

A Comparative Study for Reactive (Blue 19) Dye Adsorption Onto Polyaniline Prepared by Both Aps and Catalyzed H₂O₂ Pathways.

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IN THE present study, polyaniline (PANI), chemically synthesized using ammonium peroxydisulfate [APS] and by catalyzed hydrogen peroxide (H₂O₂), was evaluated as adsorbents for reactive (blue 19) dye. Experiments were conducted in batch mode where some parameters affecting the adsorption capacity such as contact time and concentration of dye have been investigated. An atomic absorption spectroscopy method was used for the measurement of dye contents. Adsorption equilibrium was achieved within 60 min and the adsorption capacity (q_e) was 350 mg /gm (PANI) under the optimal conditions. Adsorption equilibrium studies were carried out and showed that adsorption follows Freundlich model. The adsorption kinetics was best described by pseudo-second-order model. The results indicated that PANI, synthesized by catalyzed hydrogen peroxide (H₂O₂) can be used as a novel effective adsorbent material for the removal of the tested reactive dye with higher adsorption capacity than PANI prepared by APS.

Keywords: Conductive polymers, Polyaniline, Reactive (blue 19) dye, Sonochemical polymerization, Dye adsorption

Polyaniline is the most important and widely used type of electrically conducting polymers which has shown good stability along with simplicity of preparation⁽¹⁾. Polyaniline (PANI) exists in a variety of forms that differ in chemical and physical properties. The most common green protonated emeraldine has conductivity on a semiconductor level of the order of 1-10 S/cm which is many orders of magnitude higher than that of common polymers (<10⁻⁹ S/cm). Protonated PANI, (e.g., PANI hydrochloride) converts to a nonconducting blue emeraldine base when treated with ammonium hydroxide⁽²⁾. As shown in Fig. 1, the common synthetic process of PANI depends on oxidation of aniline hydrochloride (solution of aniline in hydrochloric acid) with APS to yield conducting polyaniline (emeraldine salt), see Fig. 2⁽³⁻⁹⁾. APS is a strong oxidant, and the polymerization of aniline is an exothermic reaction, so the reaction heat is difficult to control, leading to an agglomeration of the product and wide molecular weight distribution which worsen the mechanical properties of PANI and reduce chelating activity on its surface.^(9,10)

In our previous studies we have reported the synthesis and characterization of PANI and its derivatives via chemical oxidation pathway using APS. The study included doping agent modification ^(11,12), synthesis of PANI composites with different materials for specific applications as corrosion inhibitor for paint applications ⁽¹³⁻¹⁵⁾, antioxidant additives for rubber industry ^(16,17) and conducting nano-composites ^(18,19).

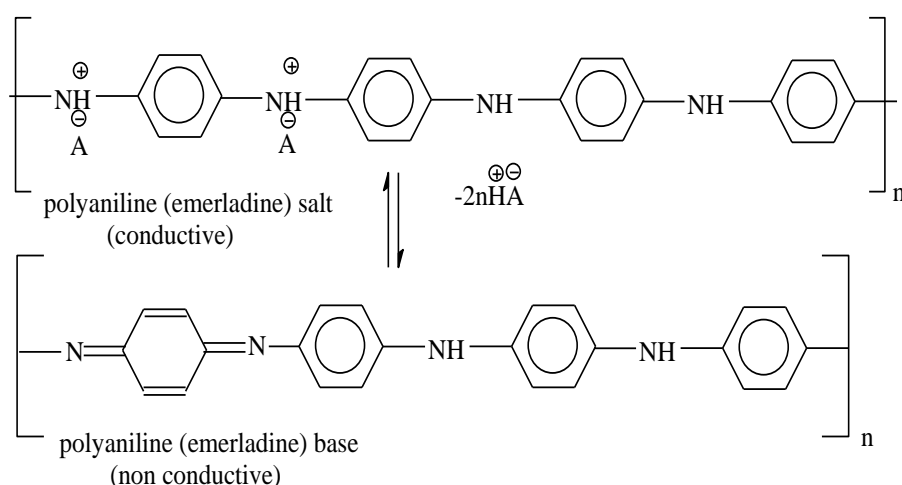


Fig. 1. Different forms of polyaniline Source: (J. STEJSKAL *et al* [6].)

