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Prefatory Evaluation on the Application of Blend Membrane of Sodium Alginate and *Moringa Oleifera* Leaves Grafted Polyacrylamide for Adsorption of Zink and Iron (ii) in Aqueous Solution

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ABSTRACT

Metal contamination of wastewater creates a need for effective adsorbents derived partly from readily available biomass. This study prepared a composite membrane comprising sodium alginate and *Moringa oleifera* leaves grafted with polyacrylamide (Na-Alg/MOL-g-PAAm) and evaluated its performance for removing zinc (Zn^{2+}) and iron(II) (Fe^{2+}) ions from aqueous solutions. The graft copolymer was synthesized using ceric ammonium nitrate as an initiator, blended with sodium alginate and crosslinked with glutaraldehyde. Batch adsorption experiments examined the effects of solution pH, ionic strength, temperature, initial metal-ion concentration and contact time. Residual metal concentrations were determined using atomic absorption spectrophotometry. The membrane achieved reported removal efficiencies of 95.7% for Zn^{2+} and 93.9% for Fe^{2+} . Metal uptake generally increased with increasing pH, initial concentration and contact time, whereas increasing ionic strength and temperature reduced uptake within the investigated ranges. Langmuir and Freundlich isotherms were examined to describe equilibrium adsorption, although incomplete fitted parameters limited definitive comparison of the models. These findings demonstrate the potential of Na-Alg/MOL-g-PAAm as an adsorbent for Zn^{2+} and Fe^{2+} removal under the tested conditions. Further characterization, regeneration studies and evaluation using real wastewater are needed to establish its suitability for practical treatment applications.

Keywords: *Moringa oleifera*; sodium alginate; polyacrylamide; composite membrane; metal-ion adsorption; wastewater treatment.

1. INTRODUCTION

Our environment is a wide and open place made natural; it includes mutual relationship between living things and non-living things. The mutual relationship in such a way that none can do without each other. The living organisms compete tremendously with the wellbeing of man, thereby meddling into human health that results to chronic illness and could also lead to sudden death of the victim (Kristin et al., 2023). Water is one of the abundantly available substances in nature. It is an important constituent of all animal and vegetable matter. Therefore, availability of safe and dependable source of water is very important for sustainable development and encouraging standard of living. However, water and its management will continue to be a major issue with

definite and profound impact on our lives and that of our planet earth (Takuissu et al., 2023). Desert is not habitable due to lack of water, yet water is the main carrier of major contaminants like heavy metals and microbes. Consequently, the quality of drinking water has been a factor in determining human welfare. And frequent pollution of drinking water has caused water borne diseases that have decimated the population of the whole cities especially in the western part of Africa. Much water polluted by natural sources has imposed a tremendous hardship to people who are compelled to use it either for irrigation or domestic purposes (Johnson et al., 2021).

Metals such as lead and cadmium are contained in the wastewater of industries such as electroplating, plastic paint manufacturing industries, mining petrochemical processes, batteries paper and pulp industries (Li et al., 2019). Moringa also known as mother's best friend grows fast and reaches up to 12 m. It has a grey bark, very thick, stiff and looks like cork peeling patches (Klimek Szykutowicz et al., 2024). *M. oleifera* being a natural tree loses its leaves from the month of December to January and new growth starts. This is for those places where dry season is observed from November to February end or March beginning example Nigeria. In ancient time, Moringa was known and used in ancient societies around the world. Report (Wendesen and Million, 2021) has the seed utilized in waste water treatment due to the high potentials of antioxidant in the seed which includes phenol. The result showed that *M. oleifera* seeds were very effective in the removal of impurities from waste water. Also (Akinteriwa et al., 2016), in the demonstration of the use of immobilized *M. oleifera* leaves in removing heavy metal ions from waste water reported how effective the biomass was. Therefore in the optimization of the leaves for waste water treatment, the leaves need to be subjected to improve form by blending it with improved polymers compound to improve in its remediation quality. The paper contributions are;

- i. **Development of a composite adsorbent membrane:** The study prepared a sodium alginate membrane blended with Moringa *oleifera* leaves grafted with polyacrylamide (Na-Alg/MOL-g-PAAm) and evaluated its application for removing Zn^{2+} and Fe^{2+} from aqueous solutions.
- ii. **Experimental evaluation of metal removal performance:** The study reported removal efficiencies of **95.7% for Zn^{2+}** and **93.9% for Fe^{2+}** and examined how pH, ionic strength, temperature, initial metal concentration and contact time influenced adsorption, providing evidence of the membrane's performance under the tested conditions.

