

Elimination of Methyl Orange Using a Novel Anionic Adsorbent Prepared by Radiation Grafting Followed by Chemical Treatment

Nazia Rahman^{1*}, Sadoara Sadika², Mahbub Kabir², Shahnaz Sultana¹, Md. Nabul Sardar¹, Md. Homayon Kabir¹

¹Nuclear and Radiation Chemistry Division, Institute of Nuclear Science and Technology, Atomic Energy Research Establishment, Bangladesh Atomic Energy Commission, Dhaka, Bangladesh

²Department of Chemistry, Jahangirnagar University, Savar, Dhaka-1342, Bangladesh

*Corresponding author: Nazia Rahman, Nuclear and Radiation Chemistry Division, Institute of Nuclear Science and Technology, Atomic Energy Research Establishment, Bangladesh Atomic Energy Commission, Dhaka, Bangladesh. E-mail: naziabaec@gmail.com

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Abstract

In the present research work, γ -ray initiated grafting of Glycidyl Methacrylate (GMA) on non-woven polyethylene fabrics was followed by amine functionalization. Dependence of amination yield on degree of grafting was investigated. Extensive examinations on the adsorption of anionic dye Methyl Orange (MO) from aqueous solution by the functionalized GMA-g-non woven PE fabrics were conducted. Influence of variation of adsorption environment likely contact time, temperature, pH and initial metal ion concentration on MO adsorption aptitude was experimented. The optimal condition that provided maximum MO adsorption was identified to be contact time 48 h, initial metal concentration 500 ppm, pH 1.759 and temperature 80°C. To understand the equilibrium between MO and the adsorbent system Langmuir isotherm model was considered. Good fitting with Langmuir isotherm model suggested that MO adsorption was restricted to one molecular layer and the highest monolayer adsorption capacity calculated was 60.60 mg/g. The adsorption kinetics was analyzed using pseudo-first-order and pseudo-second-order models. The adsorption of MO was successfully inferred with pseudo-second-order model. The bonding between different groups and the surface morphology of the adsorbent was explored by FTIR and SEM Analysis. Successful desorption of MO followed by reuse of the regenerated adsorbent fabric revealed the future prospect of the reusable adsorbent.

Keywords: Radiation grafting, Glycidyl methacrylate, Adsorption, Methyl orange, Adsorption isotherm.

Introduction

Besides textile, dyeing and printing industries, cosmetics, food colouring and papermaking industries are also responsible for discharging enormous amounts of coloured effluents in the wastewaters [1]. The quantity of dyes existing in market is over 10,000 and the volume of various kinds of dyes manufactured worldwide per year is approximately 7×10^5 tons [2]. Approximately 12% of the amount of dyes manufactured yearly goes into surrounding environment from textiles and other related industries as effluents [3]. Release of dyes into surrounding water bodies even in minor quantities can impose a massive adversative influence on aquatic life and the food cycle. Dyes may cause allergic responses, dermatitis and skin inflammation. Some dyes are even have potential to cause cancer and capable of causing mutation in aquatic organisms and human beings [4].

Then again, most of the dyes are not degradable by sunlight or microorganism. [5-7]. Hence, elimination of dye from waste water through appropriate treatment is of utmost priority. So far, several treatment procedures for instance membrane separation coagulation and electro-coagulation, oxidation, ozonation and adsorption have been practiced to treat dye waste water. Among these methods, adsorption process has been established as a competent, cost effective, reasonably simple and practically

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advantageous method for eliminating dyes and other pollutants [8-14]. Several varieties of adsorbents have been produced and employed for the exclusion of dyes from waste water. Most noticeable shortcomings of many adsorbents are lower dye adsorption capacity than desired and secondary pollution by the adsorbent itself due to nominal desorption of dyes [15].

In recent times, application of graft polymers in the dye elimination has created a new dimension. These adsorbents possess higher adsorption capability and recycling of the adsorbent after regeneration lessens the possibility of secondary pollution. Due to the covalent bonding of chosen functional monomers to the backbone of polymer chain graft polymers attains desired functional property while keeping the good mechanical performance of the parent polymer [16-17]. Grafting reaction can be initiated by ionizing radiation, plasma treatment, using chemical initiators, by oxidation methods. Among the techniques used for the initiation of grafting reaction, radiation induced grafting technique is unique because ionizing radiation penetrates extensively into the polymer matrix resulting into instant and consistent formation of radicals [18].

In present research, our intention was preparation of a radiation grafted adsorbent for methyl orange removal. Several cases of preparation of adsorbents for elimination of anionic dye are reported. Research on application of polyethylenimine nanofibrous adsorbent for highly proficient removal of anionic dyes from aqueous solution has been carried out [19]. Adsorptive removal of Methyl Orange (MO) from waste water has been examined employing modified local clay (Kaolinite) [20]. Aminated pumpkin seed powder, nanostructured proton-containing δ -MnO₂ nanoparticles of a synthetic zeolite NaA/CuO, activated carbon prepared from lignin hydrothermal synthesized Mg–Al layered double hydroxide and chitosan/diatomite composite have been investigated to adsorb methyl orange from aqueous solution [21-22]. The objective of present study was fabrication of a radiation initiated GMA grafted adsorbent for adsorptive elimination of Methyl Orange (MO). Various adsorption atmosphere were produced through varying contact time, pH and initial metal ion concentration. Kinetic data of MO adsorption got from experiments was analyzed using pseudo 1st order and pseudo 2nd order kinetic equation. Excellent fitting of experimental data with the Langmuir isotherm model represented an important feature of the MO adsorption process. The study was further extended to evaluate desorption of MO and reuse of regenerated adsorbent [23-26].