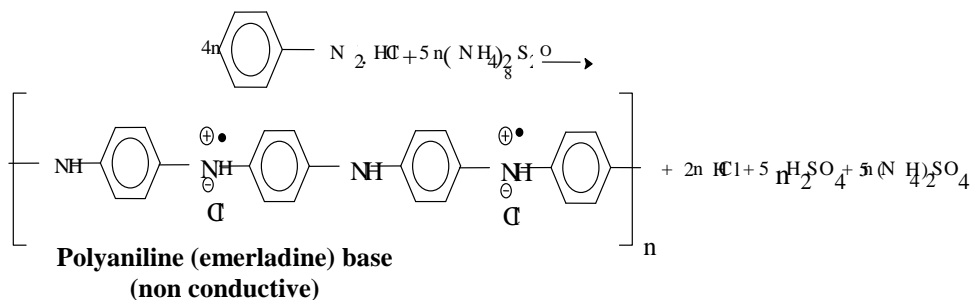


Fig. 2. Oxidation of aniline hydrochloride with APS Source: (J. STEJSKAL *et al* [7].)

H₂O₂ has been reported as an excellent oxidant for aniline polymerization because it enhances the particle size distribution and morphology of polyaniline and yields water as byproduct which greatly simplifies the industrial process and enables recycling of reaction medium because it does not contain any harmful components to aniline polymerization⁽²⁰⁾. But when H₂O₂ alone is used as oxidant, the reaction rate is extremely low. In order to increase the rate of *Egypt. J. Text. Polym. Sci. Technol.* **14**, No. 2 (2010)

reaction, the reaction may be catalyzed by using of ultrasonic waves and slight ratios of FeCl_3 catalyst which drastically increase the yield and rate of reaction to acceptable value for applications⁽²⁰⁻²⁵⁾. Recently, “polyaniline” and its composites have become under investigation as exciting new absorbing materials for water contaminants like dyes and toxic heavy metals due to the active sites on PANI surface⁽²⁶⁻³⁰⁾.

The ultimate goal of this study is to investigate and compare the effect of PANI prepared by both chemical oxidations using APS as well as H_2O_2 catalyzed by ultrasonic waves and FeCl_3 on the adsorption potency of reactive (blue 19) dye

Experimental

Materials

Commercial grade aniline ($\text{C}_6\text{H}_5\text{NH}_2$) [Adwic Co. Egypt] was purified by distillation at 180°C . Commercial Reactive (blue 19) dye ($\text{C}_{23}\text{H}_{13}\text{ClN}_8\text{Na}_2\text{O}_{10}\text{S}_2$) was supplied by Al-Alamia Co. for Modern Weaving and Textiles, Egypt. Ammonium peroxydisulfate [$(\text{NH}_4)_2\text{S}_2\text{O}_8$] and Dimethyl formamide (DMF), were purchased from Aldrich-Sigma Company, Germany, and used as received. Analytical grades of H_2O_2 (30%) and HCl (36%) were purchased from Adwic Co. Egypt. FeCl_3 salt was purchased from BDH Chemical Ltd, England.

Methods

Preparation of PANI

Two samples of polyaniline were prepared: (P1) for PANI prepared using APS as initiator and (P2) for PANI prepared by using catalyzed H_2O_2 as initiator according to the following methods:-

Preparation of (P1): 5 ml (0.55mol) of freshly distilled aniline was dissolved in 100 ml of 1M HCl solution and stirred for 10 min to ensure the homogeneity of the mixture. 12.55 gm (0.55 mol) of APS salt (Aniline : APS; 1:1 molar ratio) was dissolved in 100 ml distilled water and added dropwise to the aniline – HCl solution while stirring at room temperature for 1 hr The precipitated dark green powder of PANI was filtered and washed by hot bidistilled water and acetone till the filtrate becomes colorless. The washed precipitate was dried at 60°C overnight and ground to fine powder.

Preparation of (P2): 5 ml (0.55mol) of freshly distilled aniline was dissolved in 100 ml of 1M HCl solution and stirred for 10 min Droplets of FeCl_3 solution as catalyst was added to the solution followed by droplets of H_2O_2 solution (30%) for reaction initiation where the molar ratio of aniline: H_2O_2 : FeCl_3 is equal to 1:1:0.002 respectively. Then the reaction mixture was sonicated for 3 hr in ultrasonic bath under frequency of 30 kHz. Finally the reaction product was collected and washed as previously described in (P1).

