Methylene blue removal capabilities of activated carbon doped nanofibres

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ABSTRACT – REZUMAT

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The importance of adsorbents in wastewater treatment is related to their pollutant removal efficiency. The performance in the treatment process depends on the surface area, morphology, and chemical structure of the adsorbents. Since these properties can be controlled in both nanofibres and activated carbon, activated carbon (AC) doped nanofibres were used to remove methylene blue (MB) from the aqueous medium in this study. For this purpose, AC was synthesized from human hair by chemical activation and characterized. By this means, AC possessing 561 m^2/g Brunauer-Emmet-Teller (BET) surface area and 6.5% ash content was obtained. Thermogravimetric analysis of the AC showed that approximately. 50% of the initial weight decomposed at 750°C. The synthesized AC was doped into the silk fibroin (SF) electrospinning solution at the ratios of 1% and 5% (w/v). Scanning electron microscope and energy dispersive X-ray analyses, and BET surface area measurements were conducted for characterization. Finally, batch adsorption tests were performed under different conditions, assisted with an ultrasonic bath. According to the test results, 5% AC-doped nanofibre web with 15 mg of adsorbent amount exhibited the best performance among nanofibre webs with an adsorption amount of 262.3 mg/g. This value was achieved at pH 12, 50°C, and an ultrasonic bath-assisted process duration of 10 min. The overall results showed that the AC synthesized from human hair-doped SF nanofibre webs has the potential to remove MB from textile wastewater.

Keywords: *activated carbon, adsorption, electrospinning, nanofibre, methylene blue, wastewater*

Proprietăți de eliminare a albastrului de metilen din nanofibre dopate cu carbon activ

Importanța adsorbanților în tratarea apelor reziduale este legată de eficiența lor de eliminare a poluanților. Performanța în procesul de tratare depinde de suprafața, morfologia și structura chimică a adsorbanților. Deoarece aceste proprietăți pot fi controlate atât în nanofibre, cât și în cărbunele activ, nanofibrele dopate cu cărbune activ (CA) au fost utilizate în acest studiu pentru a îndepărta albastrul de metilen (MB) din mediul apos. În acest scop, AC a fost sintetizat din păr uman prin activare chimică și caracterizat. Prin acest mijloc, s-a obținut AC care posedă o suprafață Brunauer-Emmet-Teller (BET) de 561 m^2/g și un conținut de cenușă de 6,5%. Analiza termogravimetrică a CA a arătat că aprox. 50% din greutatea inițială s-a descompus la 750°C. AC sintetizat a fost dopat în soluția de electrofilare a fibroinei de mătase (SF) în proporții de 1% și 5% (w/v). Pentru caracterizare au fost efectuate analize la microscopul electronic cu baleiaj și cu raze X cu dispersie de energie și măsurători ale suprafeței BET. În cele din urmă, au fost efectuate teste de adsorbție pe loturi în diferite condiții de asistență cu baie cu ultrasunete. Conform rezultatelor testelor, vălul de nanofibre dopat cu 5% AC cu o cantitate de adsorbant de 15 mg a prezentat cea mai bună performanță dintre vălurile de nanofibre cu o cantitate de adsorbție de 262,3 mg/g. Această valoare a fost obținută la pH 12, 50°C și o durată a procesului asistat de baie cu ultrasunete de 10 min. Rezultatele generale au arătat că AC sintetizat din păr uman dopat cu nanofibre SF are potențialul de a elimina MB din apele reziduale textile.

Cuvinte-cheie*: cărbune activ, adsorbție, electrofilare, nanofibre, albastru de metilen, ape reziduale*

INTRODUCTION

The rise in demand for industrial products increases the use of various chemicals consumed during the production of these products, and sometimes causes these chemicals to be released into natural environments uncontrollably and in a way that harms the environment. The negative effects of the textile industry in terms of harmful chemicals released into the environment are undeniable. As the effluent discharged after domestic use or industrial processes is defined as wastewater [1], textile wastewater contains phosphates, organic dyes or heavy metals [2]. These substances harm living creatures as well as the environment. For this reason, the treatment of wastewater appears to be a very important issue. Currently, wastewater treatment is controlled by various international or national laws and regulations, and it is not possible to release wastewater into the environment without reducing the amount of harmful chemicals below a certain level. Physical, biological, and/or chemical processes are utilized to reduce the chemicals contained in the wastewater to specified values [3]. The removal of dissolved impurities from textile wastewater is often achieved by chemical or physical adsorption. The principle of adsorption is based on the accumulation of substances to be

removed in a fluid environment on solid adsorbents through chemical or physical interactions [4]. While in physical adsorption, the chemicals to be removed (adsorbates) and the surface atoms of solid adsorbents interact via van der Waals forces or electrostatic attraction forces, in chemical adsorption, surface atoms of adsorbates and adsorbents form chemical bonds [5, 6]. Activated carbon, silica, clays, metal oxides, and polymer-based materials can be used as adsorbents in both chemical and physical adsorption [7, 8].

