butyl paraben (BP), propyl paraben (PP), ethyl paraben (EP), isopropyl paraben (IPP), isobutyl paraben (IBP) and benzyl paraben (BeP). Parabens include methyl paraben (MP), propyl paraben (PP), butyl paraben (BP) and ethyl paraben (EP) are most commonly used in daily use products [12]. Methyl paraben and propyl paraben are most commonly used in cosmetics, creams pastes, fats, oil, glues and lotions. However, the emerging evidence have now revealed that parabens found to be rising toxins and their adverse effects in environment. These parabens have an endocrine disturbing impact that causes breast cancer, and this is due to the determination of these parabens attract the high concern [13]. Available methods for the determination of these preservatives in various commercial products and food items rely on the use HPLC, GC, UPLC with reverse phase system [14]. Liquid-liquid, liquid-solid extraction, and the dilution with appropriate solvents techniques were involved for sample preparation [15]. The analysis of parabens reported by HPLC-UV, with few other techniques as thin layer chromatography (TLC), capillary zone electrophoresis (CZE) with fluorescent detection. The HPLC analysis uses C18 column where in some cases cyno-propyl or C8 have been used [16]. Detection carried out at 254 nm wavelength along with UV-V detector and 245 nm is usually fascinating value for parabens [17]. Capillary zone electrophoresis (CZE) shown the shorter separation times. However, HPLC showed better detection limits as (0.02-0.05μm mL-1) than CZE (0.16-0.21 μgmL-1) [18]. For parabens analysis, HPLC-UV technique is commonly used with few other techniques as thin layer chromatography (TLC), capillary zone electrophoresis (CZE) with fluorescent detection. The HPLC analysis uses C18 column where in some cases cyno-propyl or C8. Detection carried out at 254 nm wavelength along with UV-V detector and 245 nm is most favourable value for parabens [19]. However, capillary zone electrophoresis (CZE) showed the shorter separation

times, but HPLC showed better detection limits as (0.02-0.05μm mL-1) than CZE (0.16-0.21 μgmL-1) [20]. The development of these large number of efficient extraction methods for these preservatives that allow the study of these complex cosmetics samples and other daily use products [21]. Moreover, Solid Phase Extraction is similar to liquid-liquid extraction based on a partitioning of solutes between two phases or immiscible solvents in terms of liquid and a solid (adsorbent) phase such as C18 and Oasis HLB. Graphene-based SPE and multi walled carbon nanotube, carbon nanotubes, and functionalized magnetic particles have also been projected as an alternate material for solid phase extraction. Polyaniline-coated Fe3O4 nanoparticles were used as anion exchange magnetic adsorbents of the polar compounds.

The objective of this study was to fabricate ultrafine fibrous membrane at varied concentrations using electrospinning technique. The adsorption parabens were checked by using ultrafine fibre membrane at optimized parameters as concentrations of polystyrene and the impact of solvents system.

2. Experimental

2.1 Materials and Reagents

Amorphous polystyrene (PS) with molecular weight of Mw = 280,000 g mol-1 was purchased from Aldrich (USA). Dimethylformamide (DMF), tetrahydrofuran (THF), were from Sigma-Aldrich USA and BDH Lab Suppliers Pool, BH15-LTD England. Methanol (Fisher scientific, UK), Hexane (Kosadaq listed company Korea), Sodium hydroxide (E-Merck, Germany), Hydrochloric acid (37%, Sigma-Aldrich) were used of analytical grades. Methyl paraben (MP) was purchased from Sigma-Aldrich. Freshly prepared deionized water and milli-Q water were used entire in the study.

2.2 Instrumentation

Electrospinning set-up: In order to produce electropsun ultrafine fiber of polystyrene (PS) using high potential power supply (Har-100*12, Matsusada Co., Tokyo, Japan), that is (electrospinning device) able to generate high voltage up to 100kV.

Fourier Transform Infrared Spectroscopy (FTIR): To examine the functionality of electrospun PS fibers and to investigate the residual solvent effect on the morphology of fibers. Electrospun PS were subjected to FTIR analysis (Nicolet Avatar 330, DTGS, ZnSe ATR Crystal, Mid-IR 4000-650 cm-1 and Resolution 4 cm-1).

Scanning Electron Microscope (SEM): Surface morphology of PS ultrafine fibers was observed by employing scanning electron microscope (JEOL JSM 6380 with resolution 3.0 nm, 30kV, WD8mm, SEI) (SEM).

Atomic Force Microscopy (AFM): To confirm the desired electrospun fibers morphology AFM was used. The AFM is of Agilent Technologies 5500, high frequency Si cantilever with the length of 125 µm a force constant 42 N/m and the resonance frequency 330 KHz.

High Performance Liquid Chromatography (HPLC): A Hitachi model L-6200 coupled with a UV-Vis detector (Hitachi L-4200) was used to study the parabens interaction onto the PS ultrafine fibers. HPLC equipped with C-18 Teknokroma KROMASIL 100 (15 mm 0.46 mm, 5 µm) column at reverse phase and the solvent compositions were selected as 70% of MeOH (A) and 30% of water (B), flow rate was 1mL/min and the injection volume 20 µL. UV detection was performed for paraben i.e. Methyl paraben (MP) at 254 nm wavelength. For acquisition and evaluation of data software was used Chromquest (version 4.2).

pH-meter: Using 1M solution of each HCl and NaOH. Different pHs were prepared ranging from 2-8 by inolab 7110 pH meter equipped with a glass combined rod (BNC Germany).

3. Electrospinning and Adsorption Process

Polystyrene (PS) solutions were placed in a 5 ml syringe equipped with capillary tip or micro pipette with inner diameter of 1mm, solutions were electrospun using high voltage up-to 10kV to 20kV, a grounded aluminum foil wrapped on ground rotating (100 rmp) drum as a collector was positioned about 8 cm from the tip of the syringe. The electrospinning of polystyrene performed at relative humidity of the room maintained as 250C, 40% to 45%. The resulting (PS) electrospun ultrafine fibers were dried and solidified at room temperature. A schematic process or diagram for electrospinning as shown in following Fig.1.

Preparation of polymer solutions: Polystyrene (PS) was dissolved into solvents (THF: DMF) containing different ratio i.e. 100 :0, 80 : 20, 60 : 40, 50 : 50, 20 : 80, 0 : 100 and the concentrations of polystyrene (PS) were varied 10 wt%, 20 wt% and 30 wt% [22].