Experimental

Materials and Reagents

As backbone polymer, non-woven Polyethylene (PE) fabric that was applied in this work was delivered by Kurashiki MFG Co, Japan. Sigma Aldrich Co. (USA) supplied Glycidyl Methacrylate (GMA) and also Tween 20 (Polyoxyethylene sorbitan monolaurate). Triethylamine utilized in amination of the epoxy ring of GMA (Glycidyl Methacrylate) was provided Fluka Chemika. Sodium Hydroxide and Hydrochloric acid was purchased from Merck, Germany. Methyl orange, the adsorbate of present study was collected from Sigma Aldrich Company.

Instrument and Apparatus

The reactive sites for grafting reaction onto non-woven polyethylene fabric was initiated by the Co-60 gamma-irradiator (Institute of Food And Radiation Biology (IFRB) of Atomic Energy Research Establishment (AERE), Savar, Dhaka). The pristine non-woven PE fabrics, grafted PE fabrics, amine functionalized grafted PE fabrics were effectively examined by FTIR (ATR) spectrophotometer (IR Prestige 21 provided by Shimadzu Corporation, Kyoto, Japan) to know the new bonds produced after grafting between the various groups of polymer and monomer. The analysis was carried out between wavelength range 700-4000 cm⁻¹. The change of surface morphology after grafting was detected through Scanning Electron Microscopy (SEM), the instrument used was Model JSM-6490LA, JEOL. The analysis was carried out at accelerating voltage of 20 kV.

Preparation of the adsorbent film

Preparation of non-woven polyethylene film

The non-oven polyethylene fabrics were cut into pieces of 10 cm length and 2 cm width. Then the Non-woven polyethylene fabrics were weighted and kept in polyethylene packets.

Grafting process

The non-woven PE fabrics after weighting were subjected to irradiation by the help of a Co-60 gamma radiation source. The total irradiation dose was 30 kg and irradiation was carried out at ambient temperature. After irradiation PE films were conserved in dry ice in order to maintain the free radicals. The monomer solution consists of 5% glycidyl methacrylate, 0.5% Tween-20 (as emulsifier) in distilled water. After stirring the emulsion solution for an hour, the solution was transferred into a gas passing jar. Argon gas was flown through the monomer solution for an hour for the exclusion of dissolved oxygen. Pieces

of nonwoven PE fabrics irradiated before were taken in a glass tube and instantly the de-aerated monomer solution was transferred into the tube. After pouring the monomer solution into the tube, it was securely closed with a cap. Finally, in a water bath, the grafting reaction was performed at 80°C for 4 h. Then the homo-polymers left on the surface of grafted non-woven PE fabrics were washed using the appropriate solvent. Then the grafted samples were weighed after drying.

The degree of grafting was evaluated by the subsequent equation:

$$Dg = (Wa - Wb) / Wb \times 100 \quad (1)$$

Where,

Wa=Measured weight of the PE fabric after grafting (g)

Wb=Measured weight of the PE fabric before grafting (g)

Dg=Degree of grafting of GMA (%)

Functionalization of GMA grafted non-woven PE film

To bring the presence of amine groups onto the grafted PE fabrics, the fabrics were dipped into a beaker containing 50% triethylamine in water and heated at 80°C for 6 h with continuous stirring. Amination takes place through the ring opening reaction of epoxy group of GMA. The aminated GMA-g- non woven PE films were washed thoroughly with distilled water. After washing, the fabrics were dried in an oven.

The amination yield was analyzed by the subsequent equation:

$$\text{Amination yield (\%)} = 100[\{(ma - mg) \div 101.19\} \div \{(mg - mo) \div 142.15\}] \quad (2)$$

Where,

mo=Measured weight of the PE fabric (g)

mg=Measured weight of the GMA grafted PE fabric (g)

ma=Measured weight of the aminated fabric (g)

Here, 101.19 and 142.15 represents the molecular weight of triethylamine and glycidylmethacrylate respectively.

Treatment of Aminated film with HCl

The aminated GMA-g-non woven PE fabrics were immersed in 1M solutions of HCl acid for several minutes to increase adsorption capability.

Methyl orange (MO) adsorption by aminated PE fabric

The aminated PE films were placed into 50 ml aqueous solutions of MO at room temperature (25°C). Influence of variation of adsorption environment likely contact time, temperature, pH and initial metal ion concentration on MO adsorption aptitude was experimented. pH control of the solutions was conducted with the help of HCl and NaOH solution. The MO concentrations of the solutions before and after the adsorption progression were detected using a UV spectrophotometer. The MO uptake ability of the film was analyzed using the subsequent equation:

$$Q = V(C_1 - C_2) / W \quad (3)$$

Where, Q represents the adsorption ability (mg/g of adsorbent), W represents the weight of the aminated PE fabric (g), V the volume of solution (L), and C₁ and C₂ are the MO concentrations (mg/L) prior and afterward the adsorption respectively FIG.1.

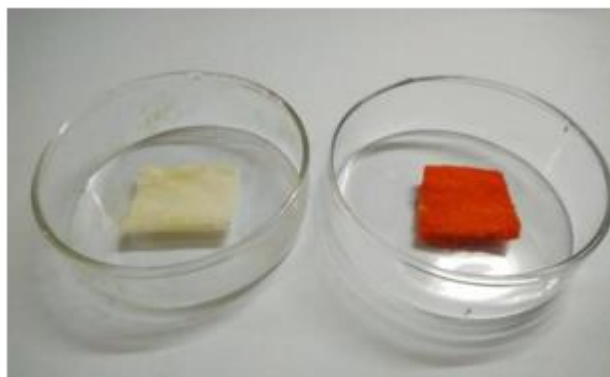


FIG.1. Aminated adsorbent before and after MO; Adsorption

Desorption of MO

The desorption of MO from the adsorbent fabrics was achieved by treating them with 2M aqueous NaOH for 24 h. The desorption proportion was computed using the subsequent equation:

$$\text{Percent desorption} = \left(\text{Ions desorbed (mg)} / \text{Ions adsorbed (mg)} \right) \times 100 \quad (4)$$

Results and Discussion

Preparation of the adsorbent fabric

Grafting of GMA on PE fabrics was performed using gamma radiation with total dose rate 30 kg. The grafted fabrics were functionalized through amination **FIG. 2**, demonstrates the influence of degree of grafting on amination yield. The amination yield exhibited upward trend up to certain percentage of grafting after which it drops. This happened because of the lessening of the accessible epoxy groups with the increased crowding between graft chains.

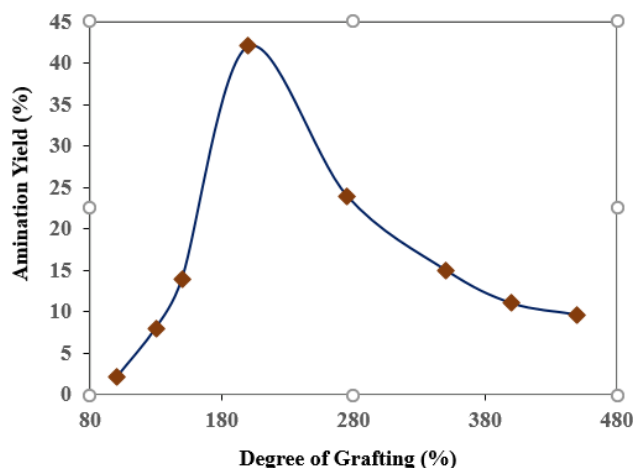


FIG. 2. Effect of degree of grafting % on amination yield.

Functionalization of GMA-g-non woven PE film was carried out with triethylamine solution. Aminated GMA-g- non woven PE film contains quaternary ammonium cation. Again MO exist as $C_{14}H_{14}N_3SO_3^-$ ion in aqueous solution. Positively charged quaternary ammonium pulls the $C_{14}H_{14}N_3SO_3^-$ ions from the aqueous solution and thus the adsorption of MO has occurred. When the aminated GMA-g-non woven PE film were treated with HCl solution, the positive H^+ ion increased the positivity on N atom which in turn enhanced the MO adsorption amount.

Characterization of adsorbent by FTIR

The IR spectrums of PE, GMA-g-PE, aminated GMA-g-PE fabric are shown in **FIG. 3a**, **3b** and **3c** respectively. The distinctive structures of the IR spectra of PE fabric are because of its C-H stretching and bending vibration. **FIG. 3A**, shows sharp peaks for C-H asymmetric and symmetric stretching at 2912 and 2846 cm^{-1} and C-H bending vibration at 1465 cm^{-1} .

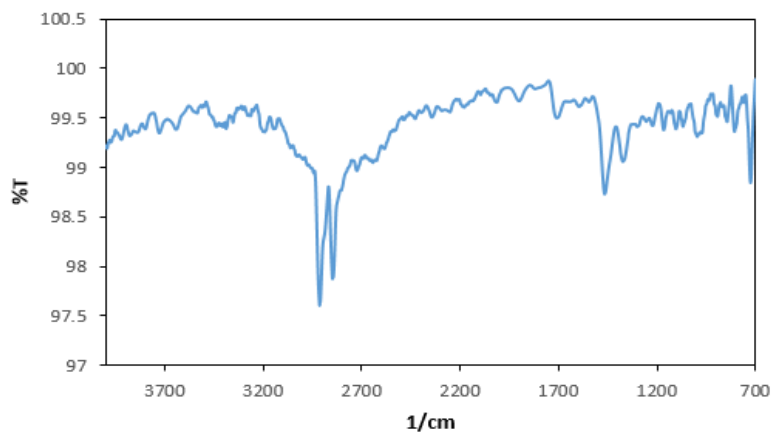


FIG. 3a. IR spectrum of PE fabric.

The FTIR spectrum shown in **FIG. 3B**, depicts indication for the grafting of GMA. In figure 3b, the peak for the stretching vibration of C=O group at 1714 cm^{-1} and the peaks for the symmetric and asymmetric vibration with CO at 1271 cm^{-1} and 1147 cm^{-1} provides proof of GMA grafting. The distinctive absorption peaks of the epoxy chain observed at 854 cm^{-1} and 912 cm^{-1} further supports the GMA grafting.

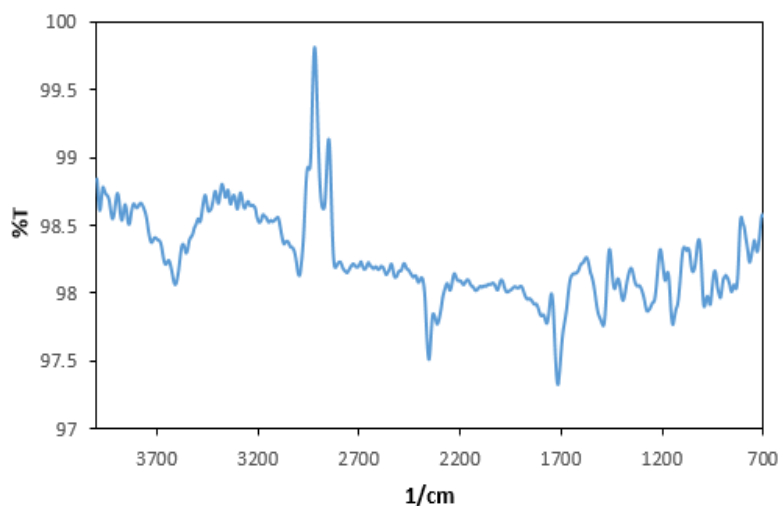


FIG.3b. IR spectrum of GMA grafted non-woven PE fabric.

The FTIR spectrum shown in **FIG. 3C**, supports successful amination of GMA grafted non-woven PE film. Since, the distinctive absorption peaks of epoxy chain at 912 cm^{-1} and 854 cm^{-1} are vanished in figure 3c due to the reaction between epoxy group and trimethylamine leading to ring opening.

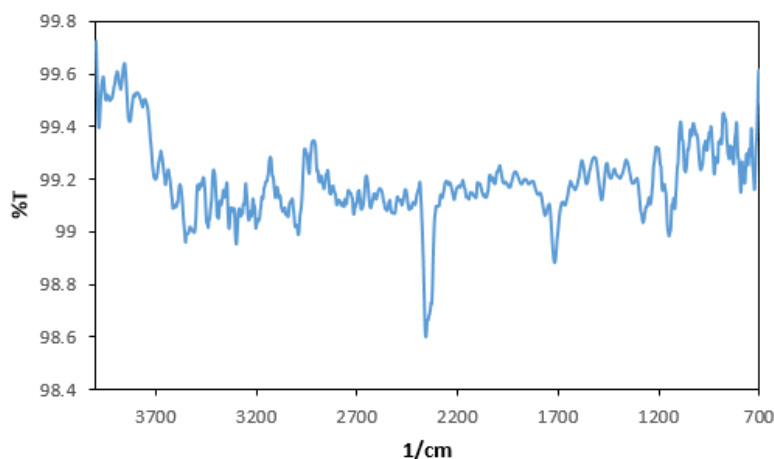


FIG. 3c. IR spectrum of aminated GMA grafted non-woven PE fabric.

SEM analysis

Non-woven polyethylene fabrics were analyzed by SEM to compare the surface morphology of nonwoven PE, grafted PE and aminated GMA grafted PE fabric. The SEM image of non-woven PE, grafted PE and aminated GMA grafted PE are shown in **FIG. 4A, 4B** and **4C** respectively. The SEM image of grafted sample clearly show new grafted and cross linked chains confirming grafting. The SEM image of aminated sample show change in physical appearance indicating further interaction

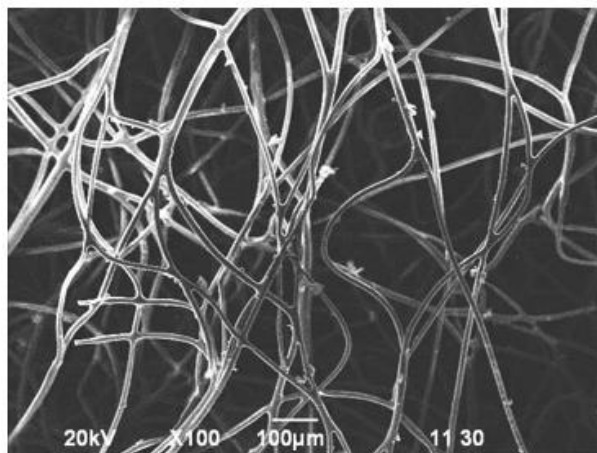


FIG. 4a. SEM image of non woven PE Fabric.

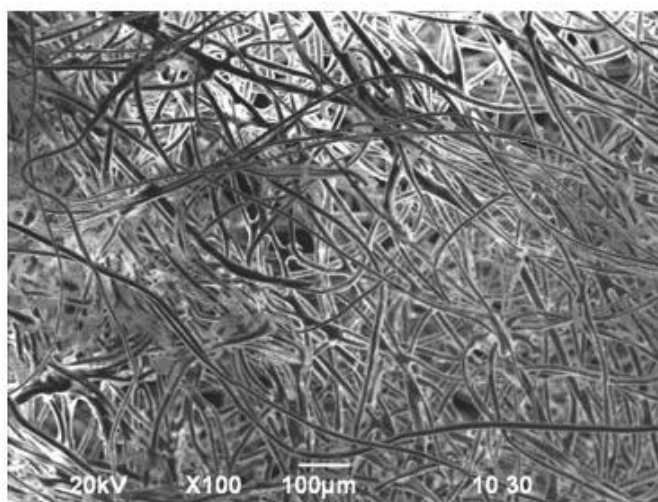


FIG. 4b. SEM image of GMA grafted non-woven PE fabric.

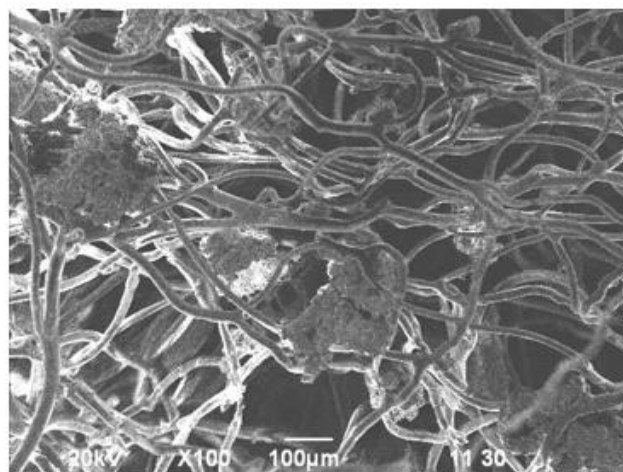


FIG. 4c. SEM image of amine functionalized GMA-g-non woven PE Fabric.

MO adsorption by the adsorbent.

