

Preparation and Application of Glycidyl Methacrylate and Methacrylic Acid Monomer Mixture-Grafted Poly(ethylene terephthalate) Fibers for Removal of Methylene Blue from Aqueous Solution

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ABSTRACT: A novel fibrous adsorbent that grafts glycidyl methacrylate (GMA) and methacrylic acid (MAA) monomer mixture onto poly(ethylene terephthalate) (PET) fibers was used for removal of methylene blue (MB) in aqueous solutions by a batch equilibration technique. The operation parameters investigated included, pH of solution, removal time, graft yield, dye concentration, and reaction temperature. The adsorption rate of MB is much higher on the MAA/GMA-grafted PET fibers than on the ungrafted PET fibers. MB was removed 99% the initial dye concentration at 10 mg L^{-1} and 93% at 200 mg L^{-1} by monomers mixture-grafted PET fibers. Pseudofirst order and pseudo-second order kinetic equations were used to examine the

experimental data of different graft yield. It was found that the pseudosecond order kinetic equation described the data of dye adsorption on fibrous adsorbent very well. The experimental isotherms data were analyzed using Langmuir and Freundlich isotherm models. The data was that Freundlich isotherm model fits the data very well for the dyes on the fibers adsorbent. The dye adsorbed was easily desorbed by treating with acetic acid/methanol mixture (50% V/V) at room temperature. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 119: 3034–3042, 2011

Key words: graft copolymerization; monomer mixture; poly(ethylene terephthalate) fibers; basic dye removal

INTRODUCTION

Basic dye contamination originated from various industries, such as dyeing, textile, cosmetics, leather, food processing, paper, and dye manufacturing are among the most important toxic pollutions in industrial wastewater and some are known to be human carcinogens.^{1,2} Removal of basic dyes from industrial wastewater before it can be discharged becomes environmentally important as well the presence of very low concentrations of dyes in effluent is highly visible and undesirable. The removal of basic dyes from industrial wastewater is a very difficult operation, as they are fairly stable to light, heat, and resist biodegradation due to their complex molecular structures.^{3–5} Hence, several technical procedures have been developed to remove dyes from the industrial wastewater. Various methods for removal of dyes in the literature have been reported, including sedimentation,⁶ filtration technology,⁷ chemical treatment,⁸ oxidation,⁹ electrochemical system,¹⁰ biological treatment,¹¹ and adsorption.¹² Among all the

aforementioned methods, adsorption process has been found to be an efficient and economic process to remove dyes.¹³ Adsorption techniques are widely used to remove dyes from wastewater, especially those that are not easily biodegradable. In adsorption process, the adsorbent plays the significant role. Several adsorbents such as alumina,¹⁴ silica gel,¹⁵ zeolites,¹⁶ activated carbon,¹⁷ rice husk,¹⁸ chitosan,¹⁹ activated clay,²⁰ and polymers²¹ has been used by many researches.

In recent years there has been an increasing interest in chelating fibers that have been used for removal of dyes,²² and heavy metals ions²³ from wastewater and enzymes immobilization.²⁴ The development of efficient chelating fibers for removal of toxic pollutants is environmental and economically important. Modified PET fibers are a preferred alternative due to their low cost, simple processing, variety of structural and surface properties, high chemical stability, high specific surface area and high adsorption capacity.²⁵ Chelating fibers consist of functional group such as $-\text{COOH}$, $-\text{C}_5\text{H}_5\text{N}$, $-\text{CONH}_2$, $-\text{NH}_2$, $-\text{COOR}$, and epoxy. The functional groups have important effects on the capacity, selectivity, and desorption of the fibers.²⁶ Poly(ethylene terephthalate) (PET) fibers are one of the most important and widely used synthetic fibers by many

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industries and has good resistance to weak mineral acids, even at boiling temperature, and to most strong acids at room temperature, oxidizing agents, sunlight, and micro organisms. However PET fibers do not contain chemically reactive groups, showing resistance to moisture, dye anions, or cations.²⁵ Certain desirable properties such as dye ability, water absorbency, and improvement in antistatic, mechanical, and thermal properties can be imparted to PET fiber by grafting with different vinyl monomers.^{25,27} Graft copolymerization of vinyl monomers from their binary mixtures is of special importance to obtain polymers having properties of both monomers in comparison to graft copolymers obtained by the grafting of individual monomers. The grafting from binary mixture of monomers has the advantage of introducing grafted chains with tailor made properties for specific applications.²⁷ Our previous studies have shown that PET fibers are good materials for the removal of different types of dyes and heavy metal ions.^{22,27,28}

In the present study, a novel fibrous adsorbent that grafts glycidyl methacrylate (GMA) and methacrylic acid (MAA) monomer mixture onto PET fibers was used for removal of basic dyes (methylene blue) in aqueous solutions by a batch equilibration technique.