Adsorption study and procedure: Interaction of methyl paraben was studied by using 10 µg/mL at batch mode and small amount of ultrafine fibers from each compositions were kept in contact with 10 mL mixture of analyte for 24 hours and then filtered. Adsorption studies were determined by high performance liquid chromatography (HPLC) and the percentage adsorptions calculated by using formulae.

$$Adorption(\%) = \frac{H_i - H_f}{H_i} \times 100$$

Where, H_i is the initial concentration of the parabens before the adsorption in solution (µg/mL) while Hf is the final concentration of the parabens after adsorption by polystyrene (PS) fibers.
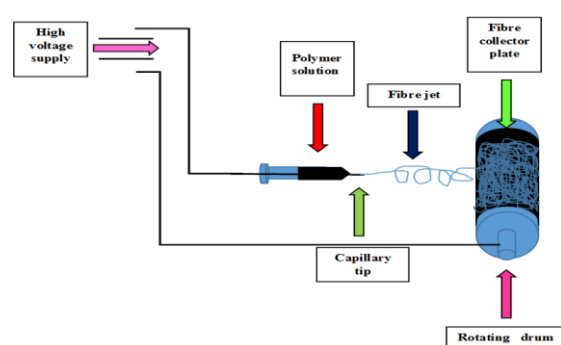

Fig. 1. Schematic diagram of electrospinning set-up for fibers

4. Characterization Electro-spun Polystyrene Fibers

FTIR-ATR Analysis: The characteristics bands of polystyrene electrospun ultrafine fibers were used 10% in solvent compositions. The stretching of C-H bond in benzene rings at 3000 cm-1 to 3106 cm-1 and C-H stretching vibration of the CH2 and CH groups present on the polystyrene chain which indicated at 2827 cm-1 and 2892 cm-1 , C-C stretch in aromatic ring around 1494 cm-1 to 1500 cm-1. The vibration of benzene ring at around 1068 cm-1 to 1141 cm-1, the band of C-H as out of plane at 694 cm-1, rocking mode of CH2 nearly at 694 cm-1. There almost no big difference was found in spectra increasing the concentration 10% to 30% but a little impact of solvents on higher concentration. Therefore, the residual solvent effect was observed in 20% and 30% due to the presence of C-O bond from THF and C=O bond and C-N bond form DMF at around 1778 cm-1, 1745 cm-1, 1035 cm-1.
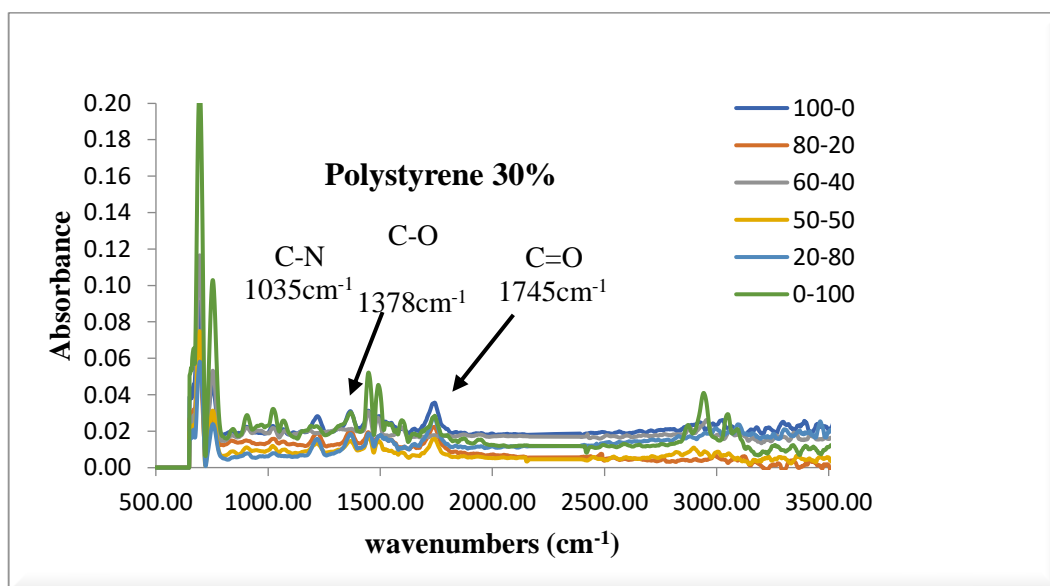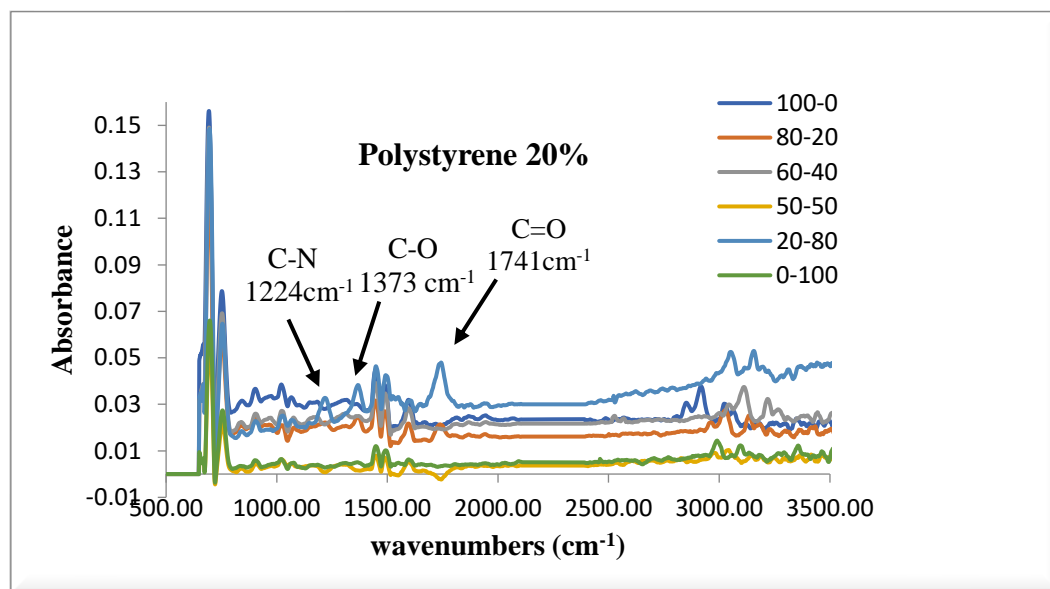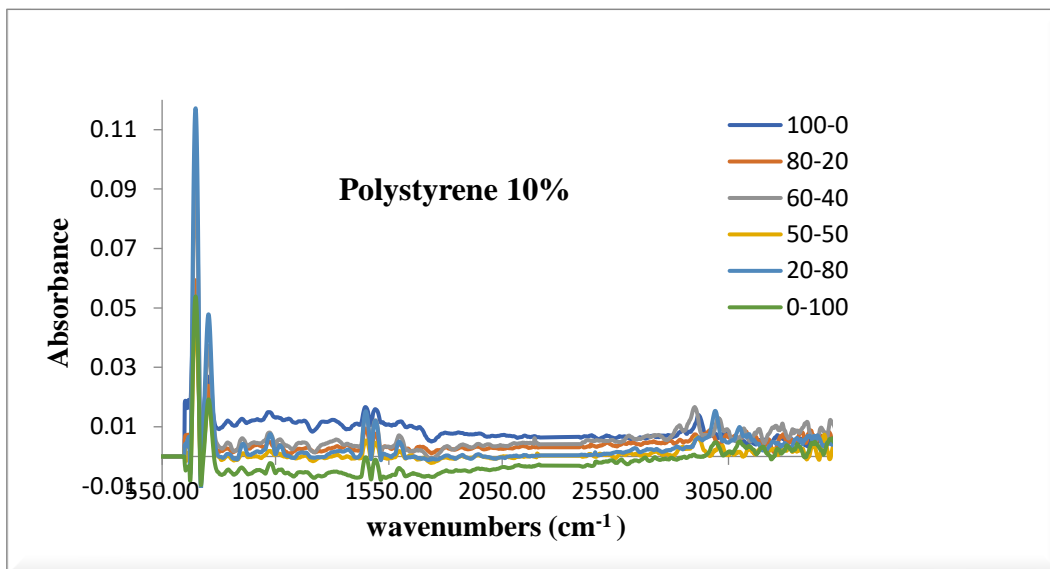
Fig. 2. FTIR-ATR Spectrum of PS 10%, 20% and 30%