It is known that there is a significant use of dyestuffs in the textile industry. These coloured substances cause some hazards and environmental problems. Approximately 10–15% of the dyestuffs are discharged as waste. One of the hazardous dyestuffs frequently encountered in textile wastewater is methylene blue (MB), also called methylthioninium chloride. The MB, a compound with a heterocyclic aromatic structure, creates a dark blue colour when dissolved in water. To protect both water bodies and aquatic ecosystems, this toxic component should be removed from wastewater [9, 10]. Since it is important to remove MB, which damages the habitat by making it difficult to transfer sunlight in waterbodies and causes the formation of single oxygen, from textile wastewater [11–14], the MB was chosen as the adsorbate in this study.

Activated carbon (AC), which can be produced from all raw materials containing at least 40–50% carbon by a (physical or chemical) activation treatment following the carbonization process, is an indispensable product for the adsorption process. The high surface area makes activated carbon significant for adsorption. In carbonization, an attempt is made to obtain the main skeleton from the structure, in which moisture and volatile substances are removed. Oxidation occurs in the activation process. Besides, the pore structure formed in the previous stage is developed through controlling parameters [15]. In this way, a product with a high specific surface area and increased adsorbate binding points is obtained. Commercial ACs, with specific surface areas of up to 1000 m^2/g, are highly preferred adsorbents in the removal of undesirable substances from wastewater due to their performance. However, its high cost poses a problem for its widespread use [16]. The use of different wastes as raw materials in activated carbon synthesis has recently started to attract attention. In studies carried out to date, textile waste [17, 18], tea waste [19], coffee waste [20], and municipal waste [21] are the wastes used in the synthesis of AC. Moreover, there are studies in which human hair is used as a raw material. In a study, which chemical activation chosen for synthesis, activated carbon with a surface area of ~36 m^2/g is obtained [22]. In another study, AC from human hair waste is used in the adsorption of reactive violet and acid green 4G from aqueous solutions [23]. This study focused on the selection of human hair, which has a carbon content of 45% [24], as a waste raw material to reduce the cost of activated carbon.

The properties developed by the production of materials at the nanoscale have led to the design and production of remarkable nanoscale products for environmental applications as well as in many application areas [25]. Nanofibre webs that are composed of nanosized fibres are utilized in various areas such as filtration, separation, sensor applications, and adsorption. Their small unit structure sizes make them effective in these applications. The reduction of the unit structure to the nanoscale results in an increase in the number of regions that will be active in adsorption, thus increasing the possibility of interaction between the nanofibrous web acting as an adsorbent and the molecules of the adsorbate. Nanofibres suitable for adsorption purposes can be obtained using different raw materials with the electrospinning method, which allows nanofibre production with simple equipment. Carbon and various polymers such as poly(acrylic acid), poly(styrene) or cellulose acetate have been tested in numerous studies to remove MB in aqueous media. The adsorption amounts varying between 40–400 mg/g have been obtained in various studies [26–29]. The maximum adsorption capacity of ~72 mg/g has been reached in a study examining MB adsorption with nanofibres formed with AC obtained from poly(acrylonitrile) [30]. There is no effective study in the literature involving the adsorption of MB by activated carbon-doped nanofibre webs. In this study, it was aimed to investigate the removal efficiency of MB from the aqueous medium by taking advantage of both the high surface areas and high adsorption capacities of nanofibres and activated carbon in a cost-effective way. In this regard, the results obtained are expected to make significant contributions to the literature.

MATERIALS AND METHODS

The hair used to produce activated carbon (AC) in the study was purchased commercially (in the form of a raw human hair wig). Human hair contains a high amount of carbon, and it is a no-cost waste material with easy availability. Silk fibroin (SF) was chosen as the polymer into which activated carbon is doped. Silk was supplied in filament form from a local company (Bursa, Türkiye) and silk fibroin was obtained according to the procedure specified in the literature [31]. Briefly, silk was degummed through boiling in 0.05% sodium carbonate aqueous solution for 30 min three times. After rinsing and drying, aqueous SF solutions were prepared by dissolving SF in Ajisawa solution ($CaCl_2$/distilled water/ethanol with molar ratios of 1:8:2). The dialysis process was performed in SnakeSkin® dialysis tubings (Thermo Scientific, IL, USA) at +4°C for three days to obtain a SF aqueous solution free from neutral salts. Then the solutions were cast, and the SF films were obtained by drying at room temperature. Medical syringes and needle tips used in electrospinning were obtained from a local company. Ethanol (with a 0.79 g/cm^3 density

