Characterization of Synthesized Zeolite from Iraqi Metakaolin and its Application in Dye Removal from Wastewater

Mohamed Ibrahim[1], Aziz Şencan[2], Mustafa Alani[2]

[1]Department of Medical Instrumentation Techniques Engineering, Dijlah University College, Baghdad, Iraq
[2]Chemical Engineering, College of Engineering, Suleyman Demirel University, Isparta, Turkey

Article Info

Article history:

Received April, 01, 2025
Revised May , 15, 2025
Accepted June 10, 2025

Keywords:

Zeolite
Metakaolin
Adsorption
Rhdamine B
Pollution

ABSTRACT

The present work aims to synthesize and characterize Zeolite using Iraqi metakaolin and to study their performance in the removal of dye from wastewater. Techniques such as scanning electron microscope SEM, X-ray diffractometer XRD, and FTIR Fourier transmission infrared spectrometer were used to synthesize zeolite characterization. A UV spectrometer determined zeolite's efficiency in removing RB dye from wastewater. The X-ray diffraction result for the zeolite prepared showed that the zeolite was identical in structure to the commercial zeolite Type A. The prepared zeolite was examined as an adsorbent to remove RB dye from wastewater. The adsorption Experiment revealed that approximately 13 ppm of dye could be removed per 2 grams of zeolite, which was deemed unsatisfactory and compatible with similar studies. Additionally, adsorption isotherms modelling indicated that the Langmuir model best described the adsorption behavior, suggesting a monolayer adsorption process.

Corresponding Author: Mustafa Alani

Chemical Engineering, College of Engineering, Suleyman Demirel University, Isparta, Turkey

Email: alanimustafa260@gmail.com

1. INTRODUCTION

Zeolites have gained significant attention due to their exceptional adsorption, ion exchange, and catalytic properties [1], [3] they are widely used in various fields such as environmental protection, water treatment, gas separation, and chemical industry [4], [5]. The unique structure and properties of zeolites make them a promising material for the removal of dyes from wastewater, offering an efficient and sustainable solution for environmental pollution [6]. Zeolites are naturally occurring minerals with a unique crystalline structure characterized by a framework of microporous cavities and channels [7]. These properties allow zeolites to selectively adsorb molecules based on size, shape, and polarity, making them highly effective in various industrial processes, including dye removal [8].These synthetic zeolites can be modified to have specific pore sizes, surface areas, and chemical compositions, making them highly versatile for various industrial applications [9]. Zeolites are a group of natural or synthetic hydrated aluminosilicate minerals characterized by a three-dimensional porous structure. This unique structure allows zeolites to selectively adsorb or exchange different molecules based on their size and charge. Zeolites can be classified into natural and synthetic types, with over 240 different known zeolite structures. The classification is based on their framework type and composition, with each type having specific properties and applications [10], [11]. Understanding the definition and classification of zeolites provides a foundational knowledge base for delving into the various production methods and application techniques in dye removal. It allows for a more comprehensive understanding of how zeolites function at a molecular level and how they can be optimised for efficient and effective dye removal processes. Zeolite has various applications, usually in removing trace metal ions, organic matter pollutants, and drug residue from wastewater. It is produced via the hydrothermal crystallisation method. Its mesoporous nature renders it extensively applicable in adsorption, ion exchange processes, zeolite membranes, and catalysis [12]**.** From the textile Industry, including dyes like Rhodamine B. The World Bank has estimated that almost 20% of world industrial water pollution comes from the treatment and dying of textiles. These pollutants impede aquatic growth and can cause the death of aquatic species by inducing anoxic conditions that disrupt photosynthesis and respiration. Moreover, their aromatic, inherently hazardous and carcinogenic structures further endanger aquatic life upon discharge. Various treatment methods were tested to safeguard aquatic environments, like adsorption, which is widely favoured for its cost-effectiveness, ease of implementation, and rapid kinetics. The efficacy of adsorption hinges on the adsorbent's capacity to remove pollutants from wastewater [13]. The present work aims to synthesise and characterise homemade Zeolite using Iraqi metakaolin and to study its performance in removing dye from wastewater. A UV spectrometer was used to determine the efficiency of zeolite in removing RB dye.