The prepared adsorbent were examined for its applicability to adsorb MO from aqueous solution. Effect of adsorption environment on MO adsorption were carefully inspected to understand the factors acting behind the adsorption and also to find out the optimal condition for adequate adsorption. A tentative mechanism of aminated adsorbent preparation and MO adsorption is shown in **FIG. 5**.

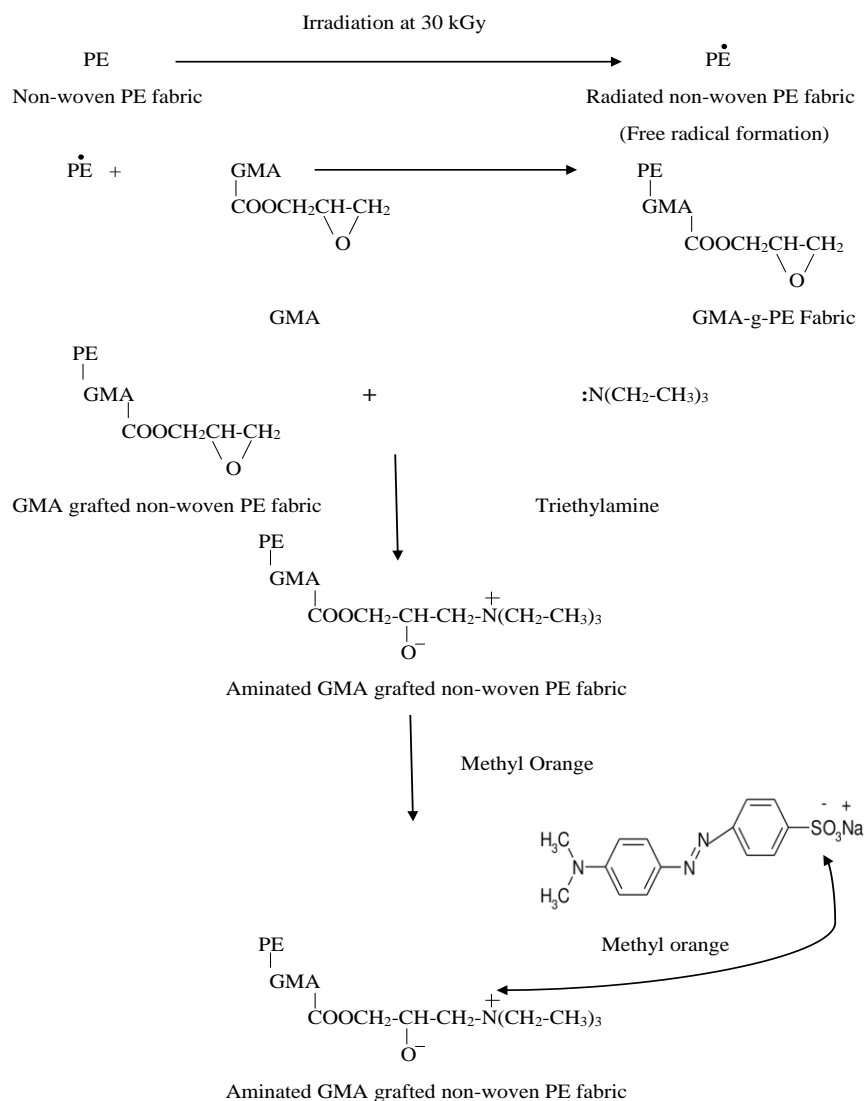


FIG.5. A tentative mechanism of aminated adsorbent preparation and MO adsorption

The effect of contact time on adsorption

It is natural that the contact time influences the adsorption quantity. How the time of contact between adsorbent and adsorbate effects the adsorption quantity is depicted in **FIG. 6**, It was quite usual that the adsorption quantity raised when the contact time between adsorbent and MO solution prolonged. Finally the, adsorption touched the equilibrium value after 48 hours when the quantity of MO adsorption was 41.73 mg/g.

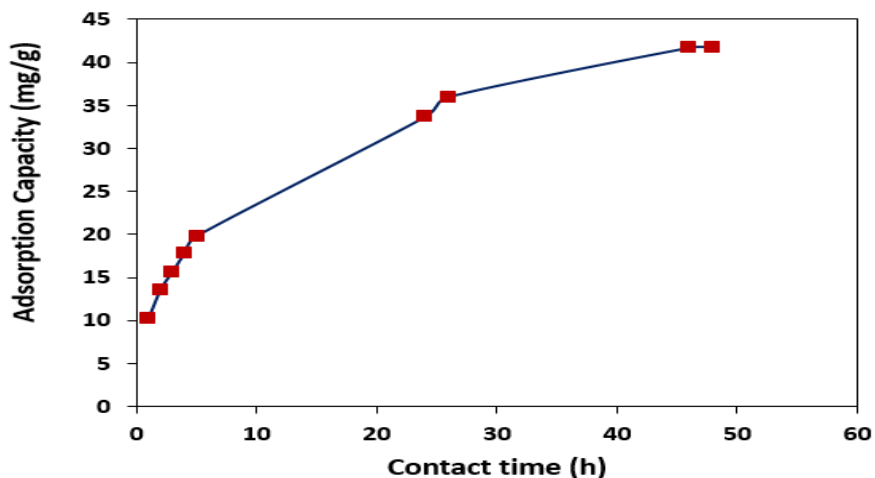


FIG.6. Effect of contact time on adsorption amount (pH 7, concentration 500 ppm, room temperature)

Kinetics study

Kinetics of the MO adsorption were analyzed. The adsorbate uptake rate markedly influences the total time of adsorbate uptake at the solid-liquid interface counting the diffusion course. The kinetics of aminated GMA-g-non woven PE film has been examined by the help of pseudo-first-order model, pseudo-second-order model. The correlation between investigational data and the model projected values was presented by correlation coefficient (R_2).

