

# Synthesis and characterization of PET fibers grafted with binary mixture of 2-methylpropenoic acid and acrylonitrile by free radical: its application in removal of cationic dye

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**Abstract** Grafting of binary vinyl monomer mixtures such as 2-methylpropenoic acid (MPA) and acrylonitrile (AN) onto poly (ethylene terephthalate) fibers (PET) was achieved in an aqueous medium with using benzoyl peroxide like free radical initiator. A new reactively fibrous adsorbent was used for removal of dye such as methylene blue (MB) from aqueous media through batch sorption method. Fibers adsorbent was swelled in solution to support the graft and the subsequent polymerization of MPA/AN onto polyester fibers. Optimum conditions for grafting were discovered and reactive fiber were characterized. Variations of graft yield with time, temperature, initiator concentration and monomer mixture ratio were investigated. The optimum initiator concentration was found to be  $8 \times 10^{-3}$  mol/L. The percentage of grafting rose steadily with the vinyl monomer mixture monomer concentration (50 %). The optimum temperature and polymerization time were found to be 80 °C and 120 min, respectively. The use of AN and MPA monomers together in grafting produce a significant increased in the graft yield. Experimental studies showed that the percentage removal of MB was a great higher on the MPA/AN grafted PET (MPA/AN-g-PET) fibers than on the original PET fibers. The adsorbed quantity of MB improved with pH and basic pH was appropriate for the elimination of MB. MPA/AN-g-PET fibers removed 98 % of cationic dye when initial concentration diverse from 10 to 80 mg L<sup>-1</sup> at pH 9.0. Almost all of the adsorbed cationic dye was eluted by ethanoic acid in methanol. Ten removal–desorption cycles indicated that the reactive fibers were favorable for repetitive use without notable change in removal capacity. Consequently, the MPA/AN-g-PET fibers have

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demonstrated potential as an effective adsorbent for the extremely effective removal of cationic dyes from aqueous media.

**Keywords** Poly(ethylene terephthalate) fibers · Grafting mixture monomers · Removal · Cationic dye · Reusing

## Introduction

Dyes are a significant class of artificial organic substances utilized in many sectors like paints, textiles, printing inks, paper, plastics and food [1–4]. Dyes are released into the surroundings from many industries. Contamination of various water resources with dyes discovered as a crucial problem owing to their harmful and toxic effects on human health and environmental. Thus, it is of nice attention to get rid of dyes from waste before they are released into the surroundings [5, 6]. The procedure of dye-contaminated water is incredibly tough since dyes are extremely soluble and stable in water. Moreover, they are non-degradable and cannot be decomposed in municipal water treatment plants [5–8]. Many technologies have been used to eliminate of dyes from waste material, that embody coagulation–flocculation [9], electrochemical treatment [10], biodegradation [11], photocatalysis [12], solvent extraction [13], reverse osmosis [14], membrane filtration [15], oxidation–reduction [16], electro-dialysis [17] and adsorption [18]. However, among these, sorption is the effective technique as a result of its top potency, low disbursement and simple operation and in most of the cases regeneration of the adsorbent is feasible [1, 19, 20]. Several adsorbents like activated carbon, clay, wood, sawdust, rice husk, fly ash, chitosan, cotton, chitin, silica, polymers, etc., are used for the removal of dyes from waste product [1, 8, 19, 21, 22].

Over the previous couple of years, use of polymeric materials as adsorbent has augmented an attention owing to their outstanding adsorption performance, low cost, wide usability, the presence of different functional groups, regenerability on site and reuse [22, 23]. Moreover, increasing attention has been centered up to the chelating fibers recently as a result of they can be suitably utilized in varied cases and possess giant specific areas that owing to an excellent adsorption capability and a major sorption rate of the fabric. Most of the modified fibers were informed by introduction of appropriate functional groups like  $-\text{COOH}$ ,  $-\text{CN}$ ,  $-\text{C}_5\text{H}_5\text{N}$ ,  $\text{CONH}_2$ ,  $-\text{NH}_2$ ,  $-\text{COOR}$ , and epoxy on fibrous polymers [18, 23, 24].

The PET fibers modification by free radical graft polymerization of monomers into fibers has exaggerated attention to induce fascinating properties in a material. The radical graft polymerization of binary vinyl mixtures monomers like 2-hydroxyethylmethacrylate (HEMA)/4-Vinyl pyridine (4-VP) [25], HEMA/acrylamide (AAM) [26], glycidyl methacrylate (GMA)/2-methylpropenoic acid (MPA) [27], Itaconic Acid/AAM [28], MAA/AMM [29] and Maleic Acid/Methacrylamide [30] onto PET fibers has been noticed to introduce appropriate functional groups within the fibers for eliminate of dyes and heavy metals ions from dirty water.