2. MATERIALS AND METHOD

Metakaolin was collected from the base of the Al-Anbar region west of Iraq. Its chemical composition is listed in Table 1. All other chemicals were of reagent grade and used without further purification purchased from Merck (Germany).

Table 1: Chemical composition (%) of Iraqi metakaolin calcined at 700^0C[ ]

SiO2	Al2O3	Fe2O3	CaO	MgO	SO3	Na2O	K2O	L.O.I
51.32	32.9	1.90	1.7	0.10	0.18	0.23	0.43	6.47

The dye used in this work was Rhodamine RhB, which is one of the most commonly used textile factories purchased from Merck, Germany. RhB has a maximum absorption wavelength of 555.6 nm, a molecular formula is

($C_{28}H_{31}N_2O_3Cl$), molecular weight 479 and has the following structural features in Figure 1.

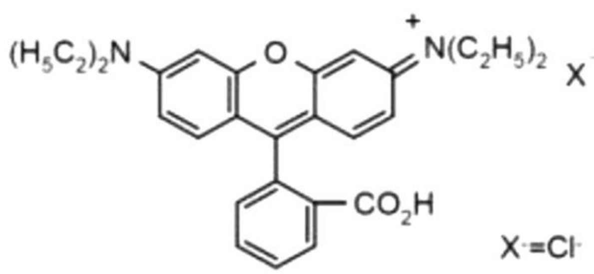

Figure.1 The chemical structure of RhB dye

Zeolite characterisation involved techniques such as X-ray diffractometer (XRD), Scanning electron microscope (SEM), and Fourier transform-infrared spectroscopy (FTIR).

2.1 Synthesis of zeolite

Metakaolin was prepared by heating raw Kaoline powder (kaolinite, dickite, nacrite, halloysite) at 700 ^{0}C in a muffle furnace for 4h. In such cases, dehydroxylation occurs, leading to the formation of metakaolin.

Zeolite was prepared following the procedure in Reference [7]. 21g of metakaolin was crushed to fine particles and heated up to 700 0C in a muffle furnace for 4 h. 30.3g of NaOH was dissolved in 300 ml of distilled water and mixed carefully with metakaolin powder using a magnetic stirrer to form a homogeneous gel. The latter gel was placed in an oven heated at 100^0C for 2h. The final resulting powder was washed several times with distilled water until the pH reached 7.

2.2 Dye Adsorption Procedure

A stock solution of RhB was prepared by dissolving 1.0 g of dye powder in 1000 mL distilled water in an appropriate volumetric flask. The stock solution was diluted to prepare a Solution with different initial concentrations for adsorption experiments. The sorption of dye was determined using a batch equilibrium method. Briefly, a known quantity of adsorbent was added in a 250-mL beaker containing 200mL of RhB solution of known concentration and ph. The mixture was stirred by a magnetic stirrer at 250 rpm at room temperature. The resulting solution was separated by centrifuge at 3000rpm for 5 minutes and filtered by filter paper. The concentration (mg/L) of RhB dye in the filtrate was measured using UV–a UV-visible spectrophotometer at 556nm, corresponding to the maximum absorbency of RhB dye.

The equilibrium adsorption capacity was calculated using the following relationship:

$$q = \frac{C0-Cf}{m} V(L) \quad [12] \tag{1}$$

Where q (mg/g) is adsorption capacity, m (g) is the mass of Adsorbent, V (L) is the volume of solution, and $C0$ and Cf (mg/L) are the dye solution concentration at initial and final Conditions, respectively. The removal efficiency (R %) of the Dye was calculated by the following relationship:

$$R(\%) = \frac{C0-Cf}{C0} x100 \tag{2}$$

3. RESULTS AND DISCUSSION

3.1. Characterization of synthesized Zeolite

Figure 2, shows the Metakaolin (KM) XRD pattern. The observed peaks of the MK structure with the broadening baseline refer to a semi crystalline structure with diffraction peaks of Aluminosilicates. Quartz and kaolinite are the main constituents of MK structure.