Pseudo-first-order model

The pseudo-first-order rate equation was described by Lagergren in 1898.

$$dQ_t / dt = k_1(Q_e - Q_t) \tag{5}$$

Where Q_e is the quantity of solute adsorbed by per unit mass of adsorbent at equilibrium (mg/g), Q_t is the quantity of solute adsorbed at any particular time (mg/g) and k_1 is the rate constant for the first order adsorption. The stated equation is combined for the marginal situations $t=0$ to $t=t$ and $Q=0$ to $Q=Q$ and then reorganized to attain the subsequent linear equation:

$$\log (Q_e - Q_t) = \log Q_e - (k_1 / 2.303)t \tag{6}$$

This is the most general formula of pseudo-first-order kinetic equation. FIG. 7, shows the data sketch and Q_e and k_1 for these plots have been described in table 1. The correlation coefficient obtained for this model is 0.973.

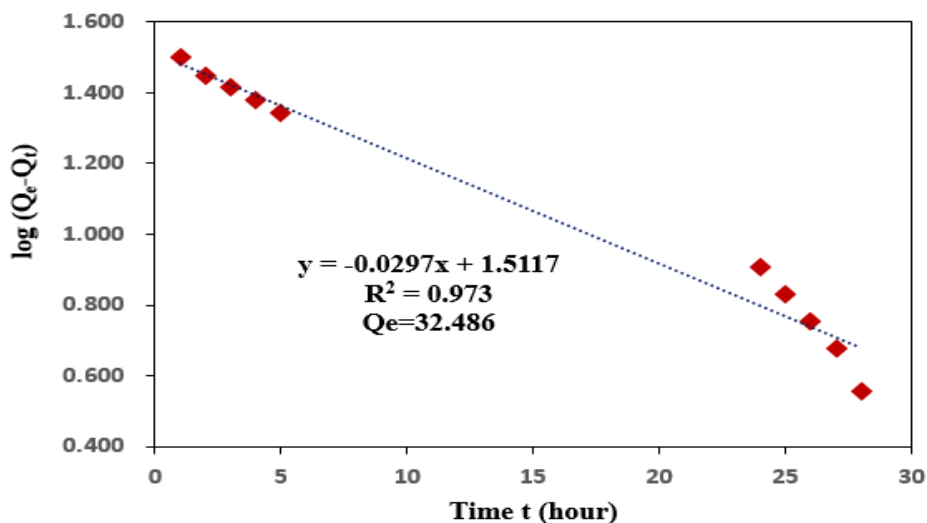


FIG.7. Pseudo-first-order kinetic plots for the adsorption of MO at room temperature. Pseudo-second-order model

The Lagergren equation was reformed by Ho and Mckay to get the differential equation:

$$dQ/dt = k_1 (Q_e - Q)^2 \tag{7}$$

Combining the above equation for the marginal conditions $t=0$ and $t=t$ and $Q=0$ to $Q=Q$ and reorganizing to attain the linearized form gives:

$$t / Q_t = (1/k_2 Q_e^2) + (1/Q_e)t \tag{8}$$

Here also Q_t and Q_e are the quantity of ions adsorbed (mg/g) at any particular time and equilibrium time respectively and k_2 (g/hmg) is the rate constant for second-order adsorption. The plot of t/Q_t vs t should give a linear relationship and k_2 and Q_e can be calculated from the slope and intercept of the plot.

The plot for MO adsorption by aminated GMA-g-non woven PE fabric is presented in **FIG. 8**, and values for k_2 and Q_e were presented in **TABLE 1**, The correlation coefficient (R_2) obtained for this model is 0.9924

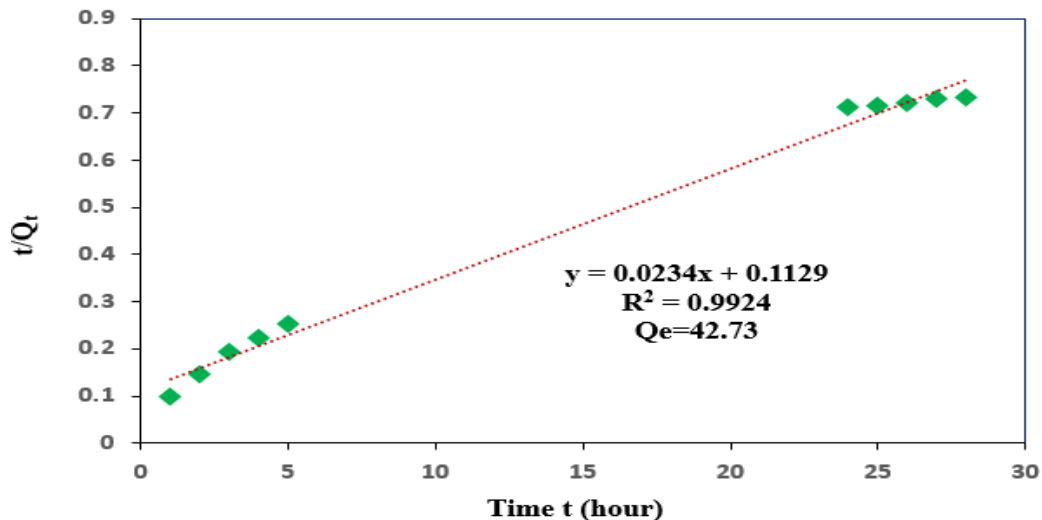


FIG. 8. Pseudo-second-order kinetic plots for the adsorption of MO at room temperature.

