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Full Paper

Adsorption performance of binary mixtures containing methylene blue and tartrazine using silica xerogel from rice husk

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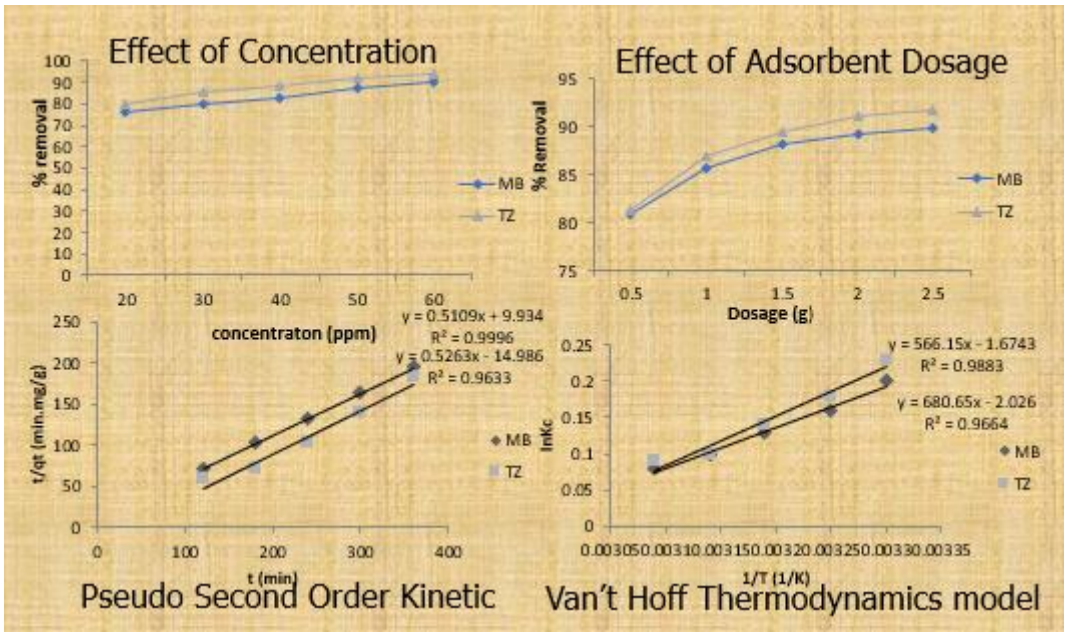

Abstract: This research work assessed the adsorption performance of silica xerogels for the removal of a binary solution of methylene blue and tartrazine dyes. Silica xerogel was extracted from rice husk using the gel method and modified with 1.0 M HNO_3 (nitric acid). Using batch adsorption methods, the effect of initial concentration, temperature, ionic strength, contact time, and adsorbent dosage on the adsorption process of methylene blue and tartrazine was studied using binary solutions of the dyes. The percentage removal of the

dyes studied increased as the initial concentration was varied from 20 ppm to 60 ppm. The removal efficiency decreased as the temperature was varied from 30 ^{0}C to 50 ^{0}C. The adsorption efficiency showed an increase at higher pH for methylene blue and at lower pH for tartrazine due to their ionic nature. The variation of contact time between 2 and 6 hours showed a sharp increase in dye removal from 2 to 5 hours, but a slow increase after 5 hours. The percentage removal of dyes increased as the adsorbent dosage increased between 0.5 to 2.5 g. The effect of ionic strength on adsorption efficiency was found to decrease as the concentration of NaCl was varied from 0.02 to 0.10 M. The experimental data were tested using the adsorption isotherms models of Langmuir, Freundlich, and Temkin. The Langmuir model was found to be the best fit among the three isotherms used for the analysis, as all the R^2 and R_L values favoured the isotherm. The data showed that the adsorption process can best be described by the pseudo-second order kinetics model, as the R^2 values for all the dyes are more than those of the pseudo-first order model. Therefore, it can be concluded that the silica xerogel is a potential adsorbent for the uptake of pollutants in wastewater.

Keywords: Adsorption, Methylene blue, Tartrazine, Silica Xerogel, Adsorbent, Rice Husk.

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Introduction

Urbanization, industrialization, population growth, and the sophistication of human activity all contribute to environmental degradation. The importance of lowering the hazardous materials found in wastewater from manufacturing and processing businesses before releasing it into the environment has become more widely recognized as a result of increased knowledge of the related issues [1]. Since color is the most evident sign of water contamination, dyes are particularly important pollutants in wastewater. Even a small amount of dyes in aquatic media can be unpleasant [2]. These enterprises release brightly colored wastewater into rivers and lakes, rendering the water unsuitable for industrial, agricultural, and residential uses [3]. The presence of dyes hurts the aesthetics of the aquatic environment, making the water unfit for drinking and other recreational uses due to its unnatural and unattractive appearance. Additionally, dyes inhibit the process of photosynthesis, which impacts the aquatic ecosystem by reducing light penetration [4]. Over the years, a variety of procedures have been used to cleanse wastewater that contains dyes. Since activated carbon has a large surface area, a microporous structure, a high adsorption capacity, and a high degree of surface reactivity, it is a simple and highly

effective adsorption method [5]. However, its comparatively high cost and production-related issues, including pore blockage, high temperature combustion, and hygroscopicity, limit its widespread application. This has prompted the hunt for novel, affordable, and effective adsorbents to clean wastewater containing dyes. Using Methylene Blue tartrazine as the model dye, a variety of substitutes, particularly agricultural by-products such as rice husk, guava leaf powder, and wheat shell, are being studied to ascertain their potential for eliminating dyes from wastewater [6]. The current work is to examine the impacts of competitive adsorption on the kinetics and thermodynamics of the adsorption process, as well as to use silica xerogel from rice husk as an unconventional, natural, low-cost adsorbent for decolorizing simulated wastewater of a binary dye system.