In first part of the study, PET fibers were grafted with MPA and AN using BPO as initiator. MPA/AN grafted PET fibers were characterized and optimized grafting

conditions such as concentration of monomer, initiator, temperature and reaction time. In second part of the study, MPA/AN-g-PET fibers were utilized for eliminate of methylene blue (MB) in aqueous media by a batch equilibration style as a new sorbent.

## Experimental

### Materials

The polyester (PET) fibers (122 dTex) was SASA Co. (Turkey) product. The models were cleared for 10 h by dimethyl ketone and dried in an oven at surroundings temperature. BPO was doubly recrystallized from chloroform in methanol and dried. Other reagents were used like received. Total chemicals were Merck products.

### Swelling procedure

The fibers were immersed in dichloroethane at 90 °C for 2 h. The swollen the fibers was wiped with cleansing tissue to remove dichloroethane and place into the polymerization medium.

### Graft copolymerization process

Graft copolymerization was allotted in a three-necked polymerization glass tube. The polymerization tube containing the PET fiber about 0.3 g, acceptable quantity of MPA/AN (50/50) monomer mixture and BPO at needed concentration in 2 mL dissolving agent (acetone) was created up to 20 mL with demineralized water. The polymerization tube was instantly placed into the water bathtub adjusted to the polymerization temperature. At the tip of the preset chemical change time, the grafted fibers were taken out. Compound and free of the homopolymers and copolymers were extracted with boiling water for 24 h and Soxhlet-extracting the PET fibers in acetone for 24 h. The modified fibers were then dried at 55 °C for 24 h and weighed. The graft yield (GY) was calculated from the weight increase in grafted fibers as follows:

$$GY (\%) = [(m_g - m_i) / m_i] \times 100 \quad (1)$$

where  $m_i$  and  $m_g$  represent the weights of the original and grafted fibers, severally.

### Determination of water uptake capability

Exactly weighed dry MPA/AN grafted fibers were immersed in H<sub>2</sub>O at 25 °C for 24 h, wiped with filter paper, and weighed. Original and MPA/AN-g-PET fibers were dried for 24 h at 55 °C and weighed immediately. Water uptake values of the fibers were calculated gravimetrically.

## FTIR spectra

FTIR spectra of MPA/AN grafted PET fibers were obtained. The fibers were cut into roughly 1 mm size, mixed with KBr, and then pressed. The spectra were recorded on a Bruker Vertex 70 V FTIR photometer.

## Scanning electron microscopy (SEM) analysis

SEM analysis was carried out to research the surface morphology of original and MPA/AN-*g*-PET fibers employing a JEOL Model JSM 5600 scientific instrument. The fibers covered a thin evaporated layer of gold and were performed.

## Sorption

A volume of 25 mL of methylene blue (MB) solution (20 mg/L) was additional onto certain amount of MPA/AN-*g*-PET adsorbent in glass container. The ingredients were agitated on shaker (Selectra) set at a 110 rpm for a fixed period at 25 °C. The charged adsorbent was separated and cleaned politely. The solution was filtered and was adjusted to 6.8 pH value, the remaining cationic dye was analyzed employing UV/Visible photometer ( $\lambda = 665$  nm MB, Perkin Elmer Lambda 25). The amount of cationic dye adsorbed by the adsorbent was analyzed using Eq. 2.

$$q(\text{mg g}^{-1}) = (C_o - C_e) V(\text{L})/w(\text{g}) \quad (2)$$

where  $C_o$  is the initial dye concentration (ppm),  $C_e$  is the last concentration of dye solution;  $V$  is the volume of the dye solution used and  $w$  is the quantity of the adsorbent used.