TABLE 1. The pseudo-first-order and pseudo-second-order rate constants for MO adsorption.

Pseudo-first-order rate constant				Pseudo-second-order rate constant		
Q_e (experimental) mg/g	Q_e (theoretical) mg/g	k_1 (h^{-1})	R_2	Q_e (theoretical) mg/g	k_2 $gh^{-1} mg^{-1}$	R_2
41.73	32.486	0.068	0.973	42.735	0.0005	0.9924

The outcomes detected that the experimental Q_e value and the theoretical Q_e value derived from first-order kinetic model are quite far from one another. Conversely, the close value of the experimental Q_e and the theoretical Q_e value calculated from second-order kinetic model are evident. Consequently the pseudo-second-order equation is suggested to be useful to describe MO adsorption of the aminated GMA-g-non woven PE fabric. Fitting of the experimental kinetic data with second order kinetic model suggests that diffusion through particles could be the rate-limiting step of the adsorption and it further recommends that the chemical bonding acts as the vital factor in the adsorption progression.

Effect of pH on adsorption

PH of the medium performs vital role in the adsorption course, increase or decrease of pH value effects the protonation and deprotonation of acidic and basic groups which in turn changes the adsorption affinity of the surface of the adsorbent. The experimental results reflecting the influence of pH on the adsorption of MO are depicted in **FIG. 9**, It can be understood that the adsorption increases considerably with the decrease of pH from 7.18 to 1.75 at 100 ppm. Functionalization of GMA-g-non woven PE fabric with trimethylamine provides a positively charged amino group. At lower pH the positivity of the amino group increase. As the amino group is responsible for MO adsorption, the greater positivity of the amino group will improve the adsorption quantity. Consequently, at a lower pH value of the solution, the MO adsorption by the adsorbent is higher.

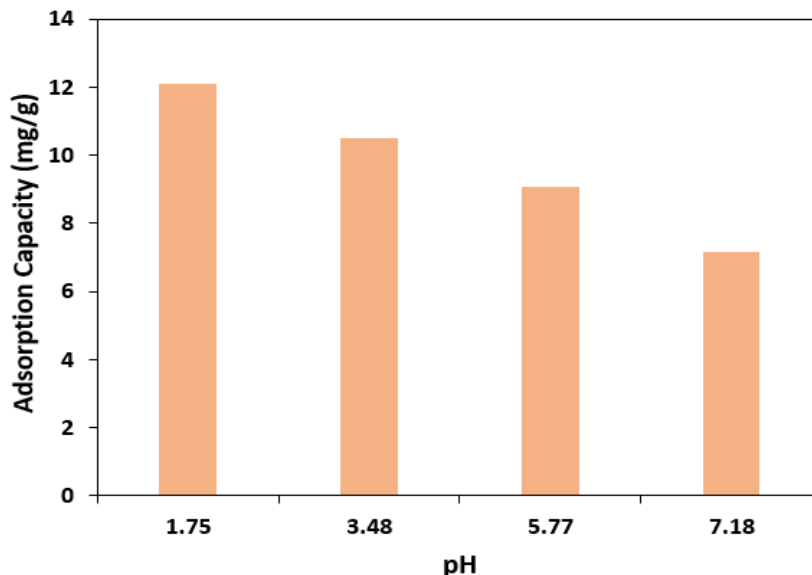


FIG.9. Effect of pH on adsorption amount (Concentration 100 ppm, time 48 h, room temperature).

Effect of initial MO concentration on adsorption

FIG. 10, displays the correlation between initial MO concentration and the volume of adsorption. The figure indicates that the quantity of adsorption of MO went on emerging with the rise of initial MO concentration and touched a flat terrain value at some raised up concentration. This can be explained as all the chelating positions of the adsorbent went through interaction with MO leaving behind no scope for further adsorption when the certain concentration was crossed. A maximum adsorption ability (40.54 mg/g) was gained at the MO concentration of 500 mg/L.

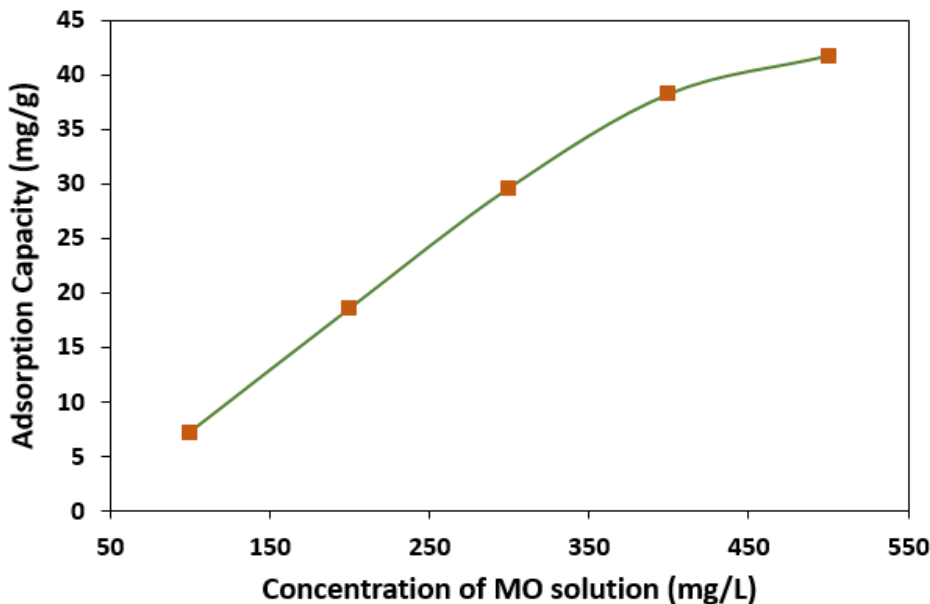


FIG.10. Effect of Concentration on MO adsorption. (pH 7, contact time 48 h)