Results and discussion

Batch Adsorption Studies

The effect of parametric factors such as concentration, contact time, ionic strength, dosage, and temperature was studied in binary solutions for the adsorption of dyes onto silica xerogel adsorbent. Figures 1 to 5 show the percentage removal of dyes under the factors mentioned in that order.

Effect of Parametric Factors on Adsorption Studies

Effect of Initial Concentration

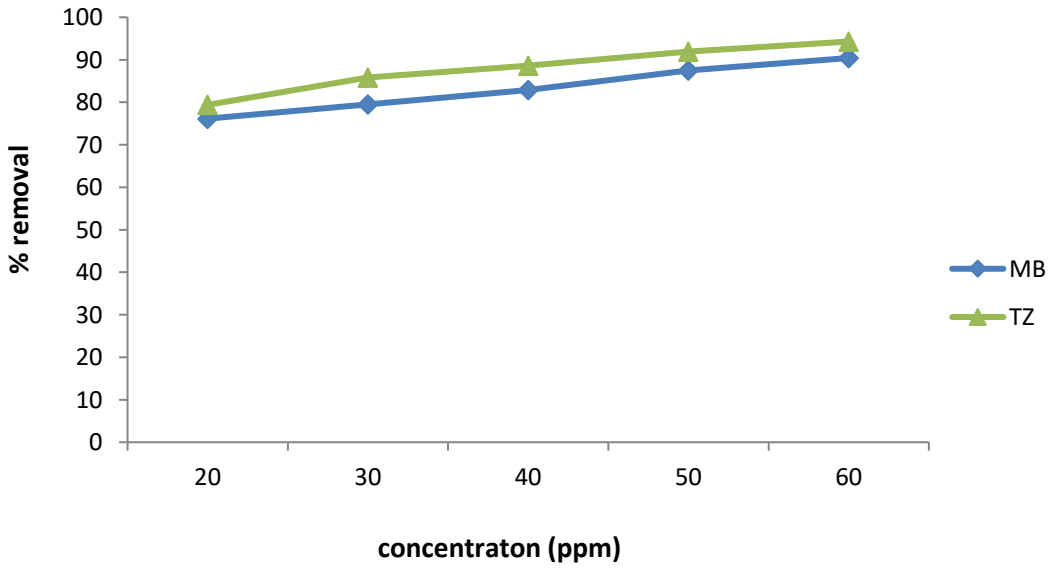


Figure 1: Effect of Concentrations on Removal Efficiency of Methylene Blue (MB) and Tartrazine (TZ) in binary solution using Silical xerogel

Figure 1 displays the results of the dye concentration effect. The percentage removal of methylene blue and tartrazine dyes was 92.43 and 94.30, respectively. For both the single and binary phases, the data indicate a general increase in percentage elimination as the dyes' starting concentration rises from 20 to 60 ppm. More dye molecules are available for the adsorbent to absorb, which results in a rise in dye concentration. Enenebeaku et al. have reported a similar tendency [7].

Effect of Contact Time

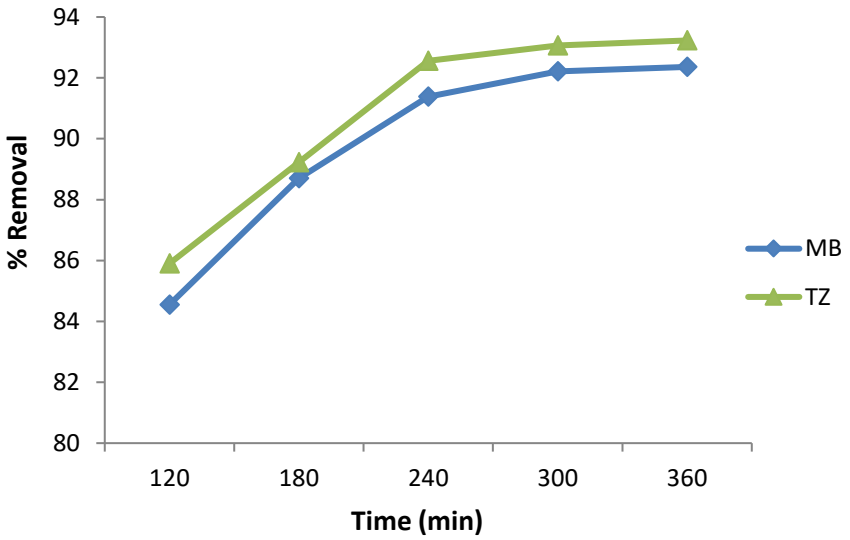


Figure 2: Effect of Contact Time on Removal Efficiency of Methylene Blue (MB) and Tartrazine (TZ) in binary solution using Silical xerogel

The effect of contact time is one of the vital factors considered when using a batch adsorption system. It is important because it gives insight into the sorption process and provides information on the maximum time required for considerable adsorption to take place [8]. Figure 2 shows the result of the adsorption of tartrazine (TZ) and Methylene blue (MB) dyes in a binary solution. It explained that the percentage removal of the dyes increased with time. The resulting percentage removal trend was TZ>MB. The increase in the adsorption was gradual after some hours, which implies that during the initial stage of adsorption, a large number of vacant sites were available for adsorption, but after some time, the surface sites were occupied, hence the difficulty in adsorption due to repulsive forces between the solute molecules on the adsorbent surface and the solution phase. These remarks have been reported by some researchers [9, 10].