SEM Imaging: Most of the fibers obtained so far were in non-woven form, the morphology of fibers with PS 10% gives maximum number of beads with wrinkled surface and ribbon like structure with small bead and smooth morphology in 20% PS. A significant change was observed in PS 30% with changing the solvent ratio. The increasing concentration of PS led to an increase in the viscosity of the polymer solution. In results, the stretching effect of the fluid jet and the instability of polymer solution jet produced electrical forces that offer the formation of uniform electrospun ultrafine fibers with beads free in micrometers diameter in Fig. 3, 4 and 5. The average diameter of all compositions was around 0.7-1.2µm (PS 10%), 1.5-4µm (PS 20%) and 2-10µm (PS 30%) in Fig.3.
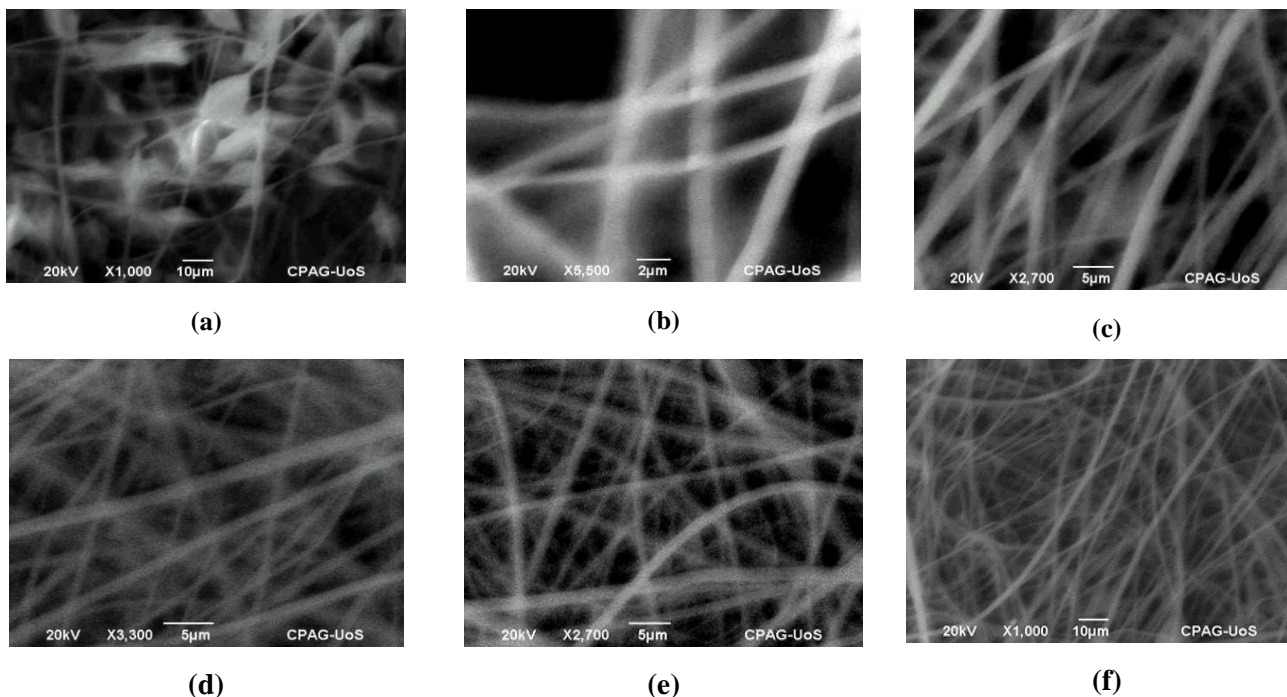

| (a) | (b) | (c) |

| (d) | (e) | (f) |

Fig. 3. (a), (b), (c), (d), (e) and (f) the PS 10% Polystyrene (PS) Electrospun Ultrafine Fibers Of Various Solvent Composition. 100:0, 80:20, 60:40, 50:50, 20:80 and 0:100
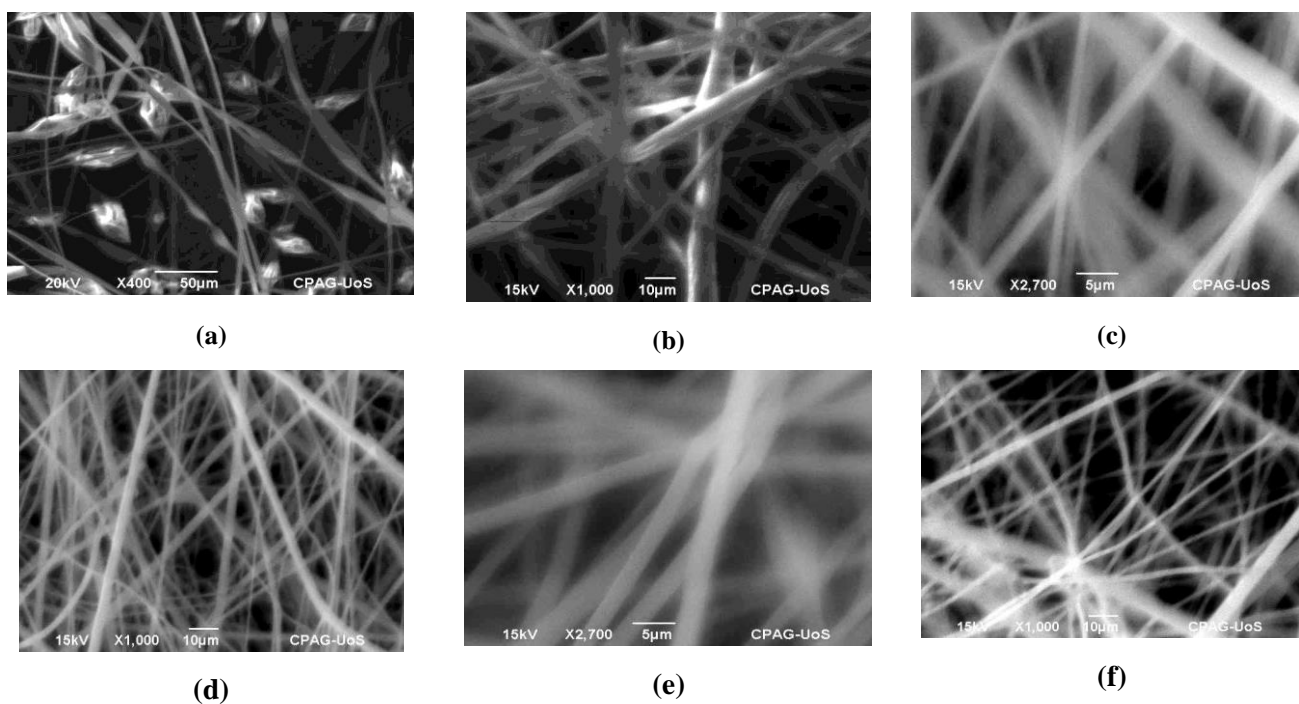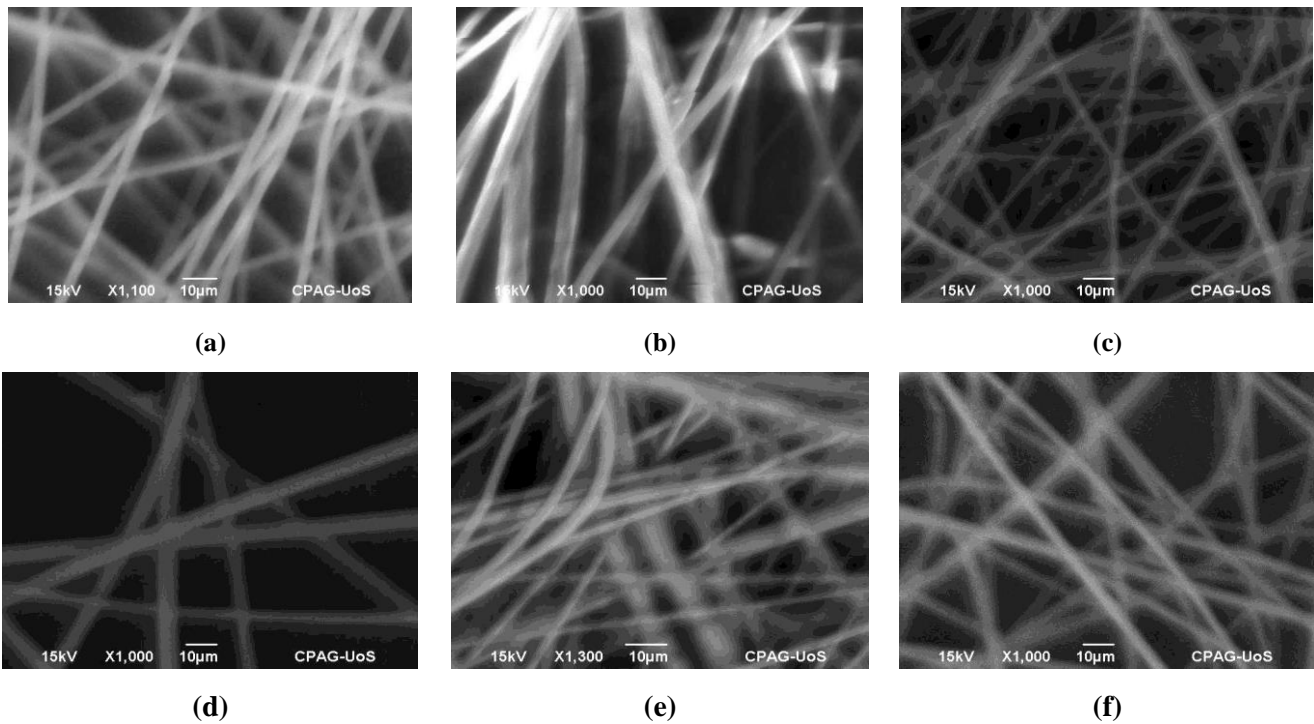

