Effects of Ultrasonic Waves on PTT Dyeing with Disperse Red 60 Dye

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ABSTRACT: *In the current study, disperse red 60 (DR60) dye was selected for dyeing of polytrimethylene terephthalate (PTT) under influence of ultrasonic radiation. For this purpose, the disperse dyes were placed under ultrasonic for 15-90 minutes and the measurements of some parameters including particle size, the influence of temperature, and time were investigated. The results illustrated that ultrasonic has a good effect to decrease particle size of the dye and the smallest particle size for the DR60 dye was obtained at the time of 45 minutes and the temperature of 20 °C. The adsorption process under ultrasonic irradiation was exhibited maximum efficiency of dye at the optimal conditions including adsorption temperature (80 °C), PTT amount (0.1 g), irradiation time (120 min), pH = 6, and initial dye concentration (40 ppm). All experiments indicate that ultrasonic has a significant effect on the adsorption of DR60 dye to PTT. As results show, the Nernst model was fitted properly to the experimental data in comparison to other isotherm models as presented. Furthermore, the achieved thermodynamic parameters including the negative value of ΔG (-0.85 to -1.58 kJ/mol) at all temperatures, positive values of ΔH (11.32 kJ/mo1) and ΔS (36.51 J/mol.K), indicate that adsorption of DR60 dye to PTT is spontaneous and endothermic in nature.*

KEYWORDS*: Disperse Red 60 dye; Ultrasonic irradiation; Polytrimethylene terephthalate; Adsorption investigations.*

INTRODUCTION

Polytrimethylene is aromatic polyester produced by polymerization of 1, 3-Propanediol, instead of ethylene glycol. According to the experimental data, the chemical properties of PTT and polyester are similar. The presence

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of Methylene groups with odd numbers of the macromolecular chain structure has caused the formation of the spiral molecular structure of the PTT, which has led to greater elasticity and increased the ability to move these chains. Thus, T_g parameter decreases and dyeing can be performed at lower pressure and temperature [1, 2]. Disperse dyes are very popular and an important class of dyes for dyeing polyester fabrics owing to their luster, wide range of color and excellent fastness properties, as well as the environmental and economic point of view [3]. Moreover, polyester is non-ionic, hydrophobic and dyeable almost only disperse dye. Therefore, when its require to dye under atmospheric conditions, carriers is used to increase inter polymer space but it remains on the fabric surface. Some problems with polyester dyeing, such as the use of carriers in dyeing, dyeing at high temperature and high pressure, and so on, cause the invention of a novel dyeing technique using ultrasonic waves[4].

Ultrasound has been used in the washing and desizing processes with regard to the ability of mass transport and stirring in textile industry. The effect of ultrasonic waves on reducing the time and temperature of dyeing operations, as well as the reduction of electrolyte consumption in the dye bath for direct dyes and the use of less water for dyeing with reactive dyes and reducing the size of the vat dye is specified [5-9].

In general, the use of ultrasonic waves in dyeing is preferable to conventional dyeing in terms of energy consumption, time, and chemical processes [10-14]. Some of these advantages include: reducing lower temperature of dyeing, exhaustion method and better fixing of dye in reactive dyes, reducing electrolyte consumption in direct dyes, not using carriers for low-temperature polyester dyeing, uniform dyeing, and less wastewater generation [15-17]. Sound frequency above 20 kHz is considered to be ultra sound (US). When ultrasound is applied to an aqueous solution or suspension an increase in mixing, shearing and mass transfer is observed [18]. It can be used for dyeing both hydrophilic and hydrophobic fibers too [19].

Recently, to improve adsorption of fabrics using disperse dye, various techniques such as UV, Gamma, Plasma, Microwave, super critical CO_2, and Ultrasonic treatment have been developed [3]. Application of mentioned techniques is some of the revolutionary ways to advance the textile wet processing. None of these methods are commercially viable due to the inherent limitations [20, 21]. The using of ultrasonic waves radiations is also one of the sources of getting energy which can be utilized in textile wet processing. Electrochemical dyeing, in which chemical reducing agents are replaced by electrons from the electric current and effluent contaminating substances can be dispensed with altogether. The plasma technology is considered to be very interesting future-oriented process owing to its environmental acceptability and wide range of applications. Since recently, however, the plasma technology is being introduced in textile industry as well. Dyeing with super critical CO_2 is still in its infancy. It has been proved time and again that its successful at laboratory scale. Large amount of research input is needed for system integration. Dyeing with this system has been found successful with synthetic as well as natural fibers [21].