2. MATERIALS AND METHODS

2.1 Materials

Materials used were sodium alginate, ceric ammonium nitrate, methanol, acryl amide, glutaraldehyde, hydrochloric acid, sulfuric acid, sodium hydroxide, n-hexane, ethyl acetate, ethanol @ 96 %, acetone which were obtained from ELISCO and OKEY ENTP. Chemical stores Enugu and all were analytical grades and all the apparatus used were not listed. The *M. oleifera* leaves

being the major raw material was obtained locally in its natural form. Hence the chemicals were utilized as supplied.

2.2 Preparation of Moringa oleifera leaves

The Moringa oleifera leaves were obtained by hand plucking directly from the tree located in umuonyiba Ituku, Awgu Local Government Area of Enugu State, Nigeria. This was achieved by drying the leaves at room temperature of about 30°C thereafter, crushed and sieved with a mesh of 100 µm so as to obtain a fine homogenous powder and was stored into polyethylene bag and kept at room temperature of (25 °C-30 °C) for the experiment. Then sodium alginate (Na-Alg) was used as supplied.

2.3 Preparation of the graft copolymer

M oleifera leaves-g- polyacryl amide (MOL-g-PAAM) was synthesized or prepared by reacting M oleifera leaves with acryl amide at 60 °C using ceric ammonium nitrate (CAN) as an initiator (Toti and Amiabhavi, 2002). To illustrate this, 2 % aqueous M oleifera leaves was prepared and kept properly for a period of one hour with 0.105mol of acryl amide at 60 °C. Then initiator concentration of 2.5×10^{-3} mol was added to the mixture and also stirred properly for another five hours (5hrs). The mass which was obtained was precipitated in excess acetone, and then same was filtered, washed with a mixture of water and methanol at ratio of 7:3 to remove the homopolymer that was formed. The resultant poly mass was then dried in an electrically controlled oven (Pickstone oven model Hp: 30) at 40 °C and weighed using mettler balance AB 204 (Ahmed et al., 2019).

2.4 Preparation of membrane

About 4 % mass of stock solution of sodium alginate (Na-Alg) was prepared in water (Sun gil et al., 2010). The stock solution with a value of 100 ml was taken in a 250 ml beaker and mixed with 0.0017 mol of glutaraldehyde (GA) or 0.1ml of 25 % in 100ml of water. Then the mixture was stirred for two hours (2 hr) at 25 °C then the mixture was poured gently and uniformly on a plastic tray and this was our membrane. The membrane formed was dried at room temperature of (25 °C-30 °C) range for twelve hours (12 hrs). Then the cast membrane on the plastic tray was cross linked by immersing it in a 1 % HCl solution taken in an equimolar mixture of methanol and water and was allowed to stand for twenty-four hours (24 hrs). Thereafter, it was washed properly in the water and dried (Osemeahon et al., 2015).

Then to prepare the blend membranes, a 4% mass of the stock solution of sodium alginate and M oleifera leaves blend (MOL-g-PAAM) was mixed in a ratio of 50:50 at 60 °C four hours (4hrs) and the solution was allowed to cool at room temperature. Then 0.1ml of 25 % in water (0.00175 mol) of GA was added onto the blend and the resultant mixture was stirred for another two hours (2 hrs). At the end of this period the mixture was poured onto a Petri dish and cross linked (Bagheripour et al., 2016).

2.5 Metal ion sorption capacity of the membrane

To determine the metal ion sorption capacity of the membrane, the three sets of samples containing 0.2 g of the dried membrane and 50ml of 200 ppm different metal ion (Cd⁺², Zn⁺² and Fe⁺²)

solution was prepared in a round bottom flask and stirred each for 24 hours at the same temperature of 25 °C. The solution was filtered and analyzed for residue metal ion (Attaso et al; 2022).

Effect of pH on sorption capacity of the membrane

The sorption behavior of the membrane was determined at different pH values ranging from 1 to 6 at room temperature of 25 °C. 0.2M sodium hydroxide and 2.0 M hydrochloric acid was used to adjust the solution of the different metal ion under study to the desired pH as the case may be the gross sample was stirred for 4 hours. Then the residue metal ion concentration was determined using AAS (Bouamama et al., 2017).

