

Kinetics and Isothermal Investigation of Composite Biosorbent for Treatment of Textile Wastewater Containing Ionic Pollutants

Doris N. Onwenna and Frederick O. Oshomogho

Department of Chemical Engineering, University of Delta, Delta State.

Department of Chemical Engineering, University of Benin, Edo State.

*Corresponding authors' email: fred.oshomogho@uniben.edu Phone: +2348055966268

ABSTRACT

This study evaluated the development of chitosan and cellulose acetate membrane-based biocomposite adsorbent for smart sorption of crystal violet dye pigments and chromium heavy metal ions from textile wastewater. Chitosan and cellulose acetate membranes were synthesized from bivalve mollusk (oyster) shells by alkalization and carboxymethylation, and from rice husk-CA by dewaxing, delignification, and acetylation. A batch adsorption experiment was conducted using a one factor at a time method. Results obtained from oyster shells yielded about 39.58% chitosan, having a porosity of 56.73%, while rice husk yielded 83.71% cellulose with a porosity of 84.69%. Surface micrographic analysis showed a highly porous structure with uneven sizes and shapes, with connected chitosan particles forming a network-like structure. The CA membrane appears to have a rough, porous surface with evenly distributed pores. Surface area analysis showed chitosan having 819.6 m²/g, while cellulose acetate had 407.1 m²/g. An isothermal study showed the Langmuir model was favoured, with a high correlation R² of 0.9936 and an adsorption capacity q_m of 21.231 mg/g for crystal violet and 28.4091 mg/g, thereby suggesting a monolayer adsorption with a localized homogeneous phase. The adsorption kinetic study suggested that the first-order adsorption process, with uptake of crystal violet dye pigments (*k_t*) as 0.0318 min⁻¹ and chromium ion uptake (*k_t*) as 0.0216 min⁻¹, suggests pseudo-first-order kinetics. Hence, a composite biosorbent consisting of rice husk-derived cellulose acetate and oyster shell-based chitosan is very effective in the simultaneous sorption of dissolved chromium ions and crystal violet dye pigments.

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INTRODUCTION

Dyes are organic compounds that impart colour to different substances, and they have many industrial applications such as textiles, ceramics, leather, printing, carpets, paper, food processing and others. They cause pollution of natural water resources and environmental problems. The dyes are the most apparent indicator of water pollution. The discharge of coloured waste may be toxic to the human body and aquatic life (Rehman *et al.*2022). Where that most of the countries specify the maximum allowable concentration for some dyes in the effluent streams less than 1 ppm (Allen *et al.*2005). The crystal violet dye is a cationic dye and a toxic material (Akbar *et al.*2020). Crystal violet is the most common material used in many industries, such as a substance for dyeing cotton, wood, silk, and other materials. Crystal violet dye pollutants that affect human health because of their many uses and their applications are considered as causing difficulty in breathing, nausea, profuse sweating and mental confusion and other effects seriously (Patel, 2022). Water is a crucial natural source for human survival. With the increase in the world's population and the increase in the volume of contaminated water, access to a clean water source is of great importance (Arivoli *et al.*2015). There are various kinds of contaminants in effluents, including toxic ions, heavy metals, pharmaceuticals, organic dyes, pesticides, and hydrocarbon compounds (Mayet *et al.*2023). The presence of these pollutants, especially organic materials, has a detrimental influence on water quality. Eliminating these toxic ions from sewage and effluents, hence, is one of the main goals of various refineries. So far, many processes have been utilized

for effluent treatment, including electrolysis, distillation, electro dialysis, gravity, solvent extraction, flocculation, ion exchange, microfiltration, reverse osmosis, sedimentation, flotation, oxidation, catalytic processes, and sorption. Among these processes, the sorption process is an efficient technique for wastewater treatment, owing to many benefits such as an abundance of adsorbents, high performance, simple operation, and low cost. With this process, the concentration of different organic pollutants can be cost-effectively diminished while improving the quality of the water resource (Brosillon *et al.*2001). One of the most significant features of adsorbents that must be considered is their reusability, which has a great influence on the economic viability of this process. The absorbed contaminant in this process must be retrieved from the sorbent surface (Dargahi *et al.*2021). Other benefits of the sorption approach that should be considered include the abundance of sorbents, a high sorption capacity, high recyclability, cost-effectiveness, and easy desorption of contaminants (Rocha & Zuquette, 2020). This research therefore studies the sorption effects of a blend of chitosan and cellulose acetate on ionic pigments and the influence of important variables in removing these contaminants in water. Sorption kinetics is another factor that will be thoroughly studied. The sorption mechanism of organic contaminants will also be studied.

Water pollution caused by toxic metals and organic compounds is still a serious problem for the general environment (Amakiri *et al.*2022). Therefore, water pollution has become a major source of concern and a priority for most industries. Heavy metal ions and dyes are common

contaminants in wastewater because they are widely used. Many of these contaminants in wastewater are known to be toxic or carcinogenic (Mrozik *et al.* 2003). Conventional treatment involves a process of coagulation/flocculation. This is a versatile process, which can be used alone or combined with biological treatments, as a way of removing suspended solids and organic material, as well as promoting the extensive removal of dyes from textile industry effluents (Mohammad Razi *et al.* 2017). Cellulose acetate is a linear, long-chain, water-soluble, anionic polysaccharide. It is the most widely used cellulose today with applications in various industries (Abe *et al.* 2018). Cellulose and chitin are the most abundant natural polymers on Earth and important biomass resources which are produced in plants and lower animals, respectively. Chitin is used as a raw material for the production of chitosan by deacetylation. Chitosan is a cationic, bioactive, natural polysaccharide, formed by glucosamine and N-acetylglucosamine subunits attached via $\beta(1 - 4)$ linkages. Chitosan has excellent properties for the adsorption of anionic dyes, principally due to the presence of protonated amino groups ($-NH_3^+$) in the polymer matrix, which interact with dyes in solution by ion exchange, at an appropriate pH (Bassyouni *et al.* 2022; Qamar *et al.* 2020). The treatment of textile effluent is a costly task for industries to meet government regulations and to practice environmentally friendly operations. The untreated effluent from these industries can be toxic to aquatic life in receiving waters bodies, hence, the need for the treatment of the wastewater before being discharged into water bodies. Various treatment procedure using different adsorbent has been studied, but getting acceptable level of ionic dye pigment removal has been a very difficult task (Rehman *et al.* 2022). However, the current problems in effluent treatment stem from the increasing pollution of waters by organic and inorganic compounds that are difficult to decompose biologically because of their resistance to the self-purification capabilities of the rivers as well as decomposition in conventional wastewater treatment plants. Consequently, conventional mechanical-biological purification no longer suffices and must be supplemented by an additional stage of processing. Among the physical and chemical processes that have proved useful for the operation is the adsorption onto activated carbon which has which has been widely applied globally (Olafadehan *et al.* 2012). The critical review of various studies conducted on the use of activated carbon has shown that the efficiency of contaminant removal varies from industry to industry.