and ACS grade), formic acid (with 98% purity), and methylene blue (possessing a solubility of 50 g/l, with a 373.9 g/mol molecular weight, and 400–600 kg/m^3 bulk density) were purchased from Merck (Germany). Sodium carbonate (in powder form, with a purity of ≥99.5% and ACS reagent), calcium chloride (in powder form), sodium hydroxide (in pellet form, ACS reagent, and with ≥97.0% purity), hydrochloric acid (ACS reagent and 37%), sulfuric acid (with 95–97% purity) were provided from Sigma Aldrich (Germany). Distilled water was used in all experiments.

AC Synthesis

The hair was subjected to a cleaning process to make it ready for synthesis. For this purpose, hairs were soaked in 70% (v/v) ethanol at room temperature for 20 minutes, and the process was repeated three times. After the final process of rinsing with distilled water, it was filtered using filter paper and then dried in an oven at 60°C. The dried hair was cut into a length of approximately. 1.0–1.5 cm with the help of scissors to facilitate the subsequent procedures.

The chemical activation method was chosen to synthesize the AC. The chemical agent used for activation was sulfuric acid. The hair was saturated with sulfuric acid by keeping it in a 20% (v/v) sulfuric acid solution for 1 h. The impregnation ratio, the ratio between the AC raw material and the chemical agent, was 1:4. Since the saturated hair should be dry before the carbonization process, the hair was dried for 24 hours in an oven set at 60°C.

A furnace (Protherm MOS 160/8, Ankara, Türkiye) was used for the carbonization process of the chemically activated and fully dried hair. To ensure that the atmosphere inside the furnace chamber is inert, the high-purity argon gas tube is tightly connected to the furnace chamber inlet. The flow rate was adjusted to approximately. 0.5 L/min with the regulator on the tube. The temperatures that can be selected according to the raw material generally vary between 500°C and 900°C [32]. In this study, 650°C was chosen as the maximum carbonization temperature. It is known that higher carbonization temperature reduces efficiency [33]. The temperature was increased from room temperature (20°C) to 650°C in 210 min with a heating rate of 3°C/min. The waiting time was set to 2 hours. After this period, the furnace was allowed to cool by itself, and the argon gas flow continued during the cooling process.

The samples taken out of the chamber were first washed with 0.5 M HCl to remove residual chemicals from them. Then they were washed with hot and cold distilled water, respectively. The drying was performed in an oven at 60°C for 24 hours. The synthesized AC was used in dry powder form for characterization and production of AC-doped nanofibre webs.

Fabrication of AC doped SF nanofibre webs

Silk fibroin is attractive due to its high biocompatibility, good thermal stability, and high mechanical strength, as well as the presence of various amino acid groups that provide great advantages for the adsorption of adsorbates [34]. Moreover, it is one of the easiest polymers to electrospin. Electrospinning is a method that allows the manufacture of nanoscaled fibres from a wide variety of materials. Additives can be easily incorporated into nanofibre webs by adding to electrospinning polymer solutions. In this study, 15% (w/v) SF/formic acid solutions were prepared. The AC was doped to these solutions in ratios of 1%, 5% and 10% (w/v). SF nanofibres without AC were also fabricated to use as control webs (blank) in adsorption tests. Although electrospinning was tried with all these solutions, no jet formation was observed with 10% (w/v) AC-doped solution. Samples doped with 1% and 5% (w/v) AC were fabricated by electrospinning for 5 hours. The samples are coded according to the amount of AC they contain. Nanofibre webs fabricated using SF solution without AC, SF solution doped with 1% (w/v) AC, and SF solution doped with 5% (w/v) AC are coded as 0ACSF, 1ACSF, and 5ACSF, respectively.

The 20–24 kV applied voltage, 0.24 mL/h flow rate, and 20 cm distance were the electrospinning process parameters. A 20 mL eccentric plastic syringe and a flattened 21G metal needle tip were used for solution feeding via a syringe pump (New Era Ne-1000, USA). A high voltage power supply (Gamma High Voltage Research Inc., USA) was used, and an aluminium foil-coated fixed plate was the grounded collector. Nanofibre spinning was performed at room temperature (20±2°C) and 30±5% relative humidity.

Characterization tests

Scanning electron microscopy (SEM, LEO 1430 VP) analyses were performed to determine the morphology of synthesized AC and nanofibre webs. The AC sample was analysed by directly sticking it on double-sided carbon tapes. On the other hand, nanofibre web samples were coated with gold for 30 s before analysis. The accelerating voltage applied in the analyses was 20 kV. The magnifications are 500 and 5000X for AC, and 2500 and 10000X for nanofibre web samples. In addition, EDX analyses were performed on the AC and the nanofibre web samples to determine elemental components. The mean diameters of the nanofibre web samples were calculated with Image-J Measurement Software by measuring 50 nanofibres' diameters from each SEM image.

