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ADSORPTION OF TARTRAZINE DYE FROM AQUEOUS SOLUTION USING SILICA EXTRACTED FROM RICE HUSK

BY

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ABSTRACT

The adsorption of tartrazine an anionic (synthetic) yellow dye using silica derived from rice husk from aqueous solution was explored. In this present work, physical and chemical properties of the synthesized material were investigated by various techniques including SEM and FTIR. The determined surface area of Rice husk ash (RHA) and activated SiO₂ were 105.8 m²/g and 226 m²/g respectively. The dye solution was made by dissolving 10 mg of dye in 1000 cm³ of deionized water. The experiments were carried out in batches, and the effect of the following parameters on dye removal was investigated, including pH, initial dye concentration, and contact time. Equilibrium was achieved in 60 min. Adsorption capacity was found to be dependent on the initial concentration of dye solution, and maximum adsorption was found to be 28.6 mg/g at 80.0 mg/L of tartrazine dye. The adsorption capacity at equilibrium (q_e) increased from concentrations from 5 to 60 mg/L. Several kinetic models were explored to fit the data, and it was discovered that the reaction mechanism may entail pseudo-second order models. Among the isotherm models studied, the Freundlich model was found to fit the equilibrium data better ($R^2 = 0.9997$) than the Langmuir ($R^2 = 0.9816$) indicating a monolayer surface process. The thermodynamic parameters, Gibbs free energy change, (ΔG) was found to be negative confirming the feasibility and spontaneity of the adsorption process. The endothermic nature of the adsorption process was confirmed from the positive value of enthalpy change, (ΔH). The positive value of the entropy change, (ΔS) indicates that the adsorption process is dissociative and more random. It can be concluded that these modified silica have very good efficiency for the removal of tartrazine dye from aqueous solution and can therefore be used in food and textile industries for the treatment of wastewater.

Keywords: Adsorption, characterization, tartrazine dye, activated silica, and rice husk

INTRODUCTION

Adsorption is one of the most effective, simple, and inexpensive techniques of eliminating organic dyes or color from textile industries (Arabkhani and Asfaram 2020; Ince and Kaplan 2017), utilizing a variety of low-cost adsorbents such as clays (Kausar *et al.*, 2018), zeolites, fly ash (Sun *et al.*, 2010), activated carbon (Belaid *et al.*, 2013) and industrial waste products. Because of its large surface area and pore volume, chemical inertness, and mechanical durability, activated carbon is the most effective adsorbent for removing organic dyes (Belaid *et al.* 2013). However, because macro dye molecules impede activated carbon micropores, large dye molecules find it more difficult to penetrate into the activated carbon's internal pore structure (Belaid *et al.*, 2013), limiting its application. Activated silica is a porous substance used as an adsorbent due to its unique surface area and pore properties, such as large surface area and high pore volume (Huang *et al.*, 2011). Unlike activated carbon, which has inaccessible pores, mesoporous silica has a pore structure that allows for the adsorption of textile color molecules (Ozturk *et al.*, 2017). Natural silica, particularly that found in agricultural waste, can be used to replace expensive commercial silica precursors in the synthesis of mesoporous silica. Rice husk is the most frequent agricultural waste that can be converted into more value silica-derived goods (Pode, 2016).

Rice (*Oryza sativa*), which occupies 1% of the earth's surface, is a staple sustenance for billions of people. Each year, over 600 million tons of rice are produced globally. Rice husk, the coating on rice grains, is composed of 50% cellulose, 25%-30% lignin, and 15-20% silica (Beidaghy Dizaji *et al.*, 2019). As the primary waste product after milling, each kilogram of rice produced yields 0.28 kg of rice husk (Deshmukh *et al.*, 2012). When rice husk is burned at temperatures exceeding 500°C, the organic components disintegrate, yielding rice husk ash. SiO₂ purity in rice husk ash ranges between 94 and 96% (Bakar *et al.*, 2016), with small impurities including K₂O, Na₂O, and Fe₂O₃.