Adsorption studies

Adsorption experiments were performed in batch equilibrium mode. All experiments were carried out by placing 0.05 g (P1) and (P2) into two conical flasks, each has 50 ml of dye solutions. The mixtures were agitated at 200 rpm at room temperature for different contact time and dye concentration. Then the solution was separated by filtration and a sample of the filtrate was analyzed to determine the equilibrium concentration of dye after adsorption. The adsorbed amounts of dye were calculated using the equation:

$$q_e = (C_o - C_e) V/m \quad (1)$$

Where C_o and C_e are the initial and equilibrium concentrations of dye (mg /L), m is the mass of PANI (g), and V is the volume of solution (L).

Characterization

-Molecular weight (M_w) of PANI was measured by gel permeation chromatography (GPC) technique using polystyrene basis [Model: Infinity-1220, Agilent, Germany] and Dimethyl Formamide (DMF) as solvent for PANI. To ensure complete solubility of PANI in DMF, samples were firstly transformed to non conductive emeraldine base form by stirring in 1 M NH_4OH for 1 hr, then washed by distilled water and dried at 60 °C overnight ⁽⁹⁾.

- Electrical conductivity of the PANI was measured on pressed pellets employing a standard two-probe method [Model: HY3020E- MASTECH DC POWER SUPPLY, USA].

-Particle size and morphology were evaluated by transmission electron microscope (TEM) [Model EM10; Zeiss, Germany].

- Equilibrium dye concentrations were analyzed by spectrophotometer [Model: HACH (DR2800)] at 540 nm maximum wavelength for Blue 19 reactive dye as resulted from experimental work.

Results and Discussion*Characteristics of PANI*

Table 1 shows a comparison between molecular weight, DC conductivity for (P1) and (P2). The results showed that PANI which was prepared by catalyzed H_2O_2 (P2) has higher molecular weight, lower polydispersity index (D) and higher DC conductivity than that prepared using APS (P1). The higher molecular weight of (P2) may be attributed to slower reaction rate of catalyzed H_2O_2 than APS as initiator which delays and reduces the number of free radicals in the initiation step compared to propagation of polymer chain ⁽²⁶⁾

DC conductivity of (P2) was increased by the catalytic effect of $FeCl_3$ combined with ultrasonic waves as it enhances the doping process of PANI prepared by H_2O_2 ^(27, 28). The addition of $FeCl_3$ in presence of ultrasonic waves

lowers the formation of by-products during the polymerization reaction and increases the polarity of PANI due to good distribution of Fe³⁺ ions in the medium by the effect of ultrasonic waves^(29, 30). The morphological properties of the prepared polyaniline were clarified via TEM analysis as shown in Fig. 3. It is clearly seen that (P2) has a uniform nano-porous structure in reverse to (P1) which has a non uniform particle size due to particles agglomeration during the aniline polymerization with APS. The nano porous structure of (P2) may be attributed to the effect of micro cavities which are generated and collapsed during ultrasonic waves in solution, Fig. 4. The collapse of cavities yields severe conditions of pressure and temperature (1000 bar, 3000 K) in thousands of hot spots inside the solution without affecting the overall conditions of solution systems⁽³⁰⁾.