Thermogravimetric analysis (TGA) of the synthesized AC was conducted by TG/DSC (Differential Scanning Calorimetry) analyser (Netzsch STA 449F3, Germany). The thermogram of the AC was recorded between 25°C and 750°C with a heating rate of 10°C/min, under a nitrogen atmosphere and using an AL_2O_3 crucible with a 10 mg AC sample.

In addition, the amount of ash in the synthesized AC was determined according to ASTM-D 2866-94.

1 g of activated carbon was placed in porcelain crucibles (50×25 mm, Isolab) and brought to constant weight at 650°C. Again, the samples placed in the muffle furnace (ELF 11/6B, Carbolite, England) at

650°C were burned for about 6 hours to reach constant weight. Then, they were cooled to room temperature in the desiccator. An analytical balance with an accuracy of 0.1 mg was used during the measurements (Radwag, Poland). The amount of ash was calculated by equation 1:

$$Ash\ content(\%) = \frac{(B - C)}{(A - C)} \cdot 100 \qquad (1)$$

where, *A* is the "crucible weight + AC amount at the beginning of the process", *B* is the "crucible weight + AC amount at the end of the process", and *C* is the "crucible weight".

Since the surface area of AC and nanofibre webs is of great importance in the adsorption process, BET surface area measurement was performed with a Micrometrics Gemini 2360 model (USA) computer-equipped device. The specific surface area measurement was based on the nitrogen (N_2) gas adsorption technique in a liquid nitrogen environment at –198°C. The device sensitivity was 0.01 m^2/g.

Batch adsorption tests were carried out to determine the potential of AC-doped SF nanofibre webs to be used as adsorbents. For this purpose, the highest absorbance wavelength of the MB was detected by a UV-visible spectrophotometer (Biochrom Libra S70, UK). Scanning was performed by considering distilled water as a blank. The highest absorbance was recorded at 670 nm by the results stated in the literature [11]. All subsequent absorbance measurements were performed at 670 nm. The MB solution with a concentration of 100 mg/l was prepared, and 8 sets of dilutions were made to obtain a calibration curve. The MB removal percent from aqueous solutions was determined using the equation derived from the calibration curve of MB ($y = 0.1348x - 0.0013$ with a R^2 of 0.9971).

Since the adsorption process is affected by parameters such as stirring type, stirring speed, adsorbent amount or process duration, several parameters were chosen for the determination of the maximum MB removal percentage. In this study, ultrasonic bath-assisted adsorption tests were performed. All tests were carried out in an ultrasonic bath (Elmasonic Easy 100 H, Elma, Germany). The ultrasonic frequency of the device was 37 kHz, and the effective ultrasonic power was 150 W. Three process durations of 2, 6, and 10 minutes, two temperatures of 30 and 50°C, and three amounts of adsorbent as 5, 10, and 15 mg, were tested. The pH values were selected as pH 5 and pH 12 to test acidic and basic aqueous media. All tests were carried out in 50 ml of MB aqueous solution with an initial concentration of 100 mg/l, and pH adjustment was made with either HCl or NaOH.

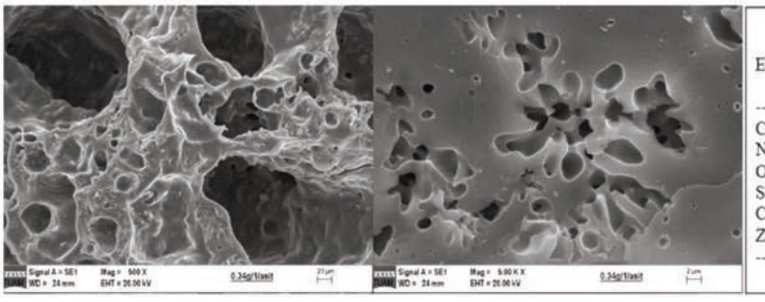
The mean of the absorbance values of the activated carbon-free (0ACSF) nanofibre web samples in pure water at 670 nm was subtracted from all absorbances recorded with the MB aqueous solution. The obtained absorbance values were converted to concentration based on the calibration curve, and the adsorption amount, *Q* (in mg/g), was calculated using equation 2:

$$Q = \frac{V(C_i - C_f)}{W} \qquad (2)$$

Where *V* is the volume of the MB aqueous solution (medium) in L, C_i and C_f are the initial and final MB concentrations in mg/l, respectively, and *W* is the weight of the adsorbent nanofibre web in g. Mean *Q* values of triplicate measurements are given.