| (a) | (b) | (c) |

| (d) | (e) | (f) |

Fig. 3. (a), (b), (c), (d), (e) and (f) the PS 10% Polystyrene (PS) Electrospun Ultrafine Fibers Of Various Solvent Composition. 100:0, 80:20, 60:40, 50:50, 20:80 and 0:100

Fig. 5. (a), (b), (c), (d), (e) and (f) the PS 30% Polystyrene (PS) Electrospun Ultrafine Fibres Of Various Solvent Composition. 100:0, 80:20, 60:40, 50:50, 20:80 and 0:100

AFM Imaging: Electrospun ultrafine fibers with all compositions, PS 30% (80-20) were found to be an optimal composition for methyl paraben sorption in fig. 6. The selected fibrous mat was coated with polyglycerol gel, dried and was placed on glass slip for AFM imaging. The obtained result shown electrospun fiber at 2.5 µm which is quite rough but further zoomed up to 1 µm image shows the grooved surface with ultrafine pores when these AMF images were zoomed at 0.5 µm to 0.25 µm, pores can be clearly observed. The average diameters were measured by using Image J software.
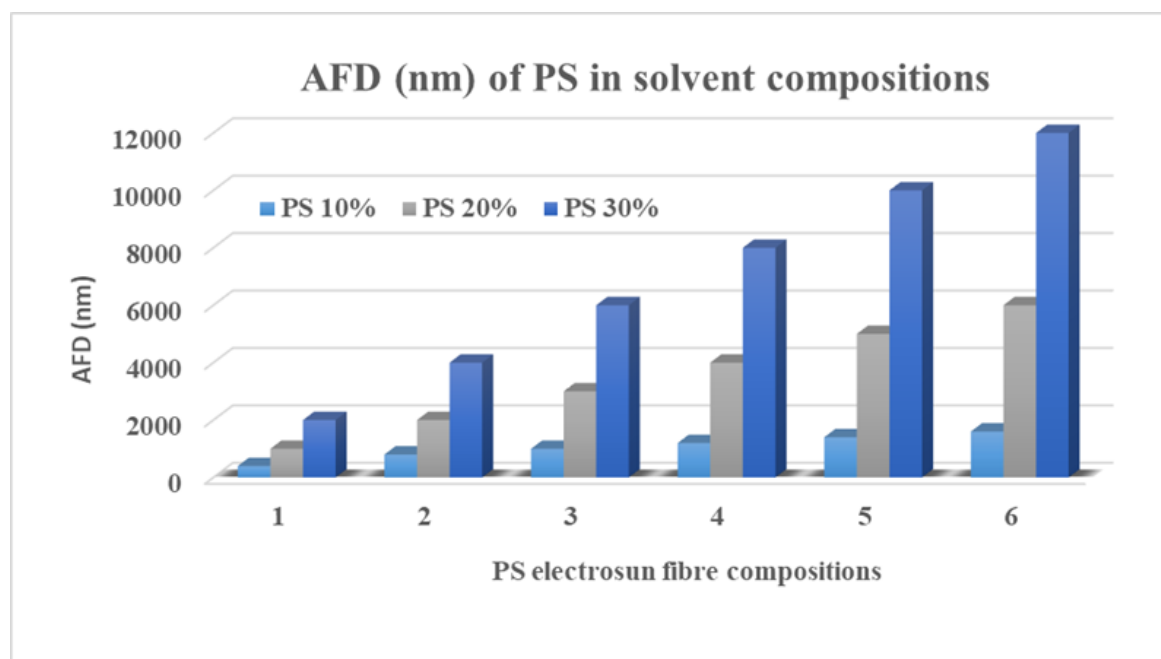