## Desorption and reuse of MPA/AN-*g*-PET fibers

Desorption of cationic dye were studied with 25 mL of ethanoic acid in methanol adjusted to different concentrations. The dye was desorbed in half an hour, then diluted with water and analyzed as above. The desorption percent was calculated employing Eq. 3.

$$\% \text{ Desorption} = \frac{\text{Amount of cationic dye (mg) desorbed}}{\text{Adsorbed amount of cationic dye (mg) by adsorbent}} \times 100. \quad (3)$$

The sorption–desorption process was repeated ten times using MPA/AN monomer mixture grafted PET fibers

## Result and discussion

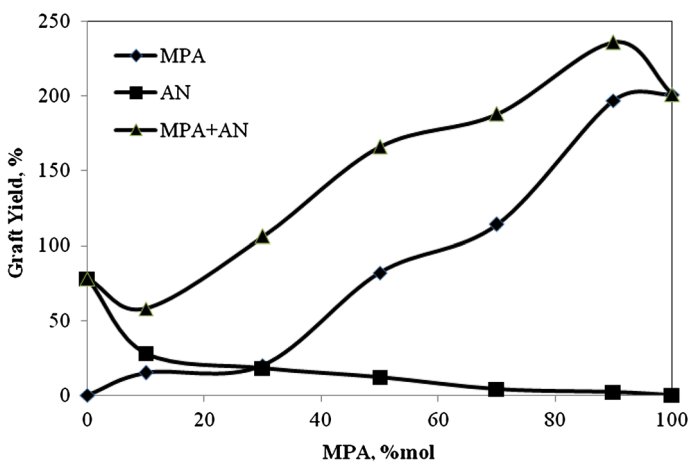
### Grafting of binary monomer mixture

In the present study, seen in Fig. 1, the % grafting achieved by the graft of PET fibers with MPA, AN and their binary monomer mixtures. Within the graft of pure MPA, the grafting increased with increasing MPA concentration, the maximum grafting is 201 %. For grafting with pure AN, the grafting increased with the AN concentration and achieved 78 %. Similarly, the use of AN and MPA binary monomer mixture in grafting manufactured an increase in the grafting. The grafting (236 %) was reached at a MPA/AN monomer mixture ratio of 90/10. The MPA has indicated a synergistic effect on AN, thus interest of AN for grafting upon PET fibers has increased. Similar results were also showed in literature [25, 30].

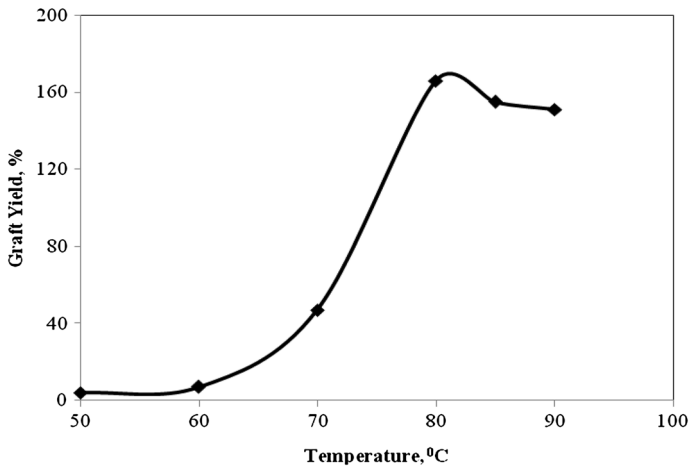
The graft copolymer chains grafted upon PET fibers. So PET fibers contains chelating groups like  $-\text{CN}$  of AN and  $-\text{COOH}$  of MPA.

### Graft copolymerization temperature study

The effect of polymerization temperature on the grafting upon PET fibers is illustrated in Fig. 2. Increase in polymerization temperature up to 80 °C causes a sharp slope in percent grafting, but beyond increase causes a decline. The rise in grafting yield up to 80 °C could be attributed to the fibers swellability, binary monomers mixture diffusion, increased rate of initiation and propagation reactions and increased number of free and macro radicals. The decrease of percentage grafting above 80 °C may be because of increased number of termination reactions.



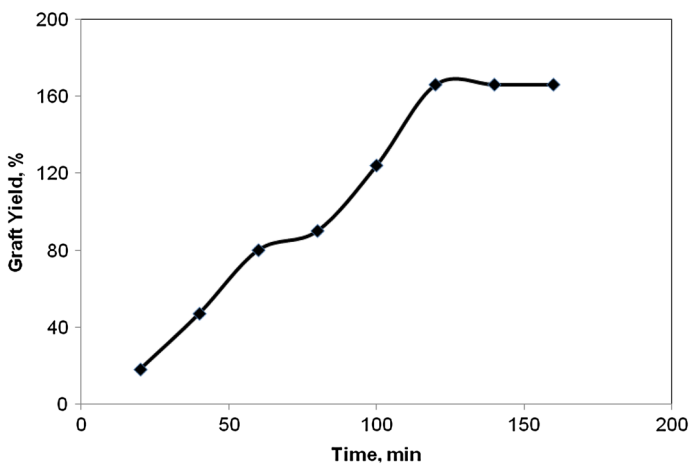
**Fig. 1** Effect of molar percentage rate on grafting percentage:  $[\text{MPA}/\text{AN}] = 1.0 \text{ mol/L}$ ;  $[\text{BPO}] = 8.0 \times 10^{-3} \text{ mol/L}$ ; temperature = 80 °C; time = 2 h



