Study the Azure A dye adsorption on the surface of the Snail shell modification

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Abstract. This study was included (Snail shell modification) snail shell melamine formaldehyde polymer (SSMFP). It is an effective adsorbent to remove Azure A dye from aqueous solution. The effects of pH, contact time, ionic strength, temperature and amount of adsorbents were evaluated. The highest adsorption efficiency was recorded at 93.90% with 5 mg/L dye according to the Beer Lambert (Dye Calibration Curve), 0.0050 g of adsorbent 10 minutes contact time and 298 K. Elimination data using Temkin, Langmuir and Freundlich models were analyzed at different temperatures ranging from 298 to 338 K, and the adsorption thermodynamic parameters were studied. Where ΔG values indicate an automatic process, ΔH indicates the exothermic property of the adsorption process and ΔS proves an increase. The adsorption surface was also studied and diagnosed by FT-IR, X-RD, AFM, and SEM technologies

Key. Adsorption, Azure A, Isotherms, Langmuir, Freundlich and Temkin, snail shell modified, FT-IR, X-RD, AFM, and SEM.

1. Introduction

The textile industries are a source of wastewater pollution. The presence of 1 mg/L of some types of dyes in wastewater produces a noticeable color (1). The textile industries have many damages to water pollution, and the reason is due to the discharge of liquid waste in the various receiving bodies, including ponds, swamps, rivers and other public sewers (2). The water used in the textile industry is almost all as wastewater. Liquid pollutants are complex because they contain surfactants, salts and toxic organic materials, etc (3) It has been estimated that the textile industries release more than 100 tons of dyes annually (4,5). The large production
of textile industries can cause a great environmental pollution, which makes it a serious source and concern for water pollution. Nowadays there is a great interest in developing wastewater treatment technologies that contain non-degradable and organic toxic materials \(^{(6)}\). Removing dyes from the environment is one of the most important things that concern researchers. Therefore, researchers resort to devising low-cost methods to remove these dyes from the water environment. Many found methods used to remove pollutants from sewage or environmental waters. Most of them can be classified as physical, chemical, or biological purification processes \((\text{Scheme 1})^{(7)}\). Among these techniques, adsorption is classified as one of the most well-known processes \((8)\). In this research, the adsorption method was used. The adsorption as a phenomenon that collects materials such as ions, atoms or molecules in their gaseous or liquid state on the surface of a solid, the adsorbing material on the surface is called the adsorbate and the surface on which the adsorption occurs is called the adsorbent surface (adsorbent). Adsorption is an economical, simple and efficient technique \((9)\). Commercial dyes are classified according to their color, application and structure. Their assortment in terms of their chemical nature is presented in Table 1 \((10)\). The dye used was Azure A and the surface used to remove Azure A dye was Snail Shell Powder modified (Snail Shell-Melamine-Formaldehyde Polymer) Snails belong to the Rostellaria family, and this type of snails is located at the edges of beaches and lakes. Samples were taken from this study from the southeastern coast of Lake Al-Raza'a located in Karbala Governorate. Melamine formaldehyde polymer It is one of the most powerful and toughest thermosome polymers and provides good properties and performance. It is an amino resin and has many advantages, such as transparency, better toughness, excellent boiling resistance, scratch resistance, anti-corrosion, insoluble in water, and moisture resistance, resulting in MF use for large industrial applications \((11)\). The reason why it is good for working as a conjugate surface with its mechanical and thermal properties as well as electrical properties showing high resistance to temperature and hydrolysis, \((12)\).

**Methods for removal of pollutants**

![Scheme (1) Method for removal of pollutants](image-url)

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Tab. [1] dividing the pigments by their nature

| Class          | Application                                      | Examples                        |
|----------------|--------------------------------------------------|----------------------------------|
| 1   Acid dyes  | “Silk, nylon, wool, modified acrylic, paper, leather, food, inkjet printing and cosmetics” | Acid red 88, Acid red 18        |
| 2   Cationic "Basic" dyes | Poly acrylonitrile, modified nylons, paper, modified polyester, cationic dyeable polyethylene terephthalate, wool, silk, tannin mordanted cotton and medicine. | Crystal Violet, Methylene Blue, Safranin, Azure (A,B,C), Basic fuschin |
| 3   Disperse dyes | Nylon, polyester, cellulose, acrylic fibers and cellulose acetate. | Disperse Red 1, Disperse Orange 37 |
| 4   Direct dyes | Rayon and cotton, leather, paper and nylon. | Congo Red, Brilliant Blue, copper blue 2R |
| 5   Reactive dyes | Wool, nylon, cotton and other cellulosic. | Reactive Black 5, Reactive Orange 16 |
| 6   Solvent dyes | Gasoline, plastics, oils, lubricants and waxes. | Solvent (Red 1, Red 49, Red 24, Red 111) |
| 7   Sulfur Dyes | Cotton and rayon, paper, leather, silk and wood. | Sulfur Brilliant Green, Sulfur black 1 |
| 8   Vat Dyes | Cotton, rayon and wool. | Vat red 10, vat violet 13 and vat orange 1 |

2. Experiments

1.2. Materials

Use materials available on the local market and of high purity. Azure-A, adsorbent Snail shell Melamine Formaldehyde Polymer (SSMFP). was used.

