The application of modified polyethyleneterphthalate (PET) nanofibers; characterization and isotherm study

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Abstract. In this study, dibenzo crown ether (DB18C6) is used to chemically modify the Electrospun Polyethylene Terephthalate (PET) nanofibers. The main parameters influence on the PET nanofiber’s surface modification with crown ether concentration and other solvents has been tested. The PET nanofibers with and without (DB18C6) are put into comparison by scrutinizing FTIR, TGA, SEM, and EDX results. It is to be mentioned that the modification of the PET nanofibers with crown ether is utilized for the extraction of methylene blue (MB) from aqueous solutions. For the purpose of evaluating the potential of the chemically modified PET nanofibers as adsorbents to remove MB from aqueous solutions, experiments of batch adsorption are carried out. The effect of MB concentration, solution pH, and shaking time are optimized. Several isotherm models such as Langmuir, Freundlich, Temkin, and Dubinin - Radushkevich (D-R) equations are utilized to test the equilibrium results. Owing to the high correlation coefficient (R2), the adsorption of MB into the modified PET is detected through Langmuir model. ΔGo, ΔHo, and ΔSo quantities are estimated. The negative ΔG° values, the positive ΔHo, and ΔSo values indicate that the adsorption process is spontaneous and endothermic.

Keywords: Electrospinning, Nanofibers, Polyethylene terephthalate (PET), Adsorption, Dibenzo-18-Crown-6 (DB18C6).

Graphical Abstract. Experiments of batch adsorption are executed for the potential evaluation of the modified PET nanofibers as adsorbents to remove MB from aqueous solutions.
1 Introduction

Electrospinning is an effective technique for the construction of versatile nanofibers from a diverse range of polymers. These polymers may range from polymers that are water-soluble to biopolymers and polymers of liquid crystalline [1,2]. By using this technique, ultrafine nanoscale fibers can be obtained by releasing the polymer solution in a high voltage electrical field [3,4]. The properties of electrospun nanofibers represented by fine diameter, high surface-to-volume[5], being high porous and versatile to physical or chemical functionalization, turn them into effective attractive alternatives to the traditional adsorbents in wastewater treatment[6]. They are further suitable for use in a wide range of industries viz ophthalmology, medicine, food, textiles, paper coatings, and automotive applications[5,7–11]. Additionally, interest in increasing the selectivity of nanofibers has led to additional work on this topic at large and on nanofiber modification with crown ether in particular[12]. Crown ether, that is modified as a novel hydrophobic porous organic copolymer, is used for the selective adsorption of dyes[13].

Dyes form are consider one of the most hazardous industrial materials that can cause severe problems to human health[14,15]. As such the removal of dyes from colored effluents has attracted much attention[16]. Methylene blue (MB) has a variety of applications, as in paper, and hair coloring, and dyeing various types of textile fabrics[17]. Although it is not particularly hazardous, it may have some harmful effects on humans [18,19]. A set of physical and chemical processes are used to remove dyes from wastewater. Biological oxidation and chemical precipitation methods can be the most salient methods used in color removal. However, such processes are effective and economical only when the concentrations of solutes are relatively high[20]. Among all the common techniques/treatments, adsorption is one of the simplest and most efficient methods for extracting dyes from wastewater resources[21,22].

Polyethylene terephthalate (PET), the most popular polyester thermoplastic, is commonly used in apparel, food and liquid packaging, and in materials thermoforming[23]. Recently, there has been researches conducted on the generation of nanofibers from bottle-grade PET[24–26]. Given the widespread availability of plastic recycling facilities, only about 5% of the 32 million tons of plastic has been recycled in the United States. Water bottles constitute the main proportion of plastic in the waste stream[27,28]. In addition to the significant environmental advantages of recycling, recycled PET filament is an outstanding candidate source for manufacturing[29].

