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Adsorption Optimization of Congo Red Dye onto Electrospun Nanofibers of Polyacrylonitrile functionalized with Fe₃O₄ Nanoparticles

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Abstract

Ferric oxide nanoparticles Fe₃O₄NPs have been prepared by the coprecipitation method, which were used to functionalize the surface of electrospun nanofibers of polyacrylonitrile to increase their effectiveness in adsorption of Congo red (CR) dye from their aqueous solutions. The effect factors of adsorption were systematically investigated such as adsorbent mass, initial concentration, contact time, temperature, ionic strength and pH. The maximum adsorbed amount of the dye was at 0.003g of adsorbent. The adsorption of dye increased with increasing initial dye concentration and the system reaches to the equilibrium state at 150 min. The adsorbed dye capacity decreases with increasing temperature which indicates to the exothermic nature of adsorption system. The results referred that the adsorption capacity increases with increasing ionic strength and it was in natural medium has a greatest value. So, the desorption process was examined to demonstrate the possibility of recycling of the adsorbent surface. The desorbed dye from the studied adsorbent surface in basic solution was better than acidic solution.

Keywords: Adsorption, Congo red, Nanofibers, Electrospinning, Polyacrylonitrile, Ferric oxide nanoparticles, Desorption.

Introduction

There are many pollutants that contribute to environmental pollution, such as phenols, heavy metals, pesticides, radionuclides, etc [1]. However, dyes are an important class of pollutants that are produced in large quantities from textiles, tanneries, dyeing, leather, plastic, paper, plastic, cosmetic and paint industries [2, 3]. In general, dyes reduce light penetration and thus greatly affect photosynthesis activity [4]. Moreover, many dyes threaten aquatic organisms and human health due to their toxic and carcinogenic nature [5].



To this day, Congo red is a typical anionic dye used in a wide range of industries, due to its surface adhesion force and low cost [6]. It is a known carcinogen due to the possibility of its metabolism to benzidine [7]. There are many water treatment technologies such as chemical oxidation [8], coagulation [9], electrochemical oxidation [10], coagulation [11] and adsorption [12]. It is a viable and helpful approach to remove dyes from water where adsorbents are continually developed [13, 14]. Therefore, nanofibers offer excellent adsorbents because of the high surface area per mass [15]. Since the ability to modify the surface of nanofibers with active substances, the incorporation of nanoparticles on the surface of nanofibers increases their capacity to remove ionic dyes from aqueous media [16].

In this work Fe_3O_4 nanoparticles was prepared and characterized by different techniques, such as infrared spectroscopy (FTIR), Atomic Force Microscopy (AFM), Scanning Electron Microscopy (SEM), X-ray dispersion spectroscopy (EDS), X-Ray Diffraction (XRD), Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Holland (BJH). The surface of electrospun polyacrylonitrile nanofibers functionalized with Fe_3O_4 nanoparticles to investigate the adsorption optimization of Congo red dye (CR) from aqueous solution. The effect of various factors were studied such as adsorbent mass, initial concentration, contact time, temperature, ionic strength and pH. The desorption of CR from adsorbent surface also was examined.

Experimental

Chemicals

All used chemical materials (Polyacrylonitrile, dimethylformamide, ferrous sulfate heptahydrate, Ferric chloride, ammonium hydroxide, hydrochloric acid, sodium chloride, potassium chloride, sodium hydroxide, sodium carbonate and Congo red dye as shown in Figure 1.) have been purchased from Sigma-Aldrich.

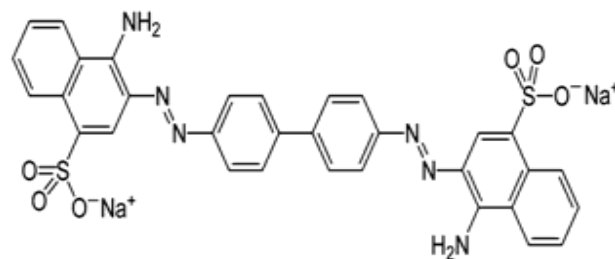


Figure 1. Chemical structure of (CR) dye.

Instruments

Hotplate stirrer LMS-1003 LabTech, Electronic balance CPA 22 Sartorius, Mechanical stirrer HS-30D Wisest iv, Water Bath Shaker SWB-25 HYSC, spectrophotometer S8400 Shimadzu, X-ray diffraction spectrophotometer XRD-6000 Shimadzu, Atomic force microscope AA-3000 Phywe, Scanning electron microscope S8000 TESCAN, Thermo gravimetric analyzer TT-1000 STA and Electro spinning instrument NE300 Invoke nose LTD.

Prepared of Ferric oxide nanoparticles $\text{Fe}_2\text{O}_3\text{NPs}$

Ferric oxide nanoparticles $\text{Fe}_2\text{O}_3\text{NPs}$ were prepared using the coprecipitation method [17]. 0.02M (500 mL) of ferrous sulfate $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ aqueous solution was heated to 353 K, then

0.08M (400 mL) of ferric chloride FeCl_3 aqueous solution was added drop by drop with the temperature maintaining. After the completion of adding, 30% (20 mL) of ammonium hydroxide was added to the previous mixture in one batch with continuous stirring. The mixture was left to become at room temperature, then the product was collected and washed with distilled water several times. Finally, the resulting calcification was calcinated in the furnace at 773 K for 180 min.