2.2. Preparation of the dye

The solution of Azure A dye Name IUPAC \((N,N\text{-dimethylphenothiazin-5-ium-3,7-diamine chloride})\) was set up with concentration of \((100 \text{ mg/L})\) as a stock solution, in which \((0.0050 \text{ g})\) of the used dye was dissolved in \((100 \text{ ml})\) of D.W.

Some physical properties of the dye: Chemical formula \(\text{C14H14CIN3S}\), water Soluble, \((\text{M.Wt} \ 291.80 \text{ g/mol})\) \((\lambda_{\text{max}} 633 \text{ nm})\) \(^{(13)}\). The structures are shown in Figur.[1].
2.3. Preparation of a Snail shell Melamine Formaldehyde Polymer (SSMFP)

A. The Snail shell- Melamine Complex (SSMC) was prepared by mixing 3.4500 g of melamine with 1.3800 g of the Snail shell powder in a ceramic mortar and adding three drops of distilled water to the mixture and then mixing for half an hour, then put the mixture in a closed vial Leave for 15 days to complete the distribution of melamine particles in the snail shell powder.

B. The SSMFP polymer was prepared by taking a weight of 4.7105 g from Snail shell-Melamine Complex (SSMC), then placed it in a conical vial and adding 1.5 mL of formaldehyde and then left the mixture for half an hour. Then the mixture is placed in a water bath at 90 °C for two hours to complete the bonding process (SSMC) and formaldehyde.

3.3. Surface study

The surface was diagnosed using FT-IR, X-RD, AFM, and SEM techniques as shown in Figures 2,3 ,and 4.

FT-IR (KBr, cm\(^{-1}\)): \(\nu\) 3469.39 (N-H), 3331 (OH), 2813.21 (C-H), 1549.50 (C=N).

Figur.[ 2 ]: FT-IR infrared spectrum of the SSMFP.
AFM is a common method for diagnosing the nature of entanglements, number of layers, as well as surface thickness and roughness. Porosity and roughness of surfaces is an important feature of the topography, so it can be compared with different surfaces (17). Figure 4. Three-dimensional image AFM images represent the surface of the Snail shell (Snail shell - melamine - formaldehyde) in which the thickness appears 5.12 nm.

Use an electronic scanning microscope (SEM) to visualize the surface appearance of the SSMFP surface Figure 4. Where spherical shapes consist of well-connected groups shown in shape and size. The surface was flat with low roughness, increasing the surface area and thus increasing the effective absorption positions (18). After specifying most of the properties of the SSMFP Powder, use to remove Azure A dye.

**Batch Adsorption Experiments**

Using (0.0050 g) of adsorbed with (25 mL) of dye solution (5 mg/L) at (150 rpm) on a thermal chaker bath. Use the Shimadzu “UV-Vis 1800” digital double beam at a wavelength of $\lambda_{\text{max}}$ to analyze the non-absorbing floating liquid of the remaining dye in each solution. Also,
(pH, contact time, the effect of ionic strength and the effects of temperature) were also studied. The amount of adsorption is expressed by the ratio \((x/m)\) which is defined as the amount of adsorbent in an mg retained by the adsorption weight (g).

\[
\text{Removal}\% = \frac{C_0 - C_e}{C_0} \times 100 \quad \ldots \ldots \ldots \ldots \ldots (1)
\]

\[
Q_e = \frac{V(C_0 - C_e)}{m} \quad \ldots \ldots \ldots (2)
\]

Where:

- \(Q_e = x/m\): is the amount of adsorbed substance in (mg/g) adsorbent,
- \(V\): is the volume of pesticide solution L that was used,
- \(C_0\): Is the concentration at the initial (mg/L),
- \(C_e\): Is the concentration of the equilibrium state (mg/L),
- \(m\): is the weight of adsorbent substance in (g)

3. RESULTS AND DISCUSSION:

3.1. Effect of Equilibrium Time

The relationship between the percentage and contact time of the Azure A dye is accomplished through the combined trials to achieve balance as shown in Figure 5. The results showed that the best balance time is within 10 minutes.