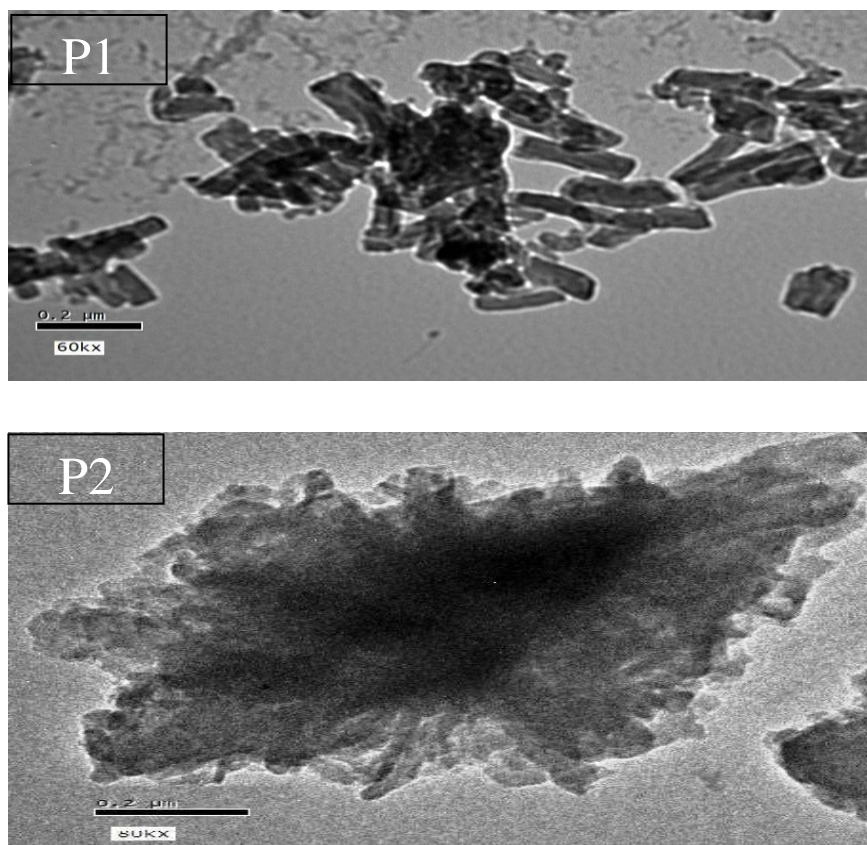


Fig. 3. TEM images for PANI prepared by APS (P1) and catalyzed H₂O₂ (P2) .

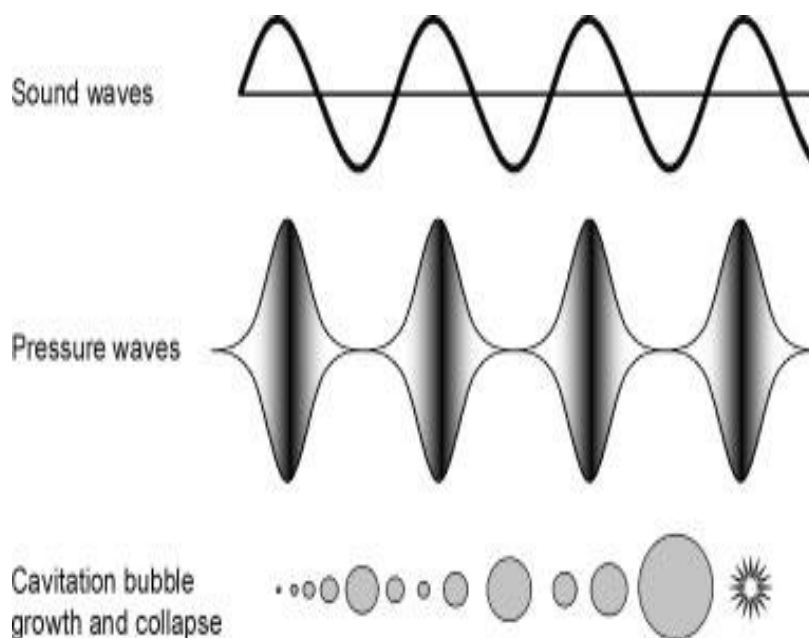


Fig. 4. The growth and collapse of cavitations bubbles. (Source: H.F. Mark *et al.* [23]).

TABLE 1. Characteristics of PANI prepared by different methods .

	(P1)	(P2)
Average Molecular weight (M_w)	40650	62487
Number Molecular weight (M_n)	34475	59164
Polydispersity Index (D)	1.17	1.05
DC conductivity (σ) [S/cm]	1.12	1.57

Adsorption studies

Determination of optimum adsorption time

The adsorption kinetics was conducted to determine the optimum adsorption time for the adsorption of dye onto synthesized PANI. Fig. 5 shows the effect of contact time on the adsorption of dye onto (P1) and (P2). The adsorption capacity increases rapidly during the initial adsorption stage and then continues at a relatively slow speed reaching equilibrium stage after 60 min. The results show firstly that, the adsorption occurs on the surface of PANI, so the adsorption rate is fast. After the surface is saturated, the adsorption gradually goes through

the inner part of PANI via the diffusion of dye onto the polymer matrix where the repulsion between adsorbate molecules, leading to a lower rate^(28, 29). It is also clearly seen that, the adsorption rate for (P2) is higher than (P1) due to the porous structure of the particles (P2) as previously revealed in TEM analysis.