2.6 Effect of ionic strength on sorption capacity of the membrane

The effect of ionic strength on sorption capacity of the membrane was determined by measuring various concentrations of metal ion solutions. 50ml of Different metal ions under study was measured and a known mass of sodium chloride range (0.20 g-1.0 g) was used to adjust the ionic strength of the measured metal ion solutions, the gross solution was stirred for 4hour, filtered and analyzed using AAS (Telma et al., 2019).

2.7 Effect of temperature on sorption capacity of the membrane

The effect of temperature on the sorption capacity of the membranes was investigated as follows. About 2.0 g of the grafted membrane was stirred for 4 hours with 50ml 200 ppm of the metal ions solution at 25 °C, thereafter, the artificial waste water was filtered and analyzed for residual metal ion concentration. And this process was repeated at different temperature range of 30 °C to 90 °C (Lukasz et al., 2022).

2.8 Effect of initial metal ion concentration on sorption capacity of the membrane

To determine the effect of initial metal ion concentration on sorption capacity of the membrane, different samples containing 50ml each of different metal ion concentrations ranging from 5 ppm to 100 ppm with each containing 0.2 g of the dried membrane was prepared and stirred for 4 hours at 25 °C. Thereafter, the synthetic waste water was filtered and analyzed for residual metal ion concentration.

2.9 Determination of effect of contact time on sorption capacity of the membrane

To determine the kinetics of the sorption for the metal ions, different set of samples consisting of 0.2 g of the dried sorbent and 50cm³ of the metal ion solution for each of the ions was prepared one after another, when the samples are steered using magnetic stirrer. These metal ions were at a time interval ranging from 2 hours to 24 hours removed and thereafter, the solution was filtered and analyzed for residual metal ion. This was done for all metal ions at 30 °C.

3. RESULTS AND DISCUSSIONS

Metal ion sorption capacity of the membrane (Na-Alg/MOL-g-PAAm)

The equilibrium sorption capacity of the membrane for Zn⁺² and Fe⁺² were 95.7 % and 93.9 % respectively as demonstrated in figure 1. The difference observed in the sorption capacity for all the metal ions could be as a result of inherent surface chemistry (functional groups like, -OH, -COOH etc), pore structure and species specific compositions (Rodrigo et al., 2023).

Pore structure and surface area; the variation in sorption of the different metal ions could be the leaf structure, particle size and activation methods influence pore size distribution (micro, meso, macropores) and total surface area affecting how easily metal ions reaches binding sites. In essence any factor changing the leaf's fundamental chemistry, structure or accessibility of binding site will alter its metal ion sorption capacity (kesinee et al., 2022).