![Figure 5: Effect of equilibrium time at Temperature 298 K, Conc.5 mg/L on adsorption of Azure A dye by SSMFP](image)

3.2. Adsorbent weight effect

A weight from 0.0050-0.0800 g (SSMFP) was taken at 298 K and the initial dye concentration was 5 mg / L. The percentage of dye removal increases with the increase in the surface weight due to the increase in the surface area and then reaching a constant value that represents the saturation of the surface (SSMFP) for the Azure A dye \(^{(21)}\), shown in Figure 6.
Figur. [6]: Effect adsorbent weight on adsorption of Azure A dye by SSMFP at 298 K.

3.3. Acidity (pH) effect
The effect of pH on the adsorption of dyes is the most important pH factors that influence the adsorption process, because it can simultaneously affect the surface charging of the adsorbent, the degree of ionization of the functional groups of the absorption, as well as the adsorption mechanism \(^{(22)}\). To study the concentration of hydrogen ion (pH) on Azure A dye by the absorbent SSMFP, the dye solutions were prepared at 5 mg/L and set to different pH values (2-12) using media. Acid and basicity of 0.1 N of Hydrochloric acid and 0.1 N of Sodium hydroxide. The results are shown in Figure 7. It is clear that the amount of dye removed at a different pH. Dye removal found in a minimum of pH=2.0 and the adsorbed dye increased with increasing pH from (2 to 10). Then there has been a noticeable pH change. For this reason, pH 10 was chosen for further trials.

Figur. [7]: Effect pH on adsorption of Azure A dye by SSMFP at 298 K.

3.4. The effect of ionic intensity
Several experiments were make at pH 10.0 to see the effect of ionic intensity on the absorption capacity and the removal rate using different concentrations ranging from (0.02-0.07) molar to each of the salts (NaCl, KCl, MgCl\(_2\), CaCl\(_2\)). The effect of these salts was with the Azure A tincture, as the percentage of removal was as high as possible with
sodium chloride salt. Figure 8 shows the result obtained in the effect of ionic intensity on dye removal.

![Figure 8: Effect of ionic intensity on the adsorption of Azure A dye by SSMFP at 298 K](image)

**3.5. Temperature effect**

Azure A has been studied using a cochlear melamine formaldehyde polymer (SSMFP) within the thermal range (298-338 K). Thermodynamic parameters such as free energy ($\Delta G$), enthalpy ($\Delta H$) and entropy ($\Delta S$) have been calculated using equations 3 to 6, which are given in table 2. Figure 9 shows an effect affecting temperature.

\[
K_{eq} = \frac{Q_E x m}{C_e x V} \quad \ldots \ldots \ldots \ldots \ldots (3)
\]

\[
\Delta G = -RT \ln K_{eq} \quad \ldots \ldots \ldots \ldots \ldots (4)
\]

\[
\ln K_{eq} = \frac{-\Delta H}{RT} + \text{con.} \quad \ldots \ldots \ldots \ldots \ldots (5)
\]

\[
\Delta S = \frac{\Delta H - \Delta G}{T} \quad \ldots \ldots \ldots \ldots \ldots (6)
\]

![Figure 9: Effect the temperature of the adsorption Azure A by SSMFP](image)
Tab. [2]: Thermodynamic parameters ($\Delta G$, $\Delta H$ and $\Delta S$) for Azure A dye on SSMFP absorption surface within the thermal range (298-338) K

| Adsorbate | Temp. K | $(-\Delta G)$ kJ mol$^{-1}$ | $(-\Delta H)$ kJ mol$^{-1}$ | $(+\Delta S)$ J mol$^{-1}$K$^{-1}$ |
|-----------|---------|-----------------------------|-----------------------------|--------------------------------|
| Azure A   | 298     | 7.1904                      | 0.0084                      | 0.0241                         |
|           | 308     | 7.2701                      |                             | 0.0235                         |
|           | 318     | 7.0204                      |                             | 0.0220                         |
|           | 328     | 7.0271                      |                             | 0.0213                         |
|           | 338     | 7.1144                      |                             | 0.0210                         |

The negative values of the free energy $\Delta G$ gives an indication on the spontaneity of the adsorption process (24). The negative sign of the $\Delta H$ refers to exothermic property of the adsorption reaction of Azure A (25). Finally, the positive signs of the $\Delta S$ values reveals the increasing the degree of freedom at the interface of solid-liquid during the adsorption of Azure A (26).

Adsorption isotherm

Adsorbed isotherms explain the adsorption process reaches equilibrium when the adsorbed particles are distributed between the liquid phase and the solid phase. Figure 10 shows the isotropic absorption of Azure A on the cochlea melamine formaldehyde polymer (SSMFP). At pH = 10.0, at a temperature from 298 to 338 K, 0.0050 g of sorbents and a time of 10 minutes, we notice that the absorption capacity of Azure A increases with increasing equilibrium Azure A, by using equation (2).