An alternative approach has been developed by using UV radiation to modify the fabric surface whilst leaving the bulk textile unaffected. Surface fibers must either absorb UV radiation directly or a suitable photo initiator must be applied to produce large number of highly reactive radicals when the textile surface is exposed to UV. Surface modification is particularly useful on natural fibers such as wool and cotton synthetic colorants. UV radiation can be successfully applied to enhance the color fastness properties as well as color strength without harming the physical characteristics of other fabrics such as wool and silk using other class of dyes [22]. Microwave dyeing takes into account only the dielectric and the thermal properties. The dielectric property refers to the intrinsic electrical properties that affect the dyeing by dipolar rotation of the dye and influences the microwave field upon the dipoles. The microwave techniques facilitate a dye-fiber contact and accelerating the rate of diffusion of the dye inside the fiber by breaking the boundary layers covering the fiber and raising the interaction between dye molecules and fibers [3, 23].

It seems apparent from the literatures that ultrasound holds promise in dyeing of variety of substrates. The ultrasonic cavitation accelerates the rate of dyeing and increases the dye uptake on fabric. The typical dyeing process involved the use of chemicals and thermal energy, which can be reduced, by using ultrasound energy. Among the wet processes, application to dyeing seems to be most advantageous, followed by finishing and preparation processes [21]. There may be a possibility of reducing the pollution load on effluent water. Many conventional

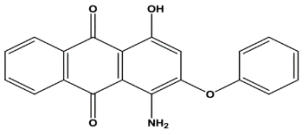
methods are being applied to uptake ability of fabric and to increase the color yield, however, ultrasonic dyeing provides cost, labour and energy effectiveness which increases the competitive atmosphere of any textile unit.

Ultrasonic offers many potential advantages in textile wet processing:

- Energy savings by dyeing at lower temperatures and reduced processing times
- Environmental improvements by reduced consumption of auxiliary chemicals
- Processing enhancement by allowing real-time control of color shade
- Slower overall processing costs, thereby increasing industry competitiveness.

Some studies reports the use of UV radiation for cotton fabric by reactive blue dye that has enhanced the strength of dye on irradiated fabric, also improved the dyeing properties [28]. Also, Microwave irradiation treatment has improved the color strength values and fastness properties of polyester fabrics by the application of disperses dyes [3] and also cotton dyeing with reactive dye [24]. Many researches using ultrasonic energy in the different fields including dyeing cellulose nanofibers with two reactive dyes [25], Polyester fabrics with disperse dyes [4], wool fabrics with a natural dye [13], microdeynier polyester fabrics with a disperse dye [26], cellulose acetate yarn with disperse dye [27], Nylon 6 nanofibers with two disperse dyes[28] and Polytrimethylene terephthalate (PTT) fibers with DR60 dye [14]. The investigation mentioned has led to the some significant results such as higher color fastness, higher dyeing performance, higher Color strength and color depth, and reduces dyeing time than conventional dyeing.

Therefore, in the present work, according to the advantages of ultrasonic treatment, the color strength properties of Polytrimethylene terephthalate (PTT) using disperse red 60 (DR60) dye was evaluated by ultrasonic assisted method. The chemical structure of the DR60 is presented in Scheme 1.

Molecular Structure	
C.I number	60756
Molecular Formula	$C_{20}H_{13}NO_4$
Molecular Weight	331.32
symbol	DR60

Scheme 1: Chemical structure of DR60.

EXPERIMENTAL SECTION

Materials and equipment

DR60 dye (low-energy) was purchased from Alvan Sabet Co. In this project, all of the chemicals were analytical grade and used without further purification. Other chemical compounds including sodium hydroxide, chloridric acid, and citric acid, were purchased from Merck Co. The measurement of pH was performed by a pHS -3B model pH Meter from REX Co. The UV–Vis absorbance spectra of samples were provided using UV-Visible S2100 spectrophotometer from Unique USA Inc equipped with quartz cell of 1 cm path length.