RESULTS AND DISCUSSIONS

Synthesizing AC from low-cost or no-cost materials has attracted attention in recent years. Since the adsorption capability of AC is high due to its characteristic properties, researchers focus on cost reduction research by selecting suitable raw materials. Human hair is one of these suitable materials with high carbon content. The SEM images of the AC synthesized by chemical activation and carbonization process are seen in figure 1, *a* and *b*. Pores with radii smaller than 0.4 nm, pores between 0.4–1 nm, pores between 1–25 nm, and pores with 25 nm and above are called sub-micropores, micropores, mesopores, and macropores, respectively [35]. When the SEM images are examined, a highly porous structure with cavities is observed, and the pores are on a macro scale. The cavities seen on the SEM images of the AC may serve as functional sites for the adsorption of the agent that is desired to be adsorbed. Moreover, according to the International Union of Pure and



EDX

Element	Series	Net [wt.-%]	unn. C [wt.-%]	norm. C [wt.-%]	Atom. C [at.-%]
Carbon	K series	28994	27.48	27.45	40.09
Nitrogen	K series	3229	18.44	18.42	23.07
Oxygen	K series	5551	22.95	22.92	25.13
Sulfur	K series	35584	6.17	6.16	3.37
Chlorine	K series	36231	7.14	7.13	3.53
Zinc	K series	14800	17.93	17.91	4.81

Total: 100.0

a *b* *c*

Fig. 1. Details of: *a* – 0.5KX SEM image; *b* – 5KX SEM image; *c* – EDX analysis result of the synthesized AC

Applied Chemistry's (IUPAC) porous solid materials classification, the AC synthesized was classified as a macroporous solid having pore sizes higher than 50 nm [36]. The SEM-EDX analysis result of the AC is given in figure 1, *c*. When the elemental content analysis of the synthesized AC was examined, 40% carbon content and 25% oxygen content were seen. The results are consistent with the studies in the literature. The carbon and oxygen content of the AC derived from human hair was found to be 49.8% and 30.3%, respectively, in another study [37].

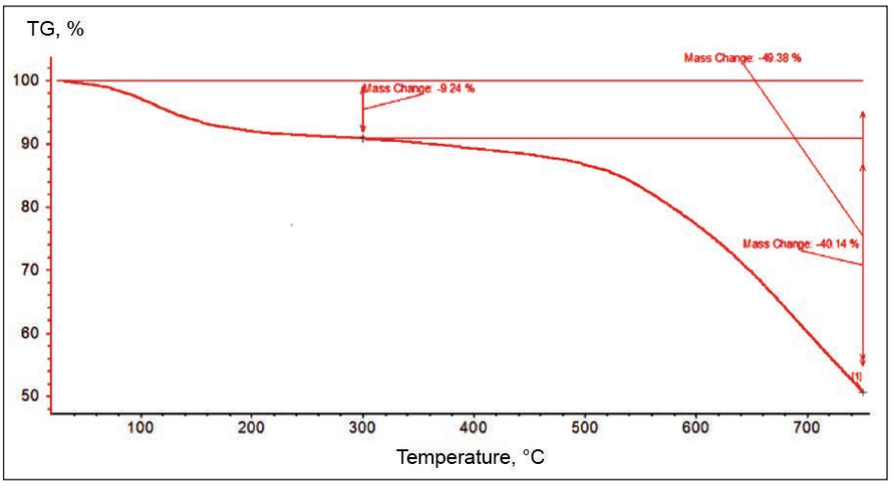


Fig. 2. Thermogram of the AC

According to the BET surface area analysis, the specific surface area of the AC synthesized was 561 m^2/g. that can be stated as a reasonable value when compared with commercial ACs. Since the higher surface area means an enhancement in the adsorption capability [38], the surface area value of the AC synthesized is important in terms of effectiveness.

The ash content of the synthesized AC was experimentally calculated, and 6.5±0.4% ash content was determined. It is necessary for ACs to have low ash content to exhibit high efficiency. This is the main disadvantage of ACs obtained from human hair.

Thermal behaviour of the synthesized AC was investigated with TGA from 25°C to 750°C. Decomposition of the AC was divided into two stages (figure 2). The first stage, which is between 25–300°C, corresponds to the slow pyrolysis and the second stage, which is between 300–750°C, corresponds to the fast pyrolysis step. In the first (slow) stage, 9.24% of the initial mass was lost due to the volatilization of volatile compounds and vaporization of moisture. The higher mass loss (approx. 40%) occurred in the fast decomposition stage up to 750°C. The TGA results proved the high thermal stability of the synthesized AC.