Figur. [10]: Isotherm absorption from the Azure A dye absorption of an aqueous solution on the surface of the (SSMFP) at (298-338) K

Langmuir isotherm:

The Langmuir model isotherms mathematical were determined as follow equations (27).

\[
\frac{C_e}{Q_e} = \frac{1}{a} + \frac{C_e}{b} \quad \text{..................(7)}
\]

Where:-

- $a$ (mg / g) maximum absorption capacity
- $b$ (mg / L) is the Langmuir constant relates to affinity and free energy of adsorption (28).
A linear plot was obtained when Ce/Qe was plotted vs Ce. as shown in Figure.11.

![Langmuir isotherms for Azure A over the studied temperatures.](image1)

**Figure [11]: Langmuir isotherms for Azure A over the studied temperatures.**

The ability of occurring and the feasibility of adsorption reaction was measured by applying the factor RL, that defined by equation (8).

\[
RL = \frac{1}{1 + b C_0} \quad \text{(8)}
\]

The value of RL refers to category of the isotherm to be either unfavorable (RL > 1), linear RL = 1, irreversible RL = 0 or favorable 0 < RL < 1.\(^{(29)}\)

**Freundlich Isotherm:**

Freundlich model is given by:

\[
\log Q_e = \log K_f + \frac{1}{n} \log C_e \quad \text{---(9)}
\]

where : \(Q_e\) and \(C_e\) are similarly to its defined above. \(K_f\) (L/mg) and \(n\) are constants of Freundlich equation which its depends on the capacity of adsorption and the intensity of adsorption respectively.\(^{(30)}\) as shown in Figure.12.

![Freundlich isotherms for Azure A dye over the studied temperatures.](image2)

**Figure [12]: Freundlich isotherms for Azure A dye over the studied temperatures.**
Temkin Isotherm model:

Temkin isotherm have a factor that give illustration about the adsorbent interactions and adsorbing species. In which Temkin isotherm assumes that the heat of adsorption for all molecules in the adsorption layer decreases linearly with increase the surface coverage due to the nature of the adsorbent– adsorbate interactions.

Temkin isotherm is given as

$$Q_e = B \ln A_t + B \ln C_e$$

where:

- $A_t$ is the binding constant at equilibrium that corresponds the maximum binding energy
- $B$ is constant which related with heat of adsorption. A linear plot was obtained when $Q_e$ was plotted vs $\ln C_e$.

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**Table 2: Langmuir, Freundlich and Temkin functions of adsorption reaction at the studied range of temperature (298 – 328) K.**

| Temp K | Langmuir isotherms | Freundlich isotherms | Temkin isotherms |
|--------|--------------------|----------------------|------------------|
|        | $a$ (mg/g) | $b$ (mg/L) | $R^2$ | $R_L$ | $K_f$ | $n$ | $R^2$ | $B$ | AT | $R^2$ |
| 298    | -2.40      | -1.84       | 0.3847 | -0.12 | 1210.59 | 0.21 | 0.8380 | 120.8 | 2.21 | 0.9881 |
| 308    | -1.87      | -1.78       | 0.3923 | -0.12 | 1467.57 | 0.18 | 0.8374 | 123.1 | 2.38 | 0.9902 |
| 318    | -1.88      | -1.65       | 0.2947 | -0.13 | 955.43  | 0.19 | 0.7775 | 115.53 | 2.48 | 0.9922 |
| 328    | -1.21      | -1.61       | 0.4134 | -0.14 | 1138.41 | 0.16 | 0.8745 | 111.75 | 2.75 | 0.9742 |
| 338    | -0.91      | -1.55       | 0.3419 | -0.14 | 1201.71 | 0.15 | 0.8090 | 113.52 | 2.98 | 0.9925 |

We note from the correlation coefficient ($R^2$) shown in Table 2 that isotherm Temkin is more applicable than isothermal Langmuir and Freundlich. This indicates that Azure A adsorption on the surface of Snail shell Melamine Formaldehyde Polymer (SSMFP), and that the absorption entrapment of all particles in the adsorption layer was linearly decreasing the surface coverage of the shell surface.
Conclusion:-

✓ This study demonstrated the possibility of using absorbent (SSMFP) to remove Azure A dye from its aqueous solutions.
✓ The removal rate was 93.90% in optimum conditions 5mg/L dye concentration, sorbent weight 0.0050 g and contact time 10 minutes at 298 K.
✓ The balance data fit very well with the Temkin isotherm model, indicating that Azure A adsorption on the surface of the modified snail shell.
✓ Thermodynamics studies indicate that the process of adsorption of the Azure A dye is automatic and exothermic, and a increase in randomness in terms of thermodynamic function values.

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