The ultrasonic irradiation was carried out with equipment operating at 20 kHz (Pars Sonic Co). The acoustic power was set at 100 W cm^{-2}, 2.5-liter transducer, the time interval of 2 - 22 minutes, and a temperature range of 20 - 90 °C, and the tip diameter of the horn was 1.1 cm.

The lab dyer-8rc model device purchased from Yazd Textile Co was performed for dyeing samples. The device consists of 12 cylinders with a capacity of 200 ml, which allowed the temperature increase in this device with a precision of ±1 until 130 °C. It should be noted that the device worked at 70 rpm. The SZ-100Z (Dynamic Light Scattering & Zeta potential analyzer) particle size analyzer from Japanese manufacturer of Horiba, Ltd, which had the ability to measure particle size in the range of 0.3 nanometers to 8 microns, was used to measurement of particle size.

Preparation of nano- disperse dyes

Initially, DR60 solution with a concentration of 1000 ppm was prepared and then the prepared dye solution was placed in ultrasonic apparatus under ultrasonic waves at time intervals of 15, 45, 60 and 90 minutes. The decreasing aggregation size of DR60 dyes was performed before dyeing. After dye preparation, it was placed in the bath.

Adsorption studies

Adsorption experiments were done by contacting different amounts of PTT (0.001-0.1g) with Batch

adsorption experiments were carried out by various amounts of PTT (0.001–0.1 g) to 100 mL DR60 dye prepared by ultrasonic waves at time intervals of 15 - 90 min with initial concentrations of 20 – 60 ppm at temperatures of 60 – 80 °C and different pH (2-8). The initial value of solution pH were adjusted by adding HCl 0.1 N and NaOH 0.1 N solutions in the range of 2 to 8.

The color concentration in the solution was measured by the UV-Vis spectrophotometer before and after the adsorption process. The instantaneous adsorption capacity (QE) and the percentage of color removal (R %) were calculated by the equations 1 and 2.

$$q_e = \frac{(C_0 - C_e)V}{W} \quad (1)$$

$$R\% = \frac{(A_1 - A_2)V}{A_1} \times 100 \quad (2)$$

Where, C_o and C_e are the initial and final concentrations of the solute in each adsorption experiment (mg / L), V is the volume of the solution (L), W is the weight of adsorbent (g), q_e is the sorption capacity (mg/g), A_1 is the initial absorbance of the color solution, A_2 is the final absorbance of the color solution, and $R\%$ is the percentage of color removal [29]. The calibration curve of disperse red 60 is presented in Fig. 1.

RESULTS AND DISCUSSION

Determination of particle size

To confirm the preparation of disperse dye in the Nano scale (Nano-disperse dye), the measurements of particle size was performed by particle size analyzer (DLS). The dye particle size was determined before and after ultra-sonication. According to the results that given in Table 1, the particle size of the dyes after ultrasound operation was obtained in the range of 0-100 nm, which confirms the effectiveness of the Nano-disperse preparation method.

The effect of irradiation time and temperature on the particle size

In order to explorer the role of ultrasonic waves on the particle size of DR60 dye, the ultrasound irradiation at the various time including 15, 45, 60 and 90 minutes, as well as at the different temperatures including 20, 40, 60 and 90 °C were performed. According to the results from Table 1 and Fig. 2, it was founded that by increasing in ultrasonic irradiation time, the particle size of the dye was changed and the smallest particle size for the DR60 dye was achieved at the time of 45 minutes and the temperature of 20 °C.

Table 1: Particle size at different ultrasonic irradiation times for DR60 dye.

Particle size (nm)	Time (min)
201.3	0
193.8	15
77.9	45
183.4	60
191.6	90

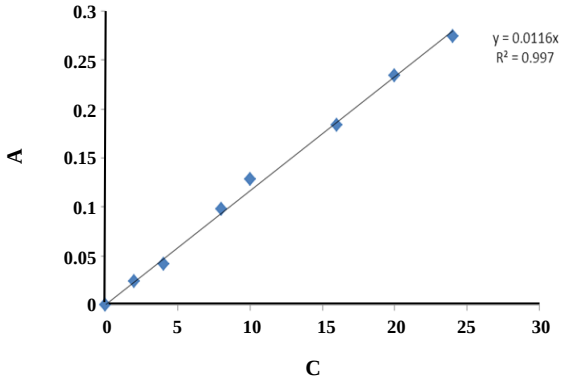


Fig. 1: The calibration curve of disperse red 60.