This study investigated the development of a biocomposite adsorbent from a blend of oyster shell-based chitosan and rice husk-based cellulose acetate for the adsorptive treatment of textile wastewater containing crystal violet ionic dye pigments and chromium heavy metal ions. The adsorption optimization, isotherm, and kinetics were evaluated. This knowledge will help develop very useful information for the establishment of efficient techniques for textile wastewater treatment.

MATERIALS AND METHODS

The major materials used in this research were oyster shells obtained from the Niger Delta region of Nigeria. Other materials, such as rice husk, were obtained from a local rice mill. The chemicals, such as Crystal violet (99.7 % pure), sulphuric acid 98% pure, and hydrochloric acid 99.7 % pure, were produced by JHD, Shatou, Guangdong, China. Others, such as Sodium Hydroxide, 96 % pure, potassium hydroxide, 96% pure, and potassium dichromate, 98% pure, were produced by CDH, New Delhi, India.

Equipment

The glassware used during the experiments includes measuring cylinders of various sizes, beakers of various sizes, conical flasks, and a separating funnel. The specific equipment utilized in the experimental processes is: MS300 Constant Temperature Magnetic Stirrer, B. Brian, England. SX-5-12 Electric Muffle Furnace, PEC Medical, US. 752N UV- Visible Spectrophotometer, PEC Medicals, USA.

Method of Chitosan Extraction and Ca Synthesis

Chitosan will be extracted from oyster shells according to the work done by Saleh *et al.* (2016), and cellulose acetate will be synthesised from rice husk according to Digitata *et al.* (2019). These methods were adopted with little modification.

Chitosan Extraction Procedure

The chitosan extraction procedure adopted would be the alkaline deacetylation of chitin from oyster shells with a strong alkaline solution at variable times, using deproteinization, demineralization, and deacetylation processes according to Saleh *et al.* (2016). Oyster shells were washed and dried in an electric air-dry oven for 6 hours at 105 °C. The completely dried shells were ground with the aid of a mechanical grinding machine, then sieved through a 100 μ m mesh size. The deproteinization was conducted by heating approximately 300 g of ground shell powder, adding 1000 ml of 2 M NaOH solution, and placing it in a water bath set to a constant temperature of 70 °C for 4 hours. The product was neutralized by washing with tap water until neutral pH, and the solid was collected and again washed with distilled water before drying in an air-dry vacuum oven.

The dried product underwent demineralization through treatment with a 1000ml solution of 3.25N HCl at an ambient temperature of approximately (30 $\pm$ 3) °C. The mixture was stirred intermittently at 30-minute intervals for a duration of 4 hours. The solid product was collected and rinsed with distilled water until it reached a pH of 7, after which it was dried. Subsequently, the demineralized sample was subjected to deacetylation by treating it with a 500ml solution of 8.75 M NaOH. The sample was then placed on a constant-temperature magnetic stirrer and heated at 100 °C for 12 hours. The solid, after being deacetylated, was filtered and washed with distilled water. It was subsequently dried in a vacuum oven, and the dried powder was sieved through a 0.4 5 μ m mesh size and kept as a nano-sized particle in an air-tight container until use.

Synthesis of Cellulose Acetate (CA)

A locally collected rice husk sample was cut manually into small pieces and dried in the sun to remove moisture. The dried sample was ground into powder using a grinding disk mill and sieved through a 100 μ m mesh size, and kept in an air-tight container until use. Ground rice husk was dewaxed in a Soxhlet apparatus using n-hexane as solvent, and then the residue was recovered and dried in the oven at 80 °C for 4 hours. The dried RH was powdered by grinding with a mechanical hand grinder. The powdered husk sample was treated with distilled water at 100°C for 2 h, filtered, and dried at 105 °C until a constant weight was obtained. The amount of aqueous extract was calculated as in Equation (1):

$$\text{Rice husk extract}(\%) = \frac{w_1}{w_0} \times 100 \quad (1)$$

where w_1 is the loss in weight and w_0 is the initial weight of the sample (before treatment).

Production of CA was carried out by two reactions: alkalization and carboxymethylation. The alkalization reaction begins after the introduction of NaOH into 5g of pure

cellulose and ethanol solution, under mechanical stirring, at room temperature for an hour. Then, the carboxymethylation reaction starts, under constant stirring, during which monochloroacetic acid (MCA) is slowly added. During this period, the reaction temperature and reaction time were controlled. The product thus obtained was then filtered and suspended in 200 mL of methanol. The slurry was neutralised using glacial acetic acid. Then the sample was washed using a 70% ethanol solution to remove undesired product. Lastly, the sample was dried at 60°C in a forced convection oven (model: FC-610, Toyo) (Lakshmi *et al.* 2017).

Cellulose Acetate (CA) Yield

CA yield was measured on a dry weight basis. The net weight of dried CA was divided by the weight of dried cellulose to get the yield value (Klunklin *et al.* 2021), as shown in Equation (2)

$$CA \text{ Yield, (\%)} = \frac{\text{weight of CA obtained}}{\text{weight of dried cellulose}} \times 100 \quad (2)$$

Preparation and Treatment of Simulated Wastewater

A 1000 mg/L standard solution of potassium dichromate, a hexavalent chromium compound often used in the textile industry, dyes primarily as a chrome mordant to fix dyes, improve colour fastness, and create specific shades in natural and synthetic fabrics, was prepared by weighing 1.362 g into a 1000 mL conical flask and digesting with the addition of 20 mL concentrated nitric acid (HNO₃), placed on a hot plate and allowed to boil until the complete dissolution of the solid. The digested solution was rinsed into a 1000 mL volumetric flask and made up to the volume mark. The prepared heavy metal solution was added, and exactly 1.00 g of crystal violet dye was added to obtain a solution containing heavy metal ions and ionic dye. Lower concentrations were prepared from the standard using the serial dilution method.

