

[Research]

Removal of brilliant blue pollution from the environment using nano polyaniline hazelnut skin composite and evaluation of effective parameters

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ABSTRACT

The entrance of household wastewater to rivers has not only brought about numerous environmental problems, which have resulted in a huge problem in the society, but also led to undesirable mental and psychological effects in the society. The removal of dye from these wastewaters before entrance to rivers is of great importance. In this research, it was discovered that by depositing a layer of polyaniline-based conductive nano polymer on hazelnut skin, a nano composite was produced which helps in removing brilliant blue (BB) dye from aqueous environments. The aim of this study was to deposit a layer of polyaniline nano polymer on the surface of hazelnut skin and produce polyaniline/hazelnut skin (Pan/Hz) composite through chemical polymerization of aniline in aqueous environments and adsorption of BB dye from aqueous environments. In this stage, polyaniline was synthesized using ammonium persulfate. In order to find the optimal conditions for the removal of the dye, the effect of a number of important parameters such as pH, contact time, initial concentration of the dye, and temperature was examined. Langmuir and Freundlich isotherms were used for adsorption studies. Furthermore, thermodynamic and kinetic studies were also conducted. This dye has the greatest adsorption at pH 2 and by optimizing other mentioned parameters, the best state has been presented for maximum adsorption. Thermodynamic studies indicate that the adsorption has been spontaneous, endothermic, and chemical, following the second order kinetics. The introduced adsorbent is a very effective one for the adsorption of BB and by using a layer of conductive nano polymer on hazelnut skin, it is possible to absorb BB dye from aqueous environments with a very high percentage.

Key words: Pollution, Nano hazelnut skin composite, Brilliant blue, Polyaniline, Environment.

INTRODUCTION

Colors are the first known contaminants in wastewaters. There are over 100,000 types of dye in the world, such that 7×10^5 tons of these dyes are produced by textile industries every year (Yu *et al.* 2017). Chemical and biological treatment of wastewaters containing these substances is difficult due to low level of adsorption and also chemical stability of dyes. For treatment of dye-containing wastewaters, nonconventional methods are usually used, including adsorption of these compounds by different adsorbents (Ncibi *et al.* 2007). Synthetic dyes include Azoaromatic groups that cause cancer, development of genetic

mutations, etc. (Ghanizadeh & Asgari 2009). The complex aromatic structure of dyes causes physiochemical, thermal, and optical stability, and in turn resistance to conventional purging of wastewaters (Vimonses *et al.* 2009). Several methods have been employed so far for the removal of dye contaminants (Crini 2006). These methods can be divided into three groups of biological, chemical, and physical (Robinson, McMullan, Marchant & Nigam 2001). Due to high cost and unavailability, many of these methods are not used for wastewater treatment (Ghoreishi & Haghighi 2003). Among synthetic polymer adsorbents, conductive polymers are the best. These

polymers have a size of nanometer scale in one of their dimensions (out of three dimensions) and have led to the use of new techniques in the production of efficient adsorbents for removal of different contaminants from the environment. These substances have had great applications for the removal of contaminants from industrial wastewaters due to their

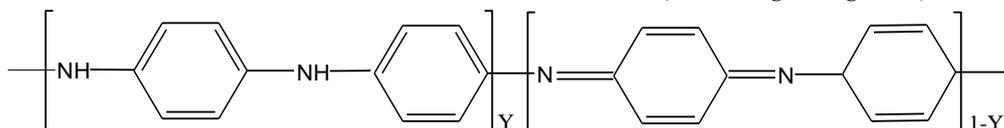


Fig. 1. The general structure of polyaniline, Y is the reduction factor and $0 \leq Y \leq 1$.