Optimization of the parameters affecting the adsorption process

Effect of pH

pH is one of the important factors in the adsorption process [30]. The adsorption experiment was performed as a function of different pH from 2 to 8 for the PTT. According to the achieved results, the optimum value for pH were carried out at pH=2.

At higher than pH=6, disperse dye was degraded and the alkaline environment has a negative effect on the adsorbent, resulting in its hydrolysis. The results show that the using of ultrasonic irradiation increases the adsorption, which is due to the loss of dye accumulation, dispersion, and eventually shrinkage of the dye and the increase of penetration of the dye into the adsorbent.

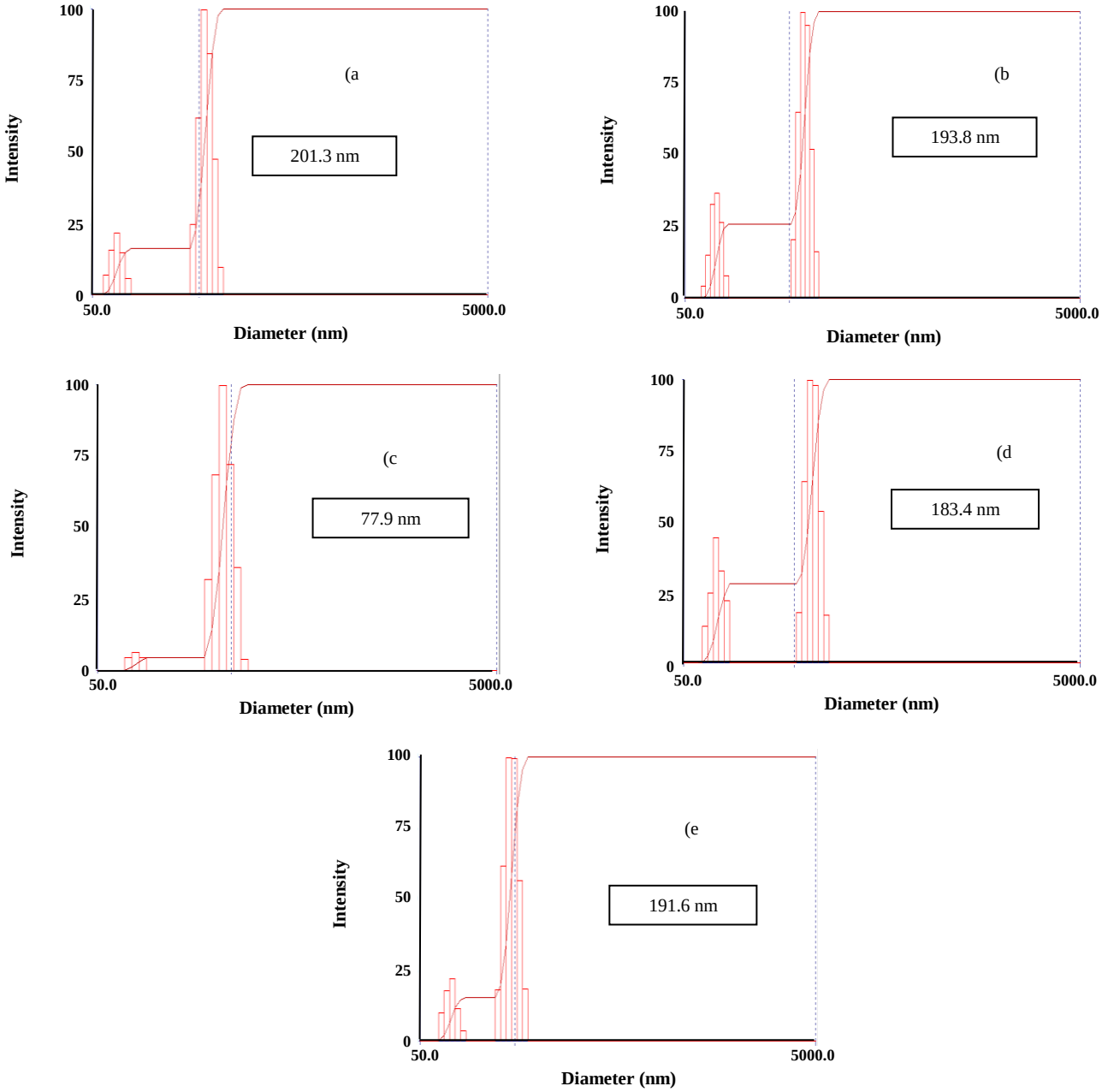


Fig. 2: Particle size diagram based on ultrasonic time a) without ultrasonic, b) with ultrasonic 15 min, c) with ultrasonic 45 min, d) with ultrasonic 60 min, and e) with ultrasonic 90 min

The study of amount of adsorbent

The effect of adsorbent amount for the dye adsorption was evaluated by varying the amount of PTT adsorbent in the range of 0.01–0.15 g. The results (Fig. 4) indicate that the maximum adsorption efficiency under ultrasound irradiation was observed at 0.1 g of sample. For both states (with and without ultrasound irradiation), the adsorption enhancement of dye with increasing