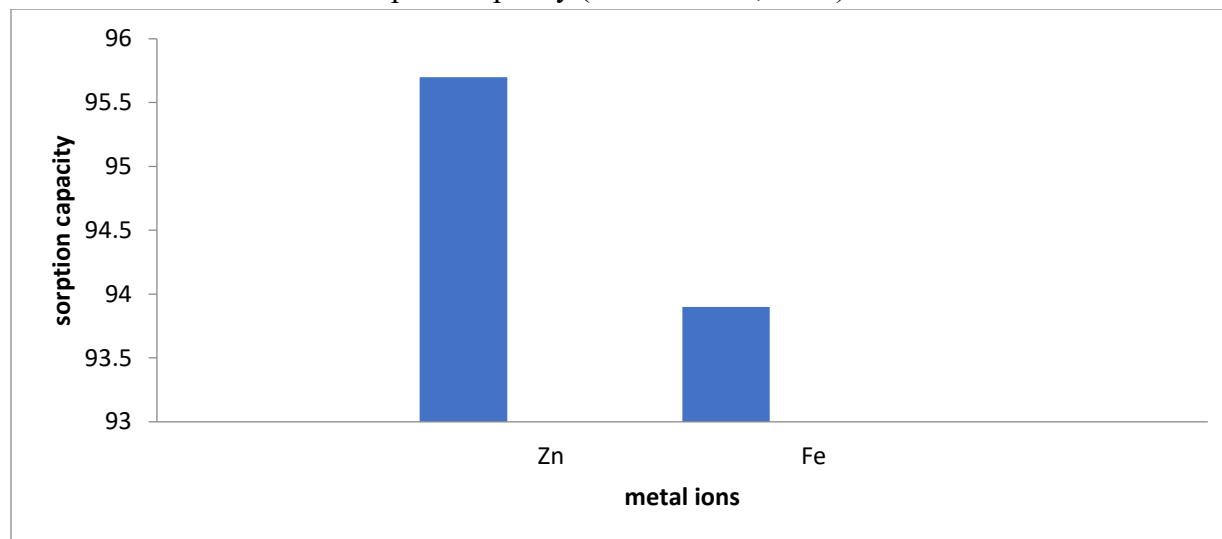


Figure 1: Metal ion sorption capacity of membrane

Effect of pH on sorption capacity of membrane

The pH of the sorption media projects the nature of the physicochemical interaction of both the ions in the solution and the nature of sorption site of the biomass under study (Andresa et al., 2021). Figure 2 shows the effect of pH on sorption capacity of the membrane.

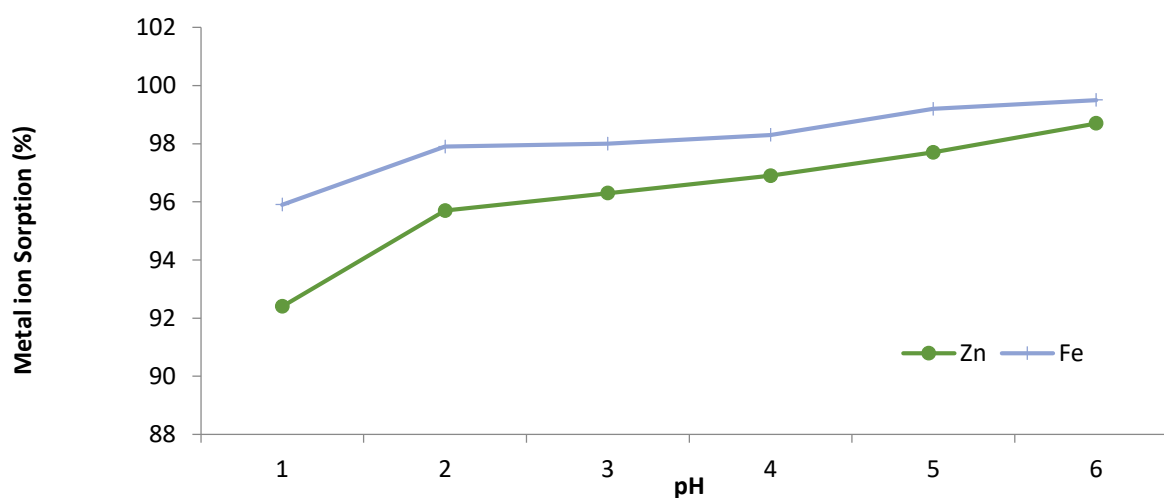


Figure 2: Effect of pH on sorption capacity of the membrane

It was observed that sorption capacity of the membrane increase with increase in pH of the solution. From the figure, a rapid increase was observed in sorption capacity from pH 1 to pH 6 having more metal ions adsorbs at higher pH and lesser metal ion at lower pH. The result is in agreement with other similar work like (Tonni, et al., 2022), (Naef et al., 2021), etc.

pH alters the equilibrium of metal ions uptake in aqueous solution by affecting the speciation of the metal ions in the solution, chemistry of the active binding site on the sorbent and the concentration of hydrogen ions in the solution, also pH changes both the adsorbent and the adsorbate (substance being adsorbed) (Valentin et al; 2017). Low pH (Acidic): adsorbent surfaces becomes positively charged due to protonation of functional group like $-\text{COOH}$, $-\text{OH}$ repelling positive ions but attracting negatively charged ions. High pH (Basic): surface becomes negatively charged (deprotonation), strongly attracting positive metal ions (cations) but repelling negative ions (Dawid and Piotr, 2022).

Effect of ionic strength on sorption capacity of the membrane

Fresh water and waste water often contains dissolved salt which compete with metal ions for binding site. Figure 3 below shows the activities of the membrane to adsorb metal ions in the presence of different concentration of normal salt (NaCl). It was observed that sorption capacity of the membrane decreases with increase in ionic strength, this scenario could be as a result of; Electrostatic shield/competition (decreases sorption): high ionic strength introduces more ions, creating a stronger electrical double layer (EDL) or screening effect around charged surfaces. These ions compete with the target metal ions (sorbate) for binding site, reducing electrostatic attraction and lowering sorption capacity of charged metal ions (Telma et al., 2019).