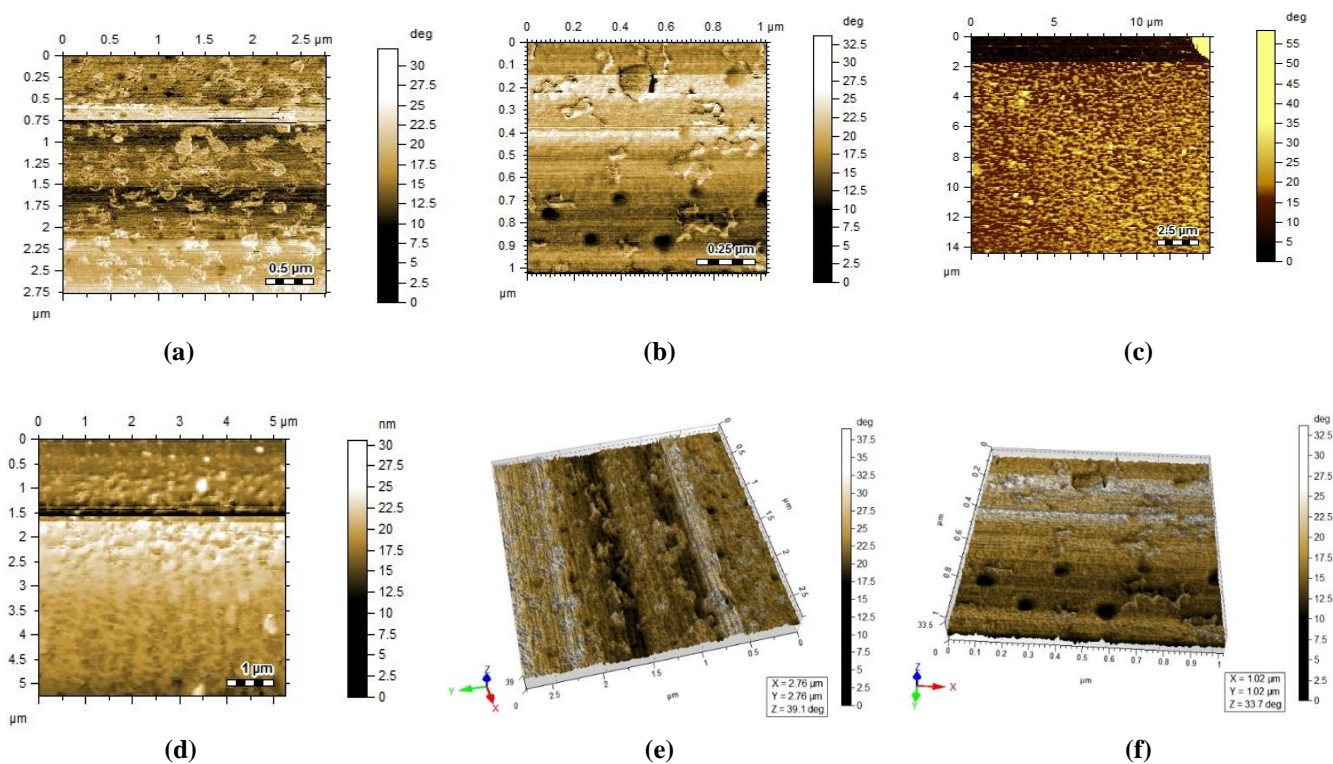**Fig. 6.** Average Diameters of Ultrafine (PS) Fiber Using Different Solvent Compositions

(a)   (b)   (c)

(d)   (e)   (f)

Fig. 7. AFM Images of PS Fibres and 3D-Images of Ultrafine Pores on Fibers Surface

5. Adsorption Studies of Methyl Paraben

Fig. 7. The adsorption efficiency of methyl paraben onto the electrospun ultrafine fiber surface was determined by adding limited amount of each fiber composition to 10 mL solution for 24 hours at room temperature. Adsorption efficiency was determined by HPLC, and the percent adsorption was calculated. At 10%, the maximum percent adsorption was obtained by a-4 (50-50) composition, where in case of 20% b-1, b-2, b-5 and b-6 compositions. The increasing the concentration PS 10% to 30% that showed better adsorption efficiency. The maximum adsorption efficiency was at c-1, c-2, and c-5 compositions where c-3, c-4 produced averaged values for adsorption.
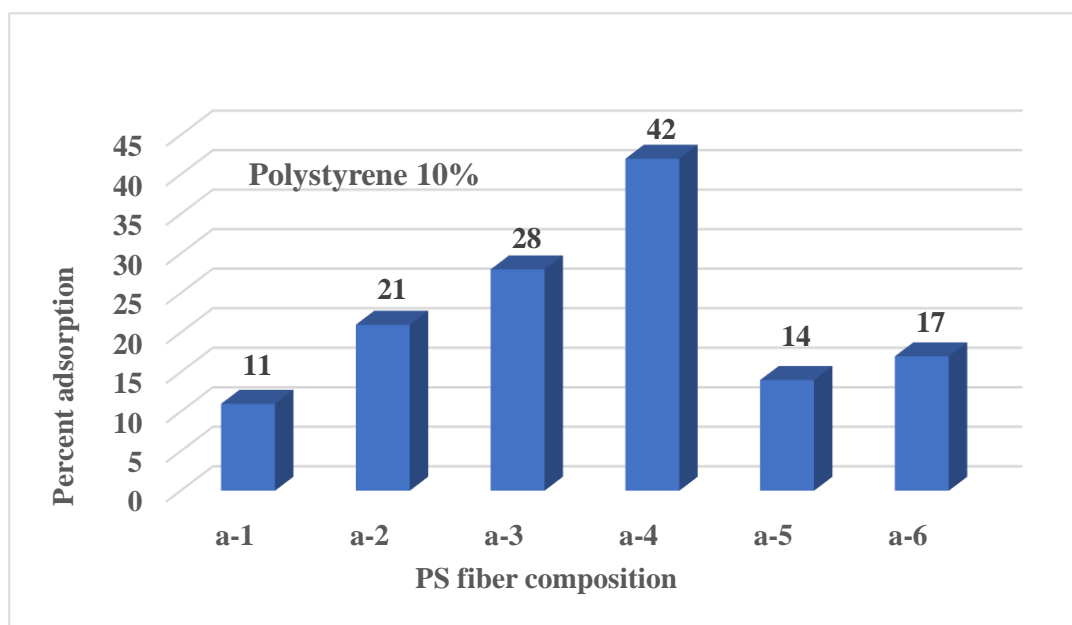

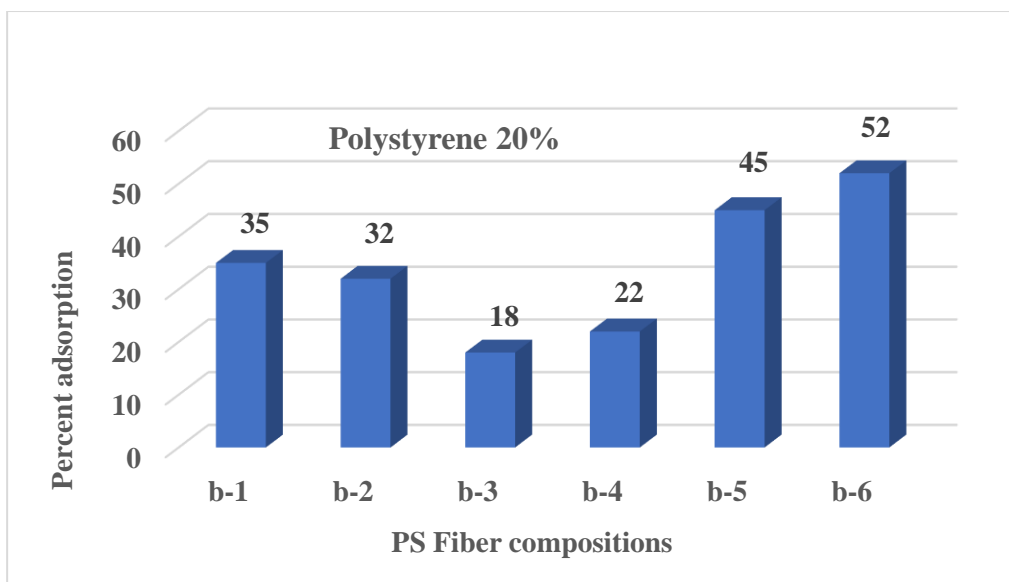**Fig. 7. (a).** Percent Sorption on Electrospun Ultrafine Fibers PS 10%