Effect of Ionic Strength

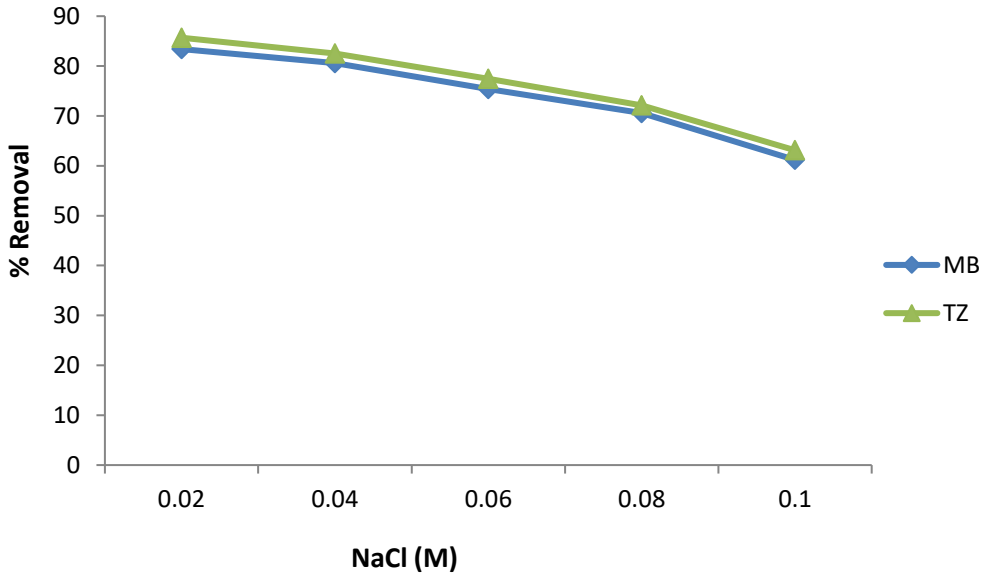


Figure 3: Effect of Ionic Strength on Removal Efficiency of Methylene Blue (MB) and Tartrazine (TZ) in binary solution using Silical xerogel

The ionic strength of a solution is a crucial parameter that controls both electrostatic and non-electrostatic interactions between the dyes and the adsorbent surface. Extensive investigations carried out on the adsorption of dyes revealed that the extent of dye uptake was strongly influenced by the concentration and nature of the electrolyte ionic species added to the dye bath [11].

The results of the percentage removal are presented in Figure 3. From the results, it appeared that the percentage removal of the tartrazine (TZ) and Methylene blue (MB) dyes decreased as the concentration of NaCl was varied from 0.02 – 0.10 M. These results can be explained as follows; the NaCl salt occupy the surface of the adsorbent sites before the dyes therefore the surface of sorbent material becomes difficult for the dyes uptake when the quantity of NaCl salt in solutions increase. The presence of salt inhibits the uptake on the surface of the adsorbent. Similar remarks have been proven by Bounmediene *et al* [12], who investigated the effect of pH and ionic strength of methylene blue removal from aqueous solution by sorption onto orange peel.

Effect of Dosage

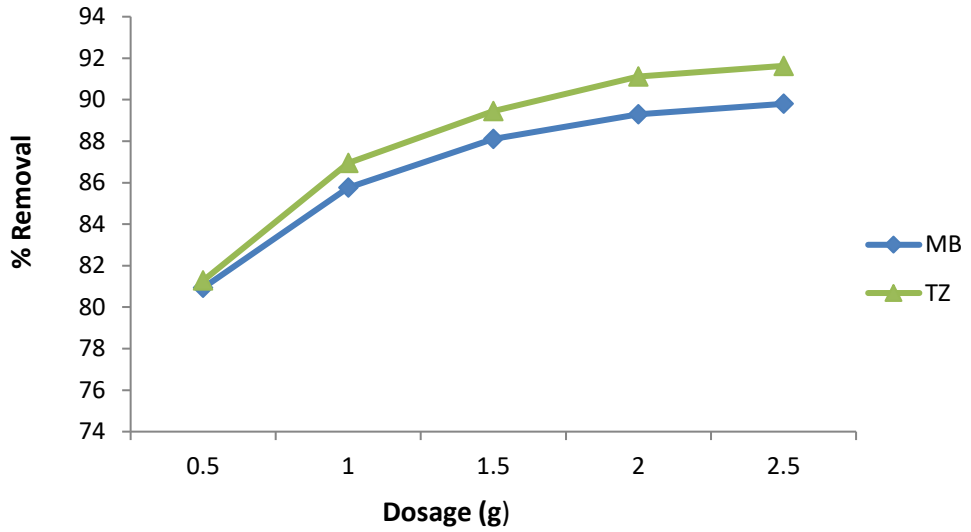


Figure 4: Effect of Adsorbent Dosage on Removal Efficiency of Methylene Blue (MB) and Tartrazine (TZ) in binary solution using Silical xerogel

Adsorbent dosage is also a vital parameter in the adsorption of dyes owing to its effects on the amount of dyes removed per unit mass of the adsorbent.