Wastewater Treatment Procedure

A batch adsorption experiment will be conducted using 100 mL of the effluent sample, and 0.5 to 2.5 g of the composite adsorbent, prepared in a 1:1 ratio of cellulose acetate and chitosan, will be weighed into a 250 mL conical flask and placed on an orbital shaker at a constant speed of 120rpm. In each experimental condition, a sample was withdrawn and filtered with Whatman's filter paper (No. 11). The filtrate was analyzed for dye pigments with the aid of a UV-Vis spectrophotometer and chromium ion concentration using the atomic absorption spectrometer (AAS). The details of the various operating conditions of the batch experimental investigations are thus highlighted and explained below;

Effect of Adsorbent Dose

In order to study the effect of adsorbent dosage, different dosages of composite adsorbent were varied from 0.5 to 2.5 g/100ml (20 mg/l), and were placed into four 150 mL bottles containing 100 mL of wastewater at 30°C. The samples were agitated for a set period, filtered, and analysed. The percentage removals, P_r , of chromium ions and dye pigment were computed by Equation (3):

$$P_r = \frac{(C_o - C_f)}{C_o} \times 100 \quad (3)$$

Also, the sorbate uptake, q (mg/g), by the adsorbent was determined with Equation (4)

$$q = \frac{(C_o - C_f) \times V}{m} \quad (4)$$

Where C_o and C_f are the initial and final concentrations in mg/l, respectively, V is the volume of effluent in litres, and m is the adsorbent dose in grams.

Effect of Contact Time

The effect of the contact time of the adsorbent on the effluent was studied with varied adsorption times of 15 to 180 minutes at about 1.0g/L of adsorbent. Samples were withdrawn, filtered using a filter, and then immediately analysed for ionic pigment and chromium ion concentration.

Effect of Temperature

The study of the adsorption isotherm is fundamental (Aneke *et al.*, 2014) and played an important role in the determination of the maximum adsorption capacity, in addition to the development of an equation that accurately represented the results and could be used for design purposes. The effect of temperature on the adsorption on the periwinkle shell was investigated under isothermal conditions in the temperature range 303 – 323 K.

Methods of Biosorbent Characterizations

Spectroscopic and microscopic analyses were used to characterize the synthesized biosorbents. Characterization of synthesized biosorbent samples was conducted with Fourier transform infrared spectroscopy (FTIR), X-ray fluorescence equipped with an energy-dispersive spectrometer (XRF-EDS), X-ray diffractometer (XRD), Brunauer-Emmett-Teller (BET), thermogravimetric analysis (TGA), and scanning electron microscope (SEM) to determine the functional groups, elemental and oxide composition, type and crystallinity, surface morphology as well as thermal stability, respectively. The total surface area of the biosorbent was measured using the nitrogen adsorption method and application of the Brunauer, Emmett, and Teller (BET) method.

Adsorption Isotherm Study

The Langmuir adsorption isotherm model (Equation 5) was adopted for homogeneous adsorption and used in monomolecular adsorption processes. The linear form of the Langmuir model is expressed as

$$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{C_e}{q_m} \quad (5)$$

where C_e is the equilibrium concentration of the adsorbate (mg/L), q_e is the amount of adsorbate adsorbed at equilibrium (mg/g), q_m is the Langmuir constant related to adsorption capacity (mg/g), and b is the Langmuir constant related to the energy of adsorption (L/mg). The linear plot of C_e/q_e versus C_e can be plotted, and the constants q_m and b can be calculated from the slope and intercept of the plot. The linear form of the Freundlich model is expressed by Equation (6).

$$\log q_e = \log k_f + \frac{1}{n} \log C_e \quad (6)$$

where q_e is the sorbate concentration in the solid at equilibrium (mg/g), C_e is the sorbate concentration in solution at equilibrium (mg/L), k_f is the Freundlich isotherm constant related to adsorption capacity (L/mg), and n is the Freundlich exponent of the Freundlich isotherm constant related to adsorption intensity. The linear plot of $\log q_e$ versus $\log C_e$ provides the values of n and k_f calculated from the slope and intercept. Temkin isotherm model predicts a uniform distribution of binding energies over the population of surface binding adsorption, presented linearly in Equation (7).

$$q_e = \beta \ln \alpha + \beta \ln C_e \quad (7)$$

where, $\beta = \frac{RT}{b}$, T is the absolute temperature in Kelvin, R is the universal gas constant, 8.314 J/mol. K and b are the Temkin constants related to the heat of sorption (J/mg). A plot of q_e versus $\log C_e$ enables the determination of the isotherm

constants α and b , and the parameters are given in the table below.

Kinetic Study

Pseudo-first-order Kinetic Model

The rate constant of adsorption is determined from the pseudo-first-order equation given by Langergren and Svenka according to Calisto *et al.* (2019) as shown in Equation (8):

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (8)$$

Where q_e and q_t are the amount of dye adsorbed (mg/g) at equilibrium and at time t (h), respectively, and k_1 is the rate constant of adsorption (h^{-1}). The pseudo-second-order equation based on equilibrium adsorption is expressed as shown in Equation (9).

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (9)$$

Where k_2 (g/mgh) is the rate constant of second-order adsorption

RESULTS AND DISCUSSION

Results of Cellulose Acetate and Chitosan Characterization

Results of the physical characterization of the sorbents are shown in Table 1. Chitosan had a higher bulk density, 1.473 g/cm^3 , than the cellulose acetate membrane, 0.358 g/cm^3 . Additionally, chitosan exhibited a lower porosity of 56.73% compared to the cellulose acetate membrane, which had a

porosity of 84.69 %. The surface area of chitosan was also found to be greater at $704.2 \text{ m}^2/\text{g}$ than that of the cellulose acetate membrane, which had a surface area of $8196 \text{ m}^2/\text{g}$. These physical characteristics play a significant role in determining the sorption capacity and efficiency of the sorbents in various applications. Micropore area and pore volume are significant features of an adsorbent (Liu *et al.* 2020). The micropore area of $400.5 \text{ m}^2/\text{g}$ for chitosan and $428.8 \text{ m}^2/\text{g}$ for cellulose acetate suggests that they are suitable for the sorption of ionic environmental pollutants. Their high micropore area allows for a greater surface area for adsorption to occur, making them effective in removing contaminants from water or air. Additionally, the pore volume of these materials indicates that they have a large capacity to hold onto pollutants, further enhancing their ability to purify the environment. Overall, the combination of high micropore area and pore volume makes chitosan and cellulose acetate ideal choices for environmental remediation applications. These results conform to chitosan and cellulose acetate membranes used in wastewater treatment to remove heavy metals like lead and mercury from contaminated water by Pooremael & Namazi (2020). The high surface area of these materials allows for efficient adsorption of the toxic metals, while their large pore volume ensures a high capacity for pollutant removal, resulting in cleaner water for discharge into the environment (Algarni & Al-Mohameed, 2022; Bassyouni *et al.*, 2022).