Langmuir adsorption isotherm model

Langmuir’s adsorption isotherm model proposes that the adsorption takes place at definite uniform positions on the adsorbent and has explained effectively numerous monolayer adsorption courses. The experimental data of the equilibrium adsorption quantity of MO onto aminated GMA-g-PE fabric were investigated whether it obey the subsequent linear form of Langmuir model:

$$C_e / Q_e = C_e / Q_m + 1 / Q_m b \tag{5}$$

Where, C_e is the equilibrium adsorbate concentration (mg/L). Q_m is the maximum monolayer adsorption quantity of the adsorbent (mg/g), Q_e is the equilibrium adsorption amount (mg/g) and b stands for Langmuir adsorption constant (L/mg). Adsorption process is assumed to follow Langmuir model when a plot of C_e / Q_e vs C_e gives a straight line with a slope of $1 / Q_m$ and an

intercept of $1/Q_{mb}$. Experiment data of MO adsorption were plotted in **FIG. 11**, and the desired linear curve and high value of correlation coefficient ($R^2=0.9842$) directs a good correlation between the parameters and point toward the monolayer adsorption of MO onto the surface of aminated GMA-g-PE fabric. The maximum monolayer saturation quantity derived from the Langmuir equation is 63.29 mg/g. A comparative picture of MO adsorption capacity of present adsorbent with some other adsorbents is presented in **TABLE 2** [27-31].

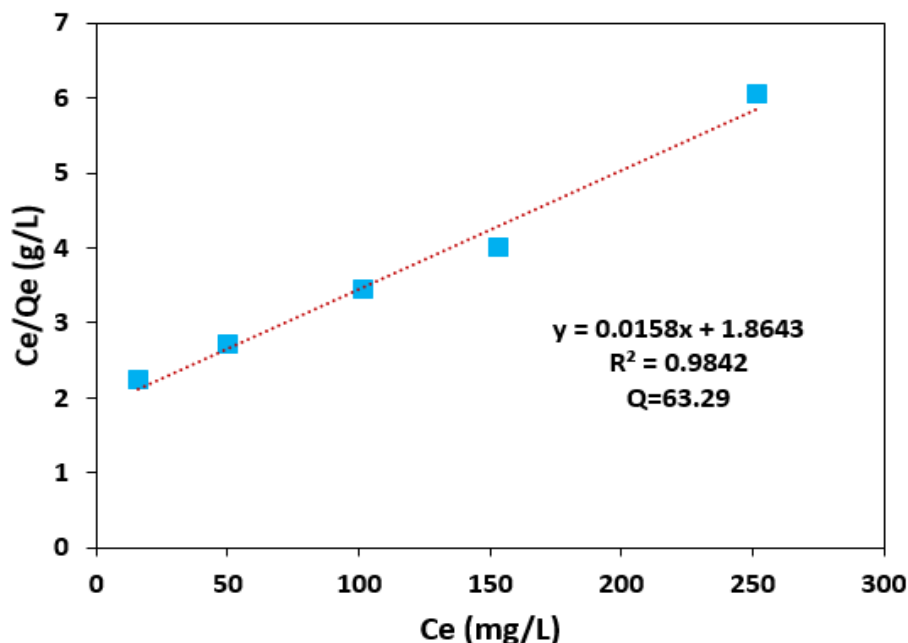


FIG. 11. Langmuir isotherm plot for MO adsorption at room temperature.

TABLE 2. A comparative picture of MO adsorption capacity of present adsorbent with some other adsorbents.

Adsorbent	Adsorption capacity (mg/g)
Aminated adsorbent [present study]	63.29
Chitosan biomass	29
Cork powder	16.66
Egussi peeling	13.89
Fe ₂ O ₃ -biochar nano-composite	20.53
Camel Thorn Plant	20.83

The effect of temperature on adsorption

Experiments revealed that the quantity of adsorption enhanced with the increase of temperature of the MO solution. At 80°C temperature, the quantity of adsorption was 36.94 mg/g, at low temperature (0°C) the amount of adsorption was 17.85 mg/g and at room temperature (25°C) the quantity of adsorption was 20 mg/g after 5 h at 500 ppm MO concentration.

Desorption and Reuse

For 1M NaOH Solution, desorption percentage of the MO adsorbed film was reached 70%. Almost same result is found for 2M and 3M NaOH solution.

Conclusions

Gamma radiation induced grafting of glycidyl methacrylate on non-woven polyethylene fabric and the amine functionalization of GMA-g-non woven PE film were investigated. The adsorbent was successfully characterized through FTIR and SEM technology. The amine functionalized GMA-g-non woven PE film was examined for the adsorption of MO from aqueous solution. How adsorption behaves under variable settings of contact time, initial metal ion concentration, pH and temperature were carefully investigated. The best condition that gave maximum MO adsorption was revealed to be contact time 48 h, initial metal concentration 500 ppm, pH 1.759 and temperature 80°C. Langmuir isotherm model was considered to realize the equilibrium between MO and the adsorbent system. Ideal fitting with Langmuir isotherm model recommended that MO adsorption was constrained to one molecular layer. The highest monolayer adsorption capacity calculated from Langmuir isotherm model was

60.60 mg/g. The adsorption kinetics was investigated using pseudo-first-order and pseudo-second-order models. The adsorption of MO was successfully concluded with pseudo-second-order model. Successful desorption of MO followed by reuse of the regenerated adsorbent fabric explored the future prospect of the practical application of the adsorbent.

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