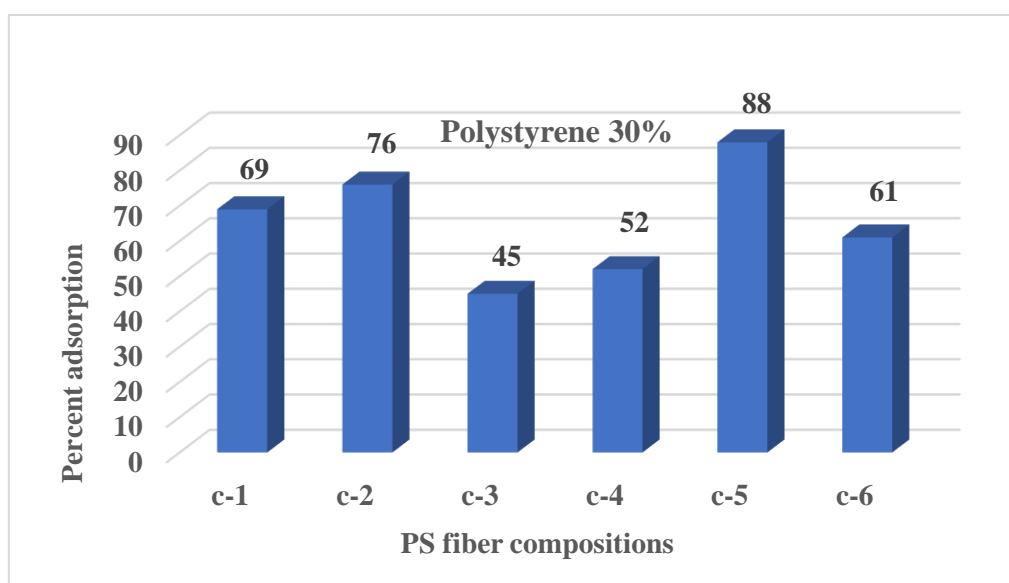**Fig. 7. (b).** Percent Sorption on Electrospun Ultrafine Fibers PS 20%

Fig. 7. (c). Percent Sorption on Electrospun Ultrafine Fibers PS 30%

6. Results and Discussions

6.1. Optimum Conditions of Adsorption

From obtained results, the adsorption was carried out by optimizing the parameters as pH, amount of fibre dose, time and then solvent elusion. 10 µg/mL solution of methyl paraben was used in optimization process. The first approach, optimization started to evaluate the pH condition from 2-8, meanwhile, using appropriate volumes of 1M solutions of NaOH and HCl. Extractions were initially performed by changing the amount of fiber 10 mg to 50 mg with sorption time of 0, 30, 60, 180, 240 minutes and 24 hours. The extraction yield was also calculated by multiple solvents.

Effect of pH: Fig. 8. The charge on the methyl parabens studied can be affected by the pH of the sample solution. Therefore, the adsorption of sample matrices may be affected by ionization of target compounds in the sample and surface charges of the sorbent material. The sample pH was investigated in the range 2.0 to 8.0 by adding appropriate volumes of HCl 1M or NaOH 1 M to the aqueous standards. According to the results, the maximum adsorption observed at pH 3.0 and a considerable decrease in sorption when the pH varied between 3.0 and 8.0. Therefore, pH 3.0 was selected as the optimum value for other parameters.
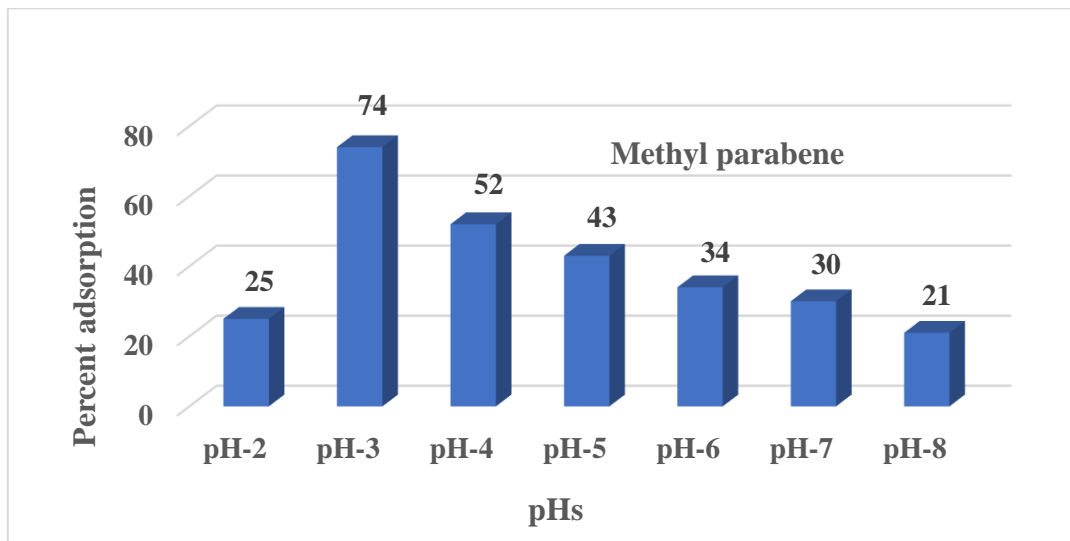
Fig. 8. Effect of pH on Adsorption of Methyl Paraben

Effect of adsorbent amount: Fig. 9. The amount of adsorbent material was evaluated in the range of 10 mg to 50 mg at pH 3.0 for adsorption studies. Therefore, all experiments were performed in an equilibrium time up-to 24 hours using batch mode. An adsorbent 10mg material was selected due to the maximum adsorption efficiency of the methyl paraben where it decreased the rest of the amounts. The porosity and the surface charge show affinity towards methyl paraben adsorption.
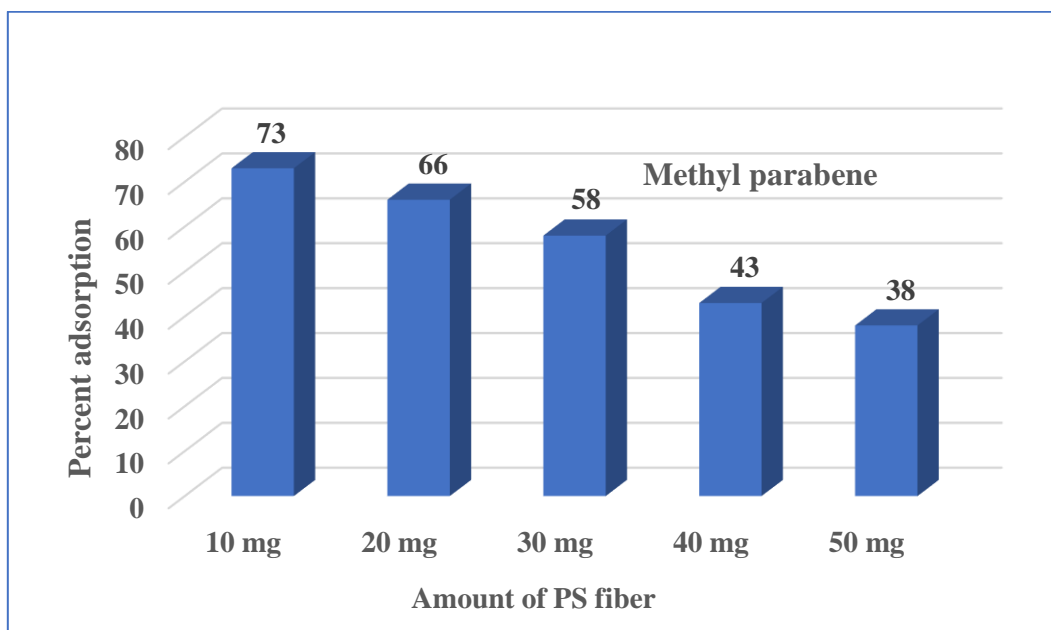