Electrospinning of Polyacrylonitrile functionalized with Fe_3O_4 Nanoparticles

A solution of polyacrylonitrile with concentration 7% wt/v was set up according previous work [18]. It is prepared utilizing dimethylformamide as a solvent at 323 K for 240 min with continuous stirring to acquire a homogeneous solution of polymer. The electrospinning solution was moved to a plastic syringe with capacity 20 mL associated with a nozzle 0.9 mm after addition 1% wt/v of the prepared Fe_2O_3 NPs. The process was done under 30 kV and the solution of polymer stretched out towards the collected drum with a rotating speed 50 rpm and following rate 0.01 mL/min, along 0.19 m distance between the two electrodes. The produced nanofibres were dried at 333 K C for 180 min in a vacuum oven.

Adsorbent surfaces

The prepared nanofibers were cut to a circular pieces with a diameter 0.02 m to be used as adsorbent surfaces in the adsorption experiments.

Experiments of adsorption

The study of adsorption of CR dye onto electrospun nanofibers of polyacrylonitrile functionalized with Fe_3O_4 NPs was performed by The amount of adsorption was explored by estimating the absorbance of the solution of Congo red dye CR with Uv-Vis spectrometer at a wavelength 497 nm.

Results and discussion

Fourier-transform infrared spectroscopy (FTIR)

Fourier-transform infrared spectroscopy (FTIR) of the prepared Fe_3O_4 NPs confirms the composition of the Fe-O bond that appeared at a frequency 686 cm^{-1} and 894 cm^{-1} [19], Figure. 2. . Moreover, a peak appears at 1654 cm^{-1} is attributed to the vibration of group C = O group which is shifted to a lower frequency, as a result to the adsorption of carbon dioxide molecules of the air at the surface of the prepared nanoparticles. It has been observed as for the polyacrylnitrile nanofibers functionalized with Fe_3O_4 nanoparticles, that the most of absorption peaks are shifted to a lower frequencies due to the interaction between the polyacrylonitrile molecules and nanoparticles, and these peaks are less intense compared to the unfunctionalized fibers, Figure. 3.

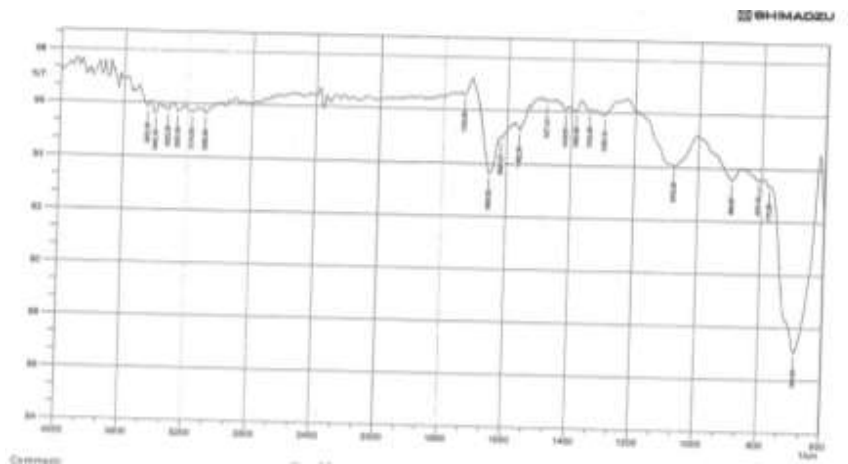


Figure. 2 FTIR spectrum of the prepared Fe_3O_4 NPs.

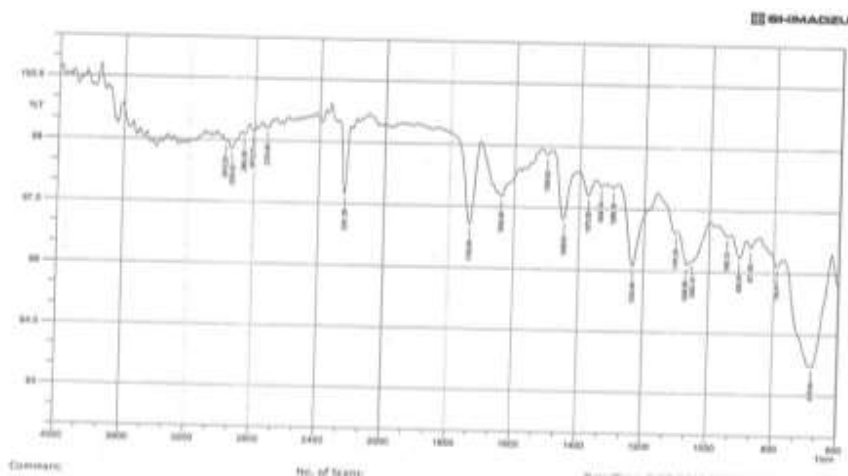
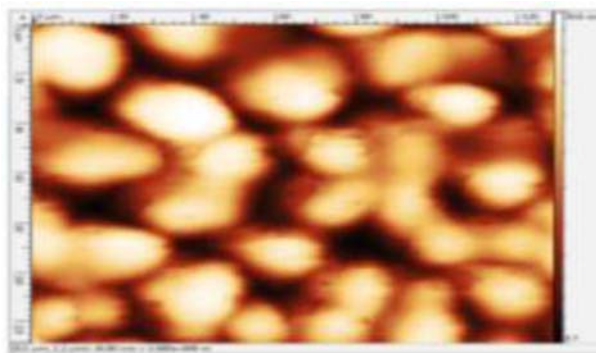


Figure. 3 FTIR spectrum of electrospun nanofibers of polyacrylonitrile functionalized with Fe_3O_4 NPs.