EXPERIMENTAL

Materials

The PET fibers (122 dTex, middle drawing) used in these experiments were provided by SASA. (Adana, Turkey). The fiber samples were Soxhlet-extracted for 6 h with acetone and dried in a vacuum oven at ambient temperature. MAA and GMA were used as supplied. Bz₂O₂ (benzoyl peroxide), as initiator, was twice precipitated from chloroform in methanol and dried in a vacuum oven for 2 days. Other reagents were used as supplied. All reagents were Merck products.

Polymerization procedure

Polymerization was carried out in a thermostated 50 mL tubes under reflux. The mixture containing the PET fiber sample (0.3 ± 0.01 g), appropriate amount of MAA/GMA mixture and Bz₂O₂ at required concentration in 2 mL acetone was made up to 20 mL with deionized water. The mixture was immediately placed into the water bath adjusted to the polymerization temperature. At the end of the predetermined polymerization time, the grafted fibers were taken out. Residual solvent, monomers, freed from the poly(MAA), poly(GMA), and poly(MAA-GMA) were removed by Soxhlet-extracting the PET fibers in methanol for 24 h and after boiling water 12 h. The grafted fibers were then vacuum-dried at 50°C

for 72 h and weighed. The graft yield (GY) was calculated from the weight increase in grafted fibers as follows:

$$GY(\%) = [(w_g - w_i)/w_i] \times 100 \quad (1)$$

where w_i and w_g denote the weights of the original (ungrafted) and grafted PET fibers, respectively.

FTIR spectrum

Fourier transform infrared (FTIR) spectra of MAA/GMA-grafted PET fibers were recorded using a Thermo-Nicolet 6700 FTIR spectrometer attached to an attenuated total reflection (ATR) apparatus, using diamond prism with an incident angle 45°.

Scanning electron microscopy

SEM studies of the original and MAA/GMA-grafted PET fibers, coated with gold, and were performed using a JEOL Model JSM 5600 microscope.

Sorption of basic dye on the fibers

A volume of 25 cm³ of each basic dye solution (20 mg L⁻¹) was added onto 0.1 g of MAA/GMA-g-PET fibers in 50 mL Erlenmeyer. The contents were shaken at 130 rpm for a predetermined period of time at 25°C using orbital shaker (Medline BS 21). The loaded adsorbent was separated by filtration and washed gently. After the cationic dye concentration of supernatant was adjusted to 6.8 pH value, it was measured by using UV/Visible spectrophotometer ($\lambda = 665$ nm MB, Pharmacia Biotech Ultrospec 2000). Calibration curves were plotted between absorbance and concentration of the standard dyes solutions. The adsorption capacity of the poly MAA-g-PET fibers was evaluated by using the following expression:

$$Q = [(C_0 - C)V]/m \quad (2)$$

where Q is the amount of dye adsorbed onto unit mass of the MAA/GMA-g-PET fibers (mg g⁻¹), C_0 and C are the concentration of the dye in the initial solution and in the aqueous phase after adsorption treatment for a certain period of time (mg L⁻¹); V is the volume of the dye solution used (L); and m is the amount of MAA/GMA-g-PET fibers used (g).

Desorption of basic dye

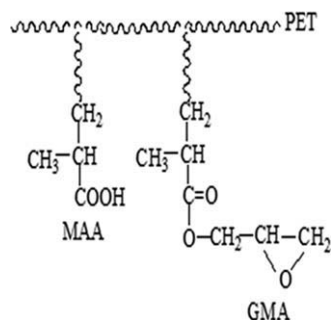
Desorption assays were carried out with the dye loaded MAA/GMA-g-PET fibers. The dye was recovered in 60 min by treating with 25 mL of acetic acid/methanol mixture (50% V/V) and 1M HCl, and then analyzed by the method mentioned above. Desorption percent was calculated using the following equations:

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

RESULTS AND DISCUSSION

PET fibers do not contain suitable functional groups that can interact with dye molecules. Thus dye ability of ungrafted PET fibers with basic dye is negligible. Hence, PET fibers were grafted with MAA and GMA monomer mixture using benzoyl peroxide as initiator in aqueous media. The polymer chains were grafted onto PET fibers. The side chains contained structural units coming from both MAA and GMA. Graft copolymerization of vinyl monomers from their binary mixtures is of special importance to obtain polymers having properties of both monomers in comparison to graft copolymers obtained by the grafting of individual monomers.²⁹ Grafting PET fibers with MAA/GMA monomer mixtures were used as a new fibrous adsorbent for removal of basic dye because it inserts into the fibers structure —COOH functional groups of MAA and epoxy, and ester functional groups of GMA, which can interact with basic dye. The structures of MAA/GMA-g-PET fibers are illustrated in Scheme 1.

