Available online at http://ijcpe.uobaghdad.edu.iq and www.iasj.net
Iraqi Journal of Chemical and Petroleum Engineering
Vol.22 No.2 (June 2021) 1 – 6
EISSN: 2618-0707, PISSN: 1997-4884

Prepared 13X Zeolite as a Promising Adsorbent for the Removal of Brilliant Blue Dye from Wastewater

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Abstract

The research discussed the possibility of adsorption of Brilliant Blue Dye (BBD) from wastewater using 13X zeolite adsorbent, which is considered a byproduct of the production process of potassium carbonate from Iraqi potash raw materials. The 13X zeolite adsorbent was prepared and characterized by X-ray diffraction that showed a clear match with the standard 13X zeolite. The crystallinity rate was 82.15% and the crystal zeolite size was 5.29 nm. The surface area and pore volume of the obtained 13X zeolite were estimated. The prepared 13X zeolite showed the ability to remove BBD contaminant from wastewater at concentrations 5 to 50 ppm and the removal reached 96.60% at the lower pollutant concentration. Adsorption measurements versus time showed 48.18% removal of the dye during just the first half-hour and the maximum removal closest to the removal at the equilibrium after one and half hour. Langmuir isotherm was described the adsorption equilibrium data with a maximum adsorption capacity of 93.46 mg/g and the kinetics data of the adsorption process was followed the pseudo-second-order.

Keywords: 13X zeolite, adsorbent, brilliant blue dye, adsorption, wastewater.

Received on 28/03/2021, Accepted on 11/05/2021, published on 30/06/2021

https://doi.org/10.31699/IJCPE.2021.2.1

1- Introduction

BBD is a disodium salt (C37H34N2Na2O9S3) produced by the condensation reaction of 2-formylbenzenesulfonic acid and aniline, followed by oxidation of the condensation product. This synthetic dye is classified as one of the triarylmethane dyes families [1]. The BBD has a color index of 42090 and is known in various commercial names such as Acid Blue 9, FD&X Blue No.1, and the most commonly used name is E133 with CAS Registry Number 3844-45-9 [2]. BBD is a commonly used dye in food industries since 1929. Until now, the use of Brilliant Blue as a food color additive at current levels does not present a safety concern to humans, but reaching this dye to water streams and accumulated it in the tissues of the marine organisms, especially fish and sea fruits and causes severe problems for these organisms, which will quickly affect the human health [2]-[4]. Different methods are applied for treating organic pollutants for water depend on treated water amount, pollutants concentration, and purity level. Generally, treating organic matter can be classified into physical, chemical, and/or biological methods [5].

However, there are many elimination methods of pollutants in wastewater; the adsorption process is the most common because of its simplicity, ease of use, and low operation cost [6], [7]. Still, the adsorption processes' most significant challenge is to find new and environmentally friendly adsorbents or improve the known adsorbents [8]. The well-known adsorbent used to adsorb different organic contaminants and/or dyes from wastewater are charcoal/activated charcoal [9]–[12], activated carbon [13]–[16], zeolites [17]–[19], starch-based environmentally friend adsorbent [20]–[24]. The present work aimed to convert a low cost locally available Iraqi potash ore to prepare a 13X zeolite adsorbent (13XZA), which is produced as a byproduct during potassium carbonate production via hydrothermal method. The prepared 13XZA was characterized by X-ray diffraction (XRD), the surface area, and the pore volume. The prepared 13XZA used to adsorb BBD and both equilibrium adsorption and kinetics were studied and discussed.

2- Experimental work

2.1. Preparation and characterization of 13XZA

The 13XZA was prepared hydrothermally from Iraqi potash ore (potassium feldspar powder) according to the previous work [25]. Potassium feldspar powder used in this study was obtained from the Department of Geological Mining of the Ministry of Industry in Baghdad.