Fig. 9. Effect of Adsorbent Dose on Adsorption Of Methyl Paraben

Contact time: Fig. 10. In order to understand adsorption time at best optimal performance, the contact time parameter was highly desirable when analyzing the adsorption since it allowed analytes retained on the surface of adsorbent material. In this study, adsorption time was also optimized 0 min, 30 min, 60 min, 180 min, 240 min and 24 hours respectively. Form the result, using variable range of times, the best efficiency was obtained after 240 min. Therefore, 24 hours were selected as the optimal adsorption time for the extraction of methyl parabens.

Solvent elution type: Fig. 11. In order to optimize the adsorption capacity of methyl parabens, different solvents were used. Therefore, selected solvents were tested to elute the methyl paraben using methanol, acetone, hexane, and then chloroform. Desorption was

carried out by using 10 mL of each solvent with solutions adjusted at pH = 0.3, experiment was conducted at room temperature and time was remain constant as 24 hours. From the obtained results, it was that the methanol is sufficient to elute sorbent on the (PS) electrospun ultrafine fibers.
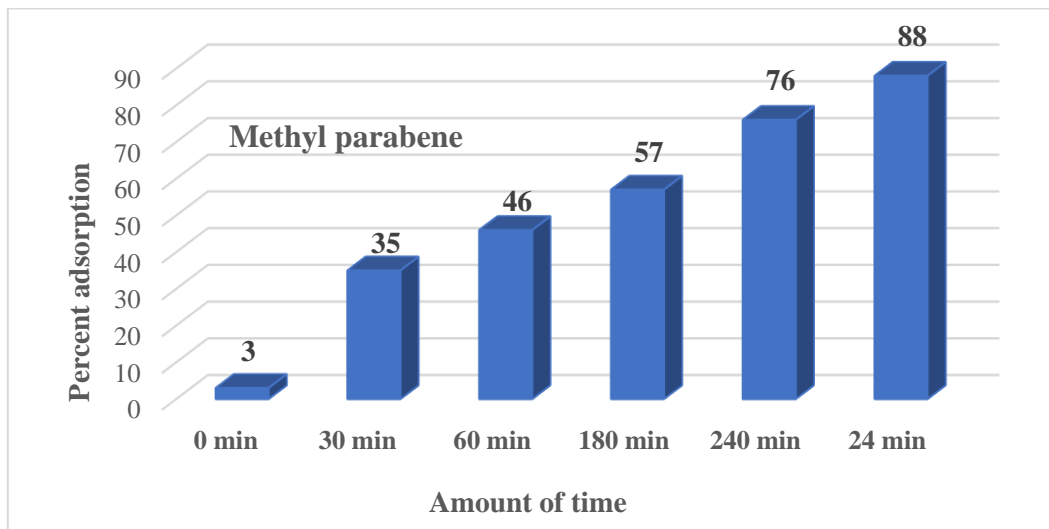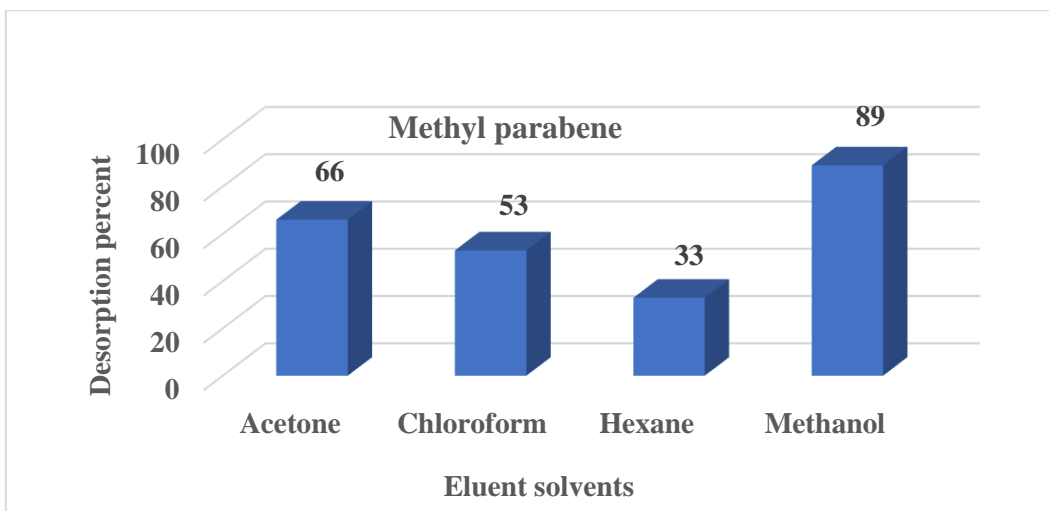

Fig. 10. Effect of Time on Adsorption of Methyl Paraben

Fig. 11. Effect of Solvent on Elution of Methanol From PS Electrospun Ultrafine Fibers

6.2. Adsorption Kinetic Study

In fig. 11. (a), (b) and (c). Kinetic studies determine the rate of methyl paraben removal from the adsorbent material, Pseudo first order, Pseudo second order and Morris-Weber applied to evaluate the data. 10 µg/mL concentration of methyl paraben was prepared and added with 0.01 g of each fibers mat for 24 hours at batch mode at room temperature, and solutions were adjusted at pH 3. The amount was calculated according to the following equations. The affinity between the experimental data and the model predicted values were expressed by the correlation

coefficients (r2). A relatively high R value was used to indicate best fit to the kinetics (close to 1).

Pseudo first order: the pseudo first-order equation is generally expressed as follows:

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303}$$

Where q_e is the amount of adsorption capacity at equilibrium time (mg/g), and q_t is the amount time in minutes as t (mg/g), then k_1 as rate coefficient of pseudo firs order adsorption (L/min). The plot of log (q_e-q_t) vs t should give a linear relationship from

which k_1 and qe can be determined from the slope and intercept of the plot, respectively.