Figure 4 shows an increase in the adsorption percentage of tartrazine (TZ) and Methylene blue (MB) dyes removal as the adsorbent mass increases from 0.5-2.5 g. The increase in the removal efficiency was more at 0.5 – 2.0 g and a slight increase at 2.5 g for all the dyes in the binary solution. This increase is due to the greater availability of exchangeable sites or surface area at the higher dose of the adsorbent. This has corresponded with the results of other researchers, Hoang *et al*, Fatimah and Luma [13, 14].

Effect of Temperature

Temperature affects spontaneity, equilibrium, rate, and randomness of the adsorption process. An increase in temperature can affect the adsorption process [13].

Figure 5 shows the result of the removal efficiency of tartrazine (TZ) and Methylene blue (MB) dyes. An increase in temperature from 30 ^{0}C -50 ^{0}C was found to result in a decrease in removal efficiency of the dyes for single and binary solutions. The decrease in the adsorption process might be due to the weakening of the attractive forces between the adsorbent and the adsorbate ions. At high temperature, the thickness of the boundary layer was expected to decrease due to the increase in the dyes to escape from the surface of the adsorbent to the solution phase. These remarks are similar to the work by Kukwa *et al* [8].

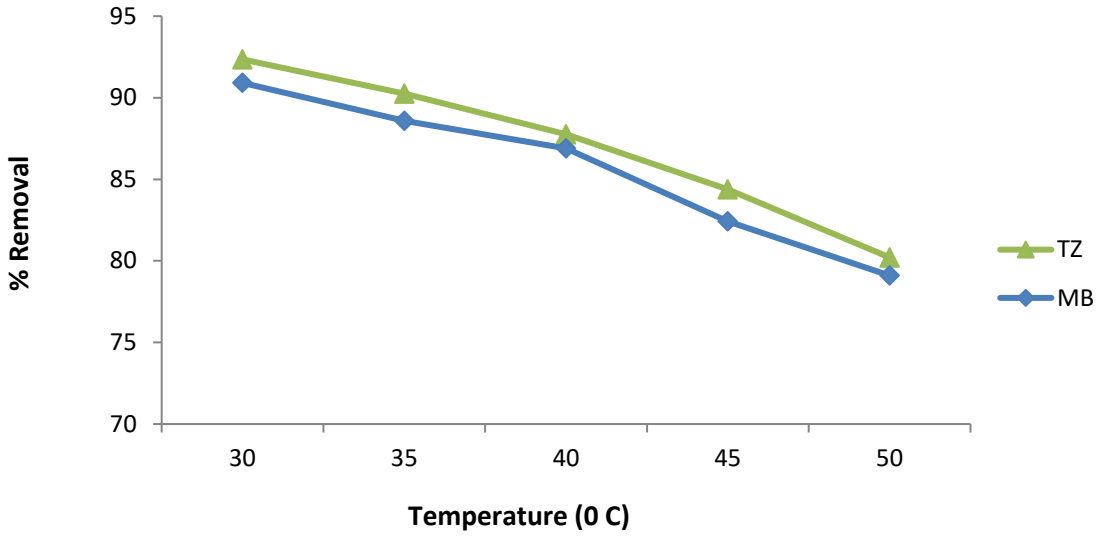


Figure 5: Effect of Temperature on Removal Efficiency of Methylene Blue (MB) and Tartrazine (TZ) in binary solution using Silical xerogel

Adsorption Kinetics

A kinetic model helps in the study of adsorption rate and predicts information about adsorbent/adsorbate interaction (physiosorption or chemisorption) [15]. Pseudo-first-order and pseudo-second-order kinetics models were employed for the adsorption kinetic behaviour of the adsorbates on the adsorbent. The accepted kinetic model for a given adsorption is based on three fundamental validity test; A good and high correlation coefficient (R^2) indicating the applicability and reliability of a given model, a close agreement between the calculated and experimental q_e values and the accepted model must have the least values for the sum of error squares (%SSE), determined using equation below [16].

$$\%SSE = \sqrt{\sum \frac{(q_e exp - q_e cal)^2}{N}} \quad (7)$$

Pseudo First and Second Order Kinetics

Table 1: Pseudo First Order Kinetics parameters for dyes adsorption

DYE	Qexp	Qcal	K_1	%SSE	R^2
MB	1.85	1.01	0.012	0.141	0.986
TZ	1.87	1.45	0.012	0.035	0.983