**Fig. 2** Effect of temperature on grafting percentage: [MPA/AN] = 1.0 mol/L (50 % mol); [BPO] =  $8.0 \times 10^{-3}$  mol/L; time = 2 h

### Graft copolymerization time study

Figure 3 shows the effect of reaction time on percent grafting. As seen, percent grafting rapidly increases with increasing graft copolymerization time and reached a maximum up to 2 h afterwards almost leveled off (166 % percent grafting). It shows that up to 2 h, the percent grafting rises because of the creation of graft copolymerization chains and the rise in length of developing chains with time. The leveling off of percent grafting with time may be mainly attributed to consuming of binary monomer mixture, formation of a diffusion barrier and increase in the medium viscosity by the formation of homo-poly(MPA), homo-poly(AN) and poly(MPA-co-AN) [26, 29].



**Fig. 3** Effect of reaction time on grafting percentage: [MPA/AN] = 1.0 mol/L (50 % mol); [BPO] =  $8.0 \times 10^{-3}$  mol/L; temperature = 80 °C

### Initiator concentration study

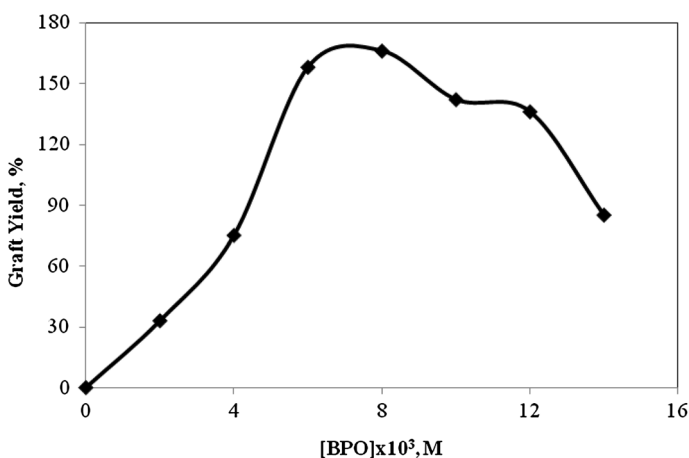
The initiator concentration has an important influence on percent grafting. Figure 4 illustrates the effect of BPO concentration variation in the range of  $0\text{--}14 \times 10^{-3}$  M. As seen the figure, maximum percent grafting can be seen at  $8 \times 10^{-3}$  M BPO then decreases with further increase in the BPO concentration. The inceptive increase is due the availability of more number of free and macro radicals created for grafting of the binary vinyl monomer mixture. Creates suitable sites for graft copolymerization is chain transfer reaction between active homo-poly(MPA), homo-poly(AN), poly(MPA-co-AN) and PET fibers. Above this limit, increase in radicals formation, possible combination reaction of the free radical species with homo-poly(MPA), homo-poly(AN), poly(MPA-co-AN) macro radicals and the fibers macro radicals. This is a typical behavior reported in many other studies [25, 26].

### Monomer mixture concentration study

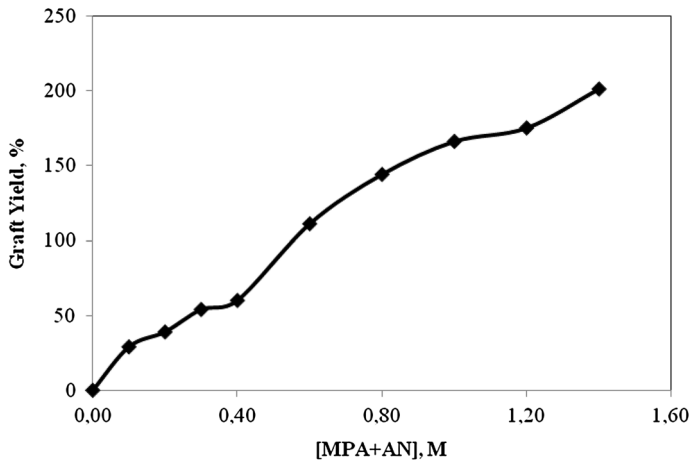
The effect of MPA/AN mixture concentration in the range of 0.0–1.4 M on percent grafting is illustrated Fig. 5. The percentage of grafting rose steadily with the vinyl monomer mixture monomer concentration (50 %). The increase in percent grafting may be attributed to the greater availability of the monomer mixture and increase in diffusion of monomer mixture upon the active site [25].