Crown ethers are among the most familiar host molecules. They have triggered growing attention owing to their potential to construct stable metal ion complexes. They contain negatively polarized oxygen atoms with varying cavity sizes. This means that they are selective in the bondage to specific ions[30]. Such selectivity has resulted in developing and synthesizing the new crown ethers in addition to their several well-defined polymers[31]. Being highly selective, crown ether has attracted a considerable research interest to form crown ether adsorbents[32]. This work deals with the modification of PET nanofibers with a crown ether to selectively remove MB from aqueous solutions, resulting in the conversion of PET-based wastes into useful products after converting them to nanofibers. The modification of the PET nanofibers with crown ether enhances the operating window of the electrically spun nanofibers and offers high selectivity. This process of modification makes them more promising and more effective for the applications of adsorption and filtration. Noteworthy, ether is chosen as chemical modification of the PET nanofibers since there are very few research studies that have used it with the nanofibers as an aid in the absorption of organic dyes.

2 Experimental and Methods

2.1 Chemicals
The polymer (PET) waste material is obtained from a local water packaging company. It is first used after washing and removing non-PET materials and later left to dry. All primary chemicals used in the research are of analytical reagent grade. Trifluoroacetic acid (TFA) is obtained from ROTH; dichloromethane (DCM) from Uni-Chem; Dibenzo-18-crown-6 (DB18C6); acetone, and methylene blue (MB) from Sigma-Aldrich.

2.2 Preparation of Solution
The PET polymer solution is prepared by mixing in (3:1) ratio of DCM and TFA; three specific PET concentrations are used in this study. To produce the desired product, 5% PET is used. PET is grinned with a grind household and then sifted with a test sieve (250 µm). A magnetic stirrer is used to stir the solution so as to dissolve PET and for four hours at room temperature to supplement the polyethylene’s solubility [33].

To obtain a stock solution of MB (1000 mg/L) from (100 ml) of deionized water, (0.1 g) of MB is dissolved. The solution is used for further experimental solution preparation. With 1 M HCL or (0.6 M) NaOH, the pH values are adjusted. Throughout this analysis, analytical grade reagents are applied and deionized water is used during the adsorption experiments.

2.3 Preparation of PET Nanofibers
The polymer solution is converted into nanofibers using an electrospinning device (NFES-100, Fig 1).

![Nanofiber electrospinning machine – NFES-100](image)

The solution is placed in a plastic syringe (5 ml) attached to a stainless-steel needle. 1 mL/h feed rate, 15 cm is the distance between the needle tip and the collector, and 15 kV is used for applied voltage, as shown in a previous study optimization[24,33].

2.4 Modification and Characterization of PET Nanofibers with DB18C6
The PET nanofiber mat is impregnated with different amounts of DB18C6: (0.008, 0.012, 0.016, and 0.02 g) dissolved in 6 mL of solvent to obtain solutions at concentration (3.70x10^{-3}, 5.55x10^{-3}, 7.40x10^{-3}, 9.24x10^{-3} mol/L). Then the weight of the best result (DB18C6) is dissolved in (6 mL) of different solvents, viz (H2O, methanol, acetone, and ethanol) to evaluate the effect of the solvent on the modification process. It is then mixed with 0.011 g of PET nanofiber mat and continuously stirred at ambient temperature. It is experimentally found out that two hours of stirring at constant room temperature is sufficient to obtain a new sorbent modified PET nanofiber that could be used in the current research to remove MB from aqueous solutions. After 2 hrs., the mat is first washed several times with deionized water to remove any free ions and then dried at room temperature for 1 hr. Comparison between morphologies and mean diameters of the PET nanofiber and the modified PET nanofiber is carried out by the field emission scanning electron microscope (TESCAN). Shimadzu is utilized to measure Fourier transforms infrared (FTIR) spectra, and the measurements of the Thermogravimetric analysis (DSC-TGA) are attested with Q600. Energy-dispersive X-ray analysis
(EDX) is used to provide elementary quantitative compositional information on C, O, and inorganic species on the surface of the PET nanofiber and the modified PET nanofiber.

2.4 Batch Adsorption Studies of MB

MB adsorption is achieved in a batch process by means of varying doses of the dried modified PET nanofiber adsorptive concentration, medium pH, and temperature. A weighed sample of the dried modified PET nanofiber is mixed with 10 ml MB solution of (5 mg/L) concentration. The mixture is shaken in a water bath of a thermostat for (30 min). It is then allowed to settle down and centrifuge. The MB concentration in the supernatant is determined by a spectrophotometer type (JANEWAY 7315 Spectrophotometer) from the contrast calibration curve (Fig.2).