Key: TZ =Tartrazine and MB = Methylene blue

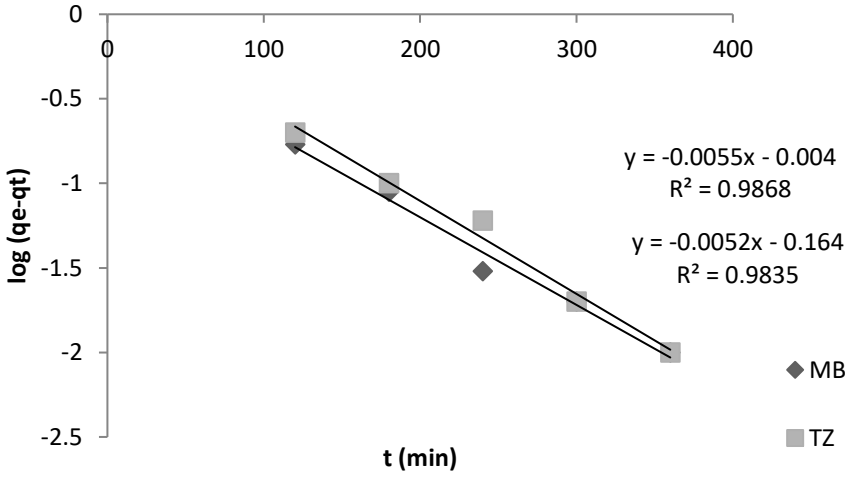


Figure 6: A plot of Pseudo First Order Kinetics for the adsorption of Methylene blue (MB) and Tartrazine (TZ) in a binary solution onto Silical xerogel

Table 2: Pseudo Second Order Kinetics parameters for dyes adsorption

DYE	Qexp	Qcal	K_2	%SSE	R^2
MB	1.85	1.96	0.026	0.002	0.999
TZ	1.87	1.89	0.019	0.018	0.963

Key: TZ =Tartrazine and MB = Methylene blue

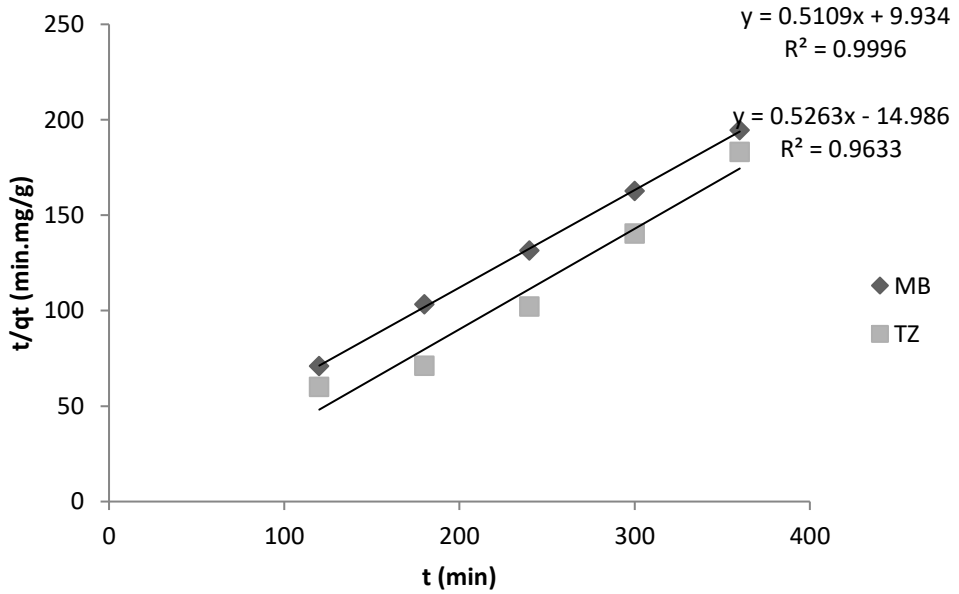


Figure 7: A plot of Pseudo Second Order Kinetic for adsorption of Methylene blue (MB) and Tartrazine (TZ) in binary solution onto Silical xerogel

Pseudo-first order kinetics models are based on the assumption that the rate of adsorption is proportional to the number of vacant sites available on the adsorbent surface and are used regularly in liquid-solid phase [17]. Table 1 shows the experimental data tested

for pseudo pseudo-first-order model. The conformity between experimental data and the model values was expressed by the coefficient of determination R^2. The R^2 values for Tartrazine (TZ) and Methylene blue (MB) were found to be 0.983 and 0.986, respectively. These values show that the model has good applicability and also explains the mechanism of adsorption kinetics. The Q_{exp} and Q_{cal} values for the dyes TZ and MB were 1.87, 1.85, and 1.45, 1.01. The Q_{exp} and Q_{cal} values are close, which implies a good fit for the adsorption.

The regression coefficient (R^2) for pseudo pseudo-second order model also suggested the applicability of the kinetic model to describe the adsorption processes of dyes removal on the adsorbent [18]. Table 2 shows the regression coefficient R^2 of Tartrazine (TZ) and Methylene blue (MB) to be 0.963, 0.999, respectively. These values indicate that the adsorption also follows second order, and the model is applicable. The Q_{exp} and Q_{cal} values for the dyes TZ, CR, and MB are 1.86, 1.85, and 1.89, 1.96, respectively. These values are also much closer, which implies that the adsorption also follows second order.