The FTIR spectra of ungrafted, 115% MAA/GMA-g-PET, and basic dye loaded GMA-g-PET fiber is given in Figure 1. It is seen that the peaks of the ungrafted PET [Fig. 1(a)] can be assigned as follows: 2960 and 2880 cm^{-1} (C—H asymmetric and symmetric in C—H, CH_2 groups), 1711 cm^{-1} (C = O), 1402 cm^{-1} (CH_2). After the grafting with GMA, the spectrum of the GMA grafted PET fiber [Fig. 1(b)] changed. The characteristic peak at 961 cm^{-1} corresponds to the asymmetric stretching vibration of the epoxy group. Hence these results provided proof for the grafting of GMA onto PET fiber. After the loading of MB on GMA-g-PET fiber [Fig. 1(c)], a strong broad band appeared at 3309 cm^{-1} , attributed to the N—H stretching vibration. The new peak at 1543 cm^{-1} fits the N—H bending vibration. These peaks



Scheme 1 MAA/GMA monomer mixture grafted PET fibers.

have supported the existence of groups MB on GMA-g-PET fiber.^{30,31}

The scanning electron micrographs of MAA/GMA-grafted PET fiber (115%) are shown in Figure 2. It is clear from the SEM results that the ungrafted PET fiber surface [Fig. 2(a)] has a smooth and relatively homogeneous appearance. The grafted side chain GMA seems to form microphages attached to the PET back-bone and causes a heterogeneous appearance in the graft copolymer [Fig. 2(b)], showing proof of grafting.²⁵

Effect of pH

One of the most important parameters in the removal of dye by MAA/GMA-g-PET fibers is pH of the solutions, which affects the surface charge of the grafted fibers and basic dye. The effect of pH on the removal of basic dye was investigated between pH 4 and 11. The MAA/GMA-g-PET fibers were incubated for 60 min with aqueous MB solution adjusted to required pH values using buffer solutions (Britton Robinson). Figure 3 illustrates the effect of solution pH on the adsorbed amount of basic dye. As shown in Figure 3, the adsorbed amount of MB significantly increased by increasing pH value of the basic dye aqueous solution from 4 to 10 increases and it remained constant at pH 10–11. The following studies were conducted at a fixed pH of 10. A similar type of behavior is also reported for the adsorption of basic dye using different adsorbents by another worker.³²

To explain the observed behavior of basic dye adsorption with varying pH, it is necessary to examine various mechanisms such as electrostatic interaction and chemical reaction which are responsible for adsorption on fibers surface. At different pH values, the protonation and deprotonation would influence the surface structure of the MAA/GMA-g-PET fibers, and dye ions would exist in different forms. As the pH is lowered, however, the surface of grafted PET fibers become positive, which prevent the approach of cationic dye. Grafted PET fibers are negatively charged at $\text{pH} \geq 5$ since the —COOH groups of MAA and epoxy, ester groups of GMA can deprotonate and negative surface charge. The surfaces of fibers are increasingly negative with increasing pH. At pH 10, a significantly high electrostatic attraction exists between the negatively charged surface of the fibers and cationic dye. The similar mechanism was suggested in our previous work.³³

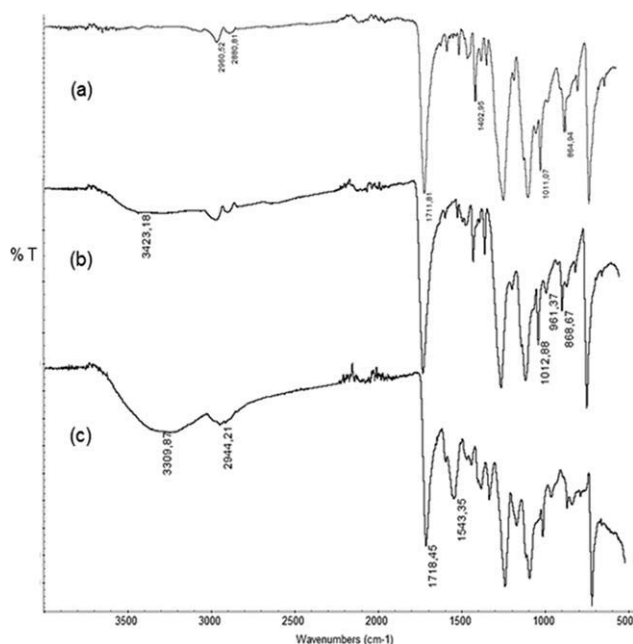


Figure 1 FTIR spectra of: (a) ungrafted, (b) 115% (50/50 mol) MAA/GMA monomers mixture grafted, and (c) basic dye loaded MAA/GMA-grafted PET fibers.

Effect of graft yield and contact time

The effect of the graft yield on the adsorbed amount of cationic dye was investigated, while keeping all other conditions constant and illustrated in Figure 4. The amount of cationic dye increased significantly by increasing the graft yield up to 70%, and then falls down upon further increase in graft yield. PET fibers which are 70% grafted have a higher adsorption capacity than the ones grafted other than 70%. Ungrafted PET fibers do not contain suitable functional groups and thus cannot interact with basic dye. Increasing graft yield increases the number of functional groups thus MAA/GMA-*g*-PET fibers have many functional groups such as carboxyl, epoxy, and ester that can increase the electrostatic interaction between the cationic groups of basic dye and the groups of MAA/GMA-*g*-PET fibers. The increase in the adsorption of basic dye on the fibers with increasing graft yield may be attributed to a higher surface area and more active sites. The results showed that the amount of basic dye is greater at smaller graft yield. This can be explained by sterical and diffusion barrier impediment at higher graft yield. As shown in Figure 4, the adsorption takes place rapidly at first, then slows down and levels off. The similar types of curves were observed in the literature.³⁴ The adsorption equilibrium was attained within 60 min. The relation between the nature of the polymer and sorption rate is generally complicated by many possible interactions on the surface. Generally, the electrostatic interaction, surface bind-

ing, and chemical reaction may be identified as the major adsorption mechanisms.