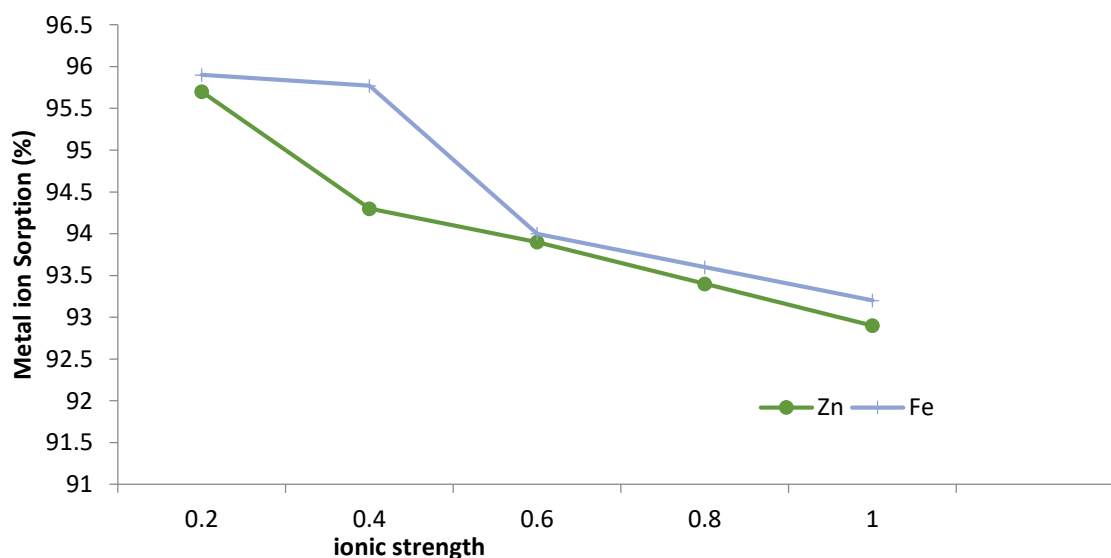


Figure 3: effect of ionic strength on sorption capacity of the membrane

Effect of temperature on sorption capacity of the membrane

The kinetics of sorption for Zn^{+2} and Fe^{+2} on the membrane was illustrated in figure 4. It was observed that there was gradual decrease in metal ion uptake by the membrane within 25 oC which

extended to 35 °C and significant drop in metal ion uptake from 45 °C to 50 °C, where we observed a kind of equilibrium for Fe²⁺, also a gradual decrease in metal ion uptake by the membrane was observed from 35 °C to 45 °C before the equilibrium was attained for Zn²⁺. These variations in sorption capacity in term of temperature could be explained by the following factors; (a) the sorption process may be physical adsorption (Xin Yang, et al., 2011). Physical adsorptions are normally exothermic; hence the degree of adsorption decreases with increasing temperature (b) inherent molecular structure (stable atomic arrangements) of the polymer made with the membrane which alters the properties of the membrane prepared from it (Seishi and Nobuyuki, 2021), (c) contraction of the membrane molecules with increase in temperature to give a more compacted form of membrane which causes the membrane pore to be narrowed and consequently hide some sorption site making them inaccessible to the metal ions (Nkwopara and Hu, 2018) and (d) bond strength; increase heat can weaken non crosslinked polymer causing them to dissolve (Lech et al., 2020).