the adsorbent dosage was attributed to an increase in the number of available adsorptive sites which favors the increase of the adsorption capacity. This result shows that there is an optimal amount dose of PTT adsorbent for the dye adsorption, which in this case is 0.1 g.

Effect of the contact time

To study the effect of contact time on dye adsorption, the time range of 5 to 180 minutes was applied. As shown

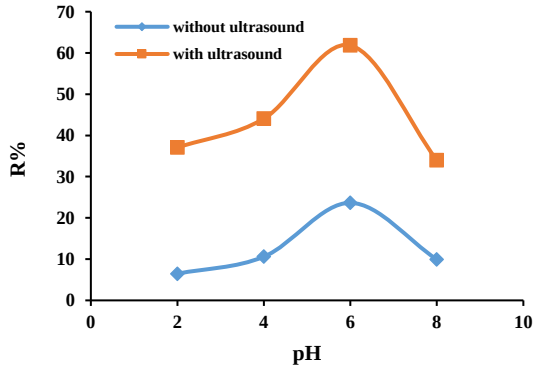


Fig. 3: Effect of pH on the adsorption of DR 60 dye using conventional and ultrasonic conditions on PTT particles (PTT dosage=0.1 g, t=120 min, Concentration of dye=40 ppm, and T=80 °C).

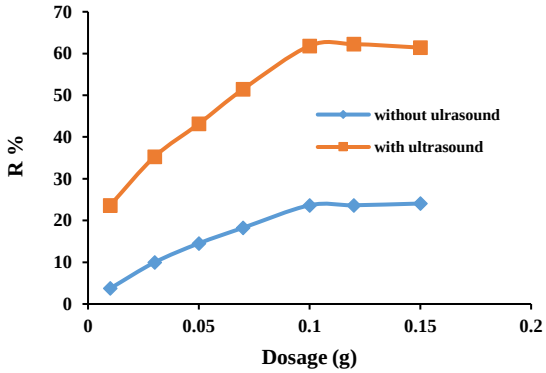


Fig. 4: Effect of amount of adsorbent on adsorption of disperse Red 60 dye using conventional and ultrasonic conditions (pH=6, t=120 min, Concentration of dye=40 ppm, and T=80 °C).

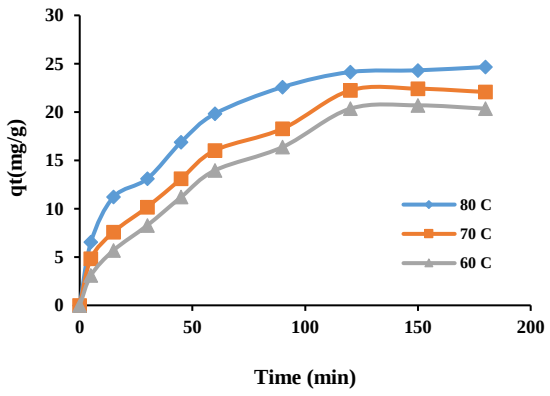


Fig. 5: Effect of contact time on the adsorption of Nano-disperse Red 60 dye on PTT (pH=6, t=120 min, Concentration of dye=40 ppm, and T=80 °C).

in Fig. 5, the optimized time was achieved at t=120 min. This process was carried out at a temperature range of 60, 70, and 80° C. As shown in Fig. 5, with increasing temperature, the adsorption rate also increases, which indicates that the adsorption process is endothermic in nature.