The Lagergren equation, a pseudofirst-order equation, describes the kinetics of adsorption process as follows.³⁵

$$\log(Q_e - Q_t) = \log Q_e - \left(\frac{k_1}{2.303} \right) t \quad (4)$$

where Q_t is the amount of dye adsorbed (mg g^{-1}) at any time, Q_e is the amount of dye adsorbed at equilibrium time per gram of adsorbent and k_1 is the rate constant (min^{-1}). According to the adsorption equation, the experimental results of Figure 4 can be converted into the plots of $\log(Q_e - Q_t)$ versus t (not

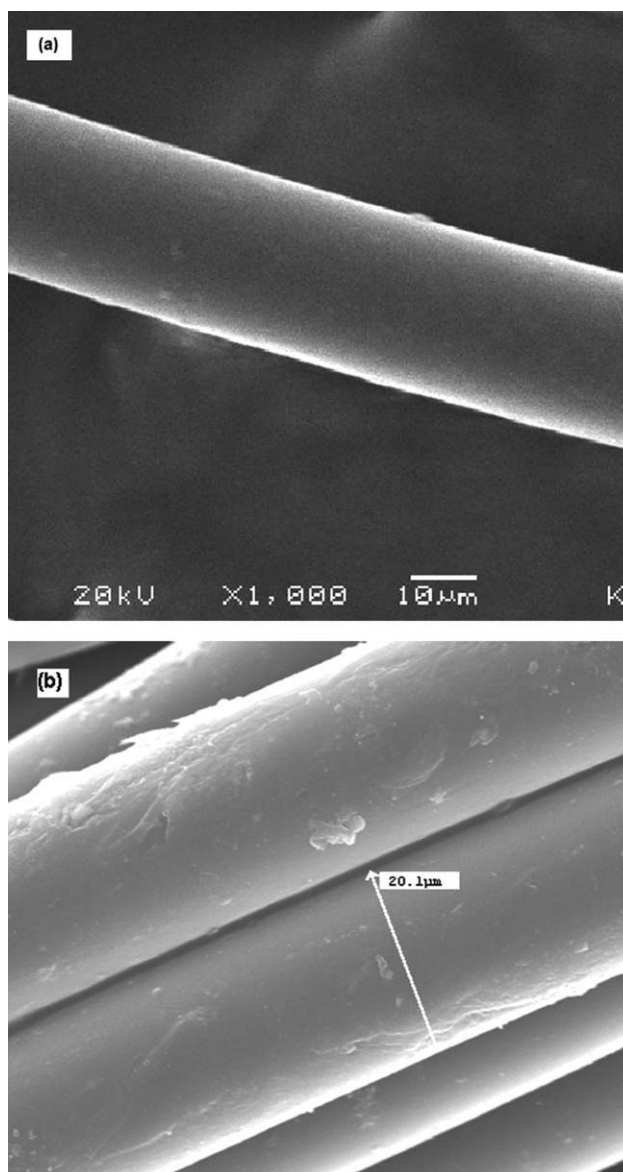


Figure 2 (a) SEM micrograph of ungrafted PET fibers, (b) SEM micrograph of MAA/GMA (115%, 50/50 mol) grafted PET fibers.

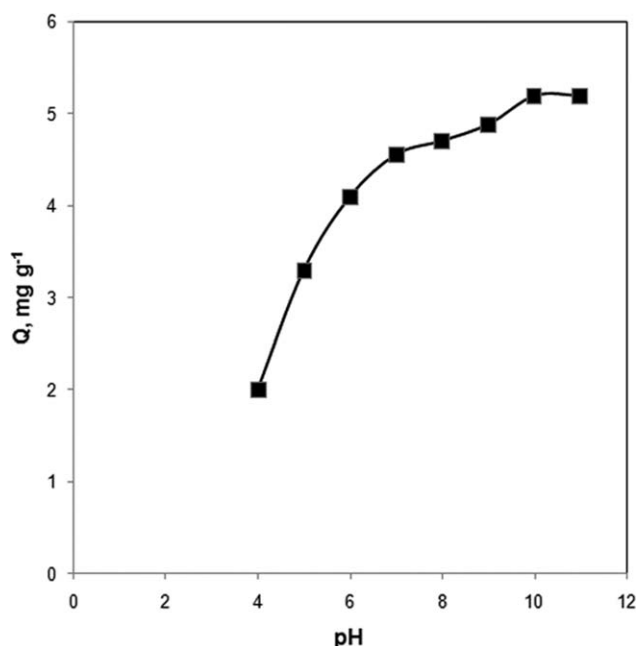


Figure 3 The pH dependence of basic dye adsorbed by MAA/GMA grafted PET fibers: Dye concentration: 20 ppm; temperature: 25°C; contact time: 60 min; graft yield: 70%.

shown Figure). Values of k_1 and Q_e were calculated from the linear plot of $\log(Q_e - Q_t)$ versus t . The experimental Q_e values do not agree with the calculated one, as shown in Table I. This shows that the adsorption of basic dye onto MAA/GMA-g-PET fiber is not a first order reaction.