![Figure 2. The calibration curve](image)

2.5 Adsorption Isotherm Investigation

The equations in (Table 1) are used to calculate the $Q_e$ (mg/g) related to the amount of MB adsorbed onto the modified PET nanofiber and the MB removal percent (R %). The adsorption results of the experiments are provided by means of the isothermic equations of Langmuir, Freundlich, Temkin, and Dubinin Radushkevich (D-R) (Table 1). The isothermic equations’ constant parameters are evaluated [34]. For each isotherm, the solution temperature and the concentration of MB varied while preserving the adsorbent weight in each sample.

| Table 1. The equations of the Present Study |
|--------------------------------------------|
| **Name** | **Equation** | **Description** |
| Q<sub>e</sub> = amount of MB adsorbed (mg/g) at equilibrium | $Q_e = (C_i - C_e) \times V / M$ | $C_i, C_e =$ initial and equilibrium concentration (mg/L) of MB in the solution. $V =$ volume of experimental solution (L), $M =$ weight of the modified PET (g). |
| Adsorption efficiency | $R\% = \frac{(C_i - C_e)}{C_i} \times 100$ | |
| Langmuir equation isotherm | $1/Q_e = (1/\bar{K}_L \times Q_m) + 1/Q_m$ | $C_e =$ equilibrium concentration in solution (mg/L). $Q_e =$ amount of MB adsorbed (mg/g). $Q_m =$ maximum monolayer capacity of the adsorbent (mg/g) $\bar{K}_L =$ adsorption equilibrium constant (L.mg<sup>-1</sup>). |
\( R_L = \text{separation factor} \)

\( R_L = 1/1+K_L \cdot C_0 \)

\( K_L = \text{constant of adsorption equilibrium} \ (\text{L.mg}^{-1}) \)

\( C_0 = \text{Initial Conc. (mg/L)} \)

Freundlich equation isotherm

\( \log Q_e = \log K_F + 1/n \log C_e \)

\( K_F = \text{constant indicating to the capacity of the adsorption} \ (\text{L.mg}^{-1}) \)

\( n = \text{constants indicating an intensity of the adsorption} \)

Tempkin equation isotherm

\( Q_e = B_T \ln A_T + B_T \ln C_e \)

\( B_T = \text{heat of adsorption} \)

\( A_T = \text{equilibrium binding constant} \ (\text{L.mg}^{-1}) \text{ corresponding to maximum binding energy} \)

Dubinin radushke vich isotherm

\( \ln Q_e = \ln Q_m - K_{D,R} \cdot \varepsilon \)

\( Q_m = \text{theoretical monolayer saturation capacity} \ (\text{mg g}^{-1}) \)

\( K_{D,R} = \text{D-R constant} \ (\text{mol}^2 \text{KJ}^{-2}) \)

\( \varepsilon = \text{Polanyi potential} \)

\( \varepsilon = RT \ln [1 + 1/C_e] \)

Energy of adsorption

\( E = 1/(2K_{D,R})^{1/2} \)

\( E = (K \text{J.mol}^{-1}) \)

Van't Hoff equation

\( \Delta G^o = \text{Gibbs Free Energy Change} \)

\( \Delta H^o = \text{Enthalpy Change} \)

\( \Delta S^o = \text{Entropy Change} \)

\( K_L = \text{Langmuir equilibrium constant} \ (\text{L/mol}) \)

\( R = \text{Gas constant} \ (8.314 \text{ J/mol K}) \)

\( T = \text{temperature} \ (\text{K}) \)

3 Results and discussion
Modification of the PET nanofiber increases the active sites on the nanofiber that increases MB adsorption from aqueous solutions.

3.1 Optimization of Experimental Conditions and Characterization of Modified PET Nanofibers.
The effect of both crown ether (DB18C6) concentration and the solvent is studied and the time required to complete the adjustment is considered.
The solvent nature plays an important role in the complex formation's stability and selectivity.