Atomic force microscope (AFM)

Figure. 4 show the micrographs of atomic force microscope (AFM) of the prepared Fe_3O_4 NPs. As shown in the two and three dimensions images have that the size of examined nanoparticles is 29.8 nm and their thickness is 30 nm.



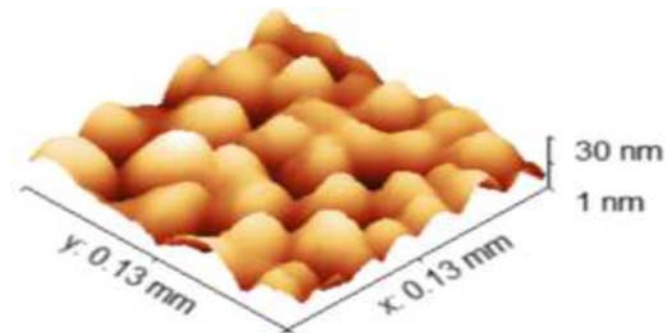


Figure. 4. AFM image of the prepared Fe₃O₄NPs.

Scanning electron microscope (SEM)

The morphology of prepared Fe₃O₄NPs surface were analyzed by scanning electron microscope, as shown in Figure. 5. It is noted that there is a high attraction between the particles, and they take the spinel shapes shape with diameters range (50-30) nm. It is noted from the surface morphology that there are swellings in the beds form in the nanofibers, which is due to the increased viscosity of the polymeric spinning solution as a result of the addition of ferric oxide nanoparticles. Fe₃O₄NPs distribution is also noticed with a small percentage, due to the small percentage of deformation, as shown in Figure. 6.

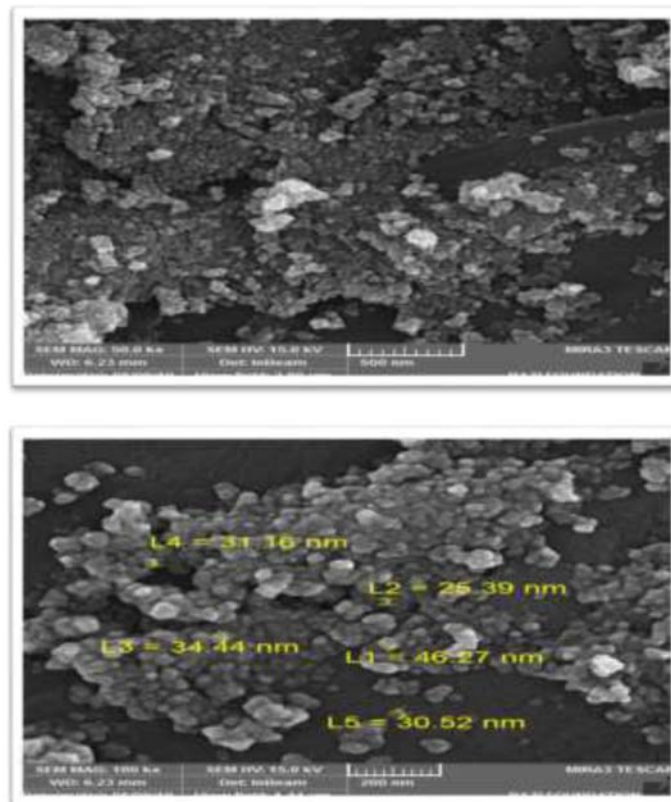


Figure. 5. SEM image of the prepared Fe₃O₄NPs.