The second-order kinetic model is expressed as:

$$\frac{t}{Q_t} = \left(\frac{1}{k_2 Q_e^2} \right) + \frac{t}{Q_e} \quad (5)$$

where k_2 ($\text{g min}^{-1} \text{mg}^{-1}$) is the rate constant of second-order adsorption.³⁶ If second order kinetic is applicable, the plot of t/Q^{-1} versus t should show a linear relationship. There is no need to know any parameter beforehand and the equilibrium adsorption capacity; Q_e can be calculated from eq. (5). Also, it is more likely to predict the behavior over the whole range of adsorption.³⁷ k_2 and Q_e were calcu-

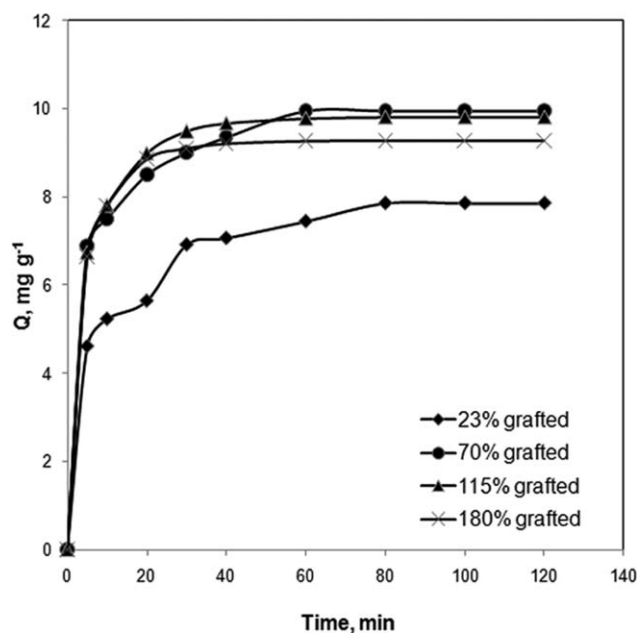


Figure 4 Relationship between adsorption time and adsorbed amount of basic dye with MAA/GMA-grafted PET fibers: Dye concentration: 40 ppm; pH: 10; temperature: 25°C.

lated from the intercept and slope of the plot of t/Q^{-1} versus t . The linear plot of t/Q^{-1} versus t (Fig. 5) shows a good agreement between experimental and calculated Q_e value (Table I). The correlation coefficients for the second order kinetic model are 0.99. These show that the adsorption of MB onto MAA/GMA-grafted PET fiber is a second order reaction. This suggest that the adsorption of MB onto grafted PET fibers is presumably a chemisorption process involving exchange and sharing of electrons mainly between dye cations and functional groups of the grafted PET fibers.³⁸ Similar phenomena are observed in the adsorption of basic dye on low-cost adsorbent.³⁹

Effect of initial dye concentration

The effect of initial MB concentration on the adsorption capacity by MAA/GMA-grafted PET fibers was

TABLE I
First-Order and Second-Order Rate Constants

Graft Yield	Q_e (exp.) (mg g^{-1})	First-order rate constants			Second-order rate constants		
		Q_e (theor.) (mg g^{-1})	k_1 (min^{-1})	R^2	Q_e (theor.) (mg g^{-1})	k_2 ($\text{g mg}^{-1} \text{min}^{-1}$)	R^2
23%	7.85	3.1	0.0179	0.94	8.1	0.022	0.99
70%	9.95	3.8	0.0465	0.99	10.4	0.025	0.99
115%	9.81	4.8	0.0891	0.99	10.2	0.036	0.99
180%	9.28	4.1	0.1036	0.99	9.6	0.051	0.99