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The prepared zeolite was characterized by XRD in the Chemical and Petrochemical Center, Industrial Research and Development Authority, Ministry of Industry, and Minerals. The crystallinity was determined by dividing the sum of crystalline peaks area of the prepared 13XZA on the sum of all peaks area of crystalline and amorphous (Eq. 1), and the average crystal size of the prepared 13XZA was determined by Scherrer's relationship (Eq.2). Finally, the surface area and pore volume of the adsorbent were measured using Brunauer–Emmett–Teller (BET) method via surface area analyzer/Q surf series/Italy in the Oil Development and Research Center - Ministry of Oil.

$$\text{Crystallinity, \%} = \frac{\sum Area\ (\text{crystal})}{\sum Area\ (\text{crystal} + \text{amorphaus})} \times 100\% \quad (1)$$

$$D = \frac{k\lambda}{\beta \cos(\theta)} \quad (2)$$

Where, D = crystallite size in nm, k = dimensionless crystallite shape factor (typical value is 0.9), λ = wavelength of the X-ray = 1.5406 Å, β = full width at half maximum (FWHM), rad, and θ = Bragg angle, rad.

2.2. Adsorption of BBD

Batch adsorption experiments were carried out by using 0.025 g of 13XZA for every 50 ml of the wastewater containing a different initial concentration of BBD (C_o between 5 to 50 ppm). The solutions were put on a shaker with a uniform speed of 200 rpm at an ambient temperature of 25±3 °C for 24 hours to reach the equilibrium state. The amount of the equilibrium concentration (C_e) was reported by averaging the dye concentration values after treatment and then the amount of adsorbed BBD per weight of 13XZA (adsorption capacity) at equilibrium (q_e, mg/g) was calculated from Eq. (3).

$$q_e = (C_o - C_e) \times \frac{V}{m} \quad (3)$$

Where q_e is the adsorption capacity of the adsorbent (mg/g), C_o and C_e in (mg/L) refer to initial and final (equilibrium) concentrations of BBD in the adsorption solution. V (L) is the volume of adsorption solution and m (g) is the weight of 13XZA used.

The kinetics experiment carried out using a1000 ml of the 50 ppm BBD and one gram of the prepared 13XZA have been put together in the beaker and the magnetic stirrer speed kept at 200 rpm. At each interval up to 180 minutes, two samples of 5 ml of the mixture were taken and the average concentration of BBD was measured and reported. While adsorbent performance for the removal of BBD was calculated by Eq. (4).

$$\text{BBD removal, \%} = \frac{C_o - C_t}{C_o} \times 100\% \quad (4)$$

The BBD concentration (in batch and kinetics experiments) was measured at 628 nm wavelength using Shimadzu UV-160A UV-VIS Recording Spectrophotometer in the Chemical Engineering Department, College of Engineering, University of Baghdad.

2.3. Adsorption Isotherms and Kinetics Models

The adsorption isotherms demonstrate the interaction between the adsorbates and adsorbents. Most widespread two-parameter isotherms were selected to describe the adsorption of BBD on the prepared 13XZA. These isotherms models are Langmuir (Eq. (5)) [26] and Freundlich (Eq. (6)) [27].

$$q_e = \frac{q_{\max} K_L C_e}{1 + K_L C_e} \quad (5)$$

$$q_e = K_F C_e^{\frac{1}{n}} \quad (6)$$

Where q_e adsorption capacity mg adsorbate per g adsorbent, C_e is the concentration at equilibrium mg/L, q_{max} is the maximum adsorption capacity in forming a complete monolayer on the surface mg/g, K_L is Langmuir coefficient related to the affinity between the adsorbate and adsorbent (L/mg), K_F is the Freundlich coefficient and n is the number of multilayers.

Adsorption–kinetics models [28] were used to describe the adsorption capacity variation with time. These models are; Pseudo-first order (Eq. 7) [29], pseudo-second-order (Eq.8) [30], and intraparticle diffusion model (Eq. 9).

$$ln(q_e - q_t) = lnq_e - k_1 t \quad (7)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e} + \frac{t}{q_e} \quad (8)$$

$$q_t = k_3 t^{\frac{1}{2}} + C \quad (9)$$

Where q_e and q_t (in mg/g) are the adsorption capacity at equilibrium and any time (t), respectively. k_1 (1/min), k_2 (g/mg.min), and k_3 (mg/(g.min$^{0.5}$)) are the adsorption rate constants for pseudo-first order, pseudo-second order, and intraparticle diffusion kinetic models, respectively. As well as, C (in Eq. (9)) is an arbitrary constant for the intraparticle diffusion.

3- Results and Discussion

3.1. Characterization of 13XZA

XRD is an effective analysis and identification technique used to identify the phase of 13XZA crystalline during crystal formation from raw materials and can provide information on unit cell size. The results of the XRD demonstrated in Fig. **1** showed a clear correspondence in the places of the peaks with the results of the previous study [25]. The calculated crystallinity value by Eq. (1) was 82.15% and the crystal size computed by Scherrer's equation (Eq. (2)) was 5.29 nm. The current results show a slightly decrease in the crystallinity value due to fewer intensity values of the XRD peaks and the crystal size is very closed to the value of the previous work [25]. This convergence in crystal size of the 13XZA indicates that all the width of the peaks of the XRD results is in the same magnitude. However, the measured surface area and pore volume were equal to 395.48 m^2/g and 0.2405 cm^3/g, respectively.

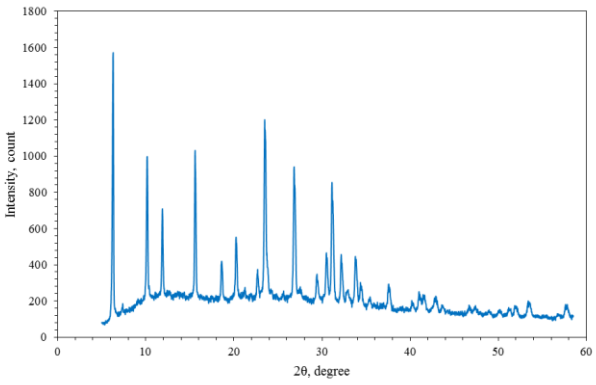

Fig. 1. XRD pattern of the hydrothermal prepared 13XZA

3.2. Adsorption of BBD on the 13XZA

a. Equilibrium and adsorption isotherms

The adsorption process at equilibrium provides important data on the effect of the initial concentration of the dye on the adsorption capacity of the adsorbent and the amount of removal at equilibrium. The equilibrium data will be important in calculations of equilibrium isotherms, which will provide important figures, including maximum adsorption capacity. As shown in Fig. **2**, the batch adsorption results of BBD on the prepared 13XZA showed a sharp increase in the equilibrium adsorption capacity from 9.66 to 80.78 mg/g of adsorbent, while the equilibrium removal values showed a decrease from 96.60 to 80.78% with an increase in the BBD concentration from 5 to 50 ppm.

The decrease in the removal values with the increasing the initial concentration is due to competition of dye molecules on the effective adsorption sites on the surface and pores of the 13XZA.

This competition decreases when using low concentrations, which cause an increase in the dye's adsorption and thus an increase in the amount of removal is observed.

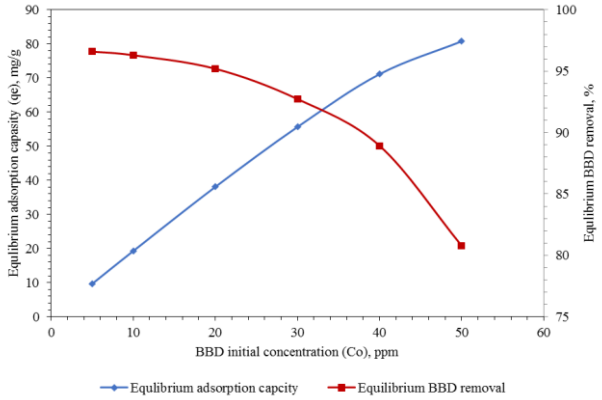

Fig. 2. Effect of initial BBD concentration on the equilibrium capacity (blue line, left y-axis) of the13XZA and equilibrium removal (red line, right y-axis)

The isotherm constants were determined using the equilibrium data and summarized in Table **1**. Obtained equilibrium data are highly correlated with both isotherms (high correlation coefficients, R^2). These isotherms are describing two extremely different phenomena, the Langmuir isotherm describing homogeneous monolayer adsorption, while the Freundlich isotherm relating heterogeneous multilayer adsorption. Therefore, both isotherms were plotted with the obtained experimental equilibrium data in Fig. **3**.

Table 1. Adsorption isotherm models constants and correlation coefficients (R^2) for the removal of BBD on the 13XZA

Isotherm model	Model constant, unit	Constant value	R^2
Langmuir	q_{max}, mg/g	93.46	0.9997
	K_L, L/mg	1.534	
Freundlich	K_F, mg^{1-n} L^n/g	31.13	0.9995
	n, -	1.98	

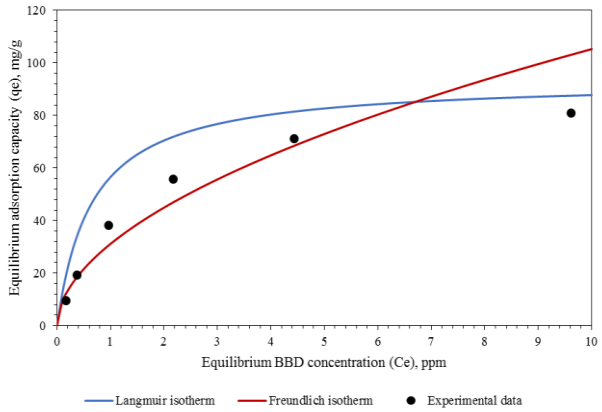

Fig. 3. Effect of the equilibrium BBD concentration on the equilibrium adsorption capacities of the 13XZA compared with Langmuir (blue line) and Freundlich (red line) isotherms

As shown from Fig. **3**, the Langmuir isotherm describing the experimentally obtained data better than the Freundlich isotherm which is deviated out the data at the values of equilibrium concentration (C_e) more than about 7. So, the Langmuir isotherm explains the obtained experimental equilibrium date of BBD adsorption on the prepared 13XZA with a maximum adsorption capacity of 93.46 mg/g (mg of BBD per g of the adsorbent), with Langmuir coefficient K_L equal to 1.534 L/mg.

b. Effect of time and the adsorption kinetics

The concentration of BBD decreasing with the time that correlated with an increase in the amount of BBD removal from the wastewater as shown in Fig. **4**.

At the first 30 minutes, the concentration of BBD was dropped sharply with about 48.18% of its initial value.

After that, the removal of BBD became slower until about 90 minutes with 77.40 % removal, and then the growth in the removal was very slightly with increasing adsorption time (80.76% removal after 180 minutes (3 hours), and 80.78% removal after 24 hours (at assumed equilibrium conditions).

The relatively fast adsorption at the beginning was because of the high concentration difference (the mass transfer driving force) between the solution and the "clean" surface of the 13XZA. Over time, more BBD adsorbed on the surface that occupied more pores and adsorption sites, which decreasing in the mass transfer driving force, and causing a slowing in the adsorption rate and stopped it at equilibrium.

The kinetic results of the adsorption of the BBD from wastewater on the prepared 13XZA according to the correlation of concentration versus time data with adsorption kinetics models (Eq. (7-9)) are summarized in Table **2**.

The obtained values of the R^2 indicate a well fit of the obtained data with the pseudo-second-order model (Eq. 8) with a rate constant equal to 0.0333 g/mg.min.

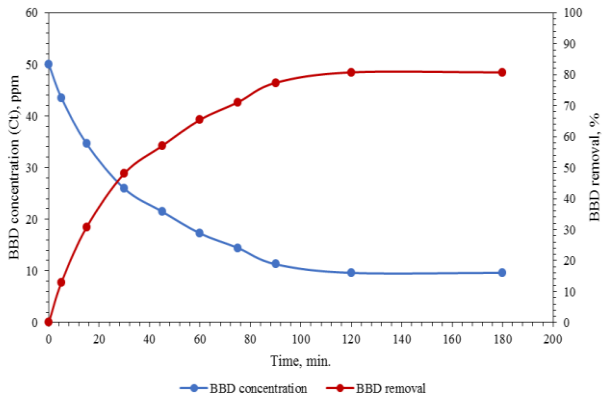

Fig. 4. BBD concentration (blue line, left y-axis) and removal (red line, right y-axis) versus the time of the adsorption over the 13XZA

Table 2. Adsorption kinetics models constants and correlation coefficients (R^2) for the adsorption of BBD on the 13XZA

Adsorption kinetic model	Model constant, unit	Constant value
Pseudo-first order	k_1, L/min	0.0533
	q_e, mg/g	84.98
	R^2	0.8661
Pseudo-second order	k_2, g/mg.min	0.0333
	q_e, mg/g	48.78
	R^2	0.9941
Intraparticle diffusion	k_3, mg/g.min$^{0.5}$	3.1971
	C, mg/g	4.7826
	R^2	0.8868

4- Conclusion

Iraqi potash ore was a good source of preparing 13XZA by the hydrothermal method as a byproduct in the production of potassium carbonate. The prepared 13XZA have 82.15% crystallinity, crystal size of 5.29 nm, surface area of 395.48 m^2/g and 0.2405 cm^3/g pore volume. Prepared 13XZA show ability to remove BBD from wastewater in the concentrations ranged between 5 to 50 ppm. The removal was decreasing from 96.60 to 80.78% by increasing the initial concentrations. The adsorption equilibrium data analysis show that Langmuir isotherm describes better the adsorption of BBD on 13XZA with maximum adsorption capacity of 93.46 mg/g. Adsorption measurements versus time recorded 48.18% removal during first 30 minutes, and the removal reached closely to the equilibrium value after 90 minutes. While the adsorption kinetics analysis shows that the adsorption of BBD on 13XZA followed the pseudo-second order with a rate constant equal to 0.0333 g/mg.min.

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تحضير زيولايت 13X كممتز واعد لازالة الصبغة الزرقاء اللامعة من مياه الصرف

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الخلاصة

ناقش البحث امكانية امتزاز الصبغة الزرقاء اللامعة من مياه الصرف الصحي باستخدام الزيولايت من نوع **13X** كمادة ممتزة والتي تعتبر منتج ثانوي لعملية انتاج كربونات البوتاسيوم من مواد البوتاس العراقية الاولية. تم تحضير مادة الامتزاز و تشخيصه بأستخدام انحراف الأشعة السينية و الذي أظهر تطابقًا واضحًا مع زيوليت **13X** القياسي. و قد بلغ معدل التبلور 82.15٪ و كان حجم الزيوليت البلوري 5.29 نانومتر، كما و تم تقدير مساحة السطح و حجم المسام للزيوليت المحضر. أظهر زيوليت **13X** المحضر القدرة على إزالة الصبغة من مياه الصرف بتركيزات 5 إلى 50 جزء في المليون و وجدت أعلى إزالة كانت بمقدار 96.60٪ عند أقل تركيز للملوث. أظهرت قياسات الامتزاز مقابل الوقت إزالة 48.18٪ من الصبغة خلال النصف ساعة الأولى فقط وحصلنا على أقصى إزالة بعد ساعة ونصف. وصف متساوي الحرارة لانجمویر بيانات توازن الامتزاز بشكل أفضل و كانت سعة الامتزاز القصوى 93.46 مجم / جم وقد كانت البيانات الحركية لعملية الامتزاز تتبع حركية الدرجة الثانية الزائفة.

الكلمات الدالة: زيوليت 13X، مادة ممتزة، الزرقاء اللامعة، امتزاز، مياه صرف.