The purity of SiO₂ produced by rice husk incineration can be increased to 98-99% (Zou and Yang, 2019). If the rice husk is pre-treated with

acids such as HCl, HNO₃, H₃PO₄, and H₂SO₄ (Xu *et al.*, 2018), the impurities mentioned above can be eliminated. Modifying the physical and chemical surface characteristics of rice husk ash may increase its adhesion qualities (Emdadi *et al.*, 2015). Because of their low production costs, these adsorbents offer the advantage of not requiring to be renewed in certain applications. Tartrazine (TART) is widely used all over the world, some studies suggest that tartrazine can lead to many allergy symptoms, anxiety, depression, migraine, asthma, urticaria, and so on. It has been recommended to be phased out in food and drink in some countries over a specified period. Its inclusion in the diet is sometimes controversial because of allergenic properties reported. Few cases of urticaria, asthma, contact dermatitis, vasculitis have been reported after consumption of food containing tartrazine (Taylor *et al.*, 2010). Hyperactivity disorders resulting in a lack of attention in children are also reported (Kamel *et al.*, 2011). So, to protect the health of consumers, a standard norm of 300 mg per kg of tartrazine in dairy products and a qualifying 7.5 mg per kg of body weight daily dose have been established (Schenone *et al.*, 2013).

Tartrazine adsorption has been investigated using a variety of adsorbents, including hen feather, bottom ash, and de-oiled soya (Kumar *et al.*, 2010), polystyrene anion exchange resins, multi-walled carbon nanotubes (Mishra *et al.*, 2010), and activated carbon (Ozsay and Van, 2010).

We discuss in detail how activated silica can be synthesized using the post-grafting approach. This adsorbent was then evaluated using Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and BET. Activated silica's ability to remove tartrazine dye from aqueous solution was tested using several adsorption parameters, including pH, initial dye concentration, and contact time. Finally, adsorption kinetics and isotherms were investigated to better understand the tartrazine dye adsorption process.

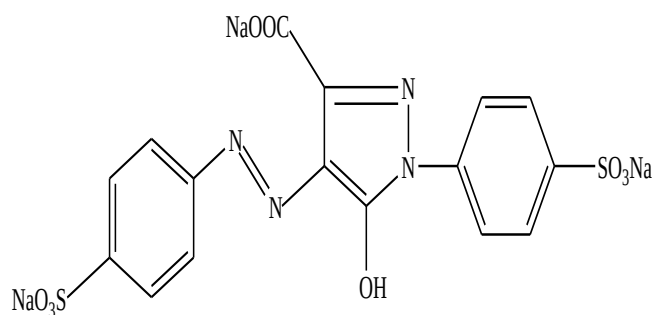


Figure 1: The structure of tartrazine coloring

MATERIALS AND METHOD

Materials

Analytical weighing balance (FA 2009), UV-Visible spectrophotometer (UV -3600 plus, Calia Technology SDN BHD 1239933-H, SHIMADZU Serial number A120155 00475), Fourier transform infrared FT-IR spectrometer (PerkinElmer Instrument Model: Spectrum Two, Serial number 114769 Malaysia). Scanning electron microscopy and energy-dispersive spectroscopy (SEM/EDX) Model: COXEM EM-30AX plus, magnification 80x- 100,00x, Accelerating Voltage 5-15KV, Centrifuge machine (MPW J2-21, Palo Alto California USA) and Sonicator (Model: Widdens).

Sample Collection

Rice husk, a byproduct from the Kwanar Dawaki rice mill industry in Kano State, was used as the starting material. The rice husk sample (RH) underwent pretreatment, following a modified version of the method described by Nur Saadah *et al.*, (2018). The pretreatment process involved, washing 250 g of raw rice husk with tap water and then double-distilled (DD) water to remove impurities. Sun drying for 48 hours, followed by air-drying in a hot-air oven at 373 K for 30 minutes to remove excess moisture. Acid leaching with 10 % HCl at 10 wt % solids for 24 hours. Washing with deionized water at pH 7 and drying at 110 °C for 24 hours. Grinding the cleaned rice husk into a fine powder using a wooden mortar and sieving through a 63µm mesh. Storing the resulting rice husk ash (RHA) in a labeled plastic container. Additionally, sulfuric acid (H₂SO₄, 95%) from Sigma-Aldrich was used for washing and cleaning, and while hydrochloric acid (37%) was used for

acidifying RH. Silica (SiO₂) was extracted from RH through heat treatment.