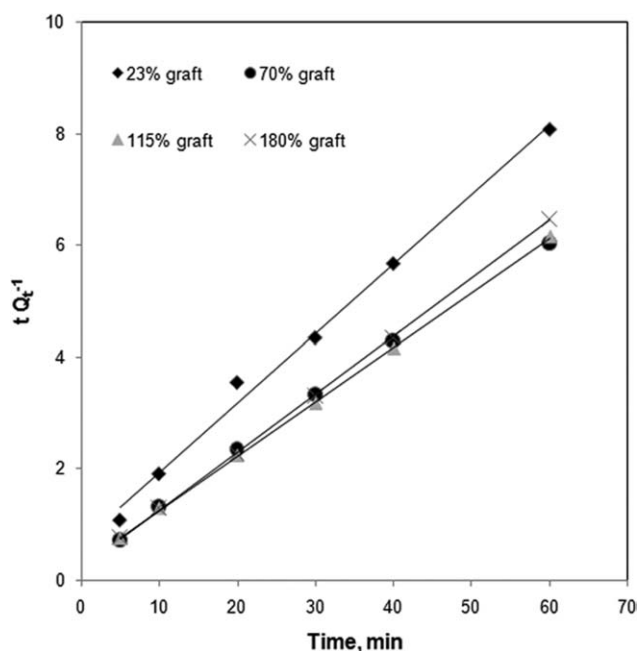


Figure 5 Pseudosecond order plots for basic dye on MAA/GMA grafted PET fibers.

systematically investigated by varying the initial concentration between 20 and 200 mg L⁻¹. Figure 6 shows the percent removal and adsorbed amount of cationic dye as a function of initial concentration at pH 10.0. It was observed that MAA/GMA-grafted PET fibers reduced from 100 to 93% when initial MB concentration varied from 10 to 200 mg L⁻¹ at pH 10.0. The adsorption increased with increasing initial dye concentration. The maximum adsorption performance for MB was achieved at 45 mg g⁻¹ using 200 mg L⁻¹ dye solution. There are reported values and some data concerning the adsorption capacities of basic dye in the literature.⁴⁰ It has been recognized that the adsorption capacity of MAA/GMA-grafted PET fibers is in good comparison with the ones in the literature; thus it seems that the grafted PET fibers would be an interesting alternative and economical industrial adsorbent.

Adsorption isotherm

The relationship between the amount of basic dye adsorbed and the MB concentration remaining in solution is described by an isotherm. The two most

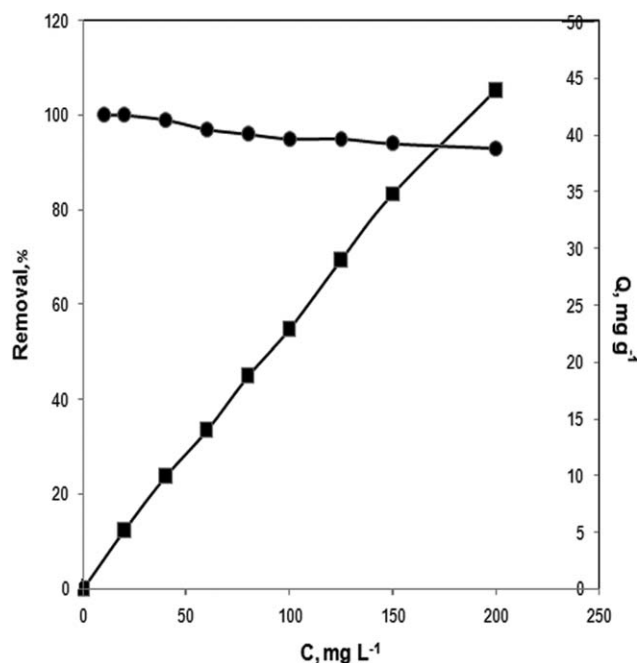


Figure 6 Effect of initial concentration of basic dye on adsorption: pH: 10; temperature: 25°C; contact time: 60 min; graft yield: 70%.

common isotherm types for describing this type of system are the Langmuir and Freundlich.^{41,42}

The Langmuir isotherm is given as:

$$Q_e = \frac{Q_o b C_e}{1 + b C_e} \quad (6)$$

The constant Q_o and b are characteristics of the Langmuir equation and can be determined from a linearized form of eq. (7).

$$\text{Langmuir: } \frac{C_e}{Q_e} = \frac{1}{Q_o b} + \frac{C_e}{Q_o} \quad (7)$$

where C_e is the concentration of basic dye (mg L⁻¹) at equilibrium, Q_o the monolayer capacity of adsorbent (mg g⁻¹), Q_e is the amount of adsorption at equilibrium and b Langmuir adsorption constant (L mg⁻¹). Thus a plot of C_e/Q_e versus C_e should yield a straight line having a slope of Q_o^{-1} and intercept of $(Q_o b)^{-1}$. Therefore, the relevant experimental data were treated and it was observed that the relationship between C_e/Q_e and C_e is linear,

TABLE II
Langmuir and Freundlich Isotherm Constants

Graft Yield	Langmuir isotherm parameters			Freundlich isotherm parameters		
	Q_o (mg g ⁻¹)	b (L mg ⁻¹)	R^2	K_F (mg g ⁻¹)	n	R^2
70%	52.1	0.25	0.88	11.5	2.0	0.98

indicating that the adsorption behavior follows the Langmuir adsorption isotherms. The b and Q_o values are presented in Table II.