Pseudo second order: Data was applied on pseudo second-order adsorption kinetics rate equation is expressed (g/mg min). The plot of (*t/qt*) and t should give linear relationship from which *qe* and k_2 can be determined from the slope and intercept of the plot.

$$t/qt = t/q_e + 1/kqe^2 + t/qe$$

Morris-Weber Model: It helped to understand the mass transfer between adsorbent material and compound. The Morris-Weber equation was applied.

q_t=k_i √t

Where qt is the amount of analyte (mg/L) at time t and k is the intra-particle diffusion rate coefficient (mg/Lmin1/2). According to the result, adsorption kinetics between methyl paraben and material was the first order and shown that the mass transfer nearly diffusion in controlled.
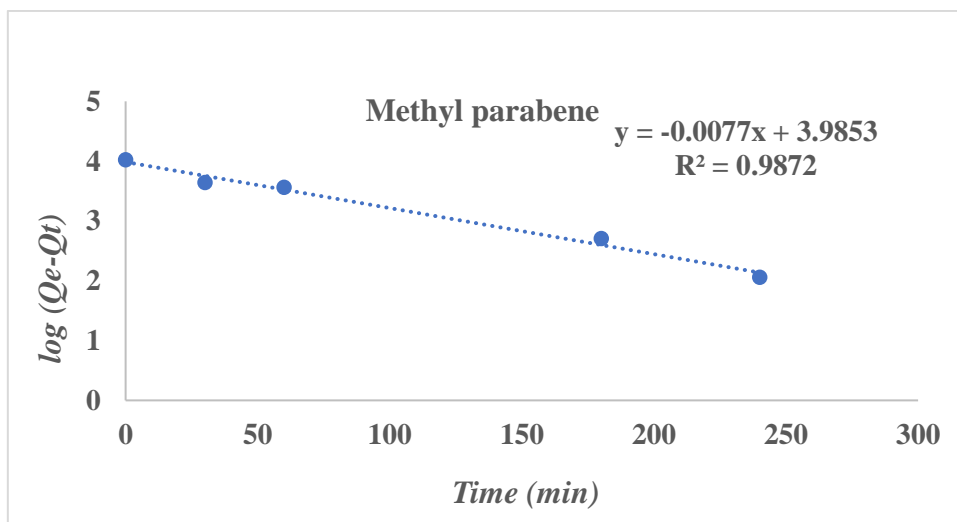

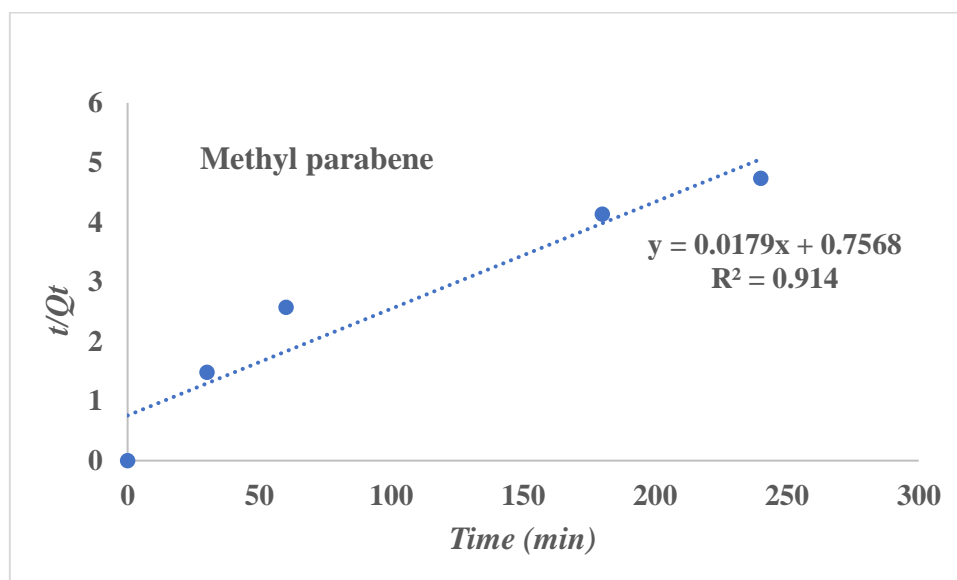**Fig. 11. (a).** Pseudo First Order Kinetics of Methyl Paraben

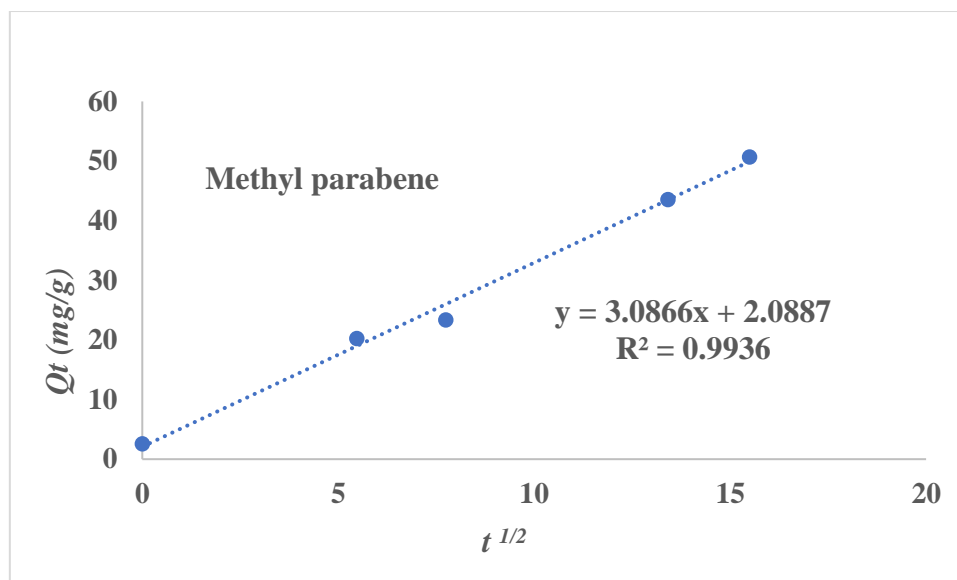**Fig. 11. (b).** Pseudo Second Order Kinetics of Methyl Paraben

Fig. 11. (c). Morris-Weber Model for Adsorption Of Methyl Paraben

7. Conclusion

Polystyrene electrospun ultrafine fibers were successfully prepared by the electrospinning method, and various solvent compositions were analyzed to assess the residual solvent effect. The study concluded that a higher polymer concentration yields the maximum residual solvent effect. Our systematic examination, employing SEM and AFM, confirmed that our electrospun polystyrene fibers have diameters of less than 10 μm. SEM images demonstrated the smooth and bead-free nature of the fibers when using a 20% DMF and 80% THF solvent composition. Additionally, AFM results revealed nano pores on the surface of the fibers. The optimized electrospun polystyrene fiber was chosen as the sorbent material for a parabens sorption study. Our findings indicated that electrospun ultrafine polystyrene fibers offer a convenient approach for parabens cleanup. The optimized conditions showed that a pH of 3.0 is the optimum range for parabens using 10 mg of fibers, and the contact time was optimized at 24 hours for the finest adsorption time, with methanol used for back extraction. Furthermore, the kinetics of parabens with the sorbent followed a first-order reaction (R2 0.914, R2 0.8984), governed by a mass transfer diffusion-controlled mechanism.

6. References

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