Silica extraction using precipitation method

Silica was extracted from rice husk ash adapting the method of Nur Saadah *et al.*, (2018). The obtained residue (RHA) was dissolved in 60 ml portions of 1N NaOH and boiled in covered 250 ml Erlenmeyer flasks for 1 h with constant stirring to dissolve the silica and produce a sodium silicate solution. The solutions was filtered through Whatman No. 41 ashless filter paper, and the carbon residues was washed with 100 ml of boiling water. The filtrates and washings were allowed to cool to room temperature and was titrated with 1N HCl with constant stirring to pH 7. Silica gels started to precipitate when the pH decreased to <10. The silica gels formed was aged for 18 hours. Deionized water (100 ml) was added to gels and then the gels were broken to make a slurry. The slurries were then be centrifuged for 15 min at 2500 rpm, the clear supernatants was discarded and the washing step was repeated. The gels was transferred into a beaker and dried in oven at 80°C for 12 h to produce xerogels. Selected silica xerogel samples was grounded and subjected to additional washing with water. All the samples were stored in airtight plastic bottles and labeled.

Preparation of the dyes' stock and working solutions

The tartrazine dye stock solutions were prepared by modifying the process reported by Mohammad and Gamal (2014). A stock solution was prepared by dissolving 1.0 g of Tartrazine dye in 1000 cm³ deionized water. Subsequently, working solutions with concentrations ranging from 0 to 60 mg/L (specifically 0, 1, 2, 5, 7, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, and 60 mg/L) were obtained through serial dilution of the stock solution.

Characterization of adsorbent

The unmodified and modified silica samples were analyzed to determine the nature of the functional groups available on the surface of the adsorbents to reveal the possible changes in the functional groups

which occurred before and after the modification. The samples were analyzed using Fourier Transform Infrared (FT-IR) spectroscopy, scanning across a wave number range of 4000 - 400 cm^{-1} with a resolution of 4 cm^{-1} , to record their FT-IR spectra. SEM/EDX were employed to evaluate surface morphology and determine silica modification percentages. Samples were coated with gold-platinum under vacuum to ensure conductivity and prevent electrostatic charging during analysis.

Batch Adsorption Studies

The dye was subjected to a batch adsorption procedure to determine the effect of experimental variables (initial concentration, contact time, and pH). The approach employed was adopted from Abdullah (2022). A dye solution with a concentration of 10 ppm was produced under pH conditions 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, and 12. Then, activated silica added and shaken at 180 rpm for 30 minutes. The dye solution, containing 10 ppm of activated silica, was shaken at 180 rpm for 5, 10, 20, 30, 40, 50, and 60 minutes. Dye solutions with concentrations of 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, and 60 ppm were produced using appropriate contact time and pH conditions. After that, the activated silica was shaken at 180 rpm to evaluate its adsorption performance. Finally, the adsorbent was centrifuged and the concentration of unadsorbed dyes was measured with a UV-vis spectrophotometer maximum. The amount of dye adsorbed onto the surface of the modified silica and the removal efficiency were measured by:

$$Q_e = (C_i - C_f) \frac{V}{m} \quad (1)$$

$$\text{Removal efficiency (\%)} R = \frac{C_o - C_e}{C_o} \times 100\% \quad (2)$$

where Q_e is the amount of dye adsorbed per unit mass of the adsorbent at time t in mg g^{-1} . C_o and C_e (in mg dm^{-3}) are the dye initial and equilibrium concentrations, respectively. C_e is the dye concentration at time t (min) in mg dm^{-3} . V is the volume of the solution in (dm^{-3}); m is the mass of the adsorbent in (g).

Optimization of Experimental Parameters

Contact time: The influence of contact time on dye adsorption was examined throughout a time span of 5-60 minutes. The contact time with the highest dye removal was chosen as the ideal (Kadhom 2020).

pH

For the determination of pH of the adsorbent, 10 mL of the analyte (tartrazine dye solutions) was taken in different Erlenmeyer flasks of 250 mL and 0.01 g of adsorbent was introduced in each of them. The pH values of these solutions were adjusted in 2 to 14 range by 0.1 M HCl/NaOH solutions (Adebayoa *et al.*, 2019).