The Freundlich isotherm is derived to model the multilayer adsorption. The Freundlich model is formulated as:

$$Q_e = K_F C_e^{1/n} \quad (8)$$

The equation may be linearized by taking the logarithm of both sides of eq. (9) and linear form of Freundlich isotherm can be given as:

$$\log Q_e = \log K_F + \frac{1}{n} C_e \quad (9)$$

where C_e is equilibrium concentration (mg L^{-1}), K_F the sorption capacity (mg g^{-1}) and n is an empirical parameter. Thus a plot of $\log Q_e$ versus $\log C_e$ should give straight line having a slope of $1/n$ and intercept of $\log K_F$ (Fig. 7). The K_F and n values are presented in Table II.

On the comparison of the R^2 values given in Table II, I can conclude that in all cases for the adsorption of target basic dye to the MAA/GMA-grafted PET fibers, Freundlich equation represents a better fit to the experimental data than the Langmuir equation. The Freundlich isotherm was used to describe heterogeneous systems and reversible adsorption [see Fig. 2(b)].

Effects of temperature

Temperature has important effect on the adsorption process. The adsorption of basic dye from an aque-

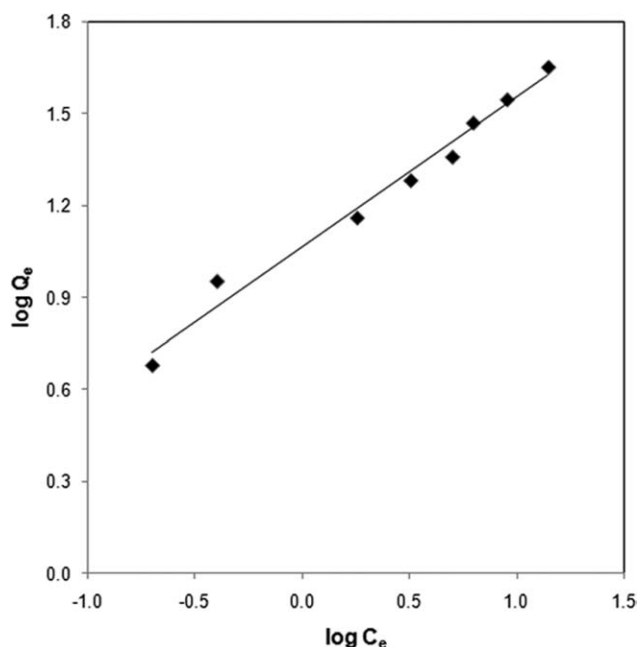


Figure 7 Freundlich plot of basic dye adsorption on MAA/GMA grafted PET fibers. pH: 10; temperature: 25°C; contact time: 60 min; graft yield: 70%.

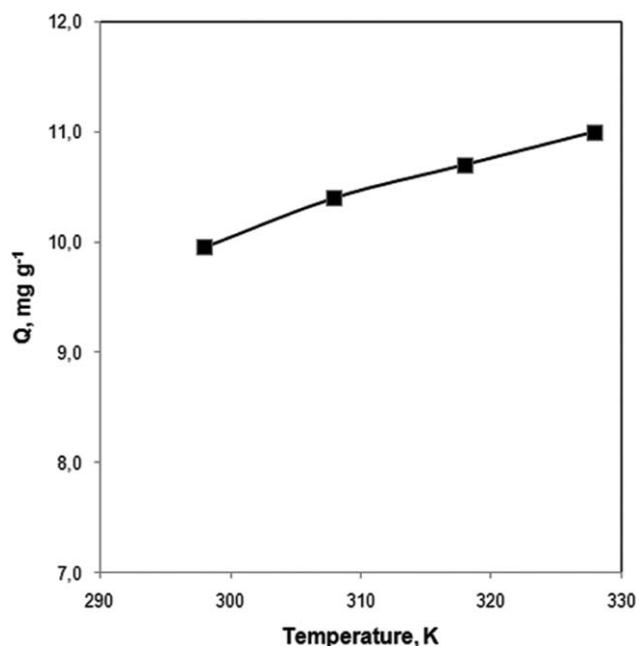


Figure 8 Effect of temperature on the adsorption amount of basic dye: Dye concentration: 40 ppm; pH: 10; contact time: 30 min; graft yield: 70%.

ous solution on MAA/GMA-grafted PET fiber was studied as a function of temperature. The results are presented in Figure 8. There is an increase in adsorption with rise in the temperature. This increase in the adsorption amount of basic dye could be attributed to cause an increase in the degree of swelling, so that the diffusion of the dye becomes much easier into the fibers.