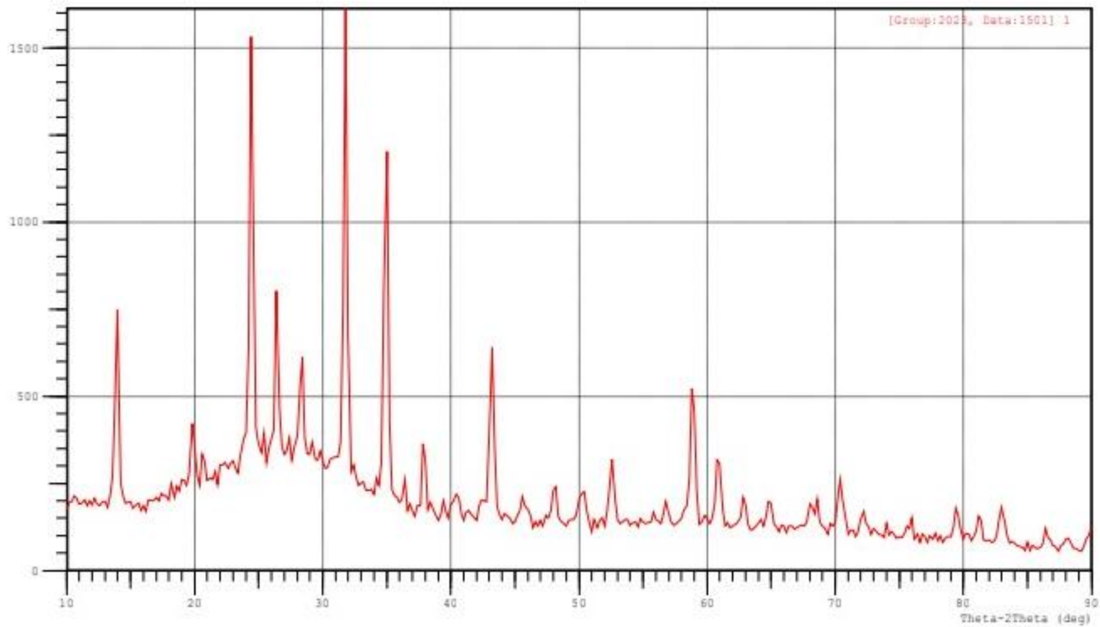

Figure 2. X-ray pattern of Metakaolin

The XRD pattern of the prepared sample is shown in Figure 3, the result clearly shows the presence of zeolite peaks.

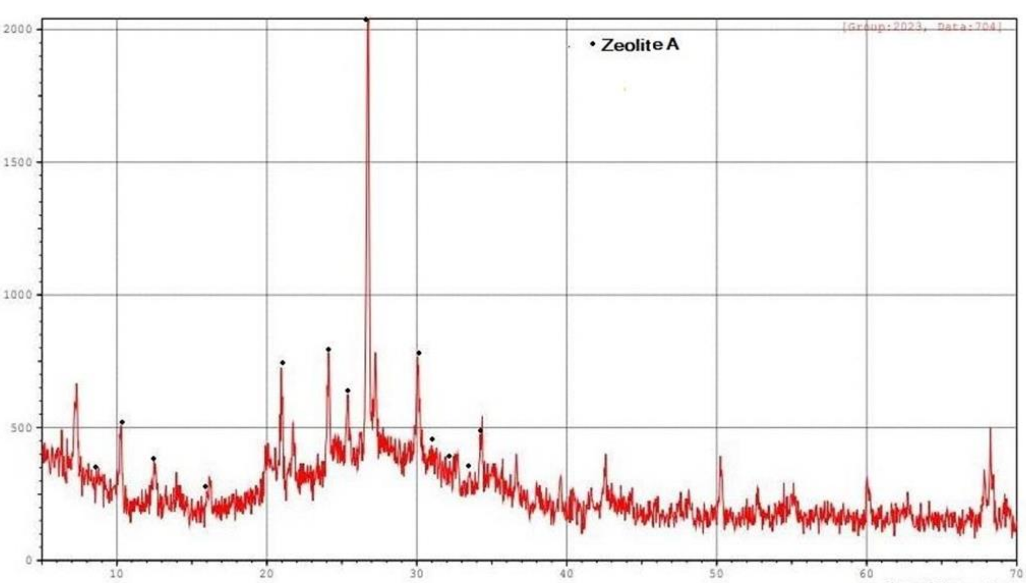

Figure 3. X ray pattern of synthesized Zeolite

The 2-theta values related to prepared zeolite peaks are tabulated in a table1, the results of other work [13] are also shown in the table for comparison. Our result agrees well with the result of the reference [13].

Table.1 Major 2- theta peaks in zeolite-A prepared in this work

Peak	2-Theta (this work)	2-Theta [13]
1	7.20	7.20
2	10.30	10.19
3	12.60	12.49
4	16.0	16.49
5	24.0	24.04

Figure 4 shows the FTIR absorption spectra for the synthesized of zeolite. The absorption band at 441 cm^{-1} corresponds to the Si, Al-O bond, and those at 995 cm^{-1} may be attributed to a symmetric stretch of the zeolite framework. The band at 1653 cm^{-1} may be attributed to the deformation band due to water absorption.

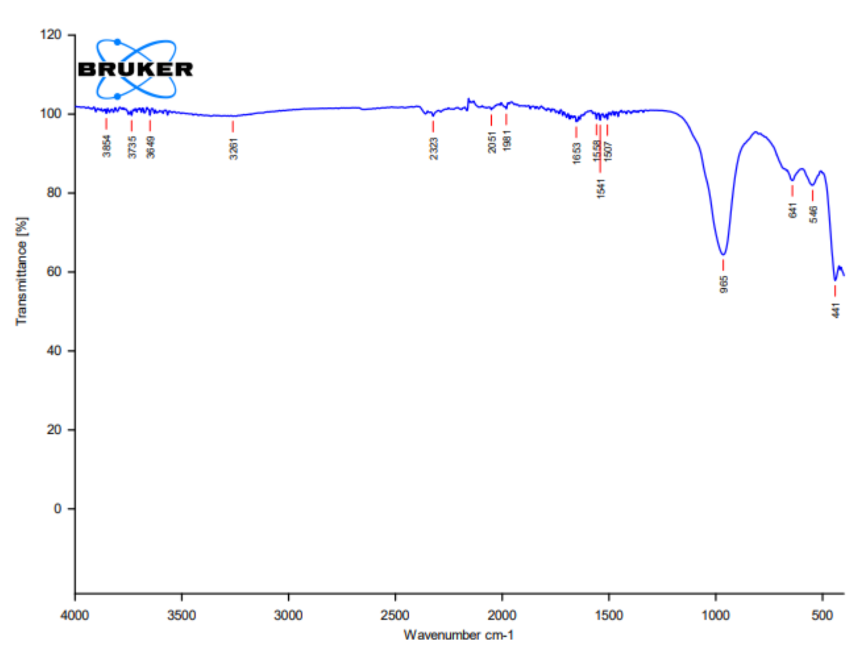

Figure 4. FTIR result of synthesized zeolite

The morphology of the synthesized sample was examined by scanning electron microscope, as shown in Figure 5. The structure reveals the presence of spheres, flakes and sometimes chunks.

Dijlah Journal of Engineering Sciences (DJES) Vol. 2, No. 2, March, 2025, pp. 23-31
ISSN: **Printed: 3078-9656, E: 3078-9664**, paper ID: 34

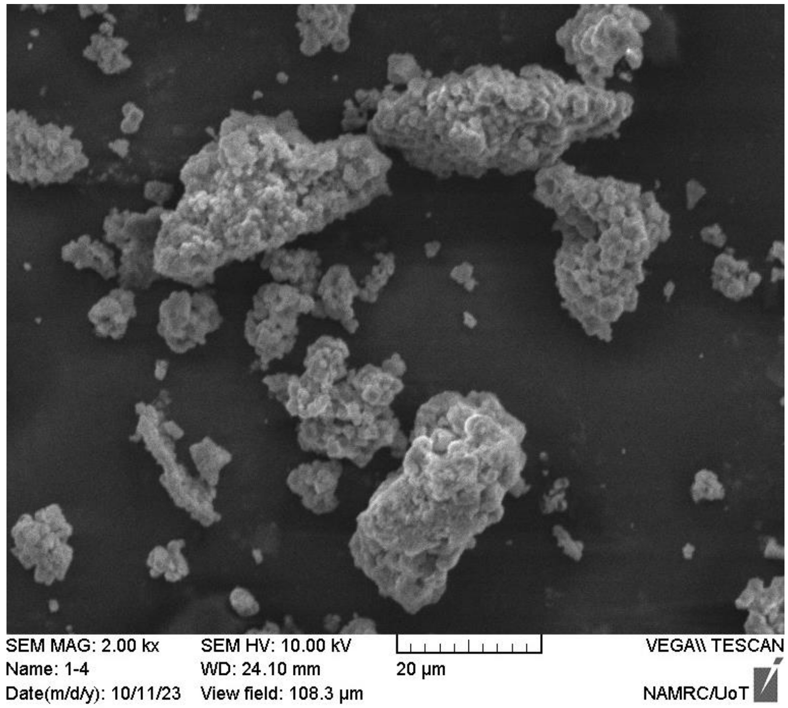

Figure 5. The morphology of the synthesized Zeolite

3.2 Adsorption of RhB Dye in Batch System on prepared Zeolite A

The adsorption of RhB Dye in Batch System was conducted to remove the dye using the zeolite prepared in this work. The following tables summarize the experiments:

Table 2. the concentration of dye as a function of zeolite mass per 15ml of dye solution

zeolite dose	Abs. at 555.6	Cons.	%q
0.5g	4.45	44.5	11
1 g	4.1	41	18
1.5 g	3.75	37.5	25
2 g	**3.43**	**34.3**	**31.4**
2.5 g	3.55	35.5	29

The maximum adsorption capacity of zeolite was 31.4 at 2g of zeolite quantity .The result of time Optimization Table 3, showed that the time required to remove the dye completely was 3 minutes.

Table 3. Time optimization

Time Min.	Abs at 556	Con.	%q
1	3.42	34.2	31.6
2	3.39	33.9	32.2
3	**3.325**	**33.25**	**33.5**
4	3.37	33.7	32.6

Finally, to determine which model better fit our results, we tested the Langmuir and Freundlich isotherm models, in Table 4; we found that the Langmuir model best fit the experimental results over the experimental range.

Table 4. Comparison between Freundlich and Langmuir results.

Langmuir	**qmax mg\g**	**KL**	**RL**	**R^2**
	15.2	**0.021025**	**0.090736**	**0.99**
freuendlish	**1\n**	**Kf**	**R^2**	
	1	**264.6671**	**0.9998**	

4. CONCLUSION

Zeolite is an important industrial material usually used in water treatment and other important chemical industries. In this work, we concluded that Zeolite type A has been synthesized successfully from metakaolin, which is already available in the region west of Iraq. The method was simple, and the cost of synthesis was very low. The material was characterized using techniques like SEM, XRD and FTIR; all of the results confirm the zeolite type A properties. An attempt to use the prepared material as an adsorbent for the removal of RhB from wastewater was found to have moderate potential for the adsorption of RhB dye from an aqueous solution in a batch adsorption system. The batch experiment showed that the percentage of RhB removed increases with the increase in the contact time and Zeolite dosage while adjusting the solution's ph. The adsorption isotherm studies demonstrated that the adsorption processes can be well-fitted by the Langmuir isotherm model, which has a higher correlation coefficient (0.990).

ACKNOWLEDGEMENTS

We would like to thank Chemical Engineering Department in the University of Suleyman demirel and Chemical Engineering Department in the University of Technology in Baghdad for their valuable and limitless Support for this research.

REFERENCES

[1] T. Abdullahi, Z. Harun, and M. H. D. Othman, "A review on sustainable synthesis of zeolite from kaolinite resources via hydrothermal process," Advanced Powder Technology, vol. 28, no. 8, pp. 1827–1840, Aug. 2017, **doi: https://doi.org/10.1016/j.apt.2017.04.028.**

[2] J. Campoverde and D. Guaya, "From Waste to Added-Value Product: Synthesis of Highly Crystalline LTA Zeolite from Ore Mining Tailings," Nanomaterials, vol. 13, no. 8, pp. 1295–1295, Apr. 2023, doi: https://doi.org/10.3390/nano13081295.

[3] A.G.Olaremu, E.O.Odebunmi, F.O. Nwosu, A.O. Adeola1, T.G.Abayomi, SYNTHESIS OF ZEOLITE FROM KAOLIN CLAY FROM ERUSU AKOKO SOUTHWESTERN NIGERIA, J. Chem Soc. Nigeria, Vol. 43, No. 3, pp 381 -786 [2018] .

[4] T. saad Algarni and A. M. Al-Mohaimeed, "Water purification by absorption of pigments or pollutants via metaloxide (Review)," Journal of King Saud University - Science, p. 102339, Sep. 2022, doi: https://doi.org/10.1016/j.jksus.2022.102339.

[5] Raja Belaabed, S. Elabed, Abdellah Addaou, A. Laajab, M. A. Rodríguez, and A. Lahsini, "Synthesis of LTA zeolite for bacterial adhesion," Boletín de la Sociedad Española de Cerámica y Vidrio/Boletín de la Sociedad Española de Cerámica y Vidrio, vol. 55, no. 4, pp. 152–158, Jul. 2016, doi: https://doi.org/10.1016/j.bsecv.2016.05.001.

[6] J. Kowalska-Kuś, A. Held, K. Nowińska, and K. Góra-Marek, "LTA zeolites as catalysts for transesterification of glycerol with dimethyl carbonate," Fuel, vol. 362, pp. 130757–130757, Apr. 2024, doi: https://doi.org/10.1016/j.fuel.2023.130757.

[7] A. Bahgaat, M. mohamed, A. Abdel Karim, A. Melegy, and H. Hassan, "Synthesis and Characterization of Zeolite-Y from natural clay of Wadi Hagul, Egypt," Egyptian Journal of Chemistry, vol. 0, no. 0, Mar. 2020, doi: https://doi.org/10.21608/ejchem.2020.23195.2378.

[8] Adeleke Abdulrahman Oyekanmi, A. Ahmad, K. Hossain, and Mohd Rafatullah, "Statistical optimization for adsorption of Rhodamine B dye from aqueous solutions," vol. 281, pp. 48–58, May 2019, doi: https://doi.org/10.1016/j.molliq.2019.02.057.

[9] M. M. Selim, D. M. EL-Mekkawi, R. M. M. Aboelenin, S. A. Sayed Ahmed, and G. M. Mohamed, "Preparation and characterization of Na-A zeolite from aluminum scrub and commercial sodium silicate for the removal of Cd2+ from water," Journal of the Association of Arab Universities for Basic and Applied Sciences, vol. 24, no. 1, pp. 19–25, Oct. 2017, doi: https://doi.org/10.1016/j.jaubas.2017.05.002.

[10] S. B. Ginting, Y. Yulia, H. Wardono, Darmansyah, M. Hanif, and D. A. Iryani, "Synthesis and Characterization of Zeolite Lynde Type A (LTA): Effect of Aging Time," Journal of Physics: Conference Series, vol. 1376, no. 1, p. 012041, Nov. 2019, doi: https://doi.org/10.1088/1742-6596/1376/1/012041.

[11] T. saad Algarni and A. M. Al-Mohaimeed, "Water purification by absorption of pigments or pollutants via metaloxide (Review)," Journal of King Saud University - Science, p. 102339, Sep. 2022, doi: https://doi.org/10.1016/j.jksus.2022.102339.

[12] M. I. Mohammed, Sitki Baytak, Synthesis of Bentonite–Carbon Nanotube Nanocomposite,

and Its Adsorption of Rhodamine Dye From Water. Arab J Sci Eng, DOI 10.1007/s13369-016-2190-7

[13] N. O. Omisanya , C. O. Folayan , S. Y. Aku and S. S. Adefila, Synthesis and characterisation of zeolite a for adsorption refrigeration application, Advances in Applied Science Research, 2012, 3 (6):3746-3754

BIOGRAPHIES OF AUTHORS

	**Prof. Dr. Muhammad Ibrahim (PhD - Glasgow) / UK**, Head of the Department of Medical Instrumentation Techniques Engineering in Dijlah College, Baghdad, Iraq. mohammed.Ibrahim@duc.edu.iq
	**Assistant professor Aziz Sencan** , PHD in Environmental Engineering from Suleyman Demirel University, Working in Chemical Engineering Department, College of Engineering, Suleyman Demirel University, Isparta, Turkey. azizsencan@sdu.edu.tr