Electrospinning is one of the most preferred methods to get nanofibre webs using versatile polymers and materials for numerous applications. The simple equipment, composed of a high voltage power supply, a syringe pump, and a grounded collector, allows for the production of nanofibres by adjusting process and solution parameters. Silk fibroin (SF) is a challenging polymer for electrospinning because of its easy electrospinnability. Besides, it preserves its thermal stability up to 250°C [39]. In this study, beadles and uniform nanofibre morphologies are obtained from SF and AC-doped SF. The SEM images of the nanofibre webs electrospun without AC, with 1% and 5% AC are given in figure 3. The mean diameters of SF nanofibres without AC, SF nanofibres containing 1% and 5% AC were measured as 263±68 nm, 276±69 nm, and 283±75 nm, respectively. As all the process parameters were kept constant during electrospinning, the mean nanofibre diameter increased with increasing AC content.

According to the SEM-EDX analysis results carbon content of the 0ACSF, 1ACSF, and 5ACSF was determined as 36.91%, 40.80%, and 41.12%, respectively. Increases in both fibre diameters and carbon content were observed with an increase in the AC amount involved. This is the sign of successful incorporation of the AC into the SF nanofibres.

The surface area enhancement of a nanofibre web is related to the nano-scaled unit structure (fibres possessing diameters in nm). The BET surface areas of 0ACSF, 1ACSF, and 5ACSF nanofibre webs were found to be 35.43 m^2/g, 42.38 m^2/g, and 43.58 m^2/g, respectively. These values are slightly different from each other. These little changes in the values are the results of morphological similarities. There was only a 20 nm difference observed (263–283 nm) between the fibre diameters of nanofibre webs. Additionally, there are studies in the literature stating similar surface area values with similar nanofibre diameters [40]. Furthermore, there are publications in the literature regarding nanofibre web applications with similar BET surface area values used for MB adsorption [41].

Since the isoelectronic point of silk fibroin is around 4.2–4.5, two pH values were chosen in this study, one slightly higher than this point (pH 5) and the other an alkaline value (pH 12). Results of batch adsorption tests performed at pH 5 and pH 12 are given in figures 4 and 5, respectively. The adsorption amounts observed at pH 12 were higher than the amounts observed at pH 5. The maximum adsorption amounts recorded were 190.5 mg/g and 262.3 mg/g at pH 5 and pH 12, respectively. SF has a net negative charge in alkaline medium (at about pH 12) [42]. Therefore, batch adsorption tests at this pH value resulted in higher adsorption amounts than at pH 5 due to the increased interactions between the adsorbent – AC doped SF nanofibres – and the adsorbate-MB, a cationic dye. Furthermore, the sample 5ACSF gave the highest adsorption amount results in all

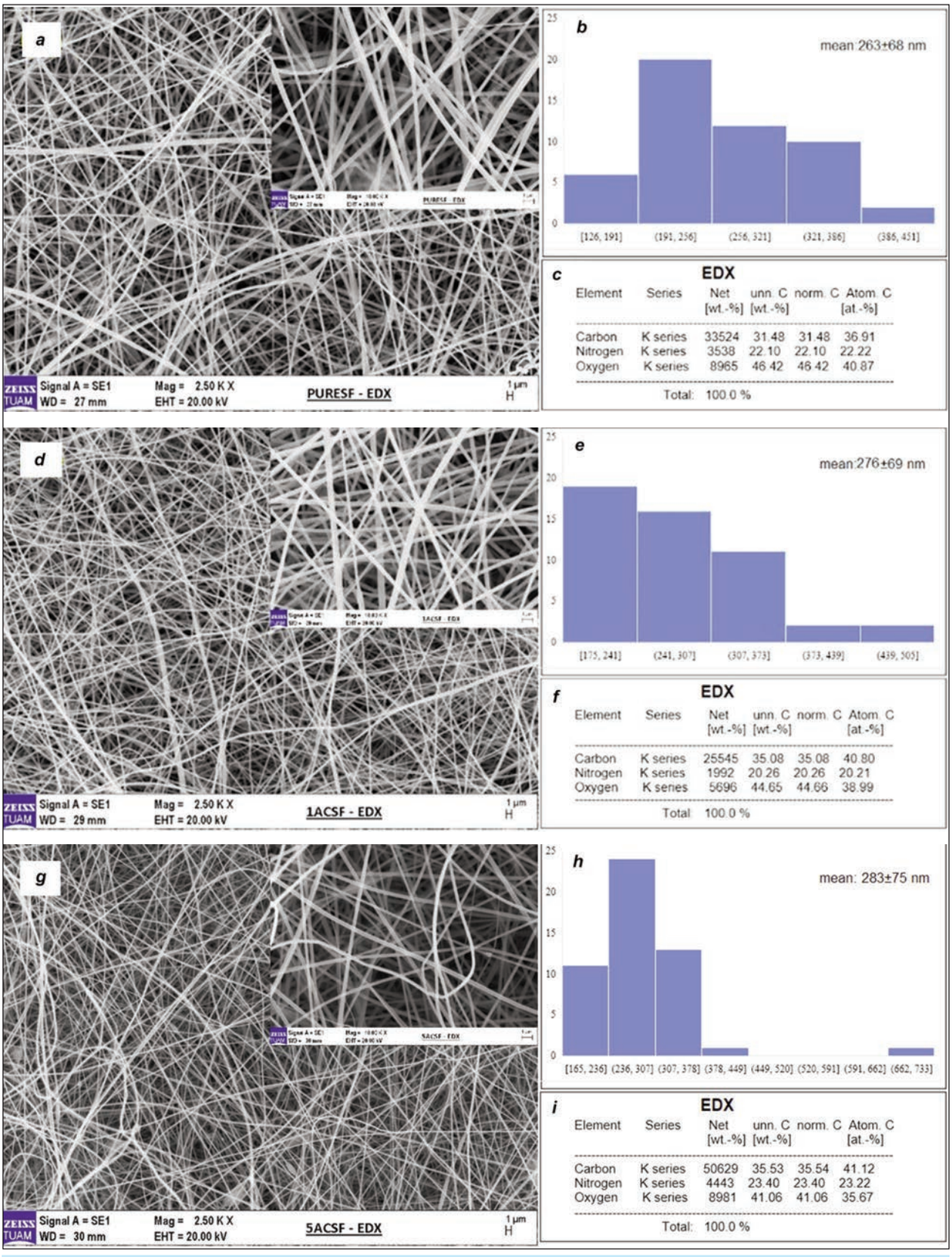


Fig. 3. SEM images (with 2.5KX and 10KX magnification), histograms of nanofibre diameters, and EDX analyses results of: *a, b, c* – 0ACSF; *d, e, f* – 1ACSF; *g, h, i* – 5ACSF

parameters tested. This can be attributed to the adsorption enhancement ability of the AC encountered at high pH values [43].

The adsorbent amount affected the adsorption capacity positively. Increasing the adsorbent amount allows more MB in the aqueous medium to be accumulated by adsorption. This is why among the 5, 10, and 15 mg adsorbent amounts, the highest values were obtained with 15 mg.

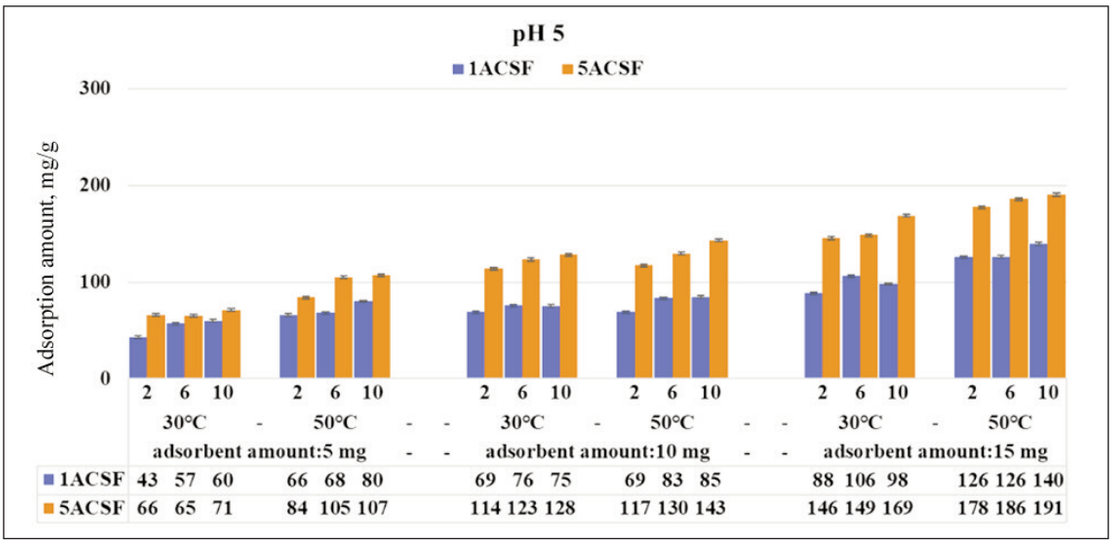


Fig. 4. Adsorption amounts of 1ACSF and 5 ACSF nanofibre webs according to the process parameters at pH 5

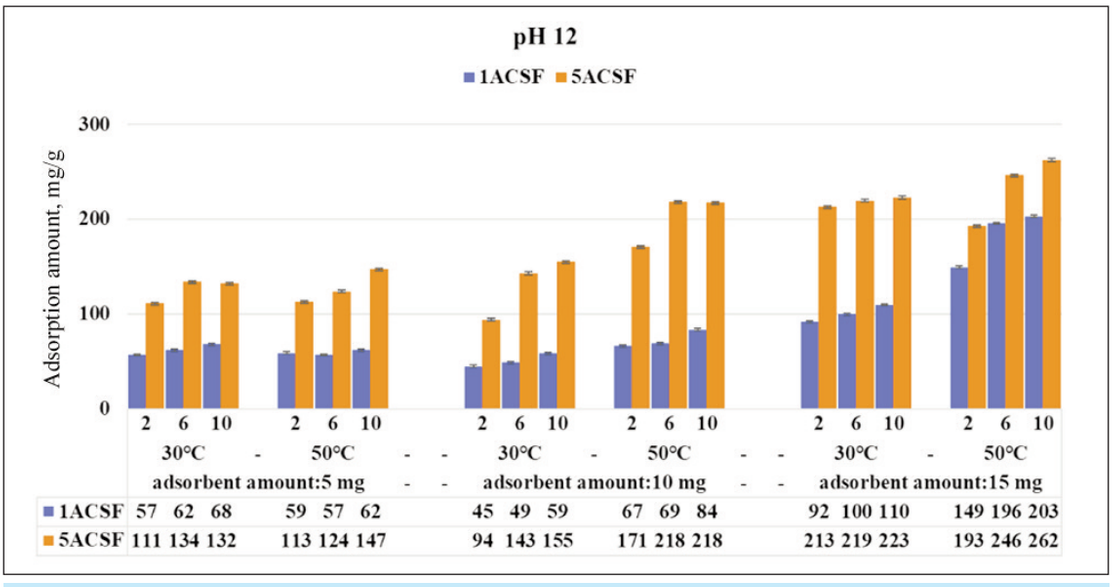


Fig. 5. Adsorption amounts of 1ACSF and 5 ACSF nanofibre webs according to the process parameters at pH 12

According to the batch adsorption test results, as the applied temperature increases, the adsorption amount increases too. Since temperature increases the mobility of molecules in physical or chemical processes, it is an expected phenomenon that adsorption efficiency increases with increasing temperature. In the literature, various temperature ranges have been studied in batch adsorption tests. In this study, two temperatures were selected as low (30°C) and high (50°C). The findings in the literature indicate that temperature appears to be a parameter that improves adsorption [27]. Moreover, the MB adsorption on the SF nanofibres is an endothermic adsorption process [44].

Increasing the processing time means extending the application time of the ultrasonic frequency applied during adsorption. Time extension improves adsorption because it also causes molecular mobility.

Moreover, significant differences between the adsorption amounts of 1ACSF and 5ACSF were observed. As the amount of AC doped increased, the adsorption amount increased. It was revealed that the AC with a higher surface area added to the nanofibres made a considerable contribution to the adsorption phenomenon.

CONCLUSIONS

The results obtained from the study can be summarized as follows: Human hair is a suitable raw material for the synthesis of AC. Chemical activation was chosen for the synthesis of AC from human hair, and macro-porous structured AC with a surface area value of 561 m^2/g was obtained. TGA of the AC denoted that ~9.24% and ~50% mass losses were observed at 300ºC and 750ºC, respectively. The 1% and 5% AC-doped SF nanofibre webs were produced.

The fibres had a regular morphology and no beads. The mean diameters of SF nanofibres without AC, SF nanofibres containing 1% and 5% AC were measured as 263±68 nm, 276±69 nm, and 283±75 nm, respectively. An increase in diameter was observed with the addition of activated carbon. The BET surface areas of nanofibre webs were found to be 35.43 m^2/g, 42.38 m^2/g, and 43.58 m^2/g for 0ACSF, 1ACSF, and 5ACSF, respectively. BET surface area values were very close to each other, and these values are considerable for adsorption. According to the results of the batch adsorption tests, a maximum of 262 mg/g adsorption amount was recorded with the 5% AC doped nanofibre web at 50°C, pH 12 and with the highest amount of adsorbent (15 mg).

The purpose of all these tests was to demonstrate the usability of activated carbon-doped nanofibres in adsorption. Thus, in the study, it has been shown that it is possible to obtain enhanced adsorption properties by the combined effect of activated carbon obtained from a waste (zero-cost) material like human hair and nanofibres, which are very attractive in wastewater treatment due to their high surface areas. Additionally, ultrasonic vibrations created by the ultrasonic bath where the adsorption media is placed aided the adsorption process and shortened the adsorption duration. Moreover, future studies may include the production of nanofibres by low-cost or recycled polymers.

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