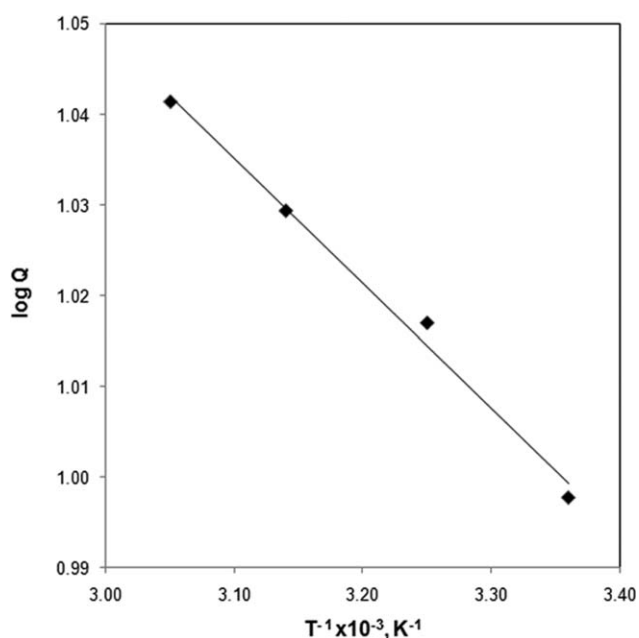


Figure 9 $\log Q_e$ versus to T^{-1} of basic dye. Dye concentration: 40 ppm; pH: 10; contact time: 30 min; graft yield: 70%.

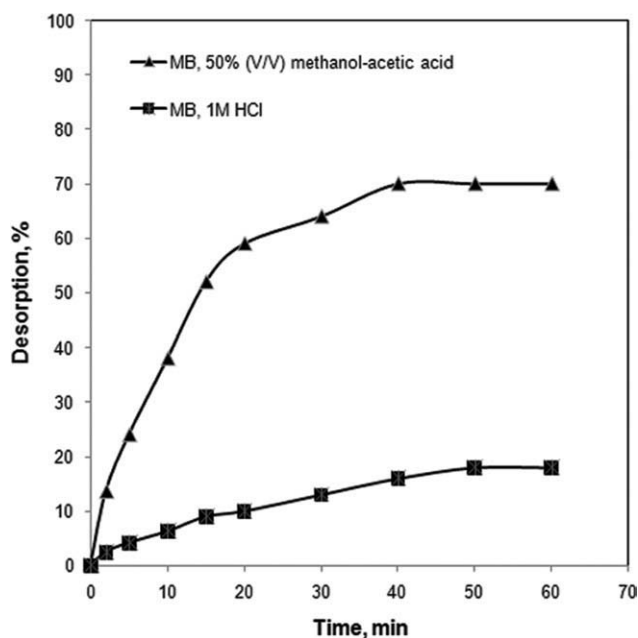


Figure 10 Desorption profile of basic dye adsorbed PET fibers: Graft yield: 70%; dye concentration: 50 ppm; temperature: 25°C.

Dye concentration: 40 ppm; pH: 10; contact time: 30 min; graft yield: 70%.

Figure 9 was obtained using the data of Figure 8 and from the Arrhenius plot of $\log Q$ versus T^{-1} . From the slopes of Figure 10, the activation energies for adsorption were calculated as 3.4 kJ mol^{-1} for MB. These low values of E_a , compared to those of typical chemical reaction of $65\text{--}250 \text{ kJ mol}^{-1}$, imply that the adsorption of grafted fibers for the basic dye is facile procedures.⁴³ As it is reflected from the positive value of the activation energies for adsorption, the adsorption process was endothermic in nature.

Desorption studies

Desorption were studied to regenerate basic dye adsorbed of MAA/GMA-grafted PET fibers. Acetic acid/methanol mixture (50%, V/V) and 1M HCl solutions were used for desorption of MB. Figure 10 shows the desorption performances and kinetics of cationic dye adsorbed grafted fibers. The desorption of the MB adsorbed MAA/GMA-grafted PET fibers was fast in the acetic acid/methanol mixture and it took 40 min to reach 70% of the desorption, on the other hand about 18% after 60 min of desorption in the 1M HCl solution. This is also substantiated by the significant adsorption of basic dyes in the alkaline pH though electrostatic attraction favors the adsorption. The investigation in the desorption studies confirm the mechanism of adsorption stated in the pH effect. It has been recognized that MAA/

GMA-g-PET fiber is stable and regenerable by acetic acid in methanol. Therefore, adsorption process should be effective for the removal of basic dyes from industrial wastewater.

CONCLUSIONS

A novel fibrous adsorbent that grafting glycidyl methacrylate (GMA) and methacrylic acid (MAA) monomer mixture onto poly (ethylene terephthalate) (PET) fibers was used for removal of methylene blue (MB) in aqueous solutions by a batch equilibration technique. The following conclusions have been obtained: adsorption process was affected by the graft yield. It was observed that pH is the most important parameter and pH 10.0 was found as the optimum pH value in the process. A 60 min of treatment time was found to be sufficient to reach the adsorption equilibrium value. MB was removed 99% the initial dye concentration at 10 mg L^{-1} and 93% at 200 mg L^{-1} by monomers mixture grafted PET fibers. The kinetics of basic dye adsorption onto MAA/GMA-grafted PET fibers followed the pseudo-second order model. A Freundlich type of adsorption was observed. The dye adsorbed was easily desorbed by treating with acetic acid/methanol mixture (50% V/V). It was recognized that MAA/GMA-grafted PET fibers should be used as adsorbent for basic dyes from aqueous medium and addressed for other cationic dyes. In the future, it could be applied in wastewater treatments, especially of textile effluents.

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