Initial dye concentration: At optimum contact duration and adsorbent dosage, the influence of initial dye concentrations in the range of 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, and 60 mg/L on removal percentages and actual amount of adsorbed dye was investigated. The concentration that eliminated the most dye was chosen as the best concentration (Zhang *et al.*, 2020).

RESULTS AND DISCUSSION

Figure 2a and b shows Fourier transform infrared spectra of activated silica before and after modification. Figure 2a, shows, three characteristic peaks at 1082 cm^{-1} , 798 cm^{-1} and 457 cm^{-1} represent different vibration modes of the Si-O-Si bond in the siloxane structural framework, specially asymmetric stretching, symmetric stretching, and bending vibrations, respectively. After modification, Fig 2b shows, a broad peak centred around 3452 cm^{-1} corresponds to the stretching vibration of hydroxyl (-OH) groups from silanol (Si-OH) and absorbed water molecules on the silica surface. A weak peak at 980 cm^{-1} indicates the presence of Si-OH bonds. A peak at 1636 cm^{-1} is attributed to the bending vibration of O-H bonds from adsorbed water molecules. Small peaks at 2996 cm^{-1} and 2355 cm^{-1} correspond to symmetric and asymmetric $-\text{CH}_2$ stretching vibrations and $-\text{CO}$ vibrations, respectively, likely due to organic impurities from the husk used in the process (Wasinton *et al.*, 2016).

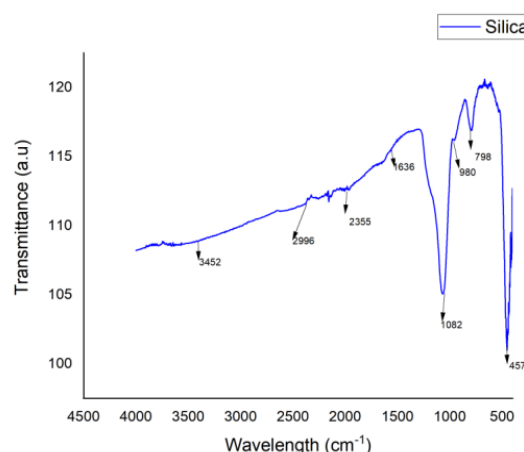


Figure 2a: shows the FTIR of activated silica before modification

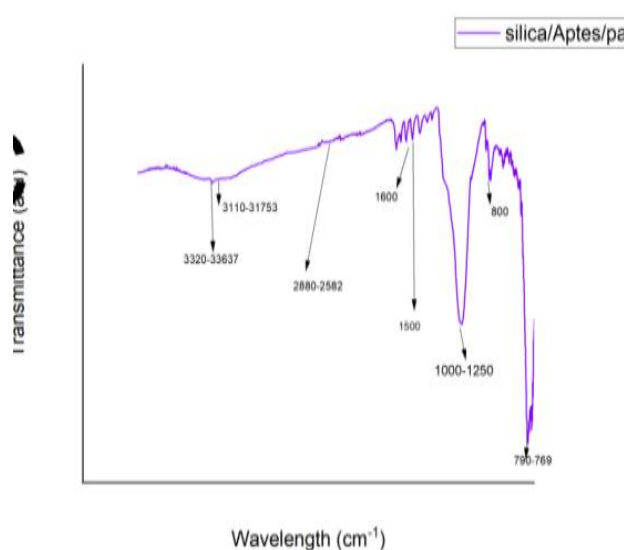


Figure 2b: shows the FTIR of activated silica after modification

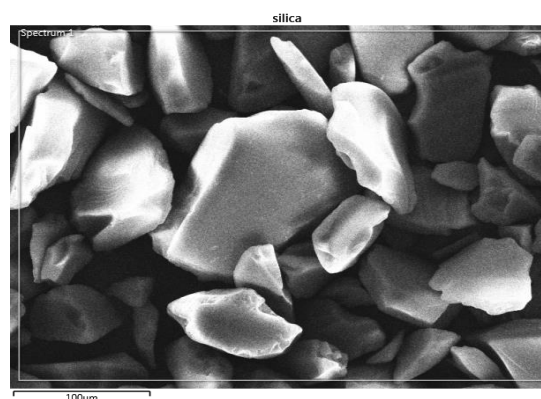


Figure 3a: showing the SEM image of activated silica before modification



Figure 3b: Shows SEM Images of activated silica after modification.