Table 1: Results of Adsorbent Characterisation

Property	Chitosan	Cellulose Acetate
Yield (%)	39.58	83.71
Bulk density (g/cm^3)	1.473	0.358
pH	7.14	7.02
Porosity (%)	56.73	84.69
Surface area (m^2/g)	704.2	8196.0
Micropore area (m^2/g)	400.5	428.8
Pore volume (cc/g)	2.015	15.24
Pore width (nm)	5.283	4.759
Pore diameter (nm)	2.64	2.647

Results of FTIR Analysis of Adsorbent

The FTIR spectrum of the adsorbents (Figures 1 and 2) reveals a complex mixture of functional groups, indicating their potential for effective ionic particulate matter removal. From Figure 4.1 of the oyster shell chitosan, the first peaks 1096.82 cm^{-1} correspond to a stretching C – O bond of aliphatic ether, and the strong stretching peak obtained at 1786.64 cm^{-1} correspond to C = O of an acid halide compound. In addition, the presence of peaks at 3612.62 cm^{-1} and 1633.15 cm^{-1} suggests the presence of alcohol (O – H) and conjugated alkene (C= C), while 1478.23 cm^{-1} showed the presence of an alkane methylene group (C – H) in the structure.

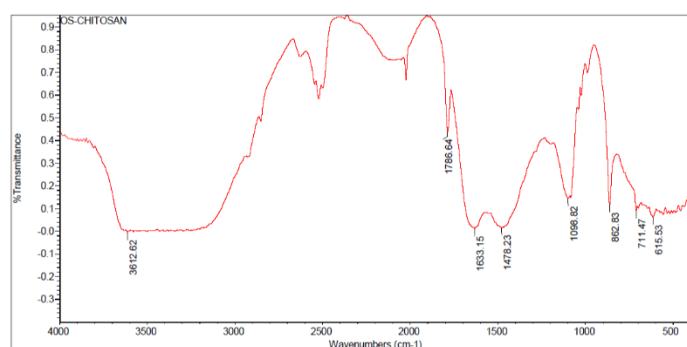


Figure 1: The Spectrum Peaks of Oyster Shell Chitosan

Figure 1 shows the FTIR spectra that were obtained for the produced adsorbent. The sample clearly displays several absorption peaks, reflecting the complex nature of the

material being examined. The prominent absorption bands have a sensitivity of 50 and an absolute threshold of 0.578 in the range of $3700\text{--}400 \text{ cm}^{-1}$. The table below provides a

summary of the experiment's results. The FTIR spectrum of the rice husk-based cellulose acetate in Figure 2 also shows various functional groups, where the first peak, 619.18 cm^{-1} , corresponds to a bending C – H of alkyne. The peaks at 1101.32 cm^{-1} and 1637.08 cm^{-1} indicate the presence of C – N and C = C, which are assigned to medium-strength amine stretching and conjugated alkene, respectively. Additionally,

the FTIR spectrum revealed a peak at 3440.49 cm^{-1} , indicating the presence of medium-stretching primary amine (N–H) stretching bonds in the cellulose acetate structure. The FTIR spectrum provides valuable information about the functional groups present in the rice husk-based cellulose acetate, allowing for a better understanding of its chemical composition.

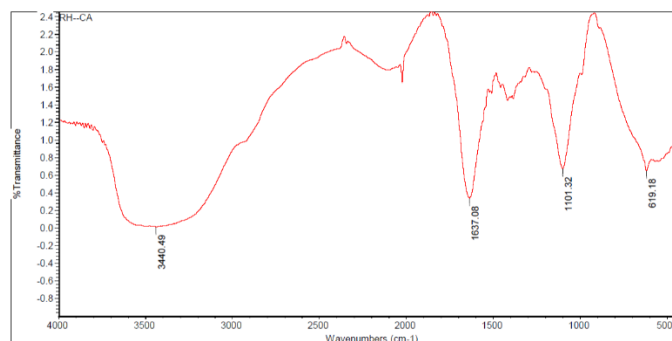


Figure 2: Spectrum of the Rice Husk Cellulose Acetate

Elemental Composition of OS Chitosan

An energy-dispersive X-ray spectrometer was used to determine the elemental composition of the chitosan component of the sorbent, and Figure 3 shows the spectrum of the oyster shell chitosan. From Table 3, it is shown that Calcium (Ca) was the most predominant element at 95.03wt%. Additionally, Carbon (C) was also present in significant amounts at 2.49wt%, followed by Aluminium (Al) at 0.58wt%. These results confirm that the oyster shell chitosan is primarily composed of Calcium, which aligns with the natural composition of oyster shells (Qamar *et al.*, 2020).

The presence of these elements further supports the effectiveness of the sorbent for heavy metal removal due to its high affinity for binding metal ions (Bayuo *et al.*, 2023). The high content of carbon suggests that the chitosan may also possess adsorption properties that can aid in removing organic contaminants from water. The presence of aluminium could potentially enhance the overall stability and mechanical strength of the chitosan material, making it a durable and efficient sorbent for various water treatment applications (Saleh *et al.* 2016).

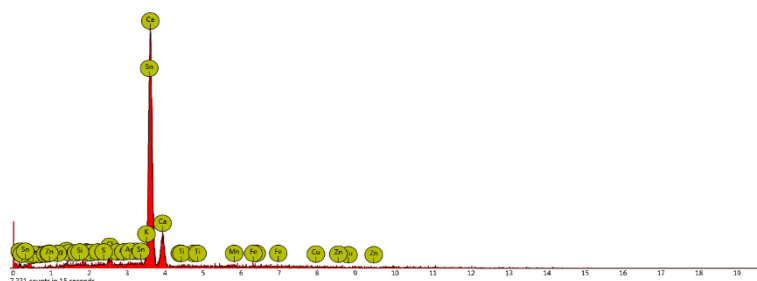


Figure 3. EDS Spectrum of OS Chitosan

Table 3: Elemental Content of Oyster Shell-Based Chitosan

Element Name	Element Symbol	Weight Conc. (wt%)
Calcium	Ca	95.03
Carbon	C	2.49
Chlorine	Cl	1.38
Aluminium	Al	0.58
Sodium	Na	0.32
Phosphorus	P	0.20
Magnesium	Mg	0.00
Sulphur	S	0.00

Elemental Composition of Cellulose Acetate

The results of EDS analysis of rice husk cellulose acetate (Figure 4) show the elemental composition, as shown in Table 4. The analysis revealed that the rice husk cellulose acetate sample contained carbon, hydrogen, and oxygen, which are typical elements found in organic compounds. The presence of these elements confirms that the cellulose acetate was derived from natural sources, specifically rice husks.