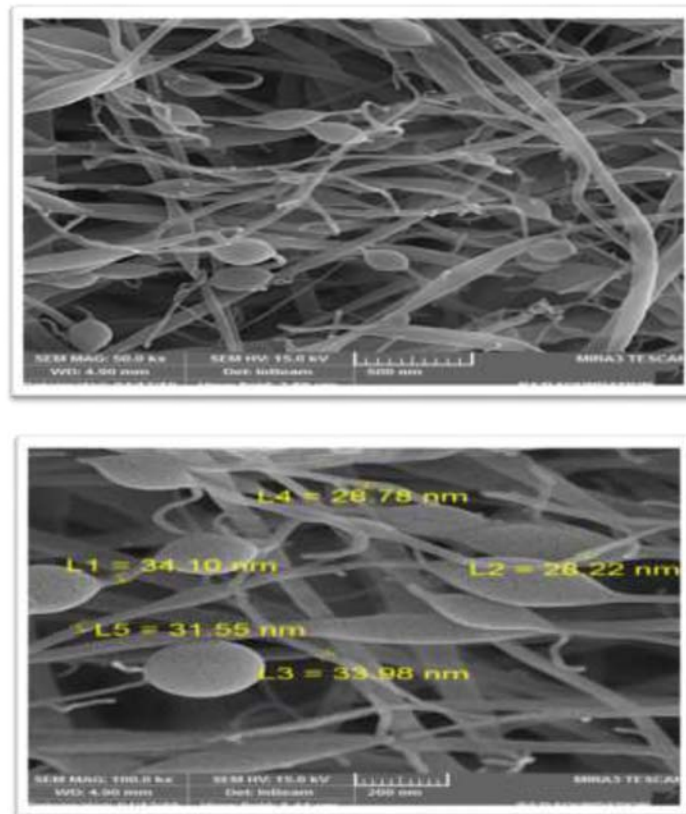


Figure. 6. SEM image of electrospun nanofibers of polyacrylonitrile functionalized with Fe_3O_4 NPs.

X-ray dispersion spectroscopy (EDS)

Figure. 7 and 8 show the X-ray dispersion spectrum (EDS) for prepared Fe_3O_4 NPs and polyacrylonitrile nanofibers functionalized with Fe_3O_4 Nanoparticles respectively, Figures (3-10). It is observed, that only iron and oxygen are present in EDX spectrum of Fe_3O_4 NPs which confirms the purity of the prepared nanoparticles. On the other side, Figure. 8 confirms the presence of iron and oxygen due to the prepared nanoparticles in addition to carbon and nitrogen as essential elements in the polymer composition.

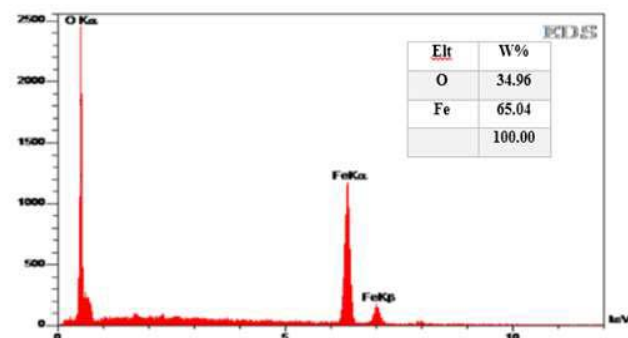


Figure. 7. EDS spectrum of the prepared Fe_3O_4 NPs.

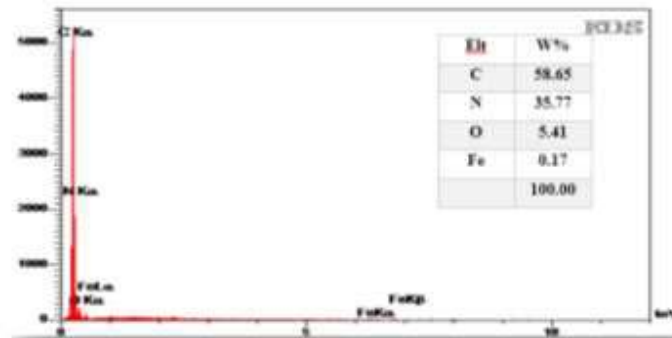


Figure. 8. EDS spectrum of electrospun nanofibers of polyacrylonitrile functionalized with $\text{Fe}_3\text{O}_4\text{NPs}$.

X-ray diffraction spectroscopy (XRD)

The XRD diffraction pattern of the prepared Fe_3O_4 nanoparticles was obtained, as shown in Figure. 9 and the diffraction peaks corresponding to 2θ were 30.2796, 35.6456, 43.3467, 53.6271, 57.3153, 62.8834 and 74.6700 which are refer to the spinel structure [20]. So, Deby Scherrer's equation formula used to calculate the crystalline size of $\text{Fe}_3\text{O}_4\text{NPs}$ [21]. It was found that the average of crystalline size is 30 nm.

$$L = \frac{0.94 \lambda}{\beta \cos \theta} \dots \dots \dots (1)$$

where D is the particle size in nm, λ is the used x-ray wavelength in (nm), β is the middle of the maximum beam width (FWHM) and θ is the angle of Bragg. Figure (3-14) shows that the polyacrylnitrile nanofibers functionalized with Fe_3O_4 nanoparticles retained with the amorphous character as diffraction patterns of $\text{Fe}_3\text{O}_4\text{NPs}$ did not appear clearly, and this is due to the low ratio of functionalization (1%) with a simple appearance of the peak feature of $\text{Fe}_3\text{O}_4\text{NPs}$ at $2\theta=45$.