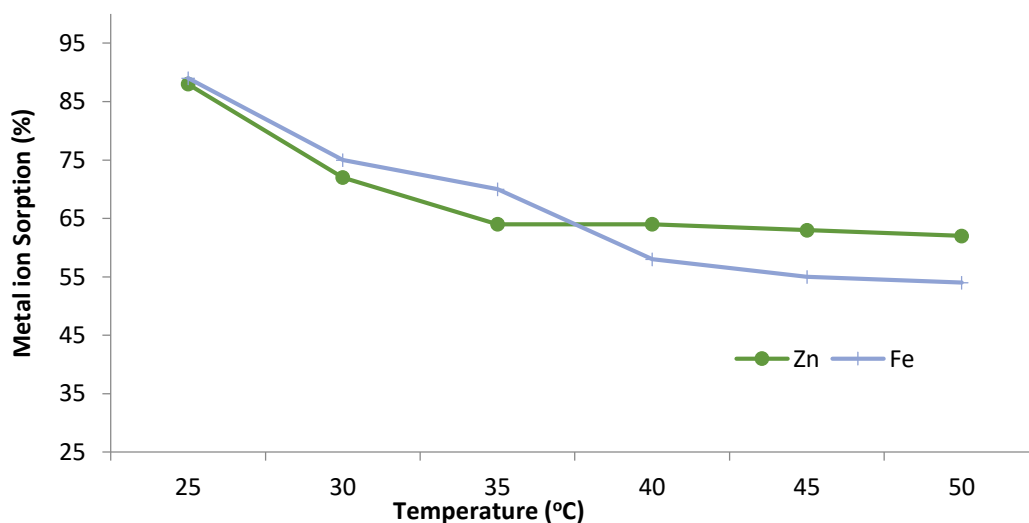


Figure 4: Effect of temperature on sorption capacity of the membrane

Effect of initial metal ion concentration on the sorption capacity of the membrane

The result of the metal ion sorption by the membrane as a function of initial metal ion concentration is shown in figure 5. It can be observed that the metal ion by (Na-Alg/MOL-g-PAAm) membrane increases with increase in initial metal ion concentration. This behavior can be explained in terms of increased driving force (concentration gradient) pushing more metal ions onto the adsorbent's available sites, overcoming repulsive forces and enhancing interactions, even as the percentage removal decreases because, sites become saturated, leading to plateaus in total capacity as observed on the graph between 50 ppm and 100 ppm perhaps. Chemically, this involves the Langmuir isotherms model where more ions fill more sites until saturation and mass transfer (film and intraparticle diffusion) steps becomes more crucial, with higher concentrations facilitating faster diffusion and overcoming energy barriers (Xiaofeng et al., 2020).

This result is a positive development especially in terms of the economy. This rapid uptake of metal ions by the prepared membrane could also be explained by the porous structure of the

membrane which accelerated metal ion diffusion to the functional groups. The flux of a cation varies in direct proportion with the metal ion concentration and hence, there should be an increase in flux with an increase in metal ion concentration (Nanev et al., 2021).

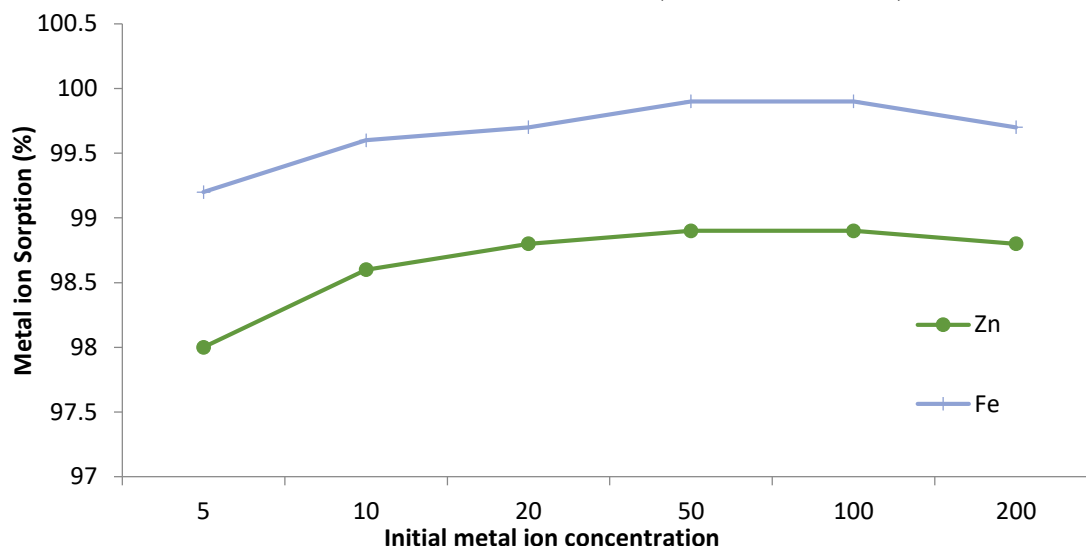


Figure 5: Effect of initial metal ion on sorption capacity of the membrane

Effect of contact time on the sorption capacity of the membrane

The result of contact time on metal ion adsorption by the membrane is illustrated in figure 6. From the graph, the sorption capacity of the membrane increased rapidly from two hours and thereafter equilibrium was reached for all the metal ions studied. A rapid increase in sorption capacity was observed within the first two hