

Synthesis of a New Nano polymer (HBNP) for the Adsorptive Removal of D-Blue Dye from Aqueous Solutions

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ABSTRACT

Many dangerous dyes can be efficiently removed from wastewater and river water by adsorption. This article discusses the synthesis of a novel Nano polymer (HBNP), which was validated by various techniques including (FT-IR, AFM, ¹H-NMR, X-ray, and ¹³C-NMR) analysis. The novel Nano polymer was employed to eliminate D-Blue dye from the aquatic ecosystem. Furthermore, varied ideal experimental circumstances (Temp., solution pH, sorbent quantity, and contact equilibrium durational) were examined and categorized, along with the adsorption equation, kinetics, and thermodynamic data of distinct sorbents about D-Blue. Ultimately, it was determined that biosorbents, notably biopolymer Nano polymer (HBNP), were highly effective in eliminating D-Blue from aqueous solutions.

Key words: Adsorption, D-Blue dye, (PETGE), Hyper branch nanocopolymer, phthalic anhydride.

INTRODUCTION

The industrial advancement in countries has precipitated environmental issues due to the accelerated production industrial progress in countries has accelerated environmental issues due to the increased production of chemicals that are

difficult to dispose of as well as the accumulation of solid wastes that are difficult to dispose of and are often expensive when needed to dispose of them (including heavy metals , phenols pollutants, dyes, pharmaceuticals and others) [1, 2], where the presence of these pollutants, even in small quantities, is considered a major threat to humans and other living organisms because they have a long half-life when they decompose, for example, synthetic dyes, which are used in various fields, one of which is the field of technology [3]. Dyes are mostly hydrophilic substances used in hair dyeing and skin tattooing, as well as coloring paper with water-based dyes. They can also be used to color some foods such as rice, and dyes are also used in cosmetics and textiles, etc. The elimination color –related contaminants derived from industrial processes or municipal waste has increasingly drawn attention great attention over recent years due to its harmful consequences [4]. Colorants were initially obtained from natural sources such as plants and animals. Then, Natural dyes were largely replaced by synthetic materials in the early twentieth century. Currently, most of the dyes available in the market are synthetic dyes, except for a limited number of inorganic dyes [5]. Due to the increasing demand for colors every year, many new color compounds are introduced into the market, leading to various applications [6]. Also, hydrophobic dyes have been manufactured, called disperse water-insoluble dyes, and are used on cellulose and various synthetic fibers through dispersion. During the dyeing process, the dye undergoes a transformation. hydrolysis, which leads to the gradual precipitation of its insoluble components as a distinction on cellulose acetate. This coloring process occurs under special conditions and with the help of long-chain dispersing agents that stabilize the dye suspension and improve the interaction with water-sensitive fibers. Which are used in synthetic dyeing textiles, such as nylon, cellulose acetate, polyamide, polyester and other synthetic polymers [7, 8]. In the twenty-first century, advanced technology has been used to eliminate pollutants, including dyes, starting at the molecular level, then the atomic level, then to nanoparticles, where it has shown its progress in production and its ability to adapt to treating pollution [9-11]. Among the dyes that cause pollution is D-Blue dye, which is a water-soluble cationic dye compound, identified as a green crystalline solid in powder form and classified as a triphenylmethane compound [12, 13]. A new

nanomaterial was created and utilized for the elimination of dye pollutants. The purpose of this research is to synthesize and describe a new nanomaterial and assess its effectiveness in the purification of D-Blue dye in aqueous solutions. The experiment also aims to identify the best operating conditions of the adsorption process and to be able to learn about the chemical and physical reactions of how the nanomaterial interacts with the contaminating dye.

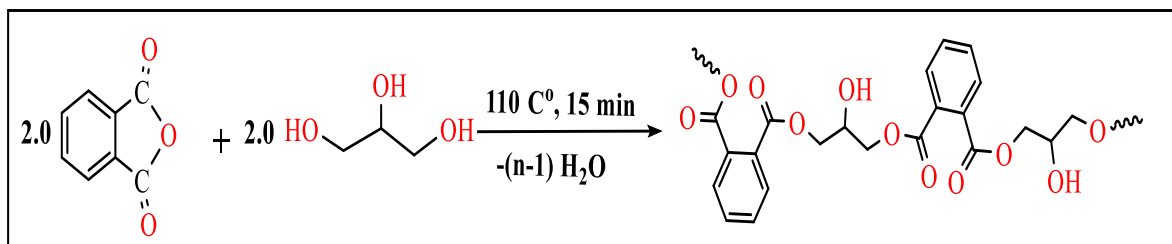
MATERIAL AND METHODS

Materials and Equipment

The chemicals used for this research, such as phthalic anhydride, glycerol, para-xylene, pentaerythritol, potassium hydroxide, dichloromethane, and the solvents used in this research were analytical grade and were sourced from reputable companies like Sigma-Aldrich and Fluka. Fourier transform infrared (FT-IR) spectroscopy was conducted using a Chemunds FT-RAffinity-1S spectrometer and proton (^1H NMR) and carbon-13 (^{13}C NMR) NMR spectroscopy was performed using a Bruker AC 400 spectrometer, at 400 MHz. (2D) and (3D) images of the Nano polymer were taken with an AFLO Company Ltd. AFM spectrometer and XRD scanning was carried out with a Japanese made Rigaku Corporation spectrometer.

Synthesis of Nanocopolymer [14]

In a 250 mL round-bottom flask at 110°C , 444 grams, equivalent to 2.0 moles of phthalic anhydride were solubilized in 35 mL of DMSO. Subsequently, 184 grams, equivalent to 2.0 moles of glycerol were included. The method yielded the nanocopolymer. To eliminate the water molecules produced because of the esterification reaction, para-Xylene was incrementally introduced at a rate of three drops every five minutes during a duration of fifteen minutes. The suspension solution was ultimately produced by including deionized water, and the resultant white precipitate was filtered and collected, as illustrated in the subsequent reaction. As stated in Scheme (1)

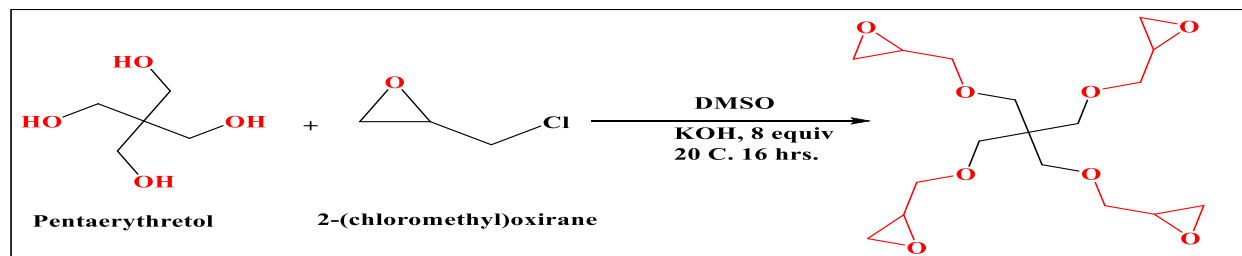


Scheme 1. Diagram of the synthesis of the nanocopolymer.

Synthesis Hyper Branched Nanocopolymer [15]

Synthesis of Pentaerythritol Tetra glycidyl Ether (PETGE):

For 15 minutes at 24°C, utilizing 50 mL of DMSO and 6.8 g (200 mmol) of Pentaerythritol. After the addition of KOH (85%) (26.25 g, 400 mmol, 2 equivalents per OH) in a single instance, the mixture was stirred for an additional 15 minutes at 24°C. An ice bath was employed to cool the reaction mixture to a temperature range of 15-20°C following vigorous agitation with a mechanical stirrer. A pressure-equalizing funnel was employed to inject EPI (55.2 g, 0.6 mol, 3 equivalents per OH) gradually over a duration of 1.5 hours. After the addition, the reaction mixture was allowed to equilibrate at 24°C while stirring for 16 hours. The mixture assumed a faint brownish tint throughout this time. An equal volume of water and DCM was utilized to extract the reaction mixture. DCM, as seen in Scheme 2.

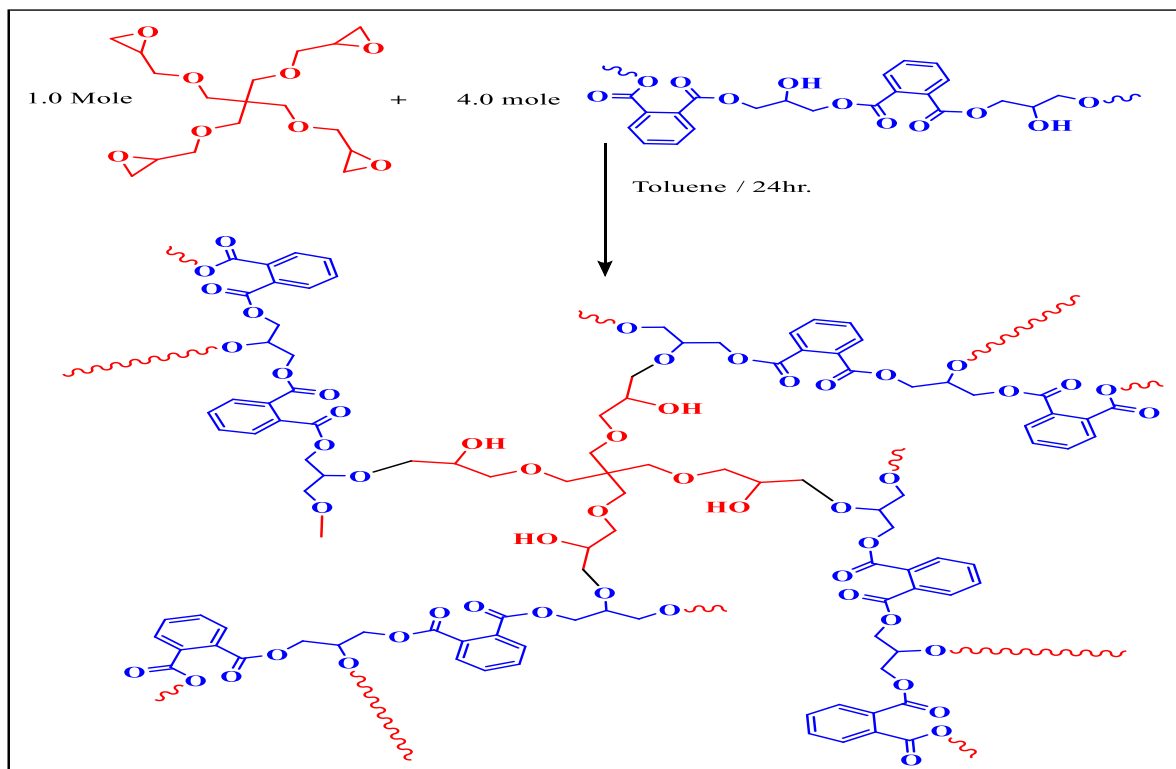


Scheme 2. The synthesis of Pentaerythritol Tetra glycidyl Ether.

Hyper branch nanocopolymer (HBNP) [14]

The synthesis included the incorporation of a nanocopolymer into the previously described reaction result. Pentaerythritol tetra epoxide, with a mass of 1.802 g (equal to 0.005 moles of epoxide groups), was subjected to mechanical agitation at ambient temperature in 100 mL of the toluene. Heating mantle was activated, and the fluid was incrementally heated to 105

°C. The nanocopolymer (0.01 mile) (6 g) was introduced into the flask. The stirrer was adjusted to roughly 200 rpm, and the reaction continued for 24 hours. The product was later separated via filtration and extensively washed with hot methanol. Consult Scheme 3.



Scheme 3. Diagram of the structure of the hyperbranched nanocopolymer.

RESULTS AND DISCUSSION

Fig. (1) depicts the FT-IR spectra. The synthesized nanocopolymer was analyzed to assess the reactive functional groups via (FT-IR) spectroscopy within the range of $(400-4000) \text{ cm}^{-1}$. The produced copolymer displays a wide band within the range of $(2400-3400) \text{ cm}^{-1}$, signifying the vibration of the acidic OH group overlapping with the alcoholic OH group. A band at 3057 cm^{-1} is aromatic ($=\text{CH}$) group, whereas bands at 2998 and 2873 cm^{-1} are aliphatic ($-\text{CH}$) group. The band at 1760 cm^{-1} is indicative of the ester ($\text{C}=\text{O}$) linkage, while the band at 1667 cm^{-1} is ($\text{C}=\text{O}$) in terminal carboxylic groups. Furthermore, the bands at $(1584$ and $1470) \text{ cm}^{-1}$ attributed to the aromatic ($\text{C}=\text{C}$) groups [16, 17].

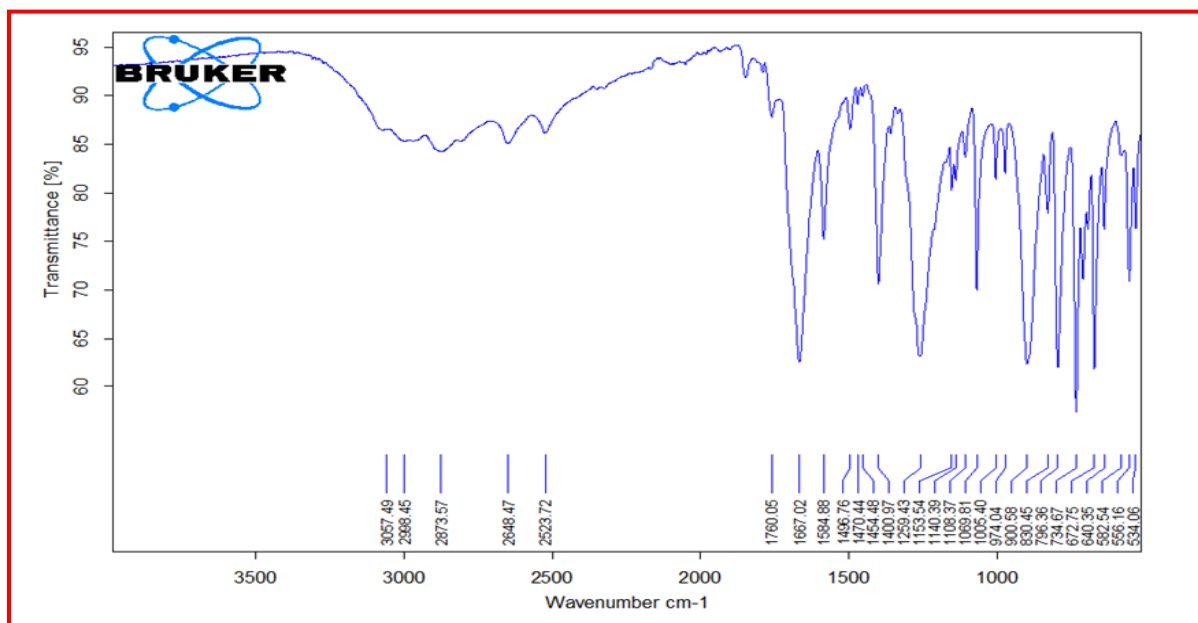


Figure 1. The peaks of the FT-IR active functional groups of the manufactured nano polymer.

Fig. (2) shows $^{13}\text{C-NMR}$ (101 MHz, DMSO- d_6) δ , the singlet broad peak at 145-160 ppm assigned to the C-OH of the carboxylic groups. Peaks from 115 ppm to 135 ppm are returns to the aromatic carbon of atoms 6-1, 20-25 and 36 - 41 . Peaks at 128 ppm to 130 ppm to the carbons of aromatic rings of atoms, singlet broad peak at 60-80 ppm returns to the C-O group (atoms 13- 32).[18, 19].

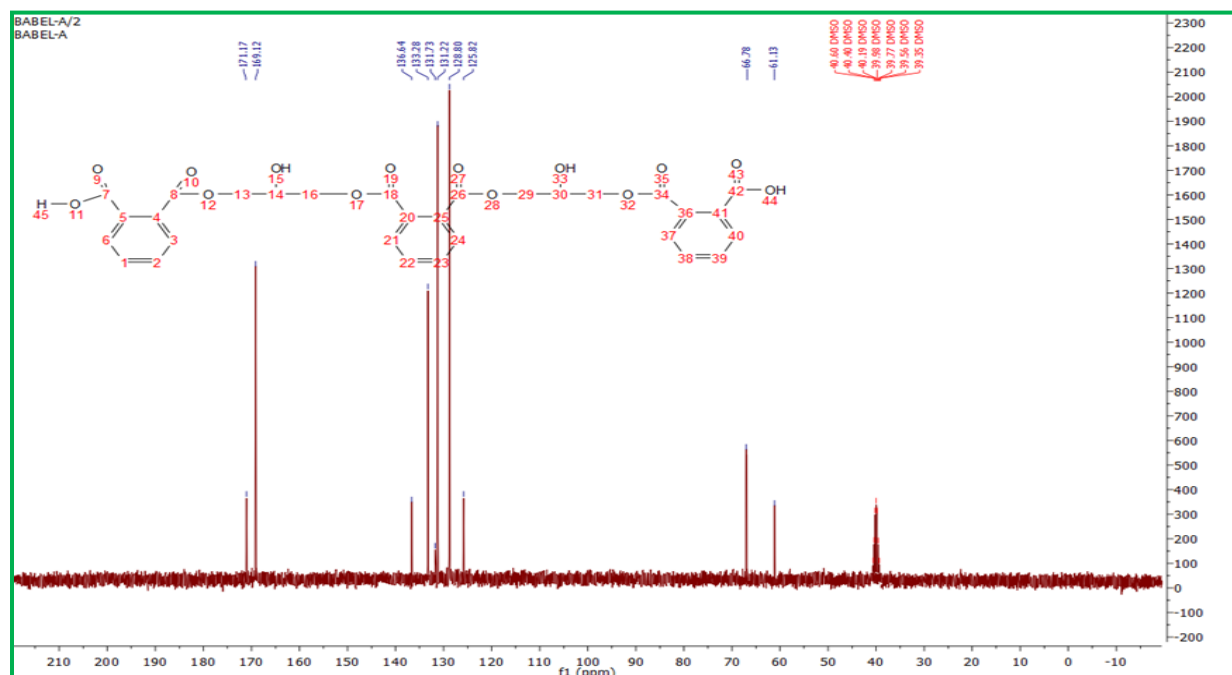


Figure 2. The $^{13}\text{C-NMR}$ of the synthesized nano polymer.

Fig. (3) shows $^1\text{H-NMR}$ (400 MHz, DMSO- d_6) δ , 13.0 ppm (for the proton of the carboxylic group), 7.5 – 8.2 ppm for (proton Aromatic), 7.7 ppm for (the aromatic protons in the polymer chain), 2.5 ppm for (d_6 -the solvent DMSO), 3.3 ppm for (the water in the NMR solvent), 2.6 ppm for (C-H aliphatic.), [20-22].

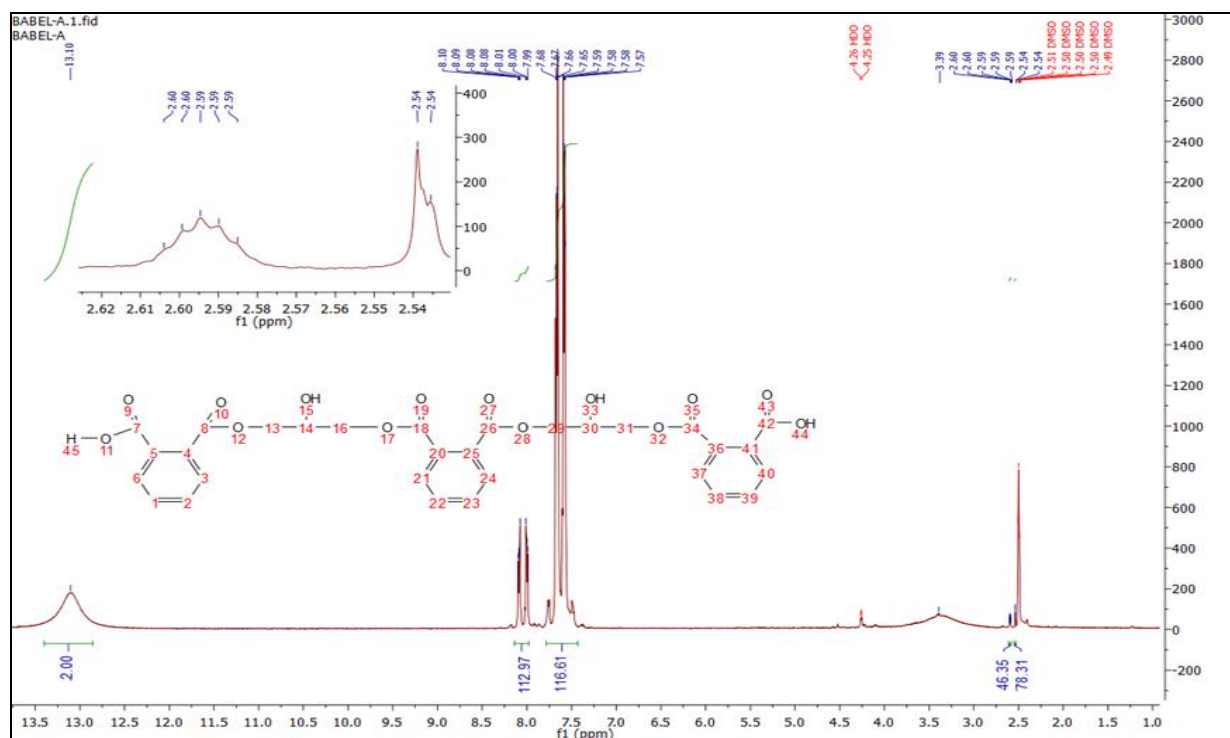


Figure 3. The $^1\text{H-NMR}$ of the synthesized nano polymer.

AFM technique was used as an initial indication that the synthesized co-polymer has a particle in the nano size range (10-100) nm. The principal work of AFM depends upon the sensing of surface by utilizing a silicon probe containing extremely sharp tips. This tip employs to image the test sample via raster scanning line by line. The above-mentioned tip is joined with a cantilever in a spring form. When the tip is in contact with the surface, the cantilever will bend, and this value of bending is detected by a laser diode and photo detector then gives the measurement. Figs. (4&5) show the (1D and 2D) images for the outer surface of the synthesized co-polymer. The roughness of the surface and the square of the root square were calculated as in below equation (1) [23].

$$R_m = \sqrt{\sum_{i=1}^n \frac{(Z_i - Z_{av})^2}{N}} \quad \dots(1)$$

Where N, Z = the number of measured points.

First evidence that the co-polymer is counted as nanomaterial is the roughness coefficient which is equal to 29.3 nm, furthermore, its square root is 35.7nm and this emphasizes that the bulk size of the co-polymer nanoparticles has an importance in its roughness, in addition to its surface homogeneity and crystalline system. AFM outcome indicates the particles size of the polymer were 81.17 nm.

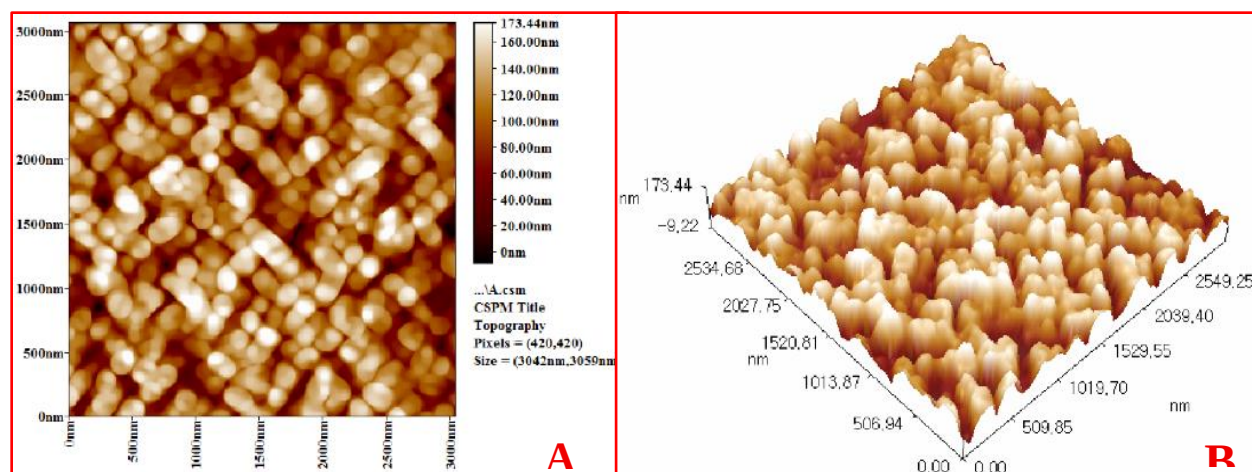


Figure 4. A (2D) and B (3D) micrograph of the nano copolymer surface.

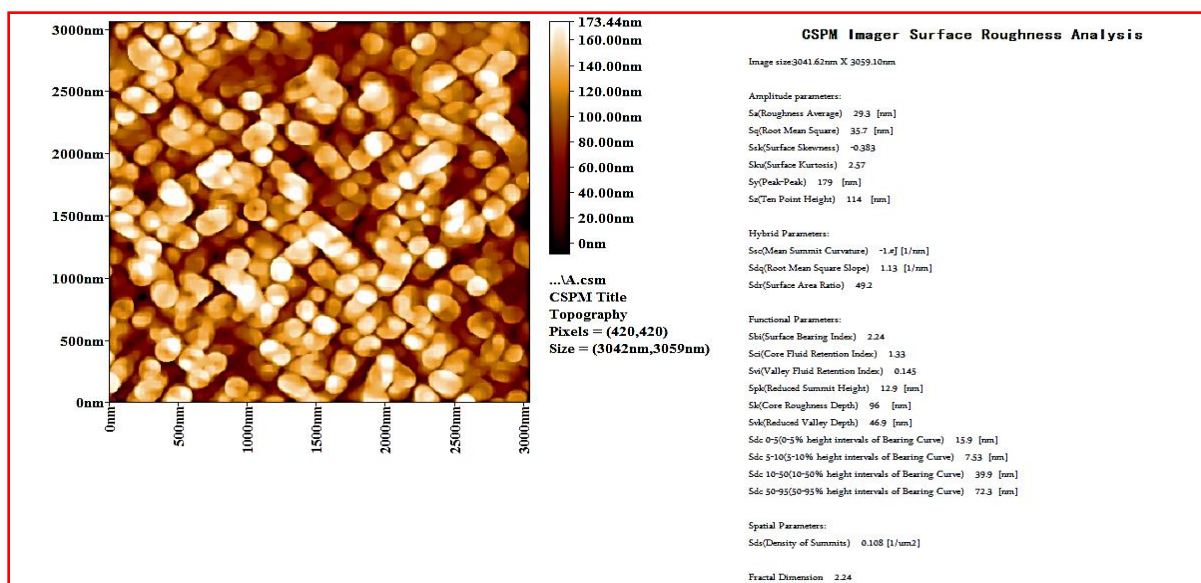


Figure 5. The (2D) micrograph and all other characteristics of the nano copolymer surface.

Table 1. The Outcomes of the AFM analysis for the synthesized copolymer.

<=10% Diameter:0 nm					Avg. Diameter: 81.17 nm			
<=90% Diameter:100.00 nm					<=50% Diameter:70.00 nm			
Volume (%)	Cumulation (%)	Diameter (nm) <	Volume (%)	Cumulation (%)	Diameter (nm) <	Volume (%)	Cumulation (%)	Diameter (nm) <
3.67	88.57	145.00	0.82	95.51	65.00	15.10	15.10	100.00
2.86	91.43	150.00	0.82	96.33	70.00	22.45	37.55	105.00
0.41	91.84	155.00	0.82	97.14	75.00	17.96	55.51	110.00
0.41	92.24	160.00	0.41	97.55	80.00	10.20	65.71	115.00
1.22	93.47	165.00	0.41	97.96	85.00	7.76	73.47	125.00
0.82	94.29	170.00	0.82	98.78	90.00	6.53	80.00	130.00
0.41	94.69	175.00	1.22	100.00	95.00	4.90	84.90	135.00

Structure of the solid state for the synthesized co-polymer was tested by X-ray diffraction (XRD) to determine its properties like purity, crystalline size and crystalline phase. Fig. 6; displays the spectra of XRD for the co-polymer nanomaterial which refers that all the diffraction peaks appear in excellent matching with the previously prepared polyester. When the size of the synthesized crystals is very small, a diffraction peak must appear, so the appearance of these diffraction peaks is the graph of the studied co-polymer refers to formation of this nano compound, it has been reported that the decreasing in the crystal size is accompanied by increase in the intensity of these peaks. The space area of the XRD peaks attributed to four reasons: micro strain for instance lattice deformation, crystalline fracture which is caused by defects of crystallization field size and the crystal domain size. Debye-Scherrer equation [24] was utilized to calculate the particle size for the copolymer nanomaterials.

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

D and k represent the average of crystals size and shape factors respectively which is equal to 0.9, λ is the wavelength of the X-ray radiation (0.1056 nm), β is the full width of half maximum height FWHM, finally θ is the deviation angle. This equation is used to the average size of the polymer particles which are around (nm) [25, 26].

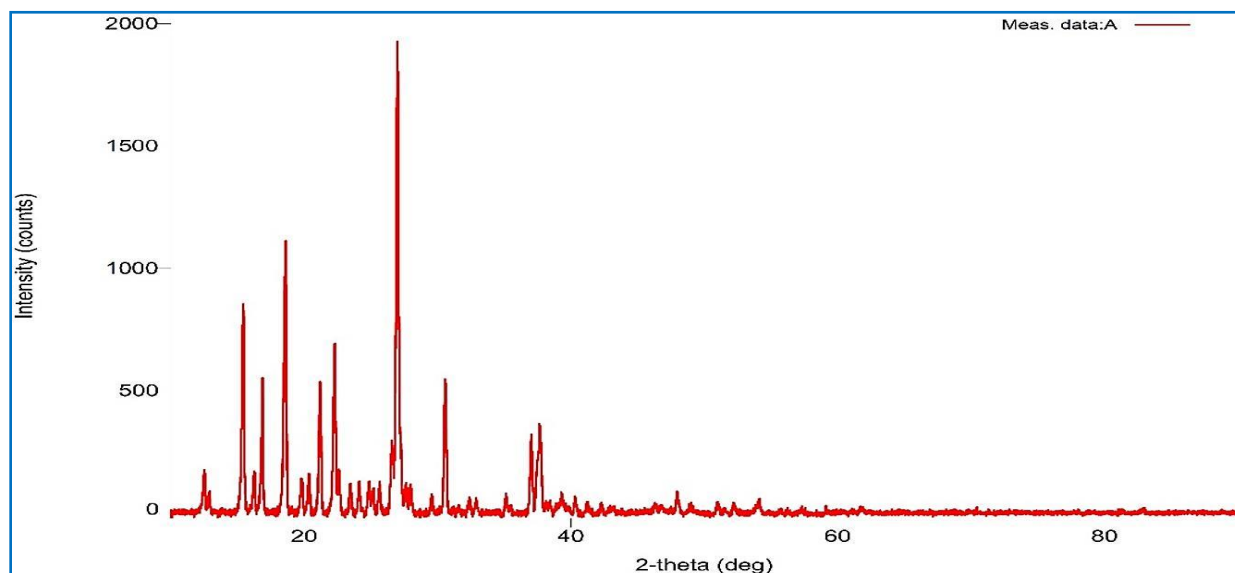


Figure 6. X-Ray spectrograph of the nanocopolymer.

Fig. 7, FT-IR spectrum for HBNP: showed several prominent absorption bands in the, specifically, the sharp band that appeared at (3432.86 cm^{-1}) resulted from the vibration of the (OH) group, while the band that appeared at (3080.23 cm^{-1}) was due to the aromatic (C-H) and also showed a band at ($2968.42 - 877.21\text{ cm}^{-1}$) resulting from the vibration of the aliphatic (C-H) bond, while the band that appeared at (1691.43 cm^{-1}) represents the ester group (O-C=O) and also showed a band at (1281.41 cm^{-1}) resulting from the vibration of (C-O) [27, 28].

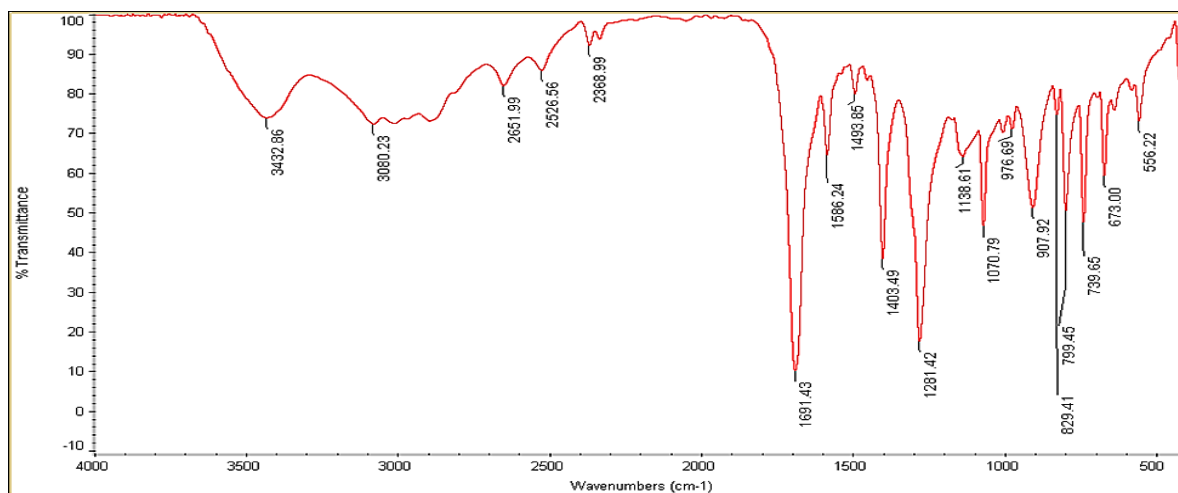


Figure 7. FT-IR of HBNP.

Fig. 8, ^1H -NMR spectrum for HBNP: it showed a set of signals; the signal that appeared at (13.16 ppm) is for the hydrogen proton in the acidic hydroxyl group, while the signals that appeared from ppm (7.62 - 7.76) are due to the hydrogen protons in the aromatic rings. As for the signal that appeared at (3.23 ppm), it indicates the hydrogen in the (OH) group, which is due to the hydrogen proton of the water molecule. As for the signal (2.5 ppm), it is due to the solvent (DMSO- d_6) [29-31].

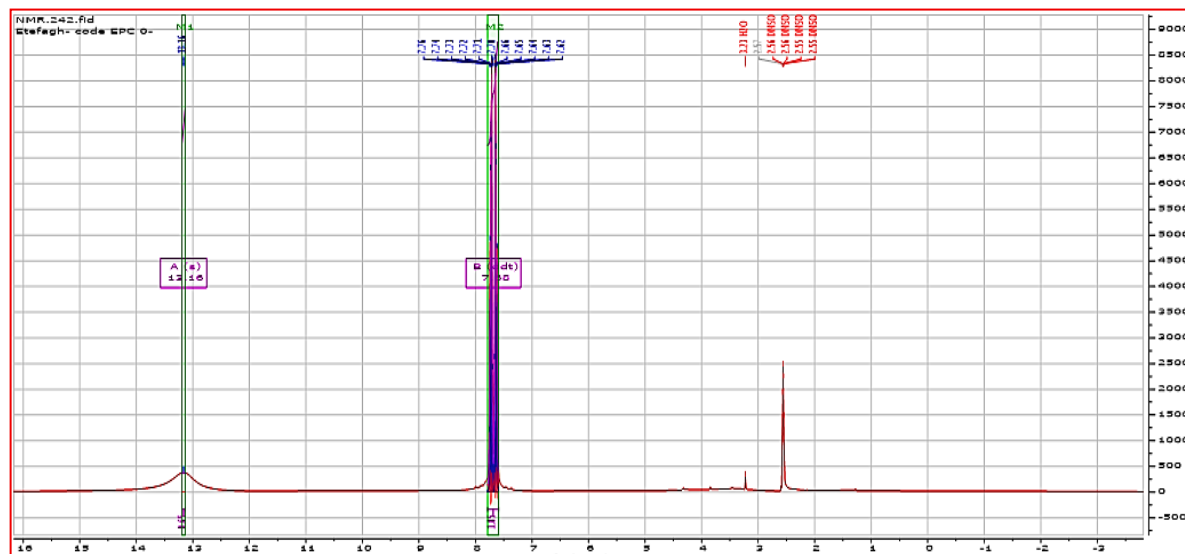


Figure 8. ^1H NMR spectrum of HBNP.

Fig. 9 (A&B) showed the outer surface of the nanoparticles of (HBNP). The rough coefficient of a linear (HBNP) surface was 1.37nm and the square root square was equal to 1.58 nm. This indicates that the bold size of the nanoparticles plays an important role in the roughness of the surface, its uniform crystalline system, and the surface homogeneity. Also, the average height of the particles was equal to 5.26 nm, as observed in Fig. (9 A). Table 2 represents the total rate of the particle sizes of the common nanoparticle and the different proportions of these volumes; the results indicate that the molecular size of (HBNP) nanoparticle was 97.53 nm.

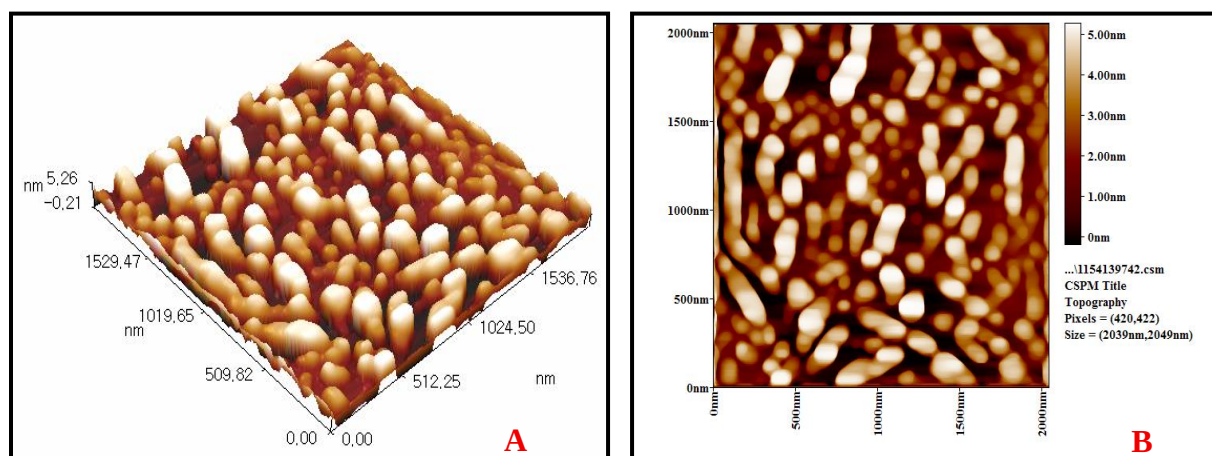


Figure 9. The A(3D) and B (2D) images displayed by the AFM for HBNP.

Table 2. The total (HBNP) nanoparticle particle size rate and the various ratios of these volumes.

Sample: 2			Code: Sample Code		
Line No.: Line no.			Grain No.:130		
Instrument: CSPM			Date: 2024-01-28		
Avg. Diameter: 97.53 nm			<=10% Diameter: 65.00 nm		
<=50% Diameter: 95.00 nm			<=90% Diameter: 125.00 nm		

Diameter (nm)<	Volume (%)	Cumulation (%)	Diameter (nm)<	Volume (%)	Cumulation (%)	Diameter (nm)<	Volume (%)	Cumulation (%)
55.00	0.77	0.77	85.00	6.92	30.77	115.00	7.69	79.23
60.00	1.54	2.31	90.00	8.46	39.23	120.00	2.31	81.54
65.00	3.08	5.38	95.00	6.92	46.15	125.00	3.85	85.38
70.00	7.69	13.08	100.00	8.46	54.62	130.00	5.38	90.77
75.00	3.08	16.15	105.00	8.46	63.08	135.00	6.92	97.69
80.00	7.69	23.85	110.00	8.46	71.54	140.00	2.31	100.00

Fig. 10: The X-ray diffraction in the nanoparticles HBNP: shows peaks at 2θ values of ($15.3^\circ, 18.5^\circ, 21.1^\circ, 22.2^\circ, 26.9^\circ, 30.5^\circ$). These peaks indicated that the new co-polymer (3) has been formed as a crystalline compound with less amorphous carbon atoms. By using origin software, the average inters planer spacing between atoms (dhkl) was 0.416 nm according to Bragg's equation (eq. 3):

$$n\lambda = 2d\sin\theta \quad \dots(3)$$

The total average crystallites size was 97.27 nm relative to Scherrer's equation(eq. 2).

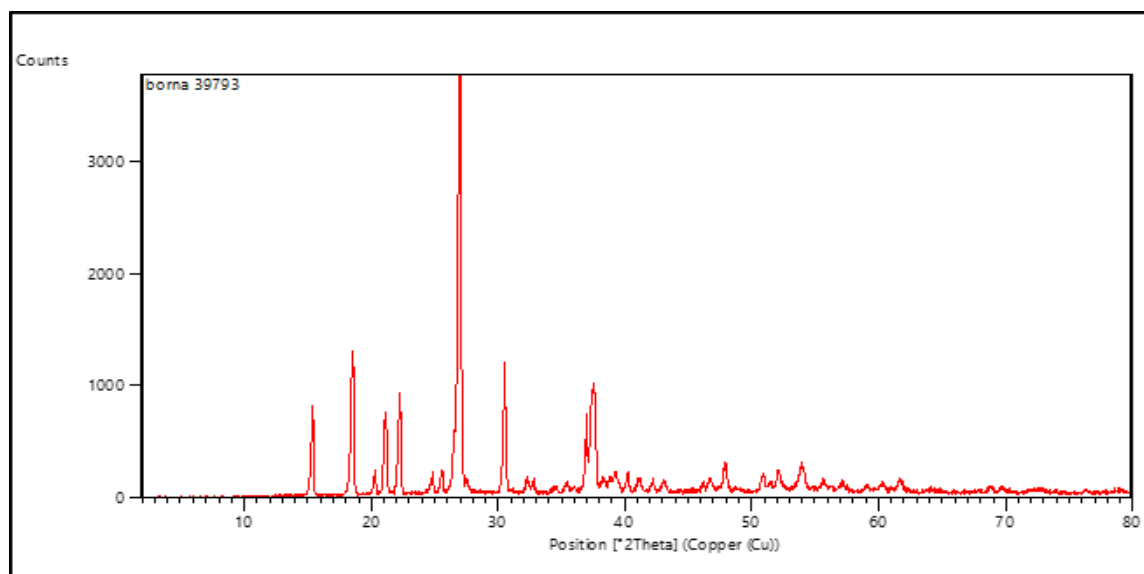


Figure10. The x-ray diffraction of HBNP.

Table 3. The ratios of crystallite dimensions and interatomic distances in the (HBNP).

2 θ	θ	FWHM	D (nm)	d _{hkl} (nm)	D (Av.) (nm)	d _{hkl} (Av.) (nm)
15.37142	7.68571	0.065976	121.5031	0.575973	97.4776	0.4163
18.5194	9.2597	0.082717	97.31015	0.478716		
21.13842	10.56921	0.086017	93.9511	0.419958		
22.21383	11.10692	0.081133	99.78578	0.399864		
26.93075	13.46538	0.102355	79.80898	0.330803		
30.50876	15.25438	0.090184	91.30647	0.292773		

D-Blue

Table 4. Adsorption of D-Blue on the surface of HBNP at 298K

Temperature	Concentration(ppm)	Equilibrium concentration	Equilibrium adsorption capacity
298K	1	0.1202	8798
	3	0.2468	27532
	5	0.3101	46899

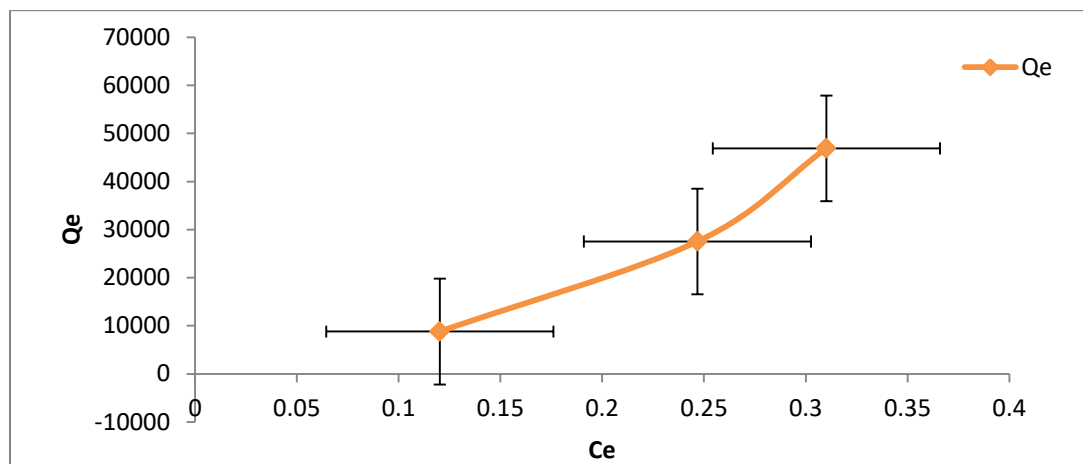


Figure 11. Adsorption isotherm study of D-Blue dye onto nanocopolymer surfaces.

Table 5. Effect of temperature (298, 308, and 318 K) on D-Blue dye adsorption onto HBNP using the Freundlich isotherm model.

Conc.	298K		308K		318K	
	LogC _e	Log Q _e	LogC _e	Log Q _e	LogC _e	Log Q _e
1	-0.92	3.9443	-0.7363	3.9119	-0.6076	3.8769
3	-0.6076	4.4398	-0.301	4.3979	-0.1612	4.3636
5	-0.5084	4.6711	-0.1612	4.6344	-0.0254	4.6082

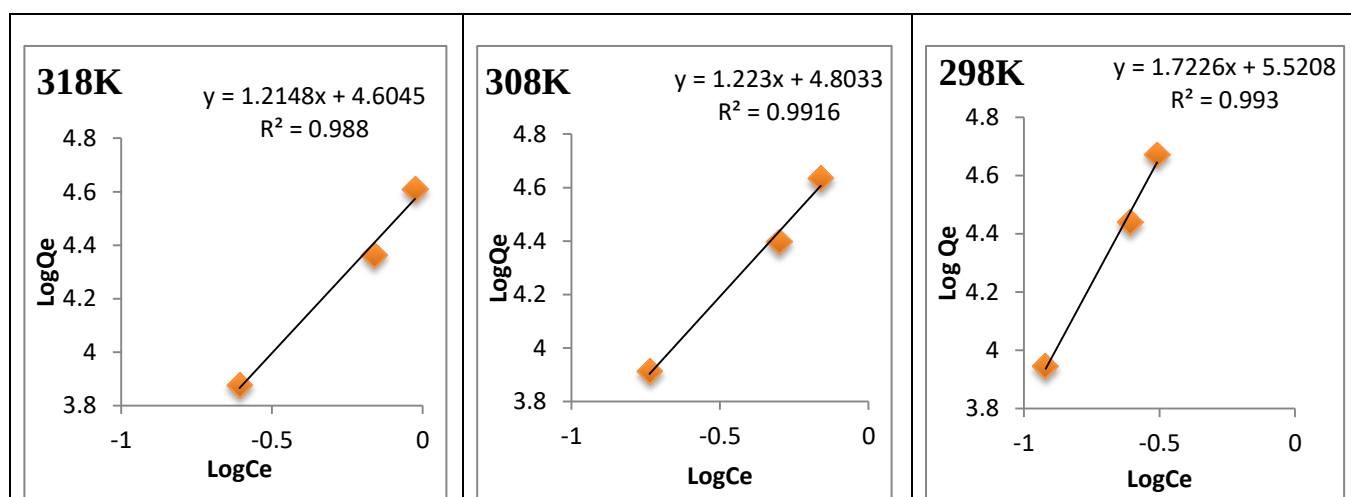


Figure 12. The application of Freundlich isotherm analysis on D-Blue dye adsorption onto HBNP at 298 K, 308Kk and 318K.

Table 6. Adsorption of D-Blue dye on the surface of HBNP at 298K, 308K and 318K (by applying Langmuir equation).

Conc. (ppm)	298K		308K		318K	
	C_e	$C_e/Q_e \times 10^{-5}$	C_e	$C_e/Q_e \times 10^{-5}$	C_e	$C_e/Q_e \times 10^{-5}$
1	0.1202	1.3811	0.1835	2.2473	0.2468	3.2766
3	0.2468	0.8964	0.500	2.000	0.6898	2.9858
5	0.3101	0.6612	0.6898	1.6003	0.943	2.3243

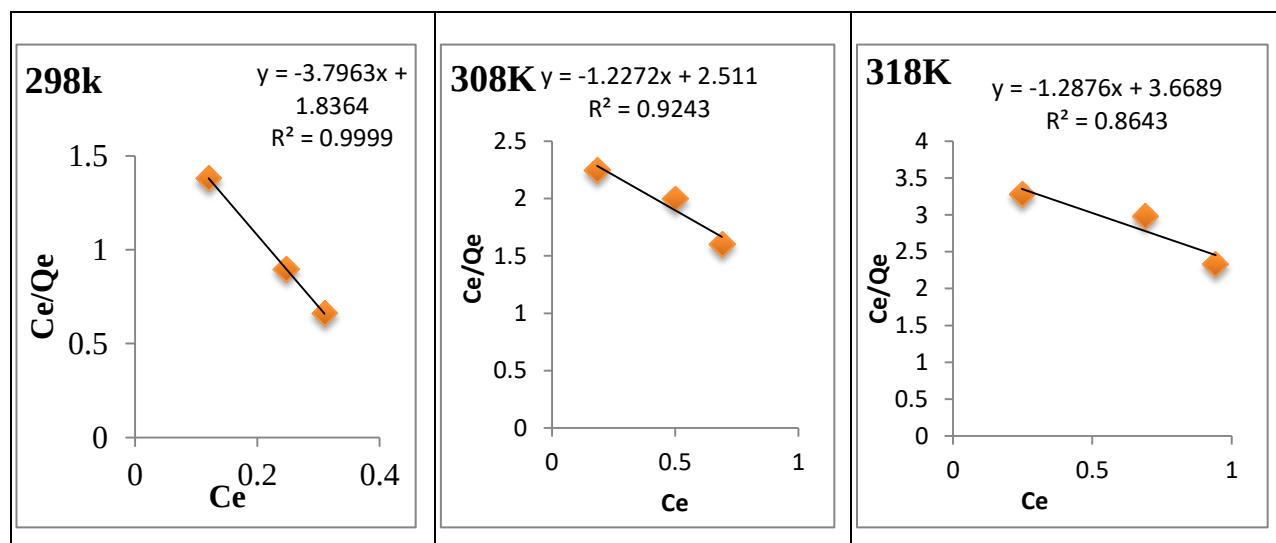


Figure13. The application of the Langmuir equation to D-Blue dye adsorption on HBNP at 298K, 308K, and 318K.

Table 8. The Temkin isotherm analysis of D-Blue dye adsorption onto HBNP at 298K, 308K, and 318K

Conc. (ppm)	298K		308K		318K	
	$\ln C_e$	Q_e	$\ln C_e$	Q_e	$\ln C_e$	Q_e
1.00	-2.1185	8798	-1.6955	8165	-1.3991	7532
3.00	-1.3991	27532	-0.6931	25000	-0.3713	23102
5.00	-1.1708	46899	-0.3713	43102	-0.0586	40570

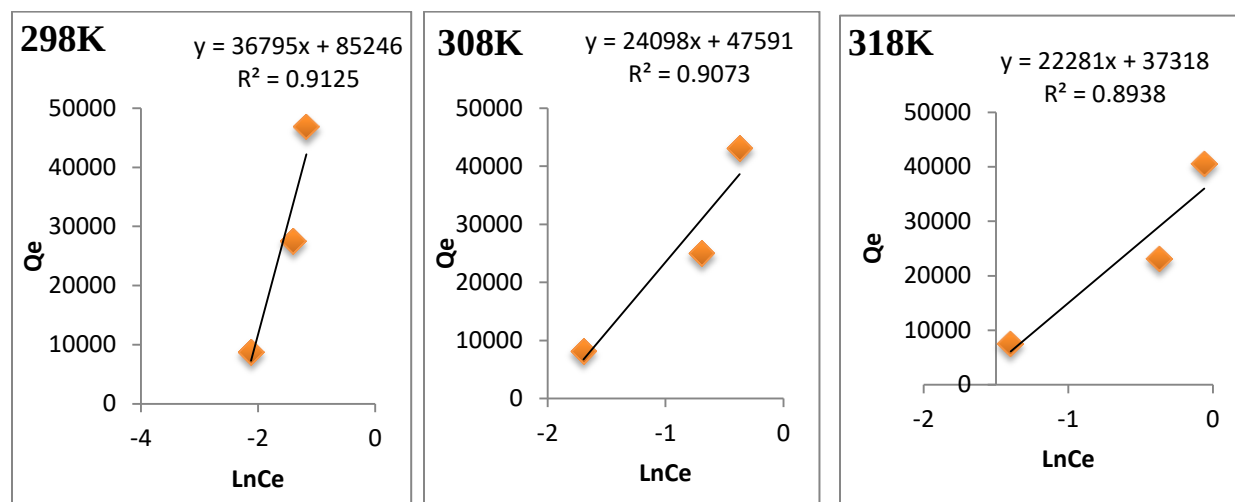


Figure 14. The Employing the Temkin equation to study D-Blue dye adsorption on HBNP at 298K, 308K, and 318K.

Conclusions

This study indicated the appropriate selection of green organic compounds and methodologies used in the synthesis of nanoparticles for different purposes in relation to environment and human health. The synthesis of HBNP Nano polymer was successfully carried out. Different techniques were used to investigate the synthesized nanoparticles, where these techniques confirmed the formation of spherical nanoparticles with good thermal stability. The effectiveness of the synthesized HBNP nanoparticles in the removal of D-Blue dye was investigated. In this article, the advantages of Adsorbents, including economic materials, nanoparticles, nanocomposites materials and composites, were utilized for the adsorption and elimination of D-Blue from aqueous solutions and sewage water. They were acknowledged as efficient and cost-effective adsorbents for the elimination of D-Blue dye. This page also discusses many experimental settings, including temperature, adsorbent, contact duration, and pH quantity, pertaining to the relevant adsorbents. This review paper indicated that the adsorption models of Freundlich and Langmuir have been commonly utilized to assess the adsorption capability of diverse adsorbents for D-Blue.

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