### Water uptake study

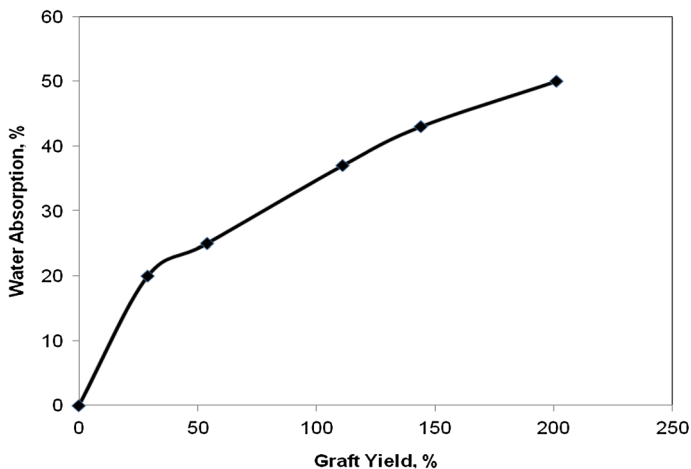
The water uptake capacity of MPA/AN-g-PET fibers was calculated gravimetrically and results are shown in Fig. 6. Pure PET fibers has very poor water uptake capacity, because it is hydrophobic and has low chemical reactivity. The water uptake of MPA/AN-g-PET fibers is increased with increasing grafting yield. This is



**Fig. 4** Variation of percent grafting with BPO concentration: [MPA/AN] = 1.0 mol/L (50 % mol); temperature = 80 °C; time = 2 h



**Fig. 5** Variation of percent grafting with MPA/AN monomer mixture concentration: MPA/AN = 50 % mol; [BPO] =  $8.0 \times 10^{-3}$  M; temperature = 80 °C; time = 2 h



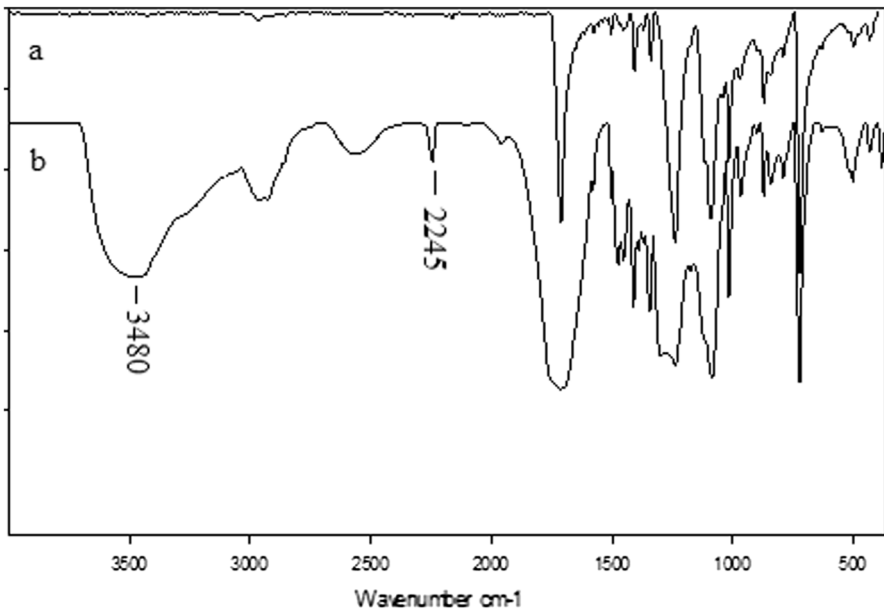
**Fig. 6** Effect of percent grafting on percent water uptake

mainly attributed to the hydrophilic groups like  $-\text{COOH}$  and  $-\text{CN}$  upon MPA/AN-g-PET fibers.