Y constantly varies between 0 for a fully oxidized form to 1 for a fully reduced form. Polyaniline, which is a kind of organic metal, can be easily prepared from inexpensive monomers such as aniline through simple chemical or electrochemical methods. Ammonium persulfate is a very suitable oxidant for chemical oxidation of aniline and its conversion to a conductive polymer. The application of conductive nano polymers including polyaniline is constantly growing. Mcdiarmid together with two of his colleagues, Shirakawa and Heger, won a Nobel Prize of chemistry in 2000. This can be ascribed to their

mechanical strength, surface area, and high adsorption capacity. Among them are polystyrene and nonconductive ester polyacrylic along with conductive polyaniline and polypyrrole. As a conductive polymer, polyaniline can be referred to as a group of conductive polymers that have the following formula (Yao-dong Liang 2018).

extensive research on polyaniline (Yu *et al.* 2017). Due to the insolubility of polyaniline in water, for its immersion, a layer of polymer is deposited on a bed of inert materials such as sawdust and agricultural wastes. In this research, hazelnut skin was used as an inert one. One of the most commonly used dyes is brilliant blue FCF (BB) dye. Its closed formula is $C_{37}H_{34}N_2Na_2O_9S_3$ and has disodium salt. Occasionally, it consists of calcium and potassium and also has a molecular weight of $792.85 \text{ g mol}^{-1}$ with a maximum adsorption at 628 nm. Its molecular structure is shown in Fig. 2. (Bogdan Tutunaru 2017).

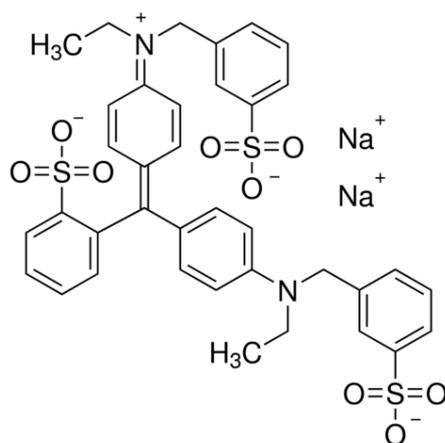


Fig. 2. The molecular structure of Brilliant Blue FCF.

Brilliant blue has been named under the international number of INS = E 133 (International Numbering System). This dye is

a powder with a color of blue to red. It has a maximum solubility in water and is considered an edible color.

If its intake into the human body exceeds 0.1 mg kg⁻¹ of body weight, it can lead to neural damage and also affect the coordination of information and dizziness. Furthermore, when consumed in excess, it can lead to severe toxicity.

In this research, we examined employing polyaniline/hazelnut skin (Pan/Hz) nano polymer adsorbent for removal and adsorption of brilliant blue dye from aqueous environments.

MATERIALS AND METHODS

Materials and instruments

The materials used in this research were purchased from Merck Co. (Germany) with high level of purity. These materials include hydrochloric acid (HCl), sodium hydroxide (NaOH), aniline (C₆H₅NH₂), ammonium persulfate [(NH₄)₂S₂O₈], ethanol (C₂H₅OH), brilliant blue (C₃₇H₃₄N₂Na₂O₉S₃), and hazelnut skin with a mesh size of 35-50. The devices and instruments consisted of pen type pH meter, magnetic stirrer, spectrophotometer JENWAY 6305, heat stirrer AREVELP/SCIENTIFICA, digital balance, and electron microscope VEGA/TESCAN.

Preparation of hazelnut skin coated with polyaniline (Pan/Hz)

A monomer solution (aniline) with a concentration of 0.2 M was brought in contact with 40 g hazelnut skin (prepared from hazelnut garden, Roudsar, Iran) with a mesh size of 35-50 for 5 continuous hours using a stirrer. A 0.4 M ammonium persulfate was added as an oxidant.

After completing addition of the oxidant, the reaction was left undisturbed for about 12 h without stirring and then filtered.

To purify and remove the monomer or extra oxidant, the obtained solid was washed with sufficient amounts of water and then with ethanol. Following complete washing, the obtained product was dried at room temperature. So that, hazelnut skin which was fully coated with a high-strength polyaniline, was prepared to perform the adsorption experiments.

The parameters influencing the extent of adsorption of brilliant blue

The effect of pH

Generally, the pH of a solution is one of the most important factors affecting the capacity of an adsorbent. This is due to the fact that the status of the active sites of the adsorbent is rapidly influenced by changes in the concentration of hydrogen ions present in the environment. To examine the effect of pH on the extent of dye adsorption and to achieve the optimal pH of the dye, the experiments were conducted within the pH range of 2-10. A total of 25 ml of the dye by an initial concentration of 10 mg L⁻¹ was employed at 293 K, followed by adding 1 g of the adsorbent nano composite. Thereafter, their pH was adjusted using diluted solutions of HCl and NaOH (0.1 M). The solution was then stirred using a stirrer for 20 min, and then filtered.