The morphological properties of the neat silica produced from rice husk ash were examined using scanning electron microscopy (SEM) to gain insights into its structural characteristics. The SEM images, shown in Figure 3a and b, reveal a layered structure in both the unmodified and modified silica samples. However, a notable difference was observed in the degree of aggregation and homogeneity. As the silicon ratio decreased, the modified silica exhibited reduced aggregation and a more uniform structure. Furthermore, the modified silica displayed irregular sizes and shapes, with a smooth surface, whereas the original silica had a non-porous and rougher structure (Faizul *et al.*, 2014).

Investigating the influence of Contact time on the adsorption of tartrazine dye from solution

Figure 4 illustrates the effect of contact time on the adsorption of tartrazine dye (10 mg/ml) onto activated silica at 25°C with constant stirring at 150 rpm. The results show that a higher percentage of tartrazine dye is removed initially, with the majority of adsorption occurring within the first 60 minutes. The optimal stirring time for maximum adsorption was determined to be 60 minutes, which was used in subsequent measurements. Prolonged stirring beyond 60 minutes led to a decrease in the amount and percentage of dye removed, with the lowest percentage (6.6%) observed at 60 minutes. This decrease is attributed to the congregation of tartrazine dye molecules on the adsorbent surface

over time, eventually reaching equilibrium (Asiagwu *et al.*, 2012).

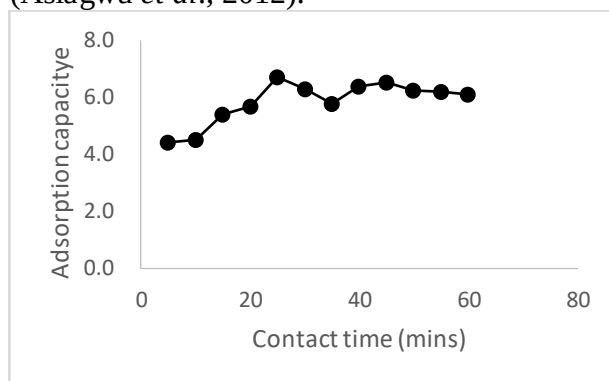


Fig. 4: Effect of contact time on tartrazine dye removal with activated silica

Effect of Initial concentration on the adsorption of tartrazine dye in solution.

Figure 5 depicts the adsorption capacity vs adsorption at varied initial dye concentrations at 25°C. It showed that the contact time required for tartrazine solutions with initial concentrations of 5-60 mg/L to reach equilibrium was 60 minutes. As shown in Fig. 5, the amount of tartrazine adsorbed onto the activated silica grew with time until it reached a fixed value beyond which no more tartrazine dye was taken from the solution. At this moment, the amount of tartrazine dye desorbing from the activated silica was equal to the amount of tartrazine dye adsorbed on the activated silica. The amount of dye adsorbed at equilibrium time represents the adsorbent's maximal adsorption capability under the given operating conditions. In this investigation, the adsorption capacity at equilibrium (q_e) increased from 22.6 to 76 mg/g when the starting dye concentrations increased from 5 to 60 mg/L. The initial dye concentration has an immediate influence on the accessible binding sites on an adsorbent surface (Gautam *et*

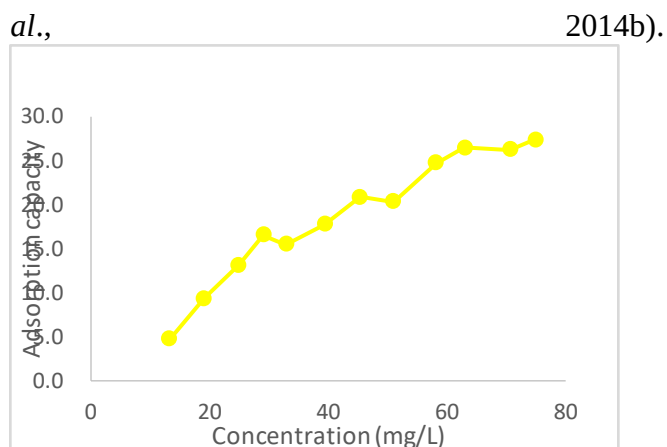


Fig. 5: Effect of initial dye concentration on tartrazine dye removal with activated silica.