Investigation of adsorption equilibrium studies

Determination of adsorption isotherm

In this section, the Nernst, Freundlich, Langmuir and Temkin equations [29, 31, 32] were used to determine the type of adsorption isotherm. Fig. 6 shows the Nano-DR60 adsorption isotherm on PTT. According to the results, the Nernst equation has the highest correlation coefficient. By plotting the graph C, in terms of q, the slope value of the adsorption isotherm (k) was obtained.

The linear form of Langmuir, Freundlich, Temkin, and Nernst isotherms are defined by the equations 3, 4, 5, and 6 respectively:

$$\frac{C_e}{q_e} = \frac{1}{k_d q_{max}} + \frac{C_e}{q_{max}} \quad (3)$$

$$\ln(q_e) = \ln K_F + \frac{1}{n}\ln C_e \quad (4)$$

$$q_e = B \ln K_t + B \ln C_e \quad (5)$$

$$q_e = kC_e \quad (6)$$

where in general, q_e is the amount of solute adsorbed per unit weight of sorbent at equilibrium (mg/g), C_e is the equilibrium concentration of solute in the bulk solution (mg/L), q_{max} corresponds to complete coverage of available sites or the maximum amount of nitrate RB5 adsorbed per unit weight of sorbent (mg/g), and k_d (L/mg) is the equilibrium adsorption constant of the Langmuir isotherm, n and K_F (mg/g) are related to adsorption intensity and adsorption capacity of the Frendlich isotherm, B and Kt (L/g) are the Temkin constants representing the heat of adsorption and the maximum binding energy, respectively.

Determination of adsorption kinetics

In order to determine the adsorption kinetics, the pseudo-first-order and pseudo-second-order kinetic equations were used to examine the kinetic data. The nonlinear and linear pseudo-first-order and pseudo-second-order equations is generally expressed in the Table 2 where q_e and q_t are the amounts of dye

Table 2: Pseudo-first and second-order adsorption kinetic equations.

Type of adsorption kinetic equation	Linear equation	Non-linear equation
Pseudo-first-order	$\ln (q_e - q_t) = \ln q_e - k_1$	$\frac{dqt}{dt} = k_1 (q_e - q_t)$
Pseudo-second-order	$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$	$\frac{dqt}{dt} = k_1 (q_e - q_t)^2$