Additionally, the EDS analysis showed small traces of other elements, suggesting that the sample may have been minimally processed or contaminated during preparation. Overall, the results provide valuable insights into the composition of the cellulose acetate derived from rice husks, which can aid in understanding its potential applications in various industries.

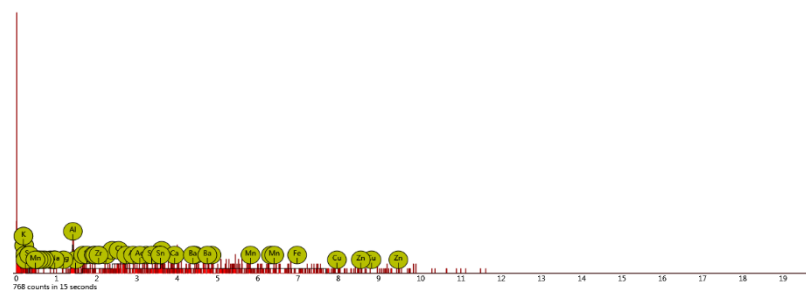


Figure 4: EDS spectrum of RH-CA

These results reveal that the rice husk cellulose acetate is composed mainly of carbon and oxygen, with small traces of other elements such as hydrogen and nitrogen. The high

carbon content suggests the material is predominantly cellulose, while the presence of oxygen indicates the presence of acetate groups.

Table 4: Elemental Content of RH-Cellulose Acetate

Element Number	Element Name	Element Symbol	Weight Conc. (wt%)
6	Carbon	C	36.02
29	Copper	Cu	23.18
30	Oxygen	O	19.90
13	Hydrogen	H	13.71
20	Calcium	Ca	3.60
12	Magnesium	Mg	1.82
14	Silicon	Si	1.14
11	Sodium	Na	0.63
15	Phosphorus	P	0.00

Surface Morphology of Chitosan and Cellulose Acetate Samples

Figure 5 shows the SEM micrograph of chitosan produced from oysters. A highly porous structure with uneven sizes and shapes is evident in the SEM image. The connected chitosan particles form a network-like structure. This distinctive shape suggests that oyster-derived chitosan may have improved

qualities for several uses, such as wastewater treatment. The porous structure of the chitosan could provide a large surface area for adsorption, making it a potentially effective material for removing pollutants from water. Additionally, the interconnected network of chitosan particles suggests that it may have enhanced mechanical strength and durability, allowing it to be used in various applications with confidence.

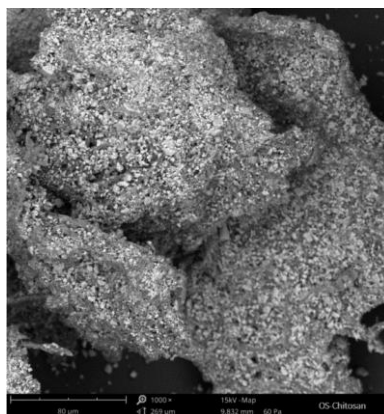


Figure 5: SEM Micrograph of Oyster Shell-Based Chitosan

The micrograph of the scanning electron microscope of the rice husk-derived cellulose acetate membrane is shown in Figure 6. The membrane appears to have a rough and porous surface, which could enhance its filtration capabilities. The pores are evenly distributed throughout the membrane, indicating uniformity in its structure. Overall, the membrane's

morphology suggests that it would be effective in separating particles and impurities from liquids. The consistent pore size and distribution allow for efficient filtration without any areas of weakness. This would make it a reliable option for various industrial applications.

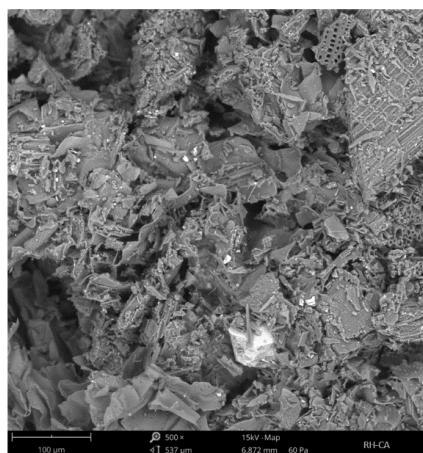


Figure 6: SEM Micrograph of Rice Husk-Derived Cellulose Acetate

SEM micrograph analysis provides efficient and detailed knowledge of the adsorbent's pore structures. This information is crucial for optimizing the adsorption process of dissolved ionic pollutants of heavy metals and ionic dissolved dye pigments. By understanding the pore structures of the adsorbent, researchers can fine-tune parameters such as temperature, pressure, and flow rate to maximize efficiency. This can lead to cost savings and improved performance in various industrial applications, such as wastewater treatment and gas separation. SEM micrograph analysis has played a vital role in understanding the effects of the adsorbent in the field of adsorption science and technology.

Surface Area and Porosity of Chitosan and Cellulose Acetate

The Brunauer-Emmett-Teller (BET) analysis of chitosan and cellulose acetate reveals their surface properties and pore

characteristics, which are crucial for efficient removal of ionic dyes and heavy metals, and are presented in Table 5. Chitosan, a biopolymer derived from chitin, had a high surface area of 819.6 m²/g due to its hierarchical structure. The surface area of cellulose acetate was 407.1 m²/g. This difference in surface area can be attributed to the unique morphology of each material, with chitosan containing more pores and rough surfaces compared to cellulose acetate (Candido & Gonçalves, 2016; Mayet *et al.*, 2023). The higher surface area of chitosan makes it a promising material for applications in water treatment, where increased surface area is advantageous. Cellulose acetate's lower surface area may make it more suitable for a less porous surface, such as in the removal of dissolved heavy metal ions.

Table 5: BET Parameters

Area/volume Parameter	Chitosan Value	Cellulose Acetate Value
Surface area (m ² /g)	819.6	407.1
Micropore area (m ² /g)	428.8	356.1
Cum Surface area (m ² /g)	90.59	73.95
Cum Ads Pore volume (cc/g)	0.2177	419.8
Micropore volume (cc/g)	0.1524	0.1266
Ads pore diameter (nm)	2.426	2.398
Micropore width (nm)	6.503	6.417
Pore diameter (nm)	2.647	2.647

Composition of OS Chitosan

The crystalline phases of the chitosan sample were determined by XRD analysis. The following weight percentages (wt%) were found by quantitative analysis from Figure 7, which shows the presence of many phases in the sample: 49 % aragonite (CaCO₃), 41 % muscovite (KAl₂(AlSiO₃O₁₀)(F, OH)₂), 7.4 % orthoclase, and 2.7 % quartz (SiO₂). These results indicate the complex nature of the chitosan sample, with multiple crystalline phases present. The dominant presence of aragonite suggests a significant amount of calcium carbonate in the sample, while the presence of

muscovite and orthoclase indicates minerals rich in potassium and aluminum. The small percentage of quartz suggests a minor presence of silicon dioxide in the sample as well (Bassyouni *et al.*, 2022). For example, the high percentage of aragonite in the sample could indicate that it was derived from a source rich in calcium carbonate, such as seashells. The presence of muscovite and orthoclase could suggest that the sample also contains minerals typically found in metamorphic or igneous rocks, adding to the complexity of its composition.

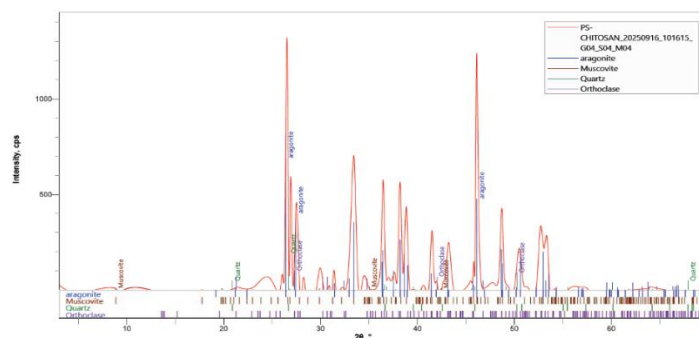


Figure 7: Crystallographic Spectrum of Oyster Shell-Based Chitosan

Isothermal Analysis of Composite Adsorbent

The results of the adsorption isotherm of the composite adsorbent conducted on the simulated wastewater containing Crystal violet as an ionic dye pigment and chromium ions as a heavy metal are shown in Figures 8 to 13. The Langmuir isotherm study on ionic dye pigment sorption is shown in Figure 8 and revealed that an increase in composite dosage

directly favours the percentage dye pigment removal. This suggests that the sorption process is dependent on the amount of composite present in the system (Oshomogho *et al.*, 2020). These results demonstrate that the adsorption capacity increases with increasing adsorbent dosage, indicating a higher affinity between the material and the adsorbate at higher dosages.

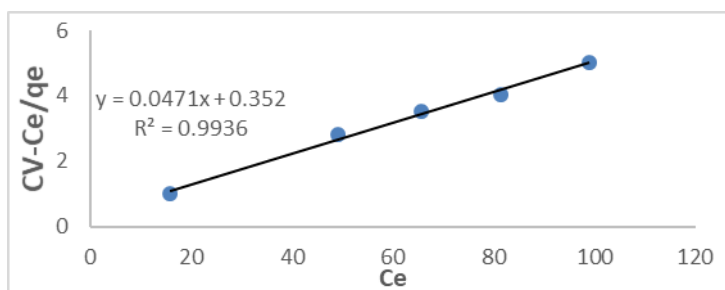


Figure 8: Langmuir Isotherm Plot of Dye Pigment (CV) Adsorption

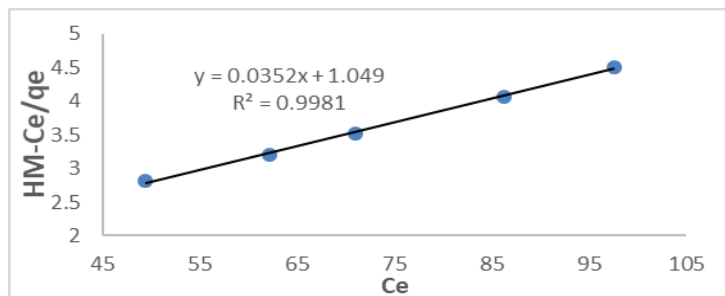


Figure 9: Langmuir Isotherm Plot of Heavy Metal (HM) Adsorption

Freundlich isotherm (Figures 10 and 11) describes the amount of crystal violet chromium ions adsorbed onto a gram of biosorbent (q_e). It determines adsorption onto heterogeneous surfaces (Awokoya *et al.*, 2016). It was observed that the batch adsorption data correlated with the linear isotherm since a straight line was obtained, with intercepts on both horizontal and vertical axes for the linear isotherm. Since $n = 4.2301$, which is greater than 1, estimated from the straight line

obtained using the Freundlich isotherm plot shown in Figure 9, the isotherm correlated with the equilibrium data, with the regression coefficient, R^2 , being 0.9451, which is very close to unity. The other constant in the Freundlich isotherm was $k_f = 6.648$, which revealed the adsorption capacity (Ji *et al.*, 2023).

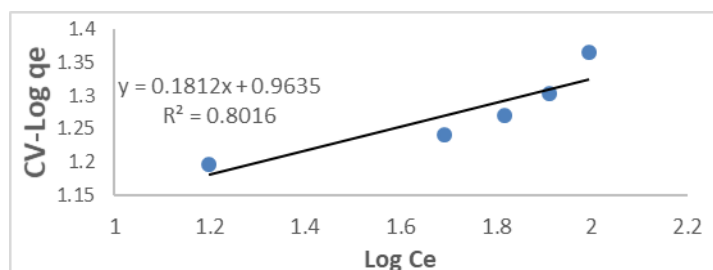


Figure 10: Freundlich Isotherm Plot of Dye Pigment Adsorption

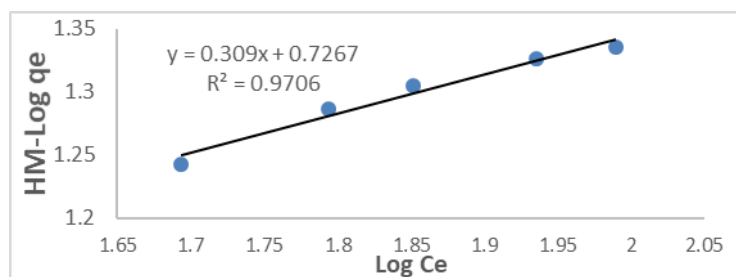


Figure 11: Freundlich Isotherm Plot of Heavy Metal Adsorption

The Temkin isotherm model predicts a uniform distribution of binding energies across the surface binding sites (Oladoja *et al.*, 2008). Figures 12 and 13 show the results of the Temkin isotherm study for crystal violet pigments and chromium ion adsorption (Ghani *et al.*, 2022). The graphs show a clear

physisorption between sorbate concentration and the amount adsorbed on the surface (Figures 12 and 13). The Temkin isotherm model accurately predicted the behaviour of the chromium ions on the surface.

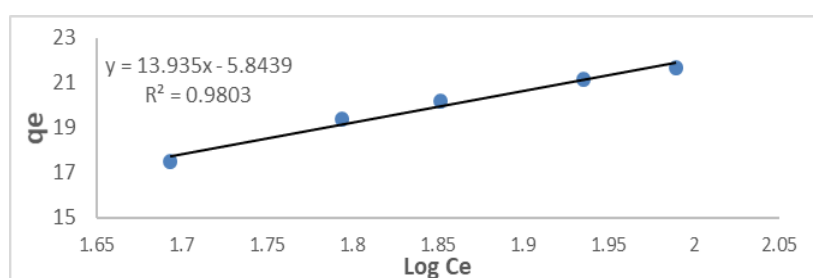


Figure 12: Temkin Isotherm Plot of Heavy Metal Adsorption

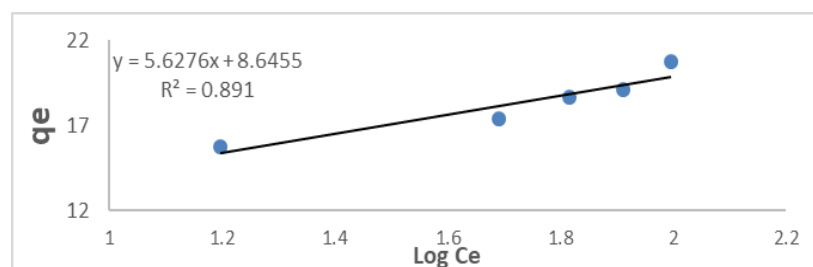


Figure 13: Temkin Isotherm Plot of Dye Pigment Adsorption

Isothermal Constants Analysis of Composite Adsorbent

The results of the Freundlich, Langmuir, and Temkin isothermal study for crystal violet dye pigment and chromium ion sorption onto biocomposite sorbent are presented in Table 6. Langmuir isotherm ($R^2 = 0.9936$), the Temkin isotherm ($R^2 = 0.8910$), and the Freundlich isotherm ($R^2 = 0.8016$) had the lowest fit for the crystal violet dye pigment sorption correlation. The chromium ion sorption isothermal study revealed a Langmuir isotherm ($R^2 = 0.9981$), a Freundlich isotherm ($R^2 = 0.9706$), and a Temkin isotherm ($R^2 = 0.9801$).

Therefore, the Langmuir isotherm showed the best fit for both crystal violet dye pigment and chromium ion sorption onto the biocomposite sorbent, with R^2 values of 0.9936 and 0.9981, respectively. This indicates that the sorption process follows a monolayer adsorption mechanism for both pollutants. The Freundlich isotherm provided the poorest fit for the sorption of the crystal violet dye pigment, suggesting that the sorption behaviour may be more heterogeneous than that of chromium ions.

Table 6: Constants of Isotherm Models

Isotherm models	Constants	Dye Pigment Value	Heavy metal value
Langmuir	b (l/mg)	0.1338	0.0335
	q_m (mg/g)	21.2310	28.4091
	R^2	0.9936	0.9981
Freundlich	k_f (mg/g)	9.1940	5.33
	n	5.5187	3.2362
	R^2	0.8016	0.9706
Temkin	α (J/mg)	34.3750	10.925
	b (J/mg)	403.3197	162.899
	R^2	0.8910	0.9801

Kinetics Analysis of Composite Sorption of Dye and Heavy Metal

The results of the kinetic study on the sorption of pollutants from contaminated water, analysed using first- and second-

order kinetic models, have been presented in Table 7 to reveal the rate of dye pigment uptake by the composite adsorbent.

Table 7: Dye Pigment Adsorption Kinetic Data

Adsorption time (minutes)	Dye removal (%) qt	qe-qt	log(qe-qt)	t/qt
60.41	76.604	19.363	1.287	0.788
65.80	80.732	15.235	1.183	0.815
70.38	83.853	12.114	1.083	0.839
75.19	86.782	9.185	0.963	0.866
80.19	89.420	6.547	0.816	0.897
85.36	91.740	4.227	0.626	0.930
90.16	93.481	2.486	0.395	0.964
95.14	94.917	1.050	0.021	1.002

The first-order kinetic model showed a higher R^2 of 0.9457 (Figure 4.33) than the second-order kinetic model with $R^2 = 0.9946$ (Figure 4.34), indicating that the sorption process followed pseudo-first-order kinetics. This suggests that the adsorption of crystal violet dye pigments onto the adsorbent is primarily controlled by surface phenomena such as pore diffusion or film diffusion (Goswami *et al.*, 2023). Additionally, pseudo-first-order kinetics indicate that sorption is dependent on the availability of sorption sites on

the adsorbent surface, and the rate decreases over time. This could be because the adsorbent has a higher affinity for chromium metal ion molecules at the beginning of the sorption process, leading to rapid initial adsorption. Furthermore, the slower rate of adsorption after the initial stage could be attributed to saturation of available sorption sites or hindrance by diffusion limitations within the pores of the bio-sorbent material.

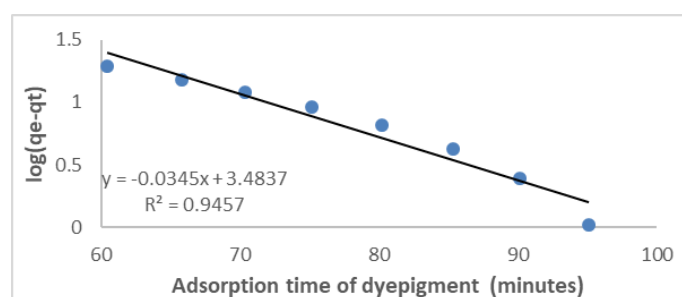


Figure 14: The Pseudo-First-Order Kinetic Plot for Crystal Violet Dye Pigment Uptake

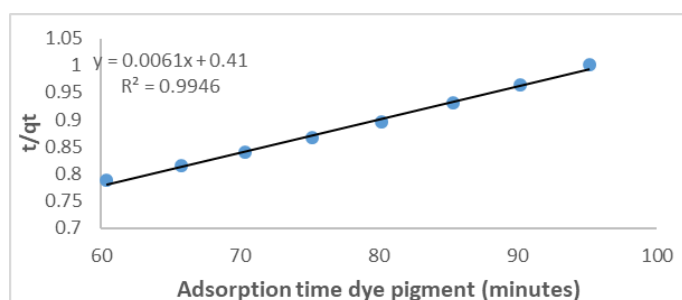


Figure 15: Pseudo-Second-Order Kinetic Plot for Crystal Violet Dye Pigment Uptake

Table 8: Chromium Ion Adsorption Kinetic Data

Adsorption time (minutes)	HM Sorption (%) qt	qe-qt	log(qe-qt)	t/qt
60.40	70.435	26.725	1.427	0.857
65.11	75.083	22.077	1.344	0.867
70.29	79.740	17.420	1.241	0.881
75.29	83.740	13.420	1.128	0.899
80.09	87.248	9.912	0.996	0.918
85.25	90.497	6.663	0.824	0.942
90.27	93.189	3.971	0.599	0.968
95.06	95.354	1.806	0.257	0.997

The kinetic study of chromium ion sorption revealed a pseudo-second-order kinetic model (Figure 4.36) with an R^2 of 0.9765 as the best fit for the sorption process, compared to the pseudo-first-order kinetic process (Figure 4.35) with an R^2 of 0.9437. This indicates that the sorption of chromium

ions onto the material is more accurately described by a pseudo-second-order kinetic model. The high R^2 value of 0.9765 suggests that this model closely represents the actual sorption process, indicating a strong correlation between the theoretical and experimental data.

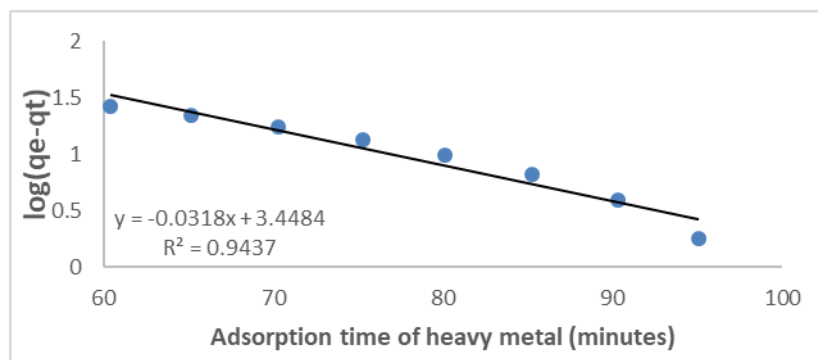


Figure 16: The Pseudo-First-Order Kinetic Plot for Chromium Ion Uptake

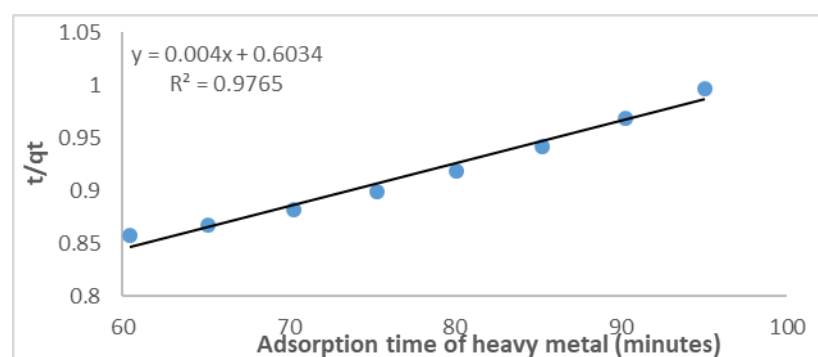


Figure 17: Pseudo-Second-Order Kinetic Plot for Heavy Metal Uptake

Pseudo-First and Second Order Kinetic Constants

Table 9: Adsorption Kinetics Constants Data

Sorbate	Rate Constant	1 st Order	2 nd Order
Crystal violet pigment uptake	k (min ⁻¹)	0.0216 (min ⁻¹)	0.000955 L/mol.min
	qe (mg/g)	72.6106	105.2632
Chromium ion uptake	k (min ⁻¹)	0.0318 (min ⁻¹)	0.0040 L/mol.min
	qe (mg/g)	250.00	0.929

Table 9 presents the kinetic constants for the first- and second-order kinetic models of the crystal violet pigments and chromium ion sorption. The first-order rate constant (k_1) is determined to be 0.0216 min⁻¹, while the second-order rate constant (k_2) is found to be 0.000955 L/mol.min. These values indicate that the first-order adsorption process proceeds at a faster rate compared to the second-order adsorption process, with a higher uptake of crystal violet dye pigments being adsorbed onto the sorbent per unit time. The rate of chromium ion uptake by the composite sorbent (Table 4.17) was seen to have a first-order rate constant (k_1) of 0.0318 min⁻¹ and a second-order rate constant (k_2) of 0.0040 L/mol.min. Therefore, the higher value of k_1 in both crystal violet pigment and chromium ion sorption suggests that the adsorption process follows pseudo-first-order kinetics, indicating that the rate-limiting step is the desorption of ionic pollutants from the solution onto the sorbent (Aktar, 2021). On the other hand, the lower value of k_2 for the second-order kinetics model implies that the adsorption process is limited by the availability of active sites on the sorbent surface for the ionic pollutant molecules to attach to (Ji *et al.* 2023).

CONCLUSION

This research investigated the treatment of industrial wastewater containing ionic dye pigment and dissolved chromium using a blend of cellulose acetate membrane and chitosan. The study revealed the effectiveness of the composite adsorbent in the uptake of the dissolved ionic pollutant. A composite biosorbent consisting of rice husk-derived cellulose acetate and oyster shell-based chitosan was very effective in the simultaneous sorption of dissolved chromium ions and crystal violet dye pigments. The Langmuir isotherm is the best fit for the sorption equilibrium data, with the regression coefficient, R^2 , being 0.9936 and 0.9981 for dye and heavy metal, respectively. The rate of chromium ion and crystal violet uptake by the composite sorbent was seen to more suited to a first-order rate constant (k_1) as 0.0318 (min⁻¹) and 0.0318 (min⁻¹) respectively.

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