Effect of pH on the adsorption of tartrazine dye

Figure 6 shows: that the adsorbed quantity is highest at pH 2. In an acidic environment, the positive charge dominates the surface of activated silica. Thus, there is an electrostatic attraction between the positive charges on the surface of the activated silica and the negative charges on tartrazine. Mezenner *et al.*, (2012) found similar results when they investigated the adsorption of a mixture of biorecalcitrant chemicals in aqueous mediums. Similarly, Akar and Uysal (2010) discovered that an acidic pH increased the adsorption capability of anionic dyes.

For $\text{pH} < 6$, there is a strong electrostatic interaction between the positively charged surface of activated silica and anionic dye, resulting in increased dye uptake. However, as pH increases, the adsorbent's positively charged sites decrease and its surface becomes negatively charged. This state inhibits the uptake of anionic dyes from the system and causes electrostatic repulsion Sushmita and Chattopadhyaya, (2013). At high pH, OH ions effectively compete with dye ions, resulting in decreased dye sorption from aqueous solutions. Changes in pH also alter the adsorptive process by causing the dissociation of functional groups on the adsorbent surface, resulting in a modification in the adsorption process's equilibrium properties. The current findings are corroborated by previous studies that revealed Tartrazine elimination from Chitin and Chitosan (Dotto *et al.*, 2012).

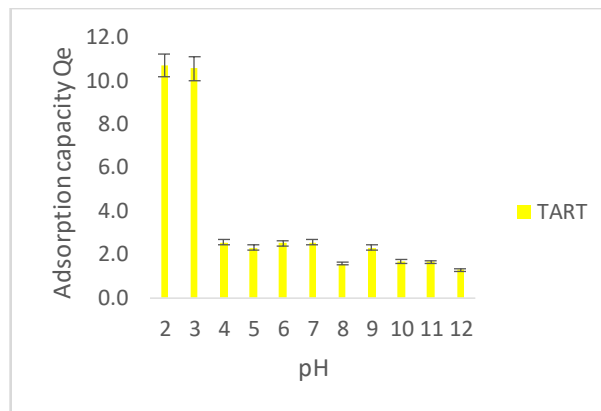


Fig. 6: Effect of pH on tartrazine dye removal using activated silica adsorbent

Langmuir Isotherm Analysis of the Sorption Process.

Figure 7 illustrates the application of the Langmuir isotherm model to determine the maximal adsorption capacity, corresponding to a complete monolayer covering of the biomass surface. The linear relationship between specific adsorption (Q_e/q_m) and equilibrium concentration (C_e) is depicted in Figure 7, with the corresponding Langmuir isotherm constants; q_m , K_L and coefficient of determination (R^2) listed in Table 1. The high R^2 value 0.9816 indicates a good fit. Additionally, Table 2 shows that the Freundlich isotherm with an R^2 value of 0.9997, provides an excellent description of the adsorption process, suggesting a monolayer surface adsorption mechanism. The favourable adsorption process is further confirmed by the Freundlich constant K_L ($0.577 < 1$), indicating a heterogeneous surface adsorption process.