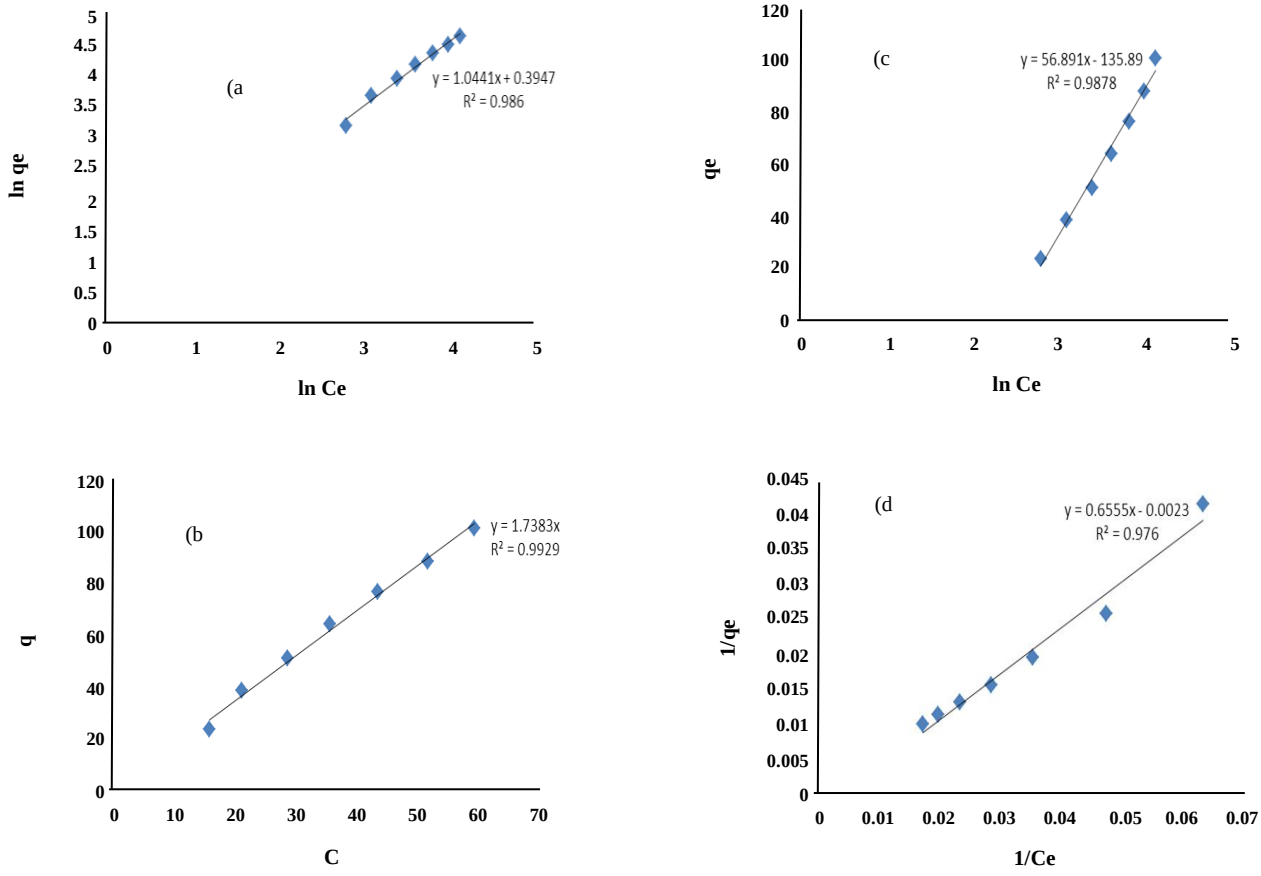


Fig. 6: Linear diagram (a) Freundlich (b) Nernst (c) Temkin (d) Langmuir Adsorption Isotherms.

adsorbed (mg/g) at equilibrium and any time during the adsorption process time *t*, respectively and k_1 and k_2 is the rate constants [29, 31-33].

As indicated in Fig. 7, and achieved data from Table 3, the correlation coefficients for the pseudo-second-order kinetic model were higher than those pseudo-first-order to dye adsorption from aqueous solution. On the other hand, it can be seen that the pseudo-second-order kinetics model suggests a good correlation for the adsorption of dye.

Determination of thermodynamic parameters of absorption

In order to the study feasibility, heat change, and spontaneous nature of the dye adsorption process under ultrasonic irradiation on PTT, determination of thermodynamic parameters including the changes in the standard free energy (ΔG°), standard enthalpy (ΔH°) and standard entropy, and (ΔS°) has been evaluated. The ΔG° thermodynamics relationship for the adsorption process was calculated from the following equation [34]:

$$\Delta G^0 = -RTLn(K_d) \quad (7)$$

$$G^0 = \Delta H^0 + T\Delta S^0 \quad (8)$$

The calculations of ΔH° and ΔS° have been evaluated from the relationship between the adsorption equilibrium

Table 3: The coefficients of pseudo-first and second-order adsorption kinetic equations.

Pseudo-second-order			Pseudo-first-order		
K_2	q_e	R^2	K_1	q_e	R^2
1.421	28.248	0.9926	0.0283	22.576	0.9761

Table 4: Thermodynamic Parameters of Nano-disperse Red 60 dye Adsorption by PTT Polymer.