Thermodynamics studies

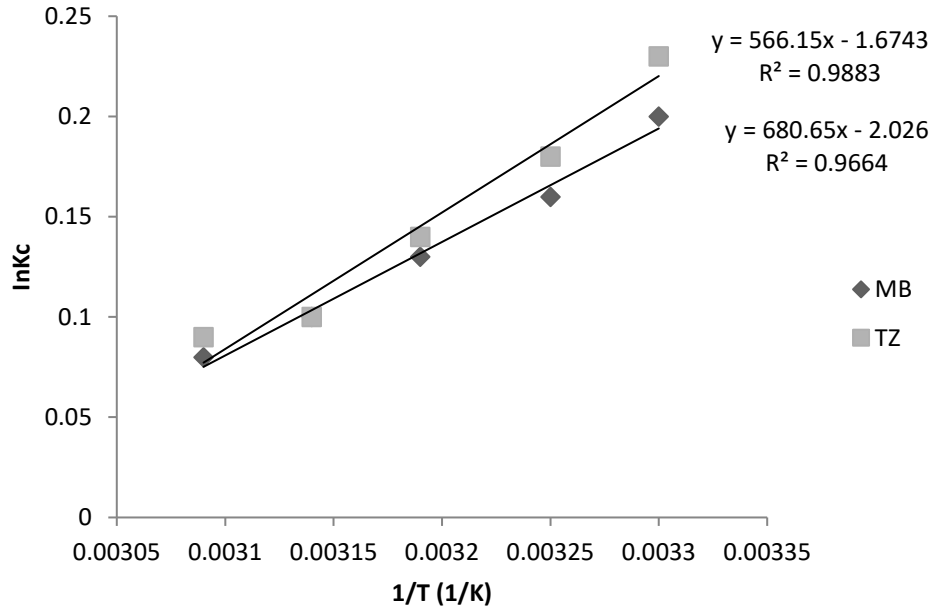


Figure 8: A plot of the Van't Hoff Thermodynamics model for Methylene blue (MB) and Tartrazine (TZ) in a binary solution

Table 3: Van't Hoff Thermodynamics parameters

DYE	ΔG (KJ/mol)	ΔH (J/mol)	ΔS (J/mol k)	R^2
MB	-489	-4705	13.917	0.988
TZ	-542	-5653	16.87	0.966

Key: TZ =Tartrazine and MB = Methylene blue

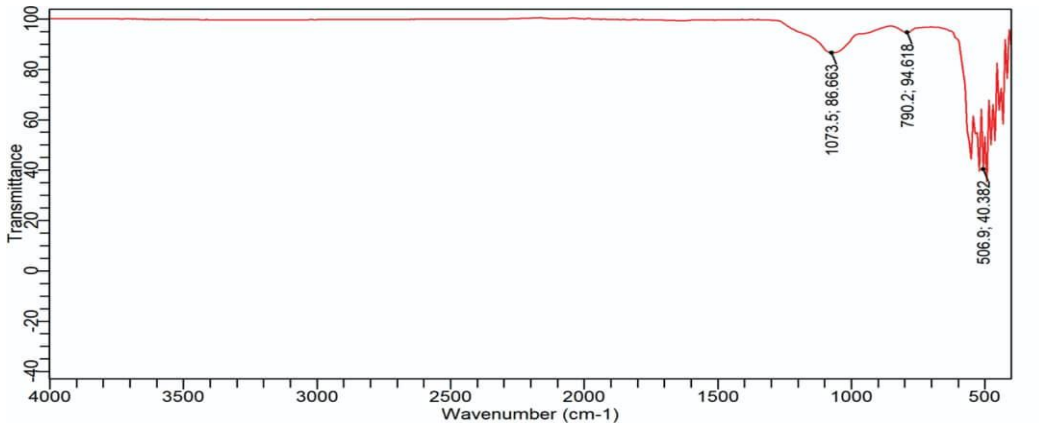
The thermodynamic parameters, such as Gibbs free surface energy change (ΔG^0), change in standard enthalpy (ΔH^0), and change in standard entropy (ΔS^0), define the feasibility of the adsorption process. From the Van't Hoff plot on Figure 8 and Table 3, the parameters were determined. The values of ΔG in KJ/mol for Tartrazine (TZ) and Methylene blue (MB) were negative (-542 and -489). ΔH had -5653 J/mol for TZ and -4705 J/mol for MB, while ΔS obtained was 13.92 for TZ and 16.87 for MB. The negative values of ΔH represent an exothermic reaction. A positive value of ΔS reflects the affinity of the sorbent towards the sorbate, the increased randomness in the solid-liquid interface, increased the degree of freedom of the sorbate, and more favourable conditions for the occurrence of the adsorption process. The negative values of the ΔG reflect the spontaneous adsorption process [19].

FTIR Characterization of silica xerogel

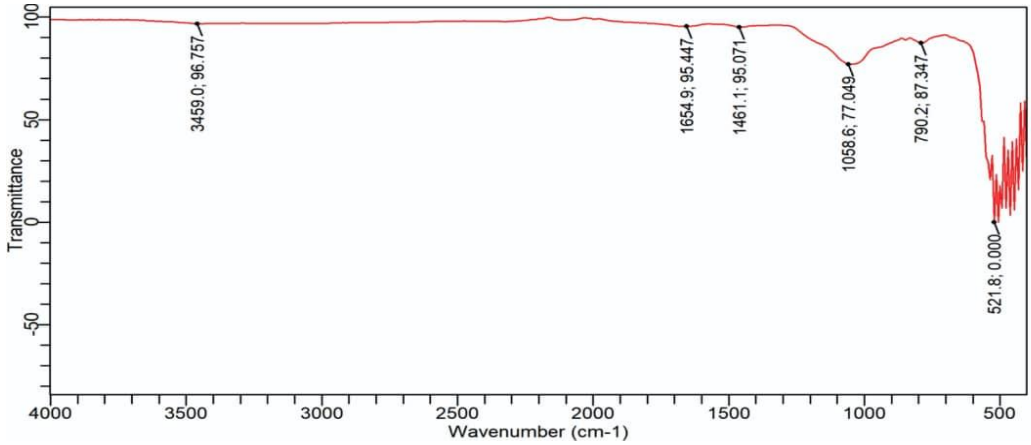
The FT-IR technique is an important tool in identifying characteristic functional groups, which are very important for adsorption. The FT-IR studies of silica adsorbent before and after adsorption are shown in Figures 9 respectively.

For the FT-IR spectra result before adsorption in Figure 9, the band at 506 cm^{-1} is due to the C-I stretching. The band at 790 cm^{-1} is assigned to the C–Cl stretch, and the band at 1073 cm^{-1} corresponds to the Si-O-Si siloxane asymmetric stretching vibration [20]. From the FT-IR spectrum, it is possible to verify that the silica adsorbent shows no peak at 2,800 and 3,000 $cm^{-1,}$ which means that there were no original organic compounds in the silica after controlled extraction and modification [7].

FT-IR spectral result after adsorption in Figure 9 shows absorption bands at 3458 cm^{-1}, which is attributed to the stretching vibration of the OH bond from the silanol group (Si-OH) and adsorbed water molecules on the silica surface. The band at 1636 cm^{-1} is the characteristic peak of the carbonyl group stretch [21]. The band at 1461 cm^{-1} is due to N-H stretching [20]. The result obtained, the result showed that there are changes in the FTIR after adsorption, which can be attributed to the possible adsorption that has taken place.



Before the adsorption of dyes

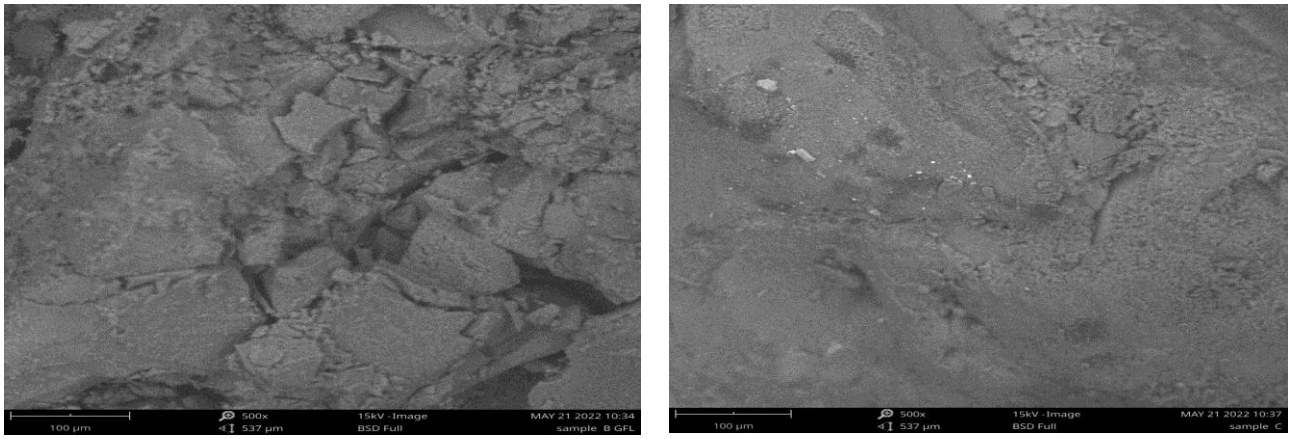


After the adsorption of dyes

Figure 9: FTIR of the Adsorbent before and after Adsorption of dyes

Scanning Electron Microscope (SEM) Characterization of Silica Xerogel

The morphology and surface structure of the adsorbent before and after the adsorption are shown in Figure 10. It has been shown that there was an adsorption on the adsorbent surface, as the morphology before adsorption changed after the adsorption experiment. The adsorbate has been shown to occupy some spaces in the adsorbent, thereby changing the morphology. These images, taken at the same magnification of 500x, show clearly the difference in morphology, which is in agreement with the work by Ezeokonkwo *et al*. [22].



Before the adsorption of dyes After the adsorption of dyes

Figure 10: SEM of the Adsorbent before and after Adsorption of dyes

Comparative study

This study deals with the removal of methylene blue/tartrazine (binary mixtures) utilizing rice husk in a batch adsorption process due to the low cost of rice husk compared with other types of adsorbents. Furthermore, in the actual industrial wastewater treatment, the adsorption process can be applied to obtain a higher removal efficiency of methylene blue/tartrazine (binary mixtures) dye because of its ability to adapt to many-sided processes, which can reduce the costs of operating and processing. Table 4 shows the comparison between this study and others for the removal of other dyes, and shows that rice husk was a promising adsorbent to remove the methylene blue/tartrazine (binary mixtures) for the first time in batch adsorption.

Table 2 Comparison of the adsorption capacity of rice husk for methylene blue/tartrazine (binary mixtures) with other reported materials

Adsorbent	Dye	*Q*e (mg/g)	Reference
Sawdust	Tartrazine	4.71	[23]
Cassava sievate biomass	Tartrazine	20.83	[24]
Cassava sievate biomass	Sunset yellow	0.091	[24]
Rice husk	tartrazine	1.24	[25]
Chitin	black 5	30	[26]
Modified Rice Husk	Methyl orange	312.5	[27]
Modified Rice Husk	Methyl orange	690	[28]
Modified Rice Husk	methylene blue/tartrazine (binary mixtures)	1.92/3.13	This study

Material and methods

Sample collection and pretreatment

319 g of rice husk was obtained from a rice mill in Wurukum, New Bridge, Makurdi, Nigeria. Rice husk obtained after sieving was washed with distilled water to remove dust and dried at room temperature for 48 h [29].

Extraction of silica xerogel from rice husk

The 319 g rice husk obtained was ashed using a muffle furnace at 600 ^{0}C for 3 h and cooled to room temperature. The Rice Husk Ash was then treated with a 200 mL portion of 1.0 M NaOH and heated at 80 ^{0}C in 250 mL Erlenmeyer flasks for 1 h with constant stirring. The solution was allowed to cool to room temperature and filtered through Whatman filter paper (110mm); the residue was washed with distilled water. The filtrate was treated with 1.0 M HCl with constant stirring. Silica gel precipitated when the pH was less than 10. The silica gel formed was aged for 24 h. The gel was transferred into a beaker and dried at 80 ^{o}C for 12 h to produce xerogels. The silica xerogel sample obtained was subjected to washing with distilled water. All the samples were stored in airtight plastic bottles for further analysis [30]. The equations of the reactions are as follows.

$$SiO_2 + 2NaOH \longrightarrow Na_2SiO_3 + H_2O \quad (1)$$

$$Na_2SiO_3 + 2HCl \longrightarrow SiO_2 + 2NaCl + H_2O \quad (2)$$

Modification of Silica Xerogel with Acid

Using nitric acid, 50 g of the extracted silica xerogel was processed. 50 g of silica xerogel and 100 mL of the acid were mixed in a 500 mL beaker, agitated for 30 minutes with a glass rod, and then heated to 80 °C for 1 h as part of the modification procedure. The filtrate was cooled, rinsed with distilled water until its pH was neutral, and then dried [31].

Preparation of Stock Solutions of Adsorbate

In a 1000 mL standard flask, 1 g of Methylene Blue and Tartrazine dyes were dissolved in distilled water to create the stock solutions of the adsorbate, and the amount was diluted accordingly. Distilled water was used to dilute the solution to the required working concentrations, and it was then kept at room temperature [32].

Adsorption Studies

In order to investigate the modified silica xerogel's equilibrium capacity and the impact of starting concentration, ionic strength, contact time, adsorbent mass, and temperature on the adsorption of tartrazine and methylene blue, batch adsorption experiments were conducted. 50 mL of the dye solution and 1.5 g of silica xerogel adsorbent were combined in a 100 mL flask. The mixture was agitated vigorously for approximately 2 h using an electric shaker, and it was then left to equilibrate for 4 h at 28 °C in a thermostatic water bath. The suspension was then filtered through Whatman (No. 1) filter paper, and a UV-Vis spectrophotometer was used to analyze the filtrate. The amount of dyes adsorbed per unit mass was calculated as

$$\text{Adsorption}\ (\%)\ = \frac{C_{i-}\,C_f}{C_i} \times\ 100 \tag{3}$$

where C_i and C_f are the initial and final metal ion concentrations, respectively,
The equilibrium capacity of adsorption is calculated using the formula:

$$\text{Qe}\ =\ (\ \text{Co}\ -\ \text{Ce})\ \times \frac{V}{M} \tag{4}$$

Where q_e (mg/g) is the equilibrium adsorption capacity, C_o (mg/L) and C_e (mg/L) are the initial and equilibrium metal concentration, respectively, V is the volume (L), and M is the amount of the adsorbent [33].

Simultaneous determination of concentration in a binary mixture

As seen below, the percentage adsorption of the dyes in a binary mixture was calculated.
Methylene blue (MB) + tartrazine (TZ). The simultaneous equation approach was used to determine the concentrations of each dye, as indicated below.
Methylene blue (MB) + Tartrazine (TZ)

$$C_{MB}\ = \frac{(A_2 a_{TZ1} - A_1 a_{TZ2})}{(a_{MB2} a_{TZ1} - a_{MB1} a_{TZ2})} \tag{5}$$

$$C_{TZ}\ = \frac{(A_1 a_{MB2} - A_2 a_{MB1})}{(a_{MB2} a_{TZ1} - a_{MB1} a_{TZ2})} \tag{6}$$

Where A_1 is absorbance of the Methylene blue (MB) at λ_1 (662 nm), A_2 is absorbance of the Tartrazine (TZ) at λ_2 (424 nm), a_{MB1} is molar absorptivity of Methylene blue at λ_1 (662

nm), a_{MB2} is molar absorptivity of Methylene blue at λ_2 (424 nm),a_{TZ1} is molar absorptivity of Tartrazine at λ_1 (424 nm)and a_{TZ2} is molar absorptivity of Tartrazine at λ_2 (662 nm).

Conclusions

By using agricultural waste to produce silica xerogel, an environmentally sustainable solution with a technically sound and commercially appealing value-added product has been made possible. The study examined a number of variables, including the impact of concentration, temperature, time, ionic strength, and dosage. For all variables examined in a binary solution, the adsorbent exhibits a greater adsorption efficiency for the removal of tartrazine in comparison to methylene blue. According to the three validity tests (excellent correlation values, closeness in q_e exp. and q_e cal., and modest values of SSE%), only the pseudo-second-order kinetic model best predicts the adsorption of the dyes onto silica xerogel. Adsorption using inexpensive adsorbents and biosorbents is acknowledged as a successful and cost-effective substitute technique for treating wastewater.

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