Table 1: Langmuir equilibrium isotherm

Dye	q_m (mg g^{-1})	K_L (L mg^{-1})	R^2
Tartrazine	20.92	7.131	0.9816

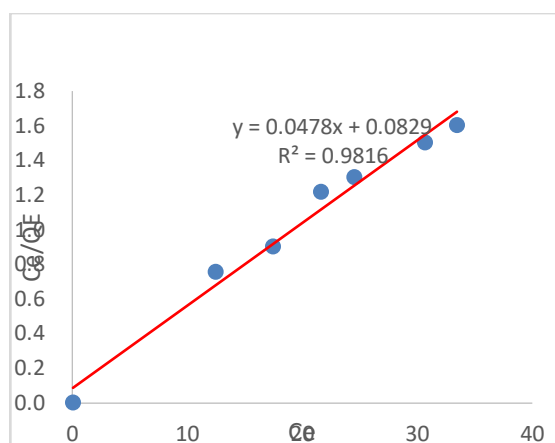


Fig 7: Showing Langmuir model

Sorption Process Evaluation using Freundlich Isotherm

The adsorbate's adsorption intensity on the adsorbent surface was evaluated using the Freundlich model. Figure 8 depicts the linear Freundlich model for the adsorption of dye ions onto activated silica. The graph shows that the Freundlich isotherm is an adequate model for the sorption process, but less so than the Langmuir isotherm, as evidenced by the n (0.969) that dipped below Table 2, implying that heterogeneous adsorption was unfavourable. Table 2 displays the linear Freundlich sorption isotherm constant and the coefficient of determination (R^2). Given the value of R^2 , the linear version of the Freundlich isotherm appears to give a plausible model for the sorption process under investigation.

Table 2: Freundlich equilibrium isotherm

Dye	I/n	K_f	R^2
Tartrazine	0.8729	7.131	0.9997

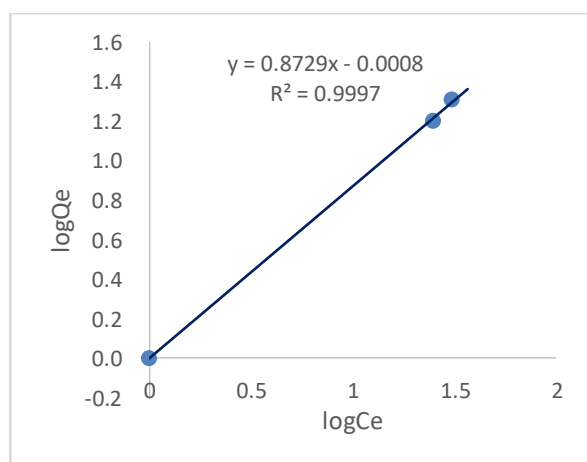


Fig 8: Showing Freundlich model

Adsorption Kinetics

Adsorption kinetics are one of the most important parameters used to determine the rate of adsorption in a particular system.

Pseudo-First Order Model

The pseudo-first order kinetics are represented by the plot of $\ln(q_e - q_t)$ against t in Figure 9. The graph shows a linear connection between dye ion diffusivity, $\ln(q_e - q_t)$, and time, indicating that the model follows a pseudo-first order model (see Table 3).

The coefficient of determination R^2 and other values reveal that the pseudo-first order model does not adequately describe tartrazine yellow dye absorption on activated silica.

Table 3: shows the values of pseudo-first order kinetics parameters.

Dye ions	K_1	q_e (mgg ⁻¹)	R^2
Tartrazine	0.0135	1.44	0.761

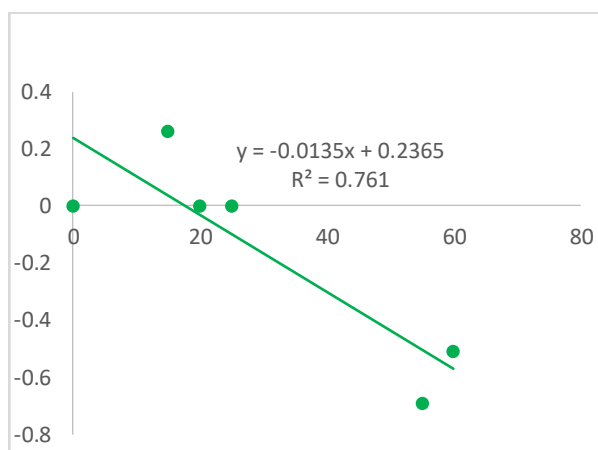
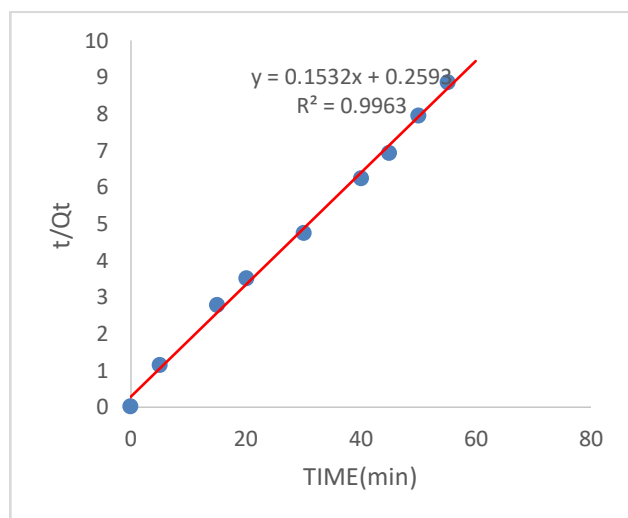