Determination of maximum adsorption capacity (q_m)

Figure 6 compares the adsorption isotherms of dye onto (P1) and (P2). The results show that the adsorption capacity of dye increases with concentration till it reaches a maximum value (q_m) which does not change by further increase in concentration. This may be attributed to the extent of a driving force of concentration gradients with the increase of dye concentration. The adsorption isotherm shows that the maximum adsorption capacity for (P1) was about 250 mg/g while it increased to about 350 mg/g for (P2). This could be attributed to the increasing of molecular weight and doping efficiency of polymer, which increase the length of chain and number of active sites thereby enhancing the conductivity and binding effect of polymer surface. This interpretation is in accordance with Maloney *et al.* assumption⁽³⁰⁾ who reported that adsorption capacity of polyaniline is mainly depending on the number of active sites on the polymer chain (amine and imine groups), the surface area of the polymer and extent of interaction with dye molecules.

Kinetics modeling study

The study of adsorption dynamics describes the solute uptake which controls the residence time of adsorbate uptake at the solid/solution interface. In this section, the results of the kinetics of dye ions adsorbed from aqueous solutions onto prepared polyaniline by different methods, as illustrated in Fig. 5 were analyzed using pseudo first-order, pseudo second-order and Elovich models, respectively as follow⁽³¹⁾:

i) Pseudo-first-order equation:

$$\text{Log}(q_e - q_t) = \text{Log} q_e - (k_1) (t) / (2.303) \quad (2)$$

ii) Intraparticle diffusion equation:

$$q_t = k_i t^{0.5} + C \quad (4)$$

iii) Elovich's equation:

$$q_t = b + a \ln t \quad (5)$$

Where q_e and q_t are the amounts of dye adsorbed (mg/l) at equilibrium and at time t (min), and t is the adsorption time (min). The other parameters are different kinetic constants, which can be determined by regression of the experimental data. The validities of these four kinetic models are checked and depicted in Fig. 7. The corresponding kinetic parameters and the correlation coefficients are summarized in Table 2. The conformity between experimental data and each model predicted values was expressed by the correlation coefficient (R^2). A relatively high R^2 values indicates that the model successfully describes the kinetics of metal ion sorption.

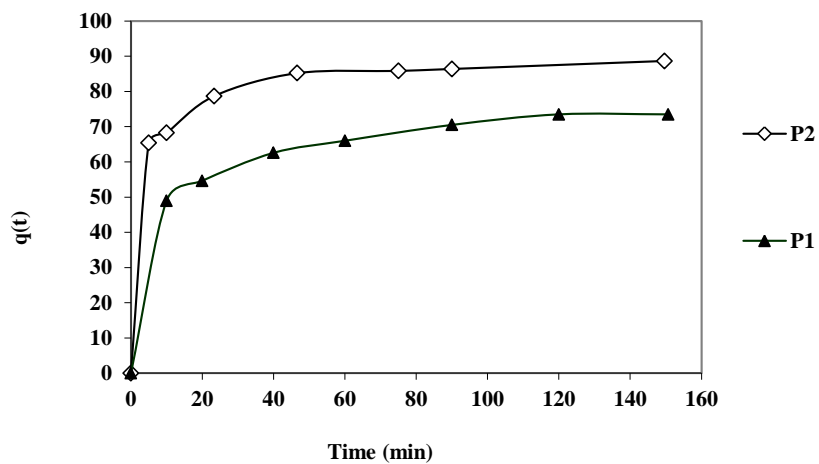


Fig. 5. Reactive blue 19 adsorption kinetics on polyaniline prepared by different methods, $C_e = 100$ mg/lit .

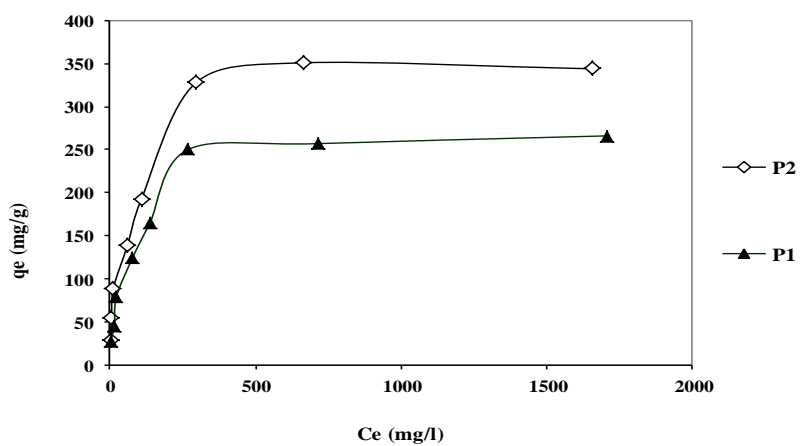


Fig. 6. Adsorption isotherm for blue K-BR dye at polyaniline prepared at different conditions