![Figure 3: The effect of solvent on modification of PET nanofibers](image)

As shown in (Fig.3), the adsorption capacity \( Q_e \) gives good results by using acetone as a solvent. Added to that, (0.02 g) of crown ether is sufficient to the modified PET nanofiber as shown in (Fig.4).
The dibenzo-18-crown-6 (DB18C6) is relatively rigid, rich in electrons, and, therefore, convenient for the complexion of aromatic gropes through pi-pi, CH-pi interaction, and charge-transfer interactions[32].

The interaction of PET nanofiber with the crown ether is confirmed by comparing the PET nanofiber FTIR spectroscopy with the modified PET nanofiber, as shown in (Fig.5). The IR spectrum band shift in the region of 4000-500 cm$^{-1}$ of PET nanofiber proves the complex formation between PET nanofiber and crown ether. The observed strong band indicates the stretching frequencies vas (Ph-O-C) in crown ether at 1241 cm$^{-1}$[35,36].
The –C=O stretching band shifted from 1719 cm\(^{-1}\) to 1711 cm\(^{-1}\). Aromatic and aliphatic –C-H band is relocated from 2957 cm\(^{-1}\) to 2960 cm\(^{-1}\), and 1409 cm\(^{-1}\) band is related to the bending of –C-H shifted to 1405 cm\(^{-1}\), whereas the band at 726 cm\(^{-1}\) (waging of aromatic hydrocarbon) is shifted to 720 cm\(^{-1}\). The disappearance of the first clear intense peak is related to –C=O asymmetry at 1260 cm\(^{-1}\) and the second peak at 2350 cm\(^{-1}\) is related to axial symmetrical deformation of CO\(_2\) [37].

SEM characterized PET nanofiber’s morphologies and the modified PET nanofibers. Micrographs and diameter distribution of PET and the modified PET are shown in (Fig.6).

![Figure 6. SEM micrographs and mean diameters of (A) PET nanofibers 9B) modified PET nanofibers](image)

The mean of neat PET nanofibers had a diameter of 31.18 nm before modifying. The mean fiber diameter after modification is (30.39 nm). Furthermore, there is no discrepancy in the fiber diameter because the addition of (DB18C6) is done after the nanofibers are produced with an electrospun. The thermogravimetric analysis assessed the thermal stability of the pure PET and the modified PET nanofibers samples as in (Fig.7).

The (TGA) curve for PET and the modified PET nanofibers sample indicate three steps of mass loss. Firstly, the PET nanofiber at about (417.89 °C) and the TG curve result approximately provide (8.244%) of mass loss and this is attributed to the initiation of the process of decomposition. Approximately (27.92) percent of the weight is lost for the modified PET nanofiber sample at (414.09 °C) because of the thermal decomposing process of (DB18C6). Secondy, the process of decomposition for the PET nanofiber and the modified PET nanofiber takes place at (454.57 °C) and (464.13 °C), respectively. The observed weight loss corresponds to about 79.01 percent of the PET nanofiber, whereas the weight loss for modified PET nanofiber is about 56.84 %.

Finally, the decomposing process occurs at 532.89 °C for the PET nanofiber and the modified PET nanofiber equally. The obtained mass loss correlate with the PET nanofiber is nearly (8.231 %), whereas the mass loss of the chemically modified PET nanofiber is approximately (13.39 %).
The charred residue is at 800 °C (4.52% ) for PET nanofiber and 1.85% for modified PET nanofibers. This ratio is thought to be attributed to the adsorption of inorganic species on the PET used mainly from isotherm bottles. This is confirmed by the elemental analysis. (Fig.8) demonstrates the EDX spectrum as well as a detailed report on the percentage of carbon and oxygen in the PET nanofibers and the modified PET nanofibers.

Figure 7. (TGA/DSC) Curves for (A) PET nanofibers 9B) modified PET nanofibers

The study findings represented by 63.31 and, 34.96 percentage of carbon and oxygen in PET nanofibers and 68.45 and, 28.85 percentage of carbon and oxygen in modified polyethylene are respectively consistent with the theoretical calculations.
3.2 Optimization of Experimental Conditions of MB Adsorption on the Modified PET Nanofiber.

The Effect of pH on the MB Adsorption

The pH value of the solution system is essential for the adsorption of MB to the modified polyethylene terephthalate (PET). The effect of pH on modified PET adsorption behavior is examined in this study by adjusting pH with dilute HCL and NaOH at (4.0, 5.0, 6.0, 7.0, 8.0, 9.0, and 10.0).

Fig. 9 shows the extent to which MB is removed as a pH function for an initial (5 mg/L) concentration using modified PET nanofiber as adsorbents. The highest adsorption capacity at pH (6) is obtained.

3.2.1 The Effect of the Initial Concentration upon the MB Adsorption.

The effect of the initial concentration upon the MB adsorption to the modified PET nanofiber is investigated by mixing 10 ml of MB solution (5, 10, 15, 20 and 30 mg/L) at pH (6) and (0.011 g) of modified PET nanofiber at room temperature and shaking the mixture at (150 rpm for 100 minutes).
The initial MB concentration provided a significant impetus to resolve any resistance of MB molecules to mass transfer between the aqueous and solid phases. Therefore, a higher initial MB concentration would increase the adsorption capacity, as in Fig. 10.

![Figure 10. The effect of initial concentration of MB on modified PET nanofibers](image1)

3.2.2 The Effects of The Adsorbent Dose
The adsorption experiment is also conducted by mixing 10 ml of MB (5 mg/L) with modified PET nanofiber doses (0.005, 0.008, 0.01, 0.02, 0.03 g) at pH (6) and shaking the mixture at ambient temperature for (100 minutes) at (150 rpm). On the one hand the adsorbent capacity $Q_e$ decreases as the adsorbent concentration is raised, while on the other hand the percentage of removal raises as the adsorbent dose is increased. This occurs because the raising adsorbent dose increases the surface area and the number of active sites for adsorption accordingly. These findings are in agreement with those presented by another researcher [38]. Fig.11 illustrates the relationship between the amount of adsorbent dose and the percentage of the adsorbent removal (R %) which is (97.35 %). This percentage is found to be the best removal by using (0.02 g) of the adsorbent. On this basis, (0.02 g) of adsorbent is selected for subsequent experiments.

![Figure 11. The effect of adsorbent dosage on the adsorption of MB on modified PET nanofibers](image2)

3.2.3 The Effect of Contact Time on Adsorption
The effect of contact time on the adsorption of MB on modified PET nanofiber is investigated by mixing (10 ml ) of the MB solution (5mg/L) at pH (6) and (0.02 g) of the modified PET nanofiber. The mixture
is then shaken with 150 rpm at ambient temperature (5, 10, 20, 30, 40, 50, 60, 70, 80, 90, 100 minutes). Fig. 12 demonstrates the contact time effect on the amounts of MB adsorbed at $Q_e$ equilibrium. It is clear that the adsorption rate at the beginning of the reaction is so rapid as about (44%) of MB is removed from the solution during the first ten minutes. Then the adsorption rate of MB on modified PET nanofiber is found to be slow as the maximum removal reaches (97%) at (100 minutes).

**Figure 12.** The effect of contact time on the adsorption of MB on modified PET nanofibers

### 3.2.4 The Effect of Temperature on Adsorption

The effect of temperature on MB adsorption onto modified PET nanofiber is investigated by mixing 10 ml of MB solution (3, 5, 10, 15, 20, 25, 30 mg/L) with pH (6) and (0.02g) of modified PET nanofiber at the temperature of (30 °C, 40 °C, 50 °C) and later shaking with 150 rpm for 100 minutes (See Fig 13).

**Figure 13.** Temperature influence on MB adsorption on modified PET nanofibers

### 3.2.5 Adsorption Isotherm

Various adsorption isotherms, namely Langmuir, Freundlich, Tempkin, and Dubinin-Radosevich isotherms have been used to study the MB interactions on the surface of the modified PET nanofiber (Table 1). For the Langmuir model, the $1/Q_e$ versus $1/C_e$ plot are used at different temperatures, as shown in Fig. 14.
Figure 14. Langmuir isotherm of the adsorption of MB on modified PET nanofibers

The linear plots of log $Q_e$ versus log $C_e$ at all adsorption doses are found to fit the Freundlich equation, as illustrated in Fig. 15.

Figure 15. Freundlich isotherm of the adsorption of MB on modified PET nanofibers

The plot of $Q_e$ versus ln $C_e$ in Fig. 16 is related to Temkin isotherm at different temperatures and has been used to study the adsorbent, whilst the plot of ln $Q_e$ vs. $\varepsilon^2$ in Fig. 17 is related to Dubinin-Radushkevich isotherm.

Figure 16. Temkin isotherm of the adsorption of MB on modified PET nanofibers
The values of Langmuir, Freundlich, and Tempkin isotherm constants and the coefficients of D-R isotherm for adsorption of MB on modified PET nanofiber are presented in Table 2. By comparing the different adsorption isotherm models, it is found that the adsorption of MB on the modified PET followed the Langmuir model due to the high correlation of coefficient ($R^2$). Accordingly, the adsorption mechanism is the monolayer coverage of the MB on the surface of the modified PET nanofibers.

**Figure 17. Dubinin-Radosevich isotherm of the adsorption of MB on modified PET nanofibers**

**Table (2):** Comparison between the coefficients isotherm parameters of MB (3, 5, 10, 20, 30 mg/L) adsorption onto modified PET nanofibers.

| Isotherm model | Isotherm parameter | Temperature °C | 30     | 40     | 50     |
|---------------|-------------------|----------------|--------|--------|--------|
|               | $Q_m$(mg/g)       |                |        |        |        |
| Langmuir      | $K_L$(L/mg)       |                | 0.2618 | 0.5357 | 13.7741|
|               | $R_L$             |                | 0.56-0.11 | 0.38-0.06 | 0.38-0.10 |
|               | $R^2$             |                | 0.9926 | 0.9371 | 0.9983 |
| Freundlich    | $K_F$             |                | 2.026  | 3.339  | 2.532  |
|               | n                 |                | 2.09   | 1.7534 | 1.6641 |
|               | $R^2$             |                | 0.9672 | 0.6805 | 0.7996 |
| Tempkin       | $A_T$             |                | 4.15   | 3.220  | 2.371  |
|               | $B_T$             |                | 1.960  | 3.197  | 3.027  |
|               | $b_T$             |                | 1285.342 | 814.053 | 887.068 |
|               | $R^2$             |                | 0.9717 | 0.8117 | 0.7219 |
| D-R           | $Q_m$(mg/g)       |                | 6.262  | 10.648 | 7.343  |
|               | $K_D.R x 10^{-7}$ |                | 5      | 4      | 2      |
|               | E KJ/mol          |                | 1000   | 1118.033 | 1581.138 |
|               | $R^2$             |                | 0.9176 | 0.7839 | 0.5994 |

In addition, the $R^2$ values indicate that the MB adsorption on the modified PET do not follow Freundlich, Tempkin, and Redlich–Peterson isotherms.

The Van’t Hoff equation is used to estimate the thermodynamic parameters. The $\Delta H^o$, $\Delta S^o$, and $\Delta G^o$, are calculated from Van’t Hoff plot, as shown in Table 3.

**Table (3):** Thermodynamic Constants Values
It is evident from the negative values of $\Delta G^o$ that the adsorption of MB on the modified nanofiber surface is spontaneous. The positive $\Delta H^o$ values indicate an endothermic character, but at the same time the positive $\Delta S^o$ values indicate an increased random nature of the system.

4 Conclusion

The modified PET nanofibers are first prepared by means of electrospinning. The PET nanofiber mat is then impregnated with different amounts of DB18C6. On comparing the results obtained from FTIR, TGA, SEM, and EDX for PET nanofiber and the modified PET nanofiber, a clear discrepancy between the two fabrics is found. The adsorption experiments indicate that the modified PET might be quite effective in removing MB from the aqueous medium at relatively low concentrations. The optimized adsorption conditions are obtained at 30 °C, pH 6, and contact time 100 min. The adsorption of MB on the modified PET is studied by adopting the Langmuir model. According to the RL value, it seems that adsorption commonly occurs under our conditions. Also, the $\Delta H^o$, $\Delta S^o$, and $\Delta G^o$ values suggest that MB adsorption represents a spontaneous and endothermic process on the modified PET nanofibers.

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