The extent of adsorption was measured using a spectrophotometer and the calibration curve was obtained. The optimal value of pH was also obtained for performing subsequent experiments.

The effect of initial concentration

The concentration of dye solution is considered an essential factor in the speed of occupation of active sites of the adsorbent. Solutions were prepared with different concentrations of 5-25 mg L⁻¹ in relation to the dye at pH = 2 (optimal value). A total of 25 ml of these solutions were placed on a stirrer for the same period of time (20 min) in contact with 1 g of the adsorbent. Then, the solutions of interest were filtered and the solution under the filter was tested to determine the amount of brilliant blue FCF using Langmuir and Freundlich patterns.

The effect of contact time and kinetic study on adsorption

The time required for achieving equilibrium plays an important role in adsorption processes. By obtaining kinetic information, the rate and mechanism of chemical reactions can be understood.

Following the determination of the controlling stage of the process, the kinetic pattern

corresponding to it can be estimated and suitable modifications can be applied to improve the rate of adsorption. At pH 2, 0.5 gram Pan Hz⁻¹ was added to 25 ml solution (10 mg L⁻¹), and then filtered. Both the filtrate and the residue were examined through spectrophotometry at $\lambda_{\max} = 628$ nm for brilliant blue FCF) at intervals of 5, 10, 15, 20, and 25 min. The data associated with the contact time of trial were evaluated along with pseudo-first-order (Eq. 6) and pseudo-second-order (Eq. 7) kinetic equations to achieve the kinetics of adsorption.

The effect of temperature and thermodynamic study of adsorption

Temperature is one of the criteria analyzed to understand the thermodynamics and trend of progression of chemical reactions, including providing useful information about changes in the standard Gibbs free energy (ΔG^0), standard enthalpy (ΔH^0), and standard entropy (ΔS^0) of the system. To examine the effect of temperature on adsorption capacity and to calculate the mentioned variables, 0.5 g of each adsorbent was brought in contact with 25 ml (10 mg L⁻¹) brilliant blue FCF at different temperatures (293, 303, 313, 323, and 333 K). The obtained results were evaluated using Eqs. 8, and 9 (Vant Hoff).

Calculations

To calculate the adsorption percentage and capacity in a batch system, the following relations were used, respectively:

$$\% \text{ Sorption} = \frac{C_0 - C}{C_0} \times 100 \quad (1)$$

$$q_t = \frac{x}{w} = \frac{(C_0 - C_e)}{w} \times V \quad (2)$$

Where C_0 is the initial concentration of the dye and C_e denotes the dye concentration remaining in the solution (mg L⁻¹), x/w shows the amount of material absorbed per unit of the adsorbent weight (mg g⁻¹), w is the value of the adsorbent (g), and V represents the volume of the sample solution (L) (Kakavand *et al.* 2014).

To evaluate the adsorption data, Langmuir (Eq. 3) and Freundlich (Eq. 4) adsorption isotherm equations were used (Ansari *et al.* 2011a).

$$\frac{1}{q_e} = \frac{m}{x} = \frac{1}{x_m} + \frac{1}{x_m b C_e} \quad (3)$$

$$\log q_e = \log k + \frac{1}{n} \log C_e \quad (4)$$

Where q_e is the absorbed body (mg) per gram of the adsorbent, C_e represents equilibrium concentration or the concentration of unadsorbed or residual brilliant blue in the solution (mg L⁻¹), and X_m denotes the maximum acceptable value of adsorption (mg g⁻¹). Parameters b , n , and k are constant. The constant number b (L mg⁻¹) is a criterion for the adsorption energy. The constant numbers k and n in Freundlich relation are considered as criteria representing the intensity of adsorption and capacity of the adsorbent, respectively. Favorable adsorption in terms of Langmuir isotherm can be examined by a constant without any unit which is known as the separation factor or equilibrium parameter R_L :

$$R_L = \frac{1}{1 + K C_i} \quad (5)$$

where, C_i is the initial concentration of the dye (mg L⁻¹). The values of this parameter are categorized into four groups, accounting for the status of adsorption. $R_L > 1$ undesirable, $R_L = 1$ linear, $0 < R_L < 1$ desirable, and $R_L = 0$ irreversible (Ansari & Mosayebzadeh 2010).

For kinetic studies, pseudo-first-order equation (Eq. 6) known as Lagergren and pseudo-second-order (Eq. 7) known as Ho & McKey was used (Ansari *et al.* 2011a).

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303} t \quad (6)$$

In this formula, q_e and q_t represent adsorption capacity at the times of equilibrium and t , respectively. By plotting $\log(q_e - q_t)$ in terms of time, k_1 and q_e values can be obtained. If adsorption kinetics follows pseudo-first-order equation, then it can be concluded that the rate of adsorption process is controlled by inter-

particle diffusion stage (Ho 2004; Ozacar & Sengil 2005).

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (7)$$

Where k_2 is the constant rate of pseudo-second-order equation (g mg min^{-1}), q_e represents the amount of agent absorbed at the time of equilibrium (mg g^{-1}), and q_t denotes the amount of agent absorbed at the time of t . By plotting t/q_t versus t , k_2 and q_e values can be obtained (Kakavand *et al.* 2014). If the adsorption kinetics follows pseudo-second-order equation, it can be claimed that the adsorption process has been conducted chemically, and chemical reaction is considered as the slowing stage of the reaction rate (Sener 2008; Sanchez *et al.* 2010).

To evaluate thermodynamic data, the following equations plus Vant Hoff equation were used (Aravindhana *et al.* 2007).

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

$$\ln K_c = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT}, \quad K_c = \frac{q_e}{c_e} \quad (9)$$

Where K_c is the thermodynamic equilibrium constant, R denotes the general constant of gases (8.314 J/mol k), and T represents the absolute temperature (K). q_e is the adsorption capacity at the time of equilibrium (mg. g^{-1}) and C_e represents the equilibrium concentration of the adsorbent (mg L^{-1}) present in the solution. By plotting $\ln K_c$ versus $1/T$ diagram, the values of changes in enthalpy and entropy can be obtained (Sener 2008; Ansari *et al.* 2011a,b).

RESULTS AND DISCUSSION

Optimal pH

In the present study, it was observed that the best pH for adsorption is 2, while at other pHs, the degree of adsorption will be diminished. The adsorption percentage for Pan Hz⁻¹ declined from 99.30 at pH 2 to 82.87 at pH 10. So that, for the rest of the experiment, pH 2 was used.

By decreasing in pH, which seems to be attributed to the presence of H⁺ ions and

development of a strong electrostatic force between the dye and polyaniline, there was an increase in the extent of adsorption. At strong alkaline pHs, the structure of polyaniline polymer was degraded. Thus at acidic pHs, greater degrees of adsorption are obtained due to preservation of the polymer structure.

Initial concentration and adsorption isotherms

To compare the composite and hazelnut skin, the extent of adsorption by hazelnut (Hz) has also been reported. The increase in the initial concentration of brilliant blue FCF reduced the adsorption percentage, such that adsorption percentage decreased from 92.27 at 5 mg L^{-1} to 75.00 at 25 mg L^{-1} for Hz. Similarly, adsorption percentage decreased from 99.50 at 5 mg L^{-1} to 98.60 at 25 mg L^{-1} for Pan Hz⁻¹.

The obtained results are presented in Table 1 and Fig. 3. R² value of Langmuir isotherm is larger for Hz and Pan Hz⁻¹, suggesting better correspondence of this model with the adsorption process. On the other hand, Pan Hz⁻¹ showed a high correlation coefficient for Freundlich isotherm (R² = 0.9924). Hence, this adsorbent also follows Freundlich isotherm to some extent. As a result of the increase in dye concentration, adsorption percentage was diminished. This is due to the constancy of the adsorbent value, though adsorption capacity or q_t increased, which is attributed to the constancy of denominator and magnification of the numerator q_t . q_m (from Langmuir isotherm) and K_F (from Freundlich isotherm) values, both of which are criteria of adsorption capacity and are far larger for Pan Hz⁻¹ confirming the superiority of the modified adsorbent in comparison with Hz. Furthermore, based on R_L values ($0 < R_L < 1$) and n ($1 < n < 10$), it can be concluded that adsorption is essential for both adsorbents and it follows the above-mentioned isotherms.

Contact time and adsorption kinetics

Adsorption was very rapid within the first five minutes, after which it became slower, and eventually the adsorbent became saturated.

The extent of adsorption did not change considerably for about 20 min. Therefore, this time was chosen as the equilibrium and optimal time for the adsorbent. Kinetic data have also been provided in Table 2 and Fig. 4. Pseudo-second-order pattern has a better correspondence with the experimental data for both adsorbents. On the other hand, q_{e2} values obtained from the pseudo-second-order model have a greater correspondence with experimental q_e values.

At primary stages of contact, a large number of surface sites are available for adsorption. However, over time the remaining adsorption

sites are largely occupied due to repulsion between solute molecules and solid phase. In this state, adsorption of the dye diffused from the surface sites to internal sites of the adsorbent.

The sum of these factors resulted to a decrease in the rate of adsorption process over time.

The q_{e2} values obtained from the pseudo-second-order model have a greater correspondence with experimental q_e values. Therefore, brilliant blue FCF adsorption follows pseudo-second-order model of Ho & McKey (1999), with the adsorption nature being of chemical type.

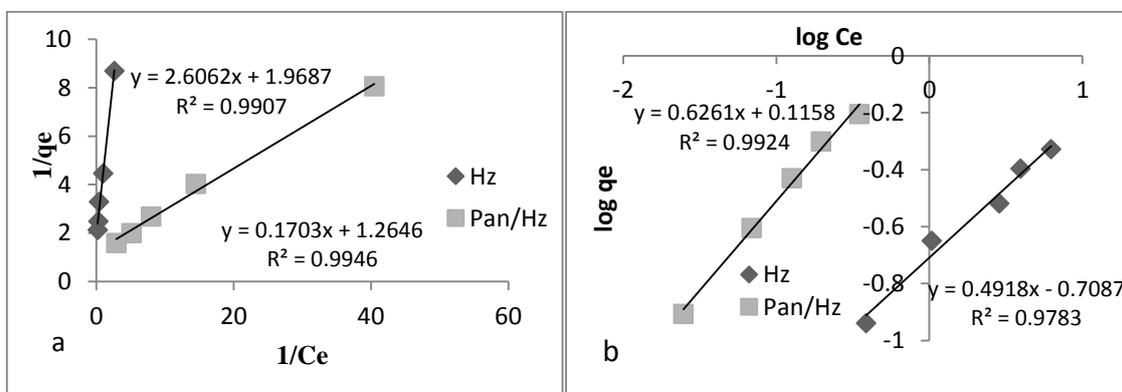


Fig. 3. Langmuir isotherm (a) and Freundlich isotherm (b) for adsorption of BB.

Table 1. Constants of Langmuir and Freundlich for adsorption of BB.

| adsorbent | Langmuir | | | | Freundlich | | |
|----------------------|---------------------------|---------------------------|--------|--------|------------|-------|--------|
| | q_m ($mg\ g^{-1}$) | K_L ($mg\ g^{-1}$) | R_L | R^2 | K_F | n | R^2 |
| Hz | 0.507 | 0.755 | 0.0502 | 0.9907 | 0.195 | 2.033 | 0.9783 |
| Pan Hz ⁻¹ | 0.790 | 7.429 | 0.0053 | 0.9946 | 1.305 | 1.597 | 0.9924 |

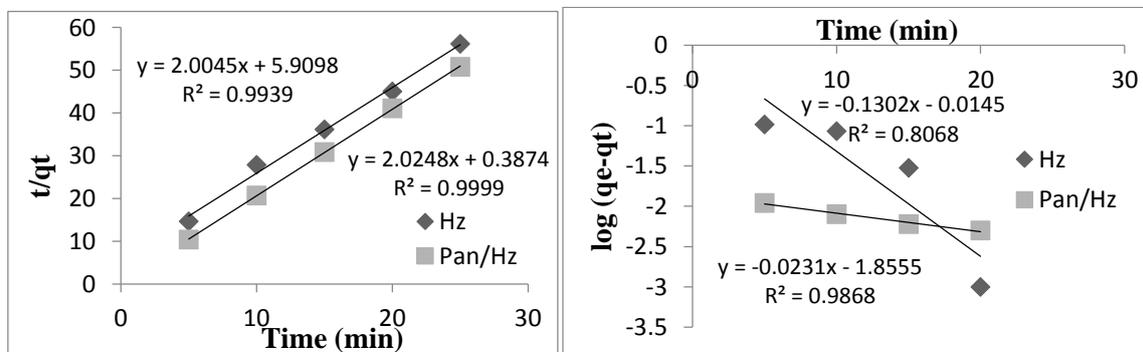


Fig. 4. Pseudo-second-order and pseudo-first-order model for adsorption of BB.

Table 2. The parameters of pseudo-first-order and pseudo-second -order for adsorption of BB.

| adsorbent | pseudo-first-order | | | pseudo-second -order | | | |
|-----------------------|--|---|----------------|--|---|--|----------------|
| | q _{e1} (mg g ⁻¹) | K ₁ (g mg min ⁻¹) | R ² | q _{e2} (mg g ⁻¹) | K ₂ (g mg min ⁻¹) | q _{e2} (exp) (mg g ⁻¹) | R ² |
| Hz | 0.967 | 0.29 | 0.8068 | 0.498 | 0.679 | 0.445 | 0.9939 |
| Pan. Hz ⁻¹ | 0.013 | 0.053 | 0.9868 | 0.493 | 10.58 | 0.492 | 0.9999 |

The effect of temperature and adsorption thermodynamics

The results are presented in Table 3 and Fig. 5. An increase in temperature from 293 to 333 K, resulted in a corresponding increase in adsorption percentage from 88.83 to 91.63 for Hz and from 97.53 to 99.43 for Pan Hz⁻¹. Based on the above data, the endothermicity of the brilliant blue FCF adsorption process is verified by both adsorbents.

At higher temperatures, the extent of displacement and rate of inter particle diffusion of the dye increased, whereby more particles of the dye per volume unit of the solution reached the surface of the adsorbent. The obtained ΔG⁰

values imply spontaneity of FCF brilliant blue adsorption process by Pan Hz⁻¹ at different temperatures. In addition, ΔH⁰ of the process is positive, suggesting its endothermicity, which is considered as an unfavorable factor. However, ΔS⁰ value is also positive (favorable factor), which is indeed the main driving force of the adsorption process, causing ΔG⁰ to become more negative at higher temperatures, thus making the adsorption process more spontaneous. In Hz adsorbent, ΔG⁰ values have proven to be positive, suggesting non-spontaneity of adsorption of brilliant blue FCF by this adsorbent.

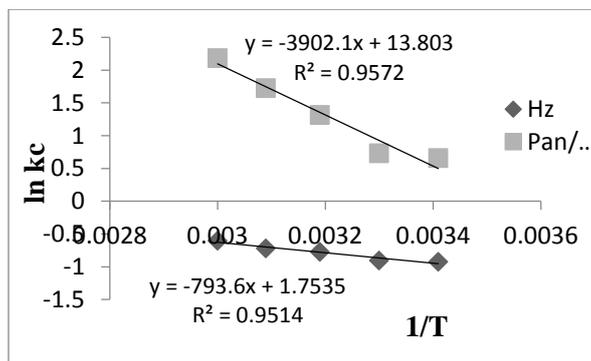


Fig. 5. Vant Hoff equation, lnK_c versus 1/T diagram for adsorption of BB.

Table 3. Thermodynamic parameters for adsorption of BB.

| adsorbent | T (K) | Kc | ΔG ⁰ (KJ mol ⁻¹) | ΔH ⁰ (KJ mol ⁻¹) | ΔS ⁰ (J molK ⁻¹) |
|----------------------|-------|-------|---|---|---|
| Hz | 293 | 0.397 | 2.327 | 6.597 | 14.57 |
| | 303 | 0.405 | 2.182 | | |
| | 313 | 0.462 | 2.036 | | |
| | 323 | 0.486 | 1.890 | | |
| | 333 | 0.547 | 1.745 | | |
| Pan Hz ⁻¹ | 293 | 1.93 | -1.181 | 32.44 | 114.75 |
| | 303 | 2.08 | -2.329 | | |
| | 313 | 3.73 | -3.476 | | |
| | 323 | 5.62 | -4.624 | | |
| | 333 | 8.87 | -5.771 | | |

Investigation of the surface morphology

Scanning electron microscopy (SEM) is a suitable instrument for the investigation of the

apparent form of adsorbents. The obtained images can be used to examine the porosity as

well as distribution of particles and polymer layers. Hazelnut skin has a rough surface and

nano scale polymer layers are formed in its pores.

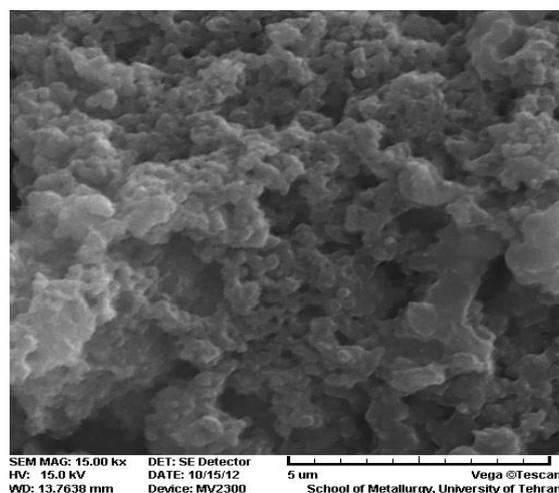


Fig. 6. SEM image for Pan/Hz/BB.

CONCLUSION

Through application of wastes such as hazelnut skin as nano polymer-keeping beds and preparation of electroactive nano composites, it is possible to deal with adsorption of organic dyes from aqueous solutions. High adsorption percentage and low contact time are among the advantages that make this method very economical. Easy preparation of hazelnut skin/polyaniline (Pan Hz⁻¹) nano composites is a huge help, making it a useful method in the preparation of organic dye adsorbent materials. By optimizing other environmental conditions including pH, initial dye concentration and contact time of the adsorbent with the organic dye, maximum adsorption of the dye can be obtained.

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حذف آلودگی بریلیانت بلو از محیط زیست با استفاده از کامپوزیت نانو پلی آنیلین پوست فندق و بررسی پارامترهای موثر

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چکیده

ورود پساب‌های رنگی به رودخانه‌ها علاوه بر مشکلات فراوان زیست محیطی که امروزه به یک معضل بزرگ جامعه تبدیل شده است، آثار بسیار نامطلوب روحی و روانی بر جامعه دارد. رنگبری این پساب‌ها قبل از ورود به رودخانه‌ها از اهمیت زیادی برخوردار است. در این پژوهش با نشان دادن یک لایه از نانو پلیمر رسانا بر پایه پلی آنیلین بر روی پوست فندق و تولید نانو کامپوزیت، برای انتقال رنگزای بریلیانت‌بلو از محیط آبی استفاده شده است. هدف، نشان دادن لایه‌ای از نانو پلیمر پلی آنیلین بر سطح پوست فندق و تهیه کامپوزیت پلی آنیلین / پوست فندق (به طور اختصار Pan Hz^{-1}) از طریق پلیمریزه کردن شیمیایی آنیلین روی پوست فندق در محیط آبی و جذب رنگزای بریلیانت‌بلو از محیط آبی است. در این مرحله پلی آنیلین با استفاده از آمونیوم پرسولفات سنتز شد. به منظور پیدا کردن شرایط بهینه در انتقال رنگزای اثرات تعدادی از پارامترهای مهم مانند pH، زمان تماس، غلظت اولیه رنگزا و دما مطالعه شد. برای مطالعات جذبی از دو ایزوترم لانگمویر و فروندلیچ استفاده شد. همچنین مطالعات ترمودینامیک و کینتیک نیز انجام شد. این رنگزا در $\text{pH}=2$ دارای بیشترین جذب بوده و با بهینه کردن دیگر پارامترهای فوق، بهترین حالت برای جذب بیشینه ارائه شده است. مطالعات ترمودینامیکی نشان می‌دهد که جذب خودبه‌خودی، گرماگیر و از نوع شیمیایی بوده و از کینتیک درجه دوم تبعیت می‌کند. جاذب معرفی شده در این مقاله یک جاذب بسیار موثر برای جذب بریلیانت‌بلو بوده و با استفاده از لایه‌ای از نانو پلیمر رسانا بر روی پوست فندق می‌توان با درصد بسیار بالا به جذب رنگزای بریلیانت‌بلو از محیط آبی پرداخت.