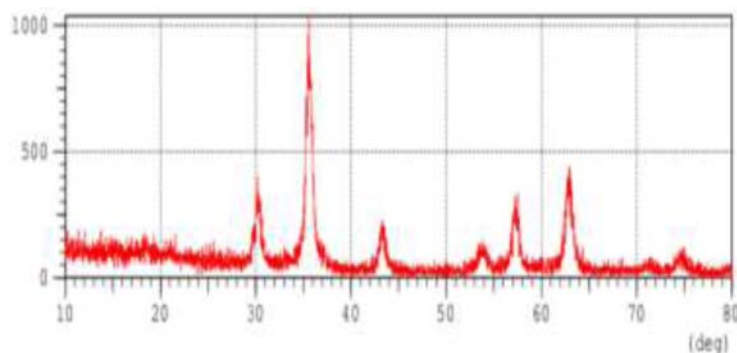


Figure. 9. XRD spectrum of the prepared $\text{Fe}_3\text{O}_4\text{NPs}$.

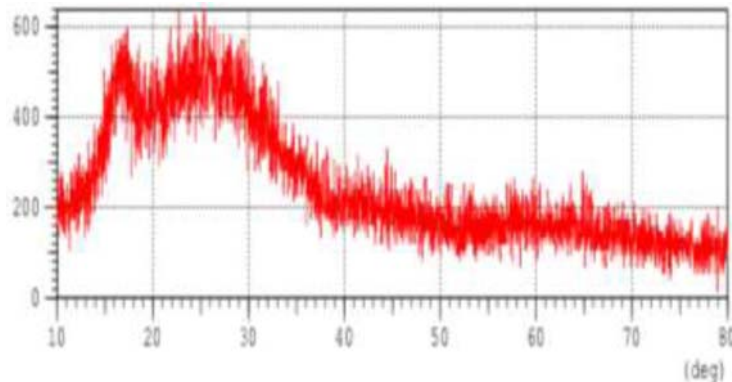


Figure. 10. XRD spectrum of electrospun nanofibers of polyacrylonitrile functionalized with $\text{Fe}_3\text{O}_4\text{NPs}$.

Thermal gravimetric analysis (TGA)

Thermal gravimetric analysis was carried out for the nanofibers of polyacrylonitrile functionalized with $\text{Fe}_3\text{O}_4\text{NPs}$ by heating them up to a temperature of 600°C and at a rate of $100^\circ\text{C}/\text{min}$ in an inert atmosphere. Figure. 11 shows that polyacrylonitrile functionalized with $\text{Fe}_3\text{O}_4\text{NPs}$ have become more stable after the functionalization, where they remained stable after heating to 334.4°C , the loss rate reached 25.9%, and when the temperature reached 385.2°C , the percentage of weight loss reached 21.8%, and the amount of loss in the degree of 417.4°C was 8.4% until it finally reached 22.8% at the degree of 594.9°C . The weight loss ratio of the functionalized nanofibers is about 78.9%, which is less than that of the pure nanofibers of polyacrylonitrile [22]. This is due to the improvement in the thermal properties of polymer due to the increase in the surface area relative to volume as a result of the functionalization with $\text{Fe}_3\text{O}_4\text{NPs}$.

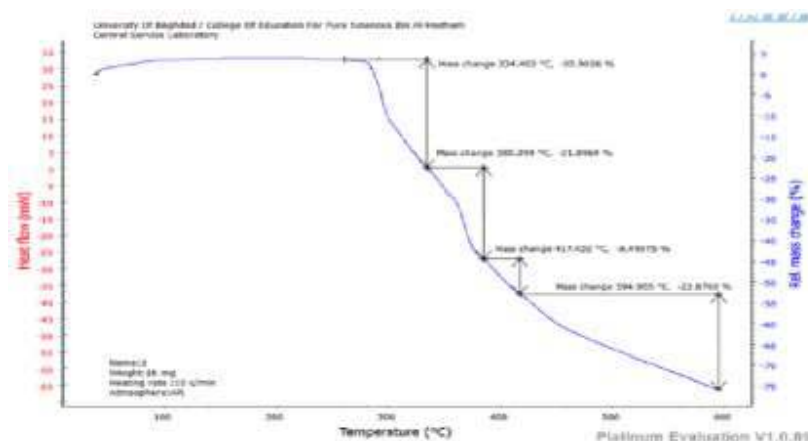


Figure. 11. TGA analysis of electrospun nanofibers of polyacrylonitrile functionalized with $\text{Fe}_3\text{O}_4\text{NPs}$.

Surface area and porosity

The surface area and porous surface of the adsorbed is one of the major factors affecting on the adsorption. So, Langmuir isotherm, Brunner-Emmett-Teller (BET) and Barrett-Joyner-Holland

(BJH) technique were used to determine the surface area and pore size the nanofibers of polyacrylonitrile functionalized with Fe_3O_4 NPs. Figure. 12 shows Langmuir isotherm of adsorption- desorption of nitrogen gas on the surface of the functionalized nanofibers PAN- Fe_3O_4 NPs. It is noted an increase in the adsorption of gas molecules is observed with an increase in the pressure applied at all points. So, the adsorption isotherm follows type II, which refers to a multi-layer adsorption. Also, a decrease in the amount of gas desorbed when the applied pressure is decreases at all studied pressures.

Figure. 13 shows BET diagram for nitrogen adsorption on the surface of the functionalized nanofibers, where it is observed that nitrogen gas molecules increase when the applied pressure increases to reach its highest value 0.1229 at a pressure of $0.355 P / P^\circ$. This means electrospun nanofibers of polyacrylonitrile functionalized with Fe_3O_4 NPs have the ability to adsorb nitrogen gas molecules at all studied pressures.

Figure. 14 shows BJH diagram for nitrogen gas adsorption on the functionalized nanofibers surface PAN- Fe_3O_4 NPs. The average pore size is 1.21nm, and the surface area of nanofiber is $12.217\text{m}^2\text{g}^{-1}$. In general, the incompatibility of the explanations between the BET and BJH diagram of the studied fiber surface is not due to the incompatibility of the BJH model to the micro pores.

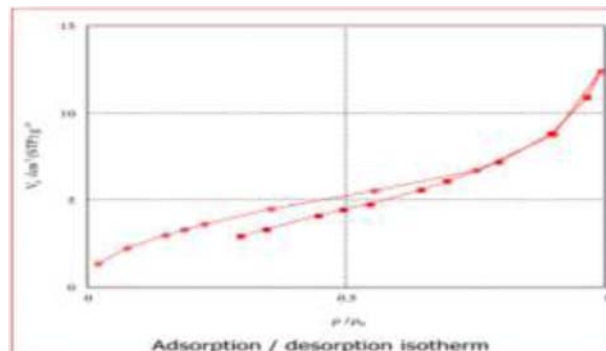


Figure. 12 Langmuir diagram of adsorption- desorption of electrospun nanofibers of polyacrylonitrile functionalized with Fe_3O_4 NPs.

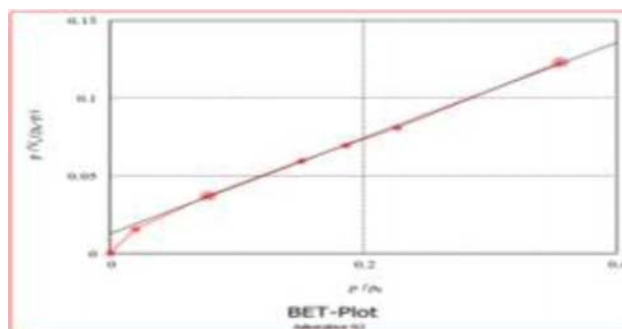


Figure. 13 BET diagram of adsorption- desorption of electrospun nanofibers of polyacrylonitrile functionalized with Fe_3O_4 NPs.

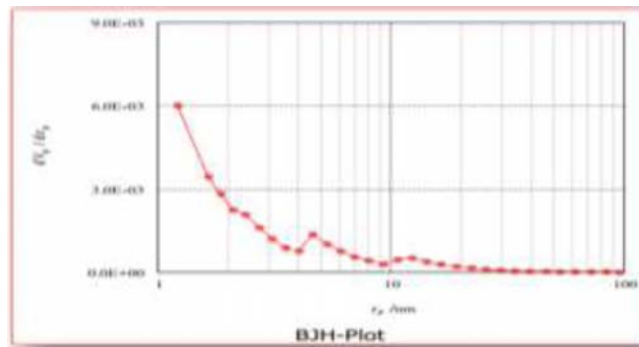


Figure. 14 BJH diagram of adsorption- desorption of electrospun nanofibers of polyacrylonitrile functionalized with Fe₃O₄NPs.

The Effect Factors on Adsorption

The amount of adsorbed of Congo red dye q_d (mg/g) was eastimated utilizing the following equation [23]:

$$q_e = \frac{(C_o - C_e)V}{w} \dots \dots \dots (2)$$

where C_o is the initial concentration of adsorbed (mg/L), C_e is the concentration of adsorbed at equilibrium (mg/L), V is the volume of solution (L) and w is the mass of adsorbent (g).

The effect of adsorbent mass

The adsorbent mass effect of nanofibers of polyacrylonitrile functionalized with Fe₃O₄NPs on the adsorption of Congo red dye at 25 °C was studied with an initial dye concentration 25mg / L, Figure 3-20. The amount of adsorbed dye increases with increasing mass of the adsorbent surface, as a result to the increase in the surface area which leads to an increase in adsorption sites [24]. As the highest adsorbed amount of the dye reached at 0.003g of adsorbent, after that the adsorbent mass became ineffective, reflecting the achievement of saturation status.

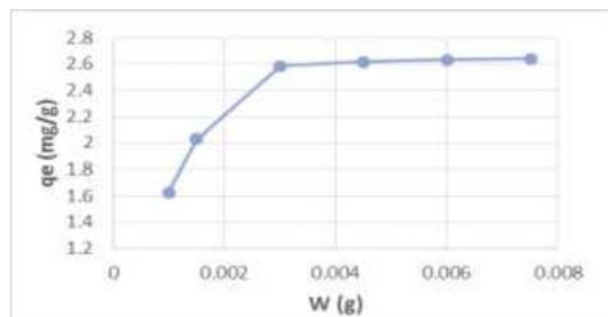


Figure. 15 Effect of adsorbent mass of CR dye adsorption onto electrospun nanofibers of polyacrylonitrile functionalized with Fe₃O₄NPs.

The effect of initial concentration

The initial concentration effect of the adsorption of Congo red dye on nanofibers polyacrylonitrile functionalized with Fe₃O₄NPs was investigated at concentrations (5, 10, 15, 20,

25, 30) mg/L, adsorbent mass 0.003g and temperature 25⁰C, Figure. 16. It is observed, that the adsorbed amount increases with increasing the dye concentration as a result to the adsorption capacity increasing. Thus, it is due to the increase in the diffusion rate, which leads to the mass transfer on the adsorbent surface [25].

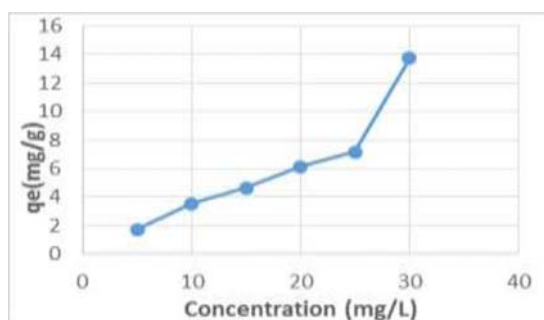


Figure. 16 Effect of initial concentration of CR dye adsorption onto electrospun nanofibers of polyacrylonitrile functionalized with Fe₃O₄NPs.

Effect of contact time

The contact time effect of the adsorption of Congo red dye on nanofibers of polyacrylonitrile functionalized with Fe₃O₄NPs was investigated at initial concentration 30 mg/L, adsorbent mass 0.003g and temperature 25⁰C, Figure. 17. The adsorbed amount increases with increasing time until it reaches a stabilized value after 150 min, it is reflect that the adsorption system has been reached to the equilibrium state at this time. This is due to the saturation of all active sites on the adsorbent surface with the adsorbed molecules of Congo red dye [26].

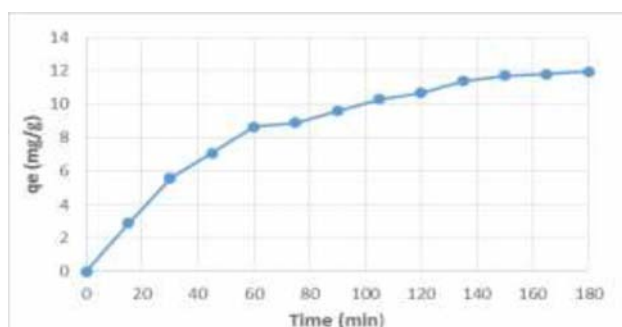


Figure. 17 Effect of contact time of CR dye adsorption onto electrospun nanofibers of polyacrylonitrile functionalized with Fe₃O₄NPs

Effect of temperature

The temperature effect of the adsorption of congo red dye on nanofibers polyacrylonitrile functionalized with Fe₃O₄NPs was examined at temperatures (298, 303, 308, 312) K, initial concentration 30 mg/L and adsorbent mass 0.003 g, Figure. 18. The adsorbed dye capacity decreases with increasing temperature, which indicates the exothermic nature of studied system [27]. This is due to the increase in the kinetic energy of the adsorbed molecules as a result to the temperature increasing, which leads to a separation of those molecules from the adsorbent surface and their return to the solution.

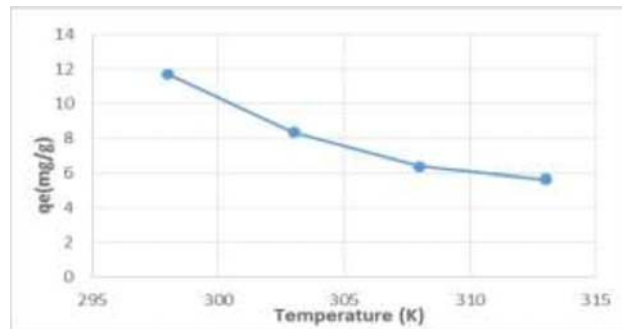


Figure. 18 Effect of temperature of CR dye adsorption onto electrospun nanofibers of polyacrylonitrile functionalized with $\text{Fe}_3\text{O}_4\text{NPs}$.

Effect of ionic strength

The ionic strength effect of the adsorption of congo red dye on nanofibers polyacrylonitrile functionalized with $\text{Fe}_3\text{O}_4\text{NPs}$ was examined with concentrations (0.1, 0.2, 0.3, 0.4) M of NaCl and KCl at initial concentration 30 mg/L, adsorbent mass 0.003 g and temperature 298 K, Figure. 19. The results showed that the adsorption capacity increases with increasing ionic strength, due to the fact that the salt collects the dye molecules in the solution. Hence, the Hydrophobicity property of dye increases and their solubility in water decrease to support the adsorption capacity increasing [28].

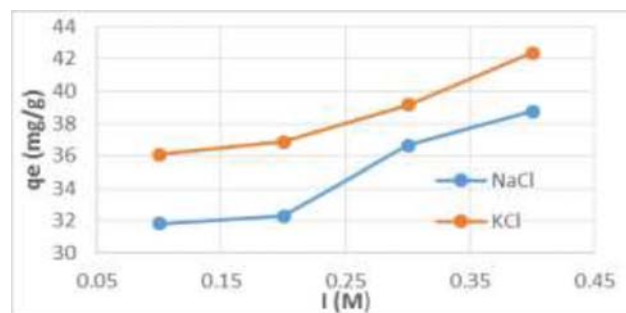


Figure. 19 Effect of ionic strength of CR dye adsorption onto electrospun nanofibers of polyacrylonitrile functionalized with $\text{Fe}_3\text{O}_4\text{NPs}$.

Effect of pH

The pH effect the adsorption of Congo red dye on nanofibers polyacrylonitrile functionalized with $\text{Fe}_3\text{O}_4\text{NPs}$ was investigated at an initial concentration 30 mg/L, adsorbent mass 0.003 g and temperature 298 K, Figure. 20. The information demonstrated that the adsorption capacity has a greatest value at natural medium, and it diminished when the pH of solution become higher. Thus, the electrostatic repulsion has been accrue at an alkaline medium which increases the negative charge of surface, which inhibits the adhesion of the anionic dye molecules onto the adsorbed surface [29].

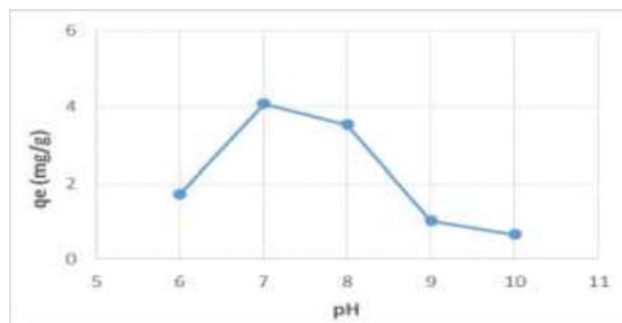


Figure. 20 Effect of ionic strength of CR dye adsorption onto electrospun nanofibers of polyacrylonitrile functionalized with Fe₃O₄NPs

Desorption investigation

The study of desorption process was carried out to demonstrate the ability to recycle absorbent surfaces after the adsorption process. 0.1 M (10 mL) of an aqueous solution of hydrochloric acid, sodium chloride, potassium chloride, Sodium hydroxide and sodium carbonate were used to determine their effect on the desorption of Congo red dye from nanofibers polyacrylonitrile functionalized with Fe₃O₄NPs for 150 min, Figure. 21.

As the amount of desorption of Congo red dye q_d (mg/g) was founded using the following equations [30]:

$$q_d = \frac{(C_d * V_{sol.})}{w} \dots \dots \dots (3)$$

where C_d is the concentration of desorbed (mg/L), C_e is, V is the volume of solution (L) and w is the mass of adsorbent (g).

It is noted from the experimental data that the highest desorption amount for the Congo red dye from the nanofibres surface is in the basic medium NaOH, which may be due to the high solubility of the dye in the basic solution compared to the acidic solution while the lowest desorption percentage is in Na₃CO₃medium. In general, the desorption process of the surface studied by various factors is low. It can be conclude that the studied nanofibers surface cannot be recycled using these factors due to the chemical adsorption of system. Hence, the disassociation of dye molecules from adsorbent surface is difficult [31].

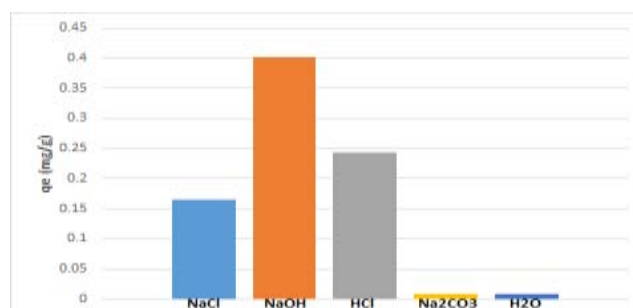


Figure. 21 Desorption of CR dye from electrospun nanofibers of polyacrylonitrile functionalized with Fe₃O₄NPs

Conclusion

The electrospun nanofibers surface of polyacrylonitrile (PAN) were functionalized with Fe₃O₄NPs successfully for utilizing as an adsorbent to congo red (CR) dye from their aqueous solutions. It was characterized utilizing Fourier-transform infrared spectroscopy (FTIR), Atomic force microscope (AFM), Scanning electron microscope (SEM), X-ray dispersion spectrum (EDS), X-ray diffraction spectroscopy (XRD), Thermal gravimetric analysis (TGA) and Brunner-Emmett-Teller (BET) technique. The effect factors such as adsorbent mass, initial concentration, contact time, temperature, ionic strength and pH were investigated. The functionalized nanofibers appeared appreciable efficiency to adsorption of (CR) dye from aqueous solutions. The greatest adsorbed amount was at 0.003g of adsorbent and it increased with increasing initial concentration. The studied system reaches to the equilibrium at 150 min. So, the decreasing in the adsorbed dye capacity with increasing temperature refers to the exothermic nature of adsorption system. The results referred that the adsorption capacity increases with increasing ionic strength and it was in natural medium has a greatest value. Additionally, the desorption process was examined to demonstrate the possibility of recycling of the adsorbent surface. It was found that the desorbed (CR) dye from the studied adsorbent surface in basic medium was better than acidic medium.

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