### FT-IR spectrum

The FT-IR spectra is known to be useful instrument to guarantee strong proof for the grafting. Thus, FT-IR spectra of original and MPA/AN-g-PET fibers have been analyzed and are displayed in Fig. 7. The FT-IR spectra of original PET fibers (Fig. 7a) displayed peaks owing to  $\text{C}=\text{O}$  (at  $1712 \text{ cm}^{-1}$ ),  $\text{C}=\text{C}$  and aliphatic  $\text{C}-\text{H}$  (at  $1411$  and  $1578 \text{ cm}^{-1}$ ) of PET fibers, while additional new peaks owing to the  $-\text{OH}$





**Fig. 7** FT-IR spectra of *a* original, *b* 201 % MPA/AN-*g*-PET fibers

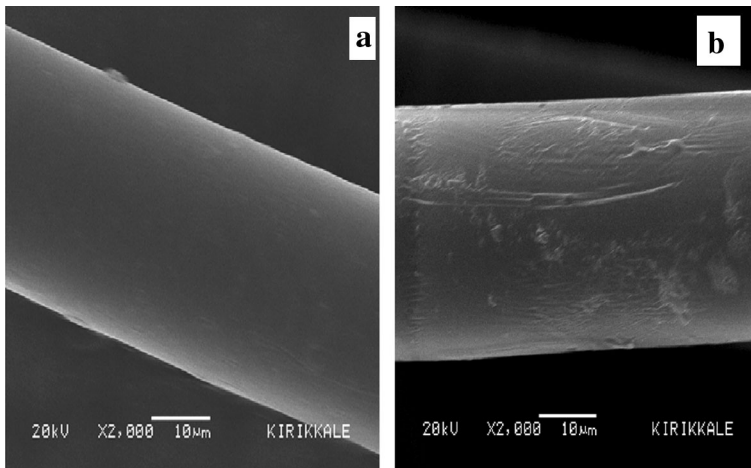
groups of MPA (at  $3480\text{ cm}^{-1}$ ) and the  $-\text{CN}$  groups of AN (at  $2245\text{ cm}^{-1}$ ) have been observed in Fig. 7b.

### Scanning electron microscopy (SEM) study

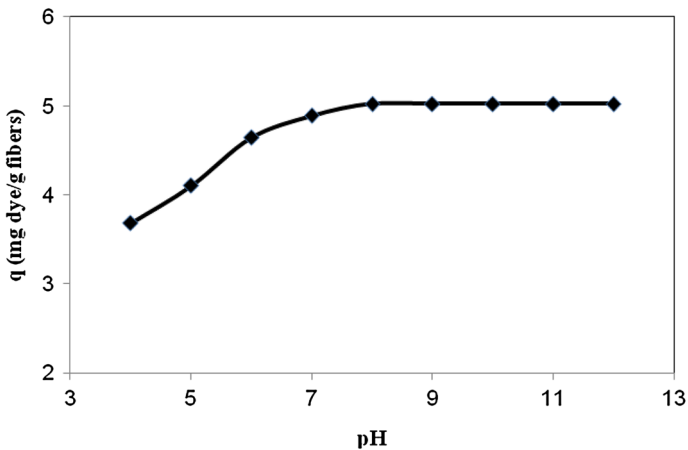
SEM study was carried out to analyze the surface morphology of original and MPA/AN-*g*-PET fibers (201 %) and is indicated in Fig. 8. SEM photograph of the original PET fiber (Fig. 8a) showed a smooth surface and relatively homogeneous surface. SEM photograph of MPA/AN-*g*-PET fibers (at 201 %), however, showed clear residual and cover of the grafting upon PET fibers.

### Effect of pH

The pH is a significant parameter in the sorption study, and thus the effect of pH on cationic dye removal was examined. Figure 9 shows that the percentage of cationic dye removal rises with increasing first pH, reaches a maximum at pH 9.0 and thereafter almost leveled off when solution pH was diversified from 1.0 to 12.0. The important rise in elute efficiency with increasing pH value may be owing to deprotonation of the carboxyl groups upon the surface of the adsorbent and thus rises the removal of cationic dye. When the pH of the solution increases; the free  $\text{COO}^-$  upon the adsorbent surface favors the sorption of cationic dye owing to the electrostatic attraction. As the low pH, the concentration of free  $\text{COO}^-$  upon the surface of modified PET fibers decreased, thus surface site of the grafted fibers do not favor the removal of cationic dye [22, 27].



**Fig. 8** SEM photographs of **a** original and **b** MPA/AN-g-PET fibers (201 %)



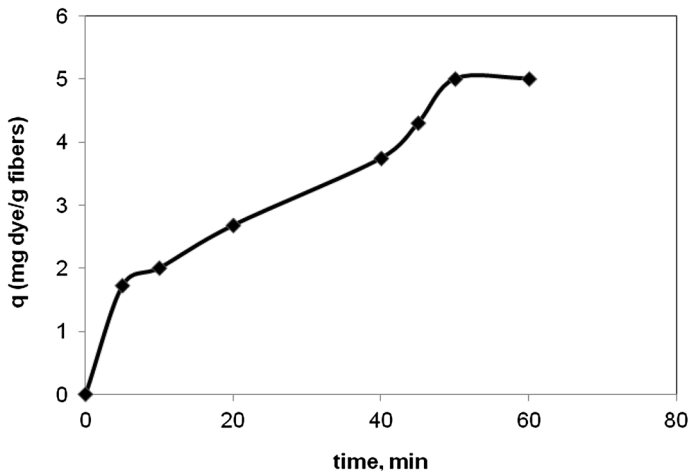
**Fig. 9** Effect of the pH on sorption: [dye] = 20 mg/L;  $T = 25\text{ }^{\circ}\text{C}$ ;  $t = 60\text{ min}$ ; % grafting = 150

In low acidic conditions, the CN of AN groups on the fibers are fully too protonated, hence the grafted fibers is inactive for adsorb of cationic dye. In basic conditions, there is negative charge on the surface the grafted fibers, hence adsorb of cationic dye rises. Interact between nitrile and cationic dye is known as Lewis acid–base interactions. The similar mechanism was suggested in our previous work [31]. This may be illustrated as in Fig. 10.

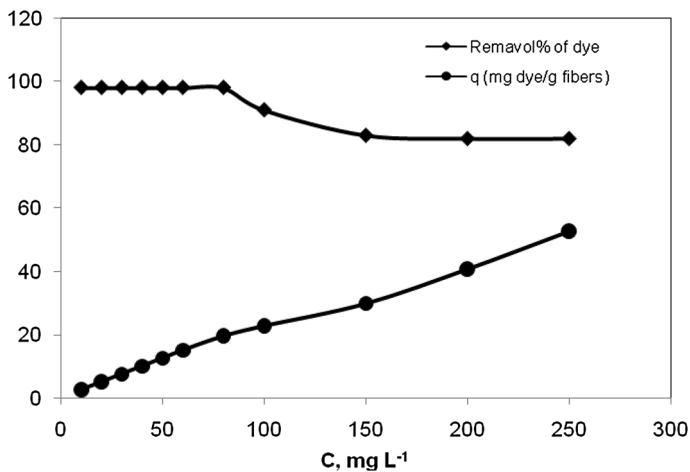
### Effect of % grafting and sorption time

Percent grafting plays a significant role on the adsorption capacity as shown Fig. 11. With raise in grafting yield from zero to 156 %, the amount of cationic dye





**Fig. 12** Adsorption time of cationic dye upon MPA/AN-g-PET fibers: [dye] = 20 mg/L; pH = 9;  $T = 25\text{ }^{\circ}\text{C}$ ; % graft = 155



**Fig. 13** Sorption of cationic dye with MPA/AN-g-PET fibers at different dye concentration: pH = 9;  $T = 25\text{ }^{\circ}\text{C}$ ;  $t = 60\text{ min}$ ; % graft yield = 155

dye when initial concentration varied from 10 to 80 mg L<sup>-1</sup> but after 80 ppm, the percentage removal of dye up down 82 %. The maximum adsorption performance for the cationic dye was achieved at 52.7 mg g<sup>-1</sup>. At lower initial concentrations, all cationic dyes present in the sorption environment could interact with the active functional groups upon the grafted PET fibers; therefore, dye removal percentage becomes higher. At higher concentrations, owing to the saturation of the active sites, the removal percentage of the dyes by the grafted fibers showed a decreasing trend. The most common active groups attached to the fibers is the carboxylate,  $-\text{COO}^-$

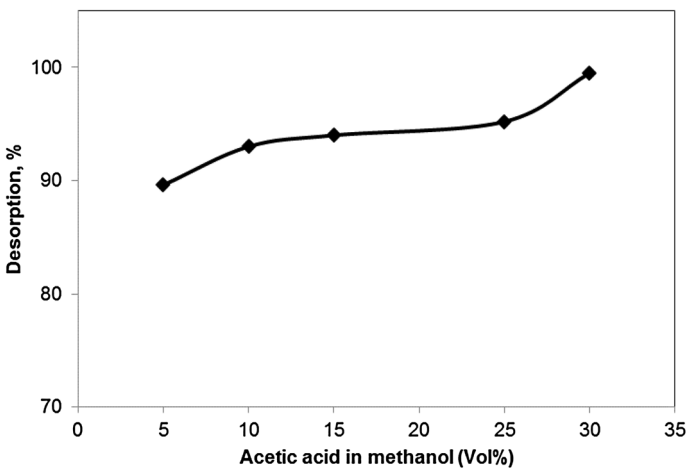
and nitrile,  $-\text{CN}$ . These sites have anionic property that is suitable for dyeing with cationic dyes. Thus, MPA/AN-*g*-PET fibers show very high adsorption capacity.