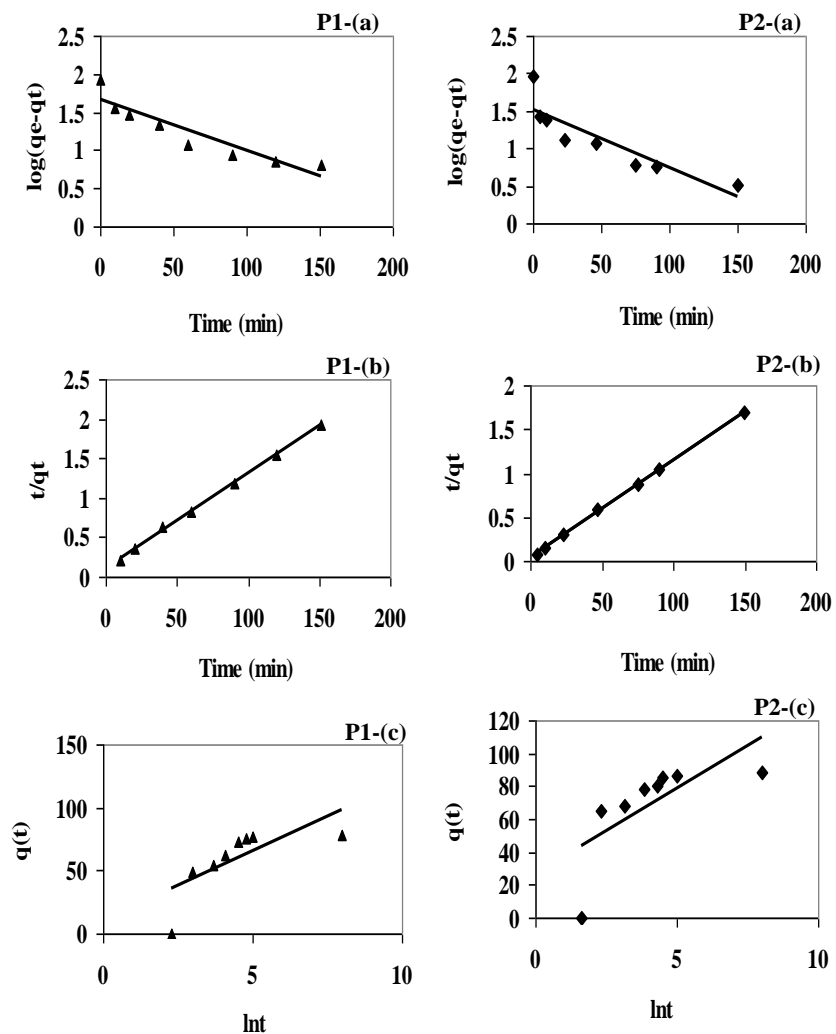


Fig. 7. Adsorption kinetics of Blue 19 reactive dye onto PANI prepared by APS (P1) and catalyzed H₂O₂ (P2): (a) pseudo-first-order, (b) pseudo-second-order, (C) Elovich models.

TABLE 2. Kinetics parameters for reactive (blue 19) dye adsorption on PANI prepared by different methods .

	(P1)	(P2)
Pseudo-first-order		
k_1 (min ⁻¹)	0.015	0.018
$q_{e,cal}$ (mg g ⁻¹)	40	33.01
$q_{e,exp}$ (mg g ⁻¹)	65	85
R^2	0.81	0.78
Pseudo-second-order		
k_2 (gmg ⁻¹ min ⁻¹)	0.00125	0.00318
$q_{e,cal}$ (mg g ⁻¹)	70.333	90.09
R^2	0.9987	0.9994
Elovich		
a	9.597	10.44
B	9.597	26.47
R^2	0.529	0.487

Based on linear regression (R^2) values, the adsorption kinetics of dye onto (P1) and (P2) could be described well (R^2 near to 1) by pseudo-second order model where the $q_{e,cal}$ value from pseudo-second order model agrees very well with the $q_{e,exp}$ value which indicates the high adsorption capacity for (P2) and (P1). Also the over all rate constant of each sorbent appears to be controlled by physical adsorption interaction on PANI surface as indicated by low linear regression (R^2) value of Elovich model.

Adsorption isotherm models study

Langmuir, Freundlich, and Temkin models were selected to investigate and compare the adsorption behavior of dye onto (P1) and (P2) polymers. The Langmuir isotherm is commonly used model for monolayer adsorption process where it assumes that the adsorption occurs at specific homogeneous sites on the adsorbent as represented by equation (6)⁽³¹⁾:

$$C_e/q_e = 1/bq_m + C_e/q_m \quad (6)$$

Where the constant b is related to the energy of adsorption (L/mg) and q_m is the Langmuir monomolecular adsorption capacity (mg/g). The value of constant b is also used for determination of dimensionless constant (R_L) according to equation (7):

$$R_L = 1 / (1 + bC_0) \quad (7)$$

Where C_0 (mg/L) is the initial dye concentration. From equation (7), the adsorption is favorable in the values of R_L lies between 0 and 1.

The Freundlich isotherm⁽³¹⁾ describes the multilayer physical adsorption systems and assumes that the adsorption process takes place on heterogeneous

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surfaces and adsorption capacity is related to the concentration of dye at equilibrium according to equation (8)⁽²²⁾:

$$\text{Log } q_e = \text{log } K_f + (1/n) \text{log } C_e \quad (8)$$

Where K_f (mg/g) is roughly an indicator of the adsorption capacity and $1/n$ is the adsorption intensity.

The Temkin adsorption model [equation (9)], considers the chemisorption of an adsorbate onto the adsorbent. This model assumes that the adsorption is characterized by a uniform distribution of binding energies related to chemical interaction on the surface⁽³²⁾:

$$q_e = A + B \text{log } C_e \quad (9)$$

Where A and B are Termkin constants.

Figure 8 shows the application of the mentioned models in dye adsorption onto (P1) and (P2) polymers. Table 3 summarizes linear regression (R^2) values and the other models parameters. The data illustrated in table 3, show that the adsorption isotherms agree well with Freundlich model for both (P1) and (P2) even at maximum adsorption capacity (q_m). In the other hand, it does not verified by Termkin as well as Elovich model which confirm that interaction between dye and polyaniline is depending on physical binding with imines and amine groups on PANI surface. The values of R_L lie between 0 and 1, indicating that the adsorption is favorable. Also, the high linear regression (R^2) for Freundlich isotherm model indicates the heterogeneous and porosity of PANI surface especially for (P2) due to the formation of multilayers of adsorbed dye.

Conclusion

Polyaniline is proved to be a new promising adsorbent for Reactive (blue 19) dye. PANI prepared by hydrogen peroxide (H_2O_2) in presence of ultrasonic waves and $FeCl_3$ as catalyst has higher removal efficiency than PANI which was prepared by the classical chemical oxidation using APS initiator. The kinetics study revealed that the adsorption follows pseudo second order model where the equilibrium was achieved after 60 min. The maximum adsorption capacity was 350 mg/g for PANI prepared by catalyzed hydrogen peroxide and 250 mg/g for PANI prepared by APS. Adsorption isotherms models revealed that the adsorption follows Freundlich model, which suggests physical sorption nature of interaction between PANI and reactive dye.

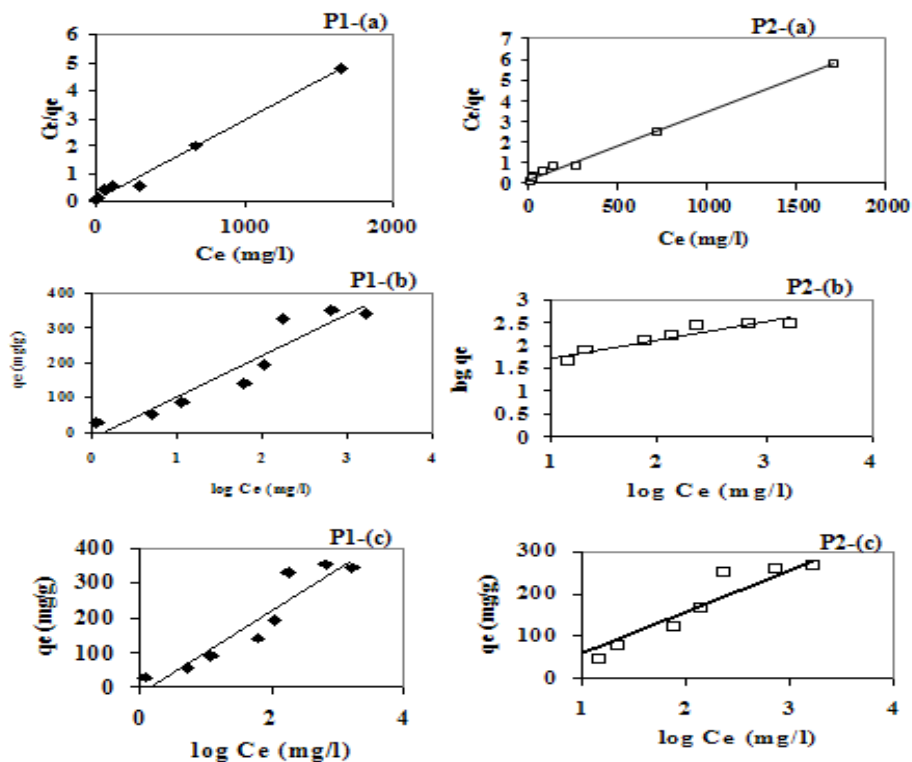


Fig. 8. Adsorption isotherm models of Blue 19 reactive dye onto PANI prepared by catalyzed H_2O_2 (P2) and APS (P1): (a) Langmuir, (b) Freundlich, (c) Temkin.

TABLE 3. Adsorption isotherms parameters for reactive (blue 19) dye adsorption on PANI prepared by different methods .

	(P1)	(P2)
Langmuir		
q_m (calc) (mg/g)	303.03	370.143
b (L/mg)	0.0157	0.0333
R_L	0.11309	0.05673
R^2	0.902	0.92
Freundlich		
n	2.81136	2.55885
K_F (mg/g)	31.952144	19.961814
R^2	0.964	0.982
Temkin		
A (L/g)	23.49	-36.4
B	117.4	91.31
R^2	0.6893	0.7258

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قسم الهندسة الكيميائية ، *قسم البلمرات والمخصلات - المركز القومي للبحوث
و**قسم الهندسة الكيميائية - كلية الهندسة - جامعة القاهرة - القاهرة - مصر .

تهدف الدراسة إلي إدمصاص الصبغة الزرقاء من النوع النشط (Reactive blue 19) بواسطة نوعين مختلفين من البولي أنيلين تم تحضير إحداهما بواسطة بيكرينات الأمونيوم كإحدى لعملية البلمرة بينما الأخر تم تحضيره بواسطة فوق أكسيد الهيدروجين المحفز بكلوريد الحديدك والموجات فوق الصوتية. وقد تم إجراء التجارب بتقليب البوليمر مع محلول الصبغة في تفاعل مغلق (Batch mode) وتم دراسة العوامل المؤثرة علي كفاءة الإدمصاص وتشمل زمن التفاعل وتركيز محلول الصبغة. و قد أظهرت الدراسة أن عملية إدمصاص الصبغة تصل إلي مرحلة الاتزان خلال ٦٠ دقيقة من بدأ التقليب وكفاءة إدمصاص تصل إلي ٣٥٠ جم من الصبغة لكل جرام من البولي أنيلين وذلك عند أحسن ظروف للتشغيل باستخدام البوليمر المحضر بواسطة فوق أكسيد الهيدروجين المحفز. و قد أجريت أيضا دراسة لنماذج الحركة والاتزان التي تحكم عملية الإدمصاص حيث ثبت أنها تخضع لقوانين الحركة من الدرجة الثانية ونموذج فريندلش للاتزان. و بمقارنة النتائج بالدراسات السابقة في هذا المجال تبين أن البولي أنيلين (المحضر بواسطة فوق أكسيد الهيدروجين المحفز) يصلح كمادة اقتصادية عالية الكفاءة لإدمصاص الصبغات الصناعية النشطة في معالجة مياه الصرف.