Temperature (K)	K(l/mol)	ΔG(kj/mol)	ΔH(kj/mol)	ΔS(j/mol k)
333.15	1.36046	-0.85224	11.32	36.51
343.15	1.50617	-1.16798		
353.15	1.71570	-1.58432		

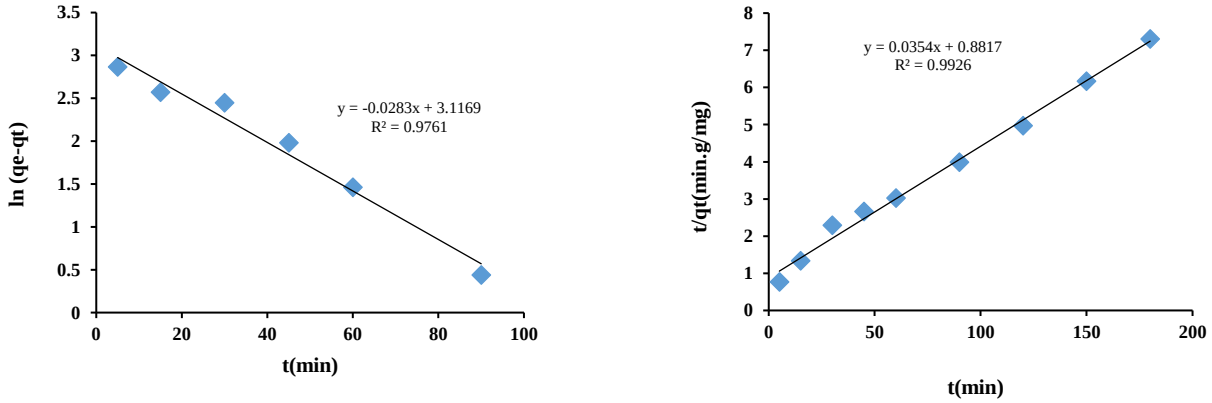


Fig. 7: The diagram of (a) Pseudo-first-order adsorption kinetics (b) Pseudo-second-order adsorption kinetics.

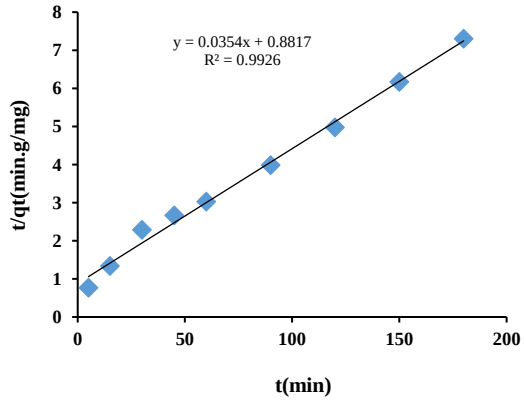


Fig. 8: Linear diagram of lnkd vs. 1/T for determining of thermodynamic parameters

constant at different temperatures by van't Hoff equation [34,35]:

$$\mathrm{Ln}(k_d) = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \qquad (9)$$

Where R is the gas constant (8.314 j/mol.K), T is the temperature (K), and K_d is the ratio of the dye adsorbed by the adsorbent to the remaining dye in the solution. The slope and intercept of the line produced by drawing Ln(K_d) versus 1/T were attributed to the ΔS° and ΔH° of sorption. The thermodynamic parameters are presented in Table 4. The negative values of the ΔG° at three temperatures indicate the possibility and spontaneous quiddity of the adsorption process of DR60 on PTT absorbent.

CONCLUSIONS

According to our achieved results, it was found that the accumulation of color molecules and the adsorption studies are affected by ultrasonic irradiation. Firstly, the effective parameters in the adsorption process such as pH, adsorbent amount, contact time, temperature, and dye concentration were optimized, and then the adsorption isotherms, adsorption kinetics, and thermodynamics of adsorption were studied. The results show that under

ultrasonic irradiation the dye particles tend to the smaller size and are defined as nanoscale particles. Our finding shows the highest fitting was achieved for Nernst's adsorption isotherm model. From the thermodynamic parameters, it was found that the free Gibbs energy is negative, which indicated that the process is endothermic in nature Also, the positive value of ΔS° can be attributed to the increases of randomness at the solid-solution interface during the adsorption process of dye to PTT. Finally, it was shown with a general overview that when ultrasonic irradiation is applied, the size of the dye particles is decreased and due to the penetration of the dyes to fiber layers is increased and the enhancement of the adsorption process was observed. In comparison with a previous study, the PTT fibers were dyeing with DR60 dye under ultrasonic waves and the penetration of the color in the fiber was increased [14].

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