### Desorption studies

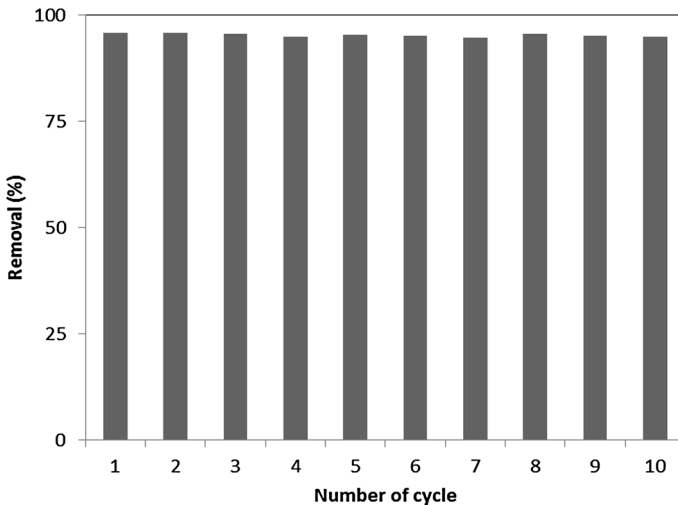
It is required for adsorbed cationic dye upon the grafted fibers to be eluted readily to use the grafted fibers repeatedly for regeneration of cationic dye. The adsorbed cationic dyes by adsorbent were recovered using the different acetic acid concentrations in methyl alcohol as shown in Fig. 14. At 30 °C, in 30 min., as the acetic acid concentrations in methyl alcohol raised from 5 to 30 %, the desorption % raised from 90 % to almost 100 % for MB adsorbed of 20 ppm. Desorption rate of dye from MPA-*g*-AN-*g*-PET fibers was elevated good and was fast. MPA/AN-*g*-PET fibers were eluted without losing their stability and activity. Thus, an adsorbent for the removal of cationic dyes should be effective.

### Reuse of grafted fibers

Reuse of the adsorbent is the most important property of the adsorbent in the industry as it is more economical. For reusability of the grafted fibers, acetic acid in methanol adjusted to different concentration values were added to the dye loaded MPA/AN-*g*-PET fibers (see Fig. 15). Adsorption–desorption of cationic dye was repeated ten times using dye concentration of 20 ppm and with the same grafted fiber adsorbent. Adsorption capacity of MPA/AN-PET fibers did not considerably decrease after ten adsorption–desorption cycles; thus, it can be used repeatedly for the sorption dyes.



**Fig. 14** Desorption profile of dye ion adsorbed PET fibers:  $T = 25\text{ }^{\circ}\text{C}$ ;  $t = 30\text{ min}$ ;  $[\text{dye}] = 20\text{ mg/L}$ , % graft yield = 155



**Fig. 15** Effect of number of cycle on the percent removal of cationic dye on MPA/AN-g-PET fibers

## Conclusion

MPA/AN-g-PET fibers were prepared as a new adsorbent and found to be effective in the sorption of cationic dye. Rate of graft polymerization of the MPA upon PET fibers raised with the presence of AN monomer owing to its synergistic. Water uptake and the removal of cationic dye increased with the increasing percent grafting. The sorption was found to be linked with parameters such as pH, concentration and time. The sorption of cationic dye depends on active groups as MPA and AN inserted in the PET fibers. MPA/AN-g-PET fibers removed 98 % of cationic dye when initial concentration diversified from 10 to 80 mg L<sup>-1</sup>. Cationic dye adsorbed upon MPA/AN-g-PET fibers was desorbed using the ethanoic acid in methyl alcohol and the adsorbent can be used repeatedly in the sorption–regeneration cycle without any loss and stability of the removal capacity. MPA/AN-g-PET fibers are considered to have potential application in industrial water for the sorption of cationic dye.

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