Fig 9: showing first-order kinetics

Pseudo-Second Order Model

Figure 10 shows the connection between t/q_t and t , which exhibits pseudo-second order kinetics. The plot shows that the relationship between t/q_t and t is linear, confirming the model. Table 4 shows the initial sorption rate, equilibrium adsorption capacity q_e , pseudo-second order rate constant K_2 , and coefficient of determination R^2 . From the value of coefficient of determination R^2 , The pseudo-second order model clearly provides a more accurate explanation of the adsorption process than the pseudo-first order model.

Table 4: Values of pseudo-second order kinetics parameters**Fig 10: showing second-order kinetics**

Thermodynamic Studies

The results are shown in Table 5. The negative value (ΔG) for the adsorbate when used with activated silica adsorbent indicates that the adsorption process is viable and spontaneous. The degree of spontaneity of the process was shown to increase with rising temperature. According to Zhang *et al.* (2014), the ranges for physisorption and chemisorption processes are -20 to 0 kJ/mol and -40 to 0 kJ/mol, respectively. The values of ΔG observed in this work are within the range of -20 to 0 kJ/mol, showing that the adsorption of tartrazine onto the surface of activated silica occurs via a physisorption mechanism. The characteristic energy shift associated with dye adsorption on the surface of the adsorbent can be calculated using thermodynamic parameters. Thus, Gibbs free energy change, ΔG (kJ mol⁻¹), enthalpy change, ΔH (kJ mol⁻¹), and entropy change, ΔS (J mol⁻¹ K⁻¹) were calculated for the adsorption of dye onto the activated silica adsorbent using four different temperatures (298, 303, 313, and 333 K) by using the following equations to identify the value at which optimum performance is observed.

$$\text{From } \Delta G = -RT \ln K_c \quad (7)$$

$$\ln K_c = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (8)$$

where R is the universal gas constant (8.314 J mol⁻¹ K⁻¹), T is the absolute temperature (K) and K_c is the

distribution coefficient ($\frac{Q_E}{C_e}$). By plotting $\ln K_c$

Dye ions	K ²	qe (mgg ⁻¹)	R ²
Tartrazine	0.28	6.78	0.9963

versus $\frac{1}{T}$, the thermodynamic parameters can be acquired then by rearrangement.

$$\Delta G = \Delta H - T\Delta S \quad (9)$$

Table 5 shows how the slope and intercept of the linear fluctuation of $\ln k$ with the reciprocal of temperature ($1/T$) were used to estimate the values of ΔH and ΔS .

Table 5: Thermodynamic Parameters for the Adsorption Process

Temp(K)	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (J/k)	R
298	-1.03			
303	-1.32	16.02	57.22	0.9947
313	-1.87			
333	-3.03			

The Table 5 shows that the R^2 value (0.9947) supports the linear fit of the data to the Van't Hoff model. Additionally, the negative values of ΔG at various temperatures indicate that the adsorption process is spontaneous and feasible. The positive value of enthalpy indicates that the process is endothermic. Meanwhile, Zhang *et al.*, (2014) revealed the same observation about the endothermic nature of the process.

CONCLUSION

The study investigated the efficacy of modified silica in removing tartrazine yellow dye from aqueous solutions. The results showed that, the adsorption capacity of activated silica increased with longer contact times. Higher pH levels led to a decrease in color removal from the solution. The Freundlich isotherm model accurately described the adsorption process, indicating a strong affinity between the modified silica and tartrazine dye. The pseudo second-order kinetics model best fit the adsorption process, suggesting a rapid and efficient removal of the dye. Overall, modified silica

demonstrated excellent potential as an adsorbent for removing tartrazine yellow dye from aqueous solutions, making it a viable option for industrial - scale applications. The optimal operating parameters for this process have been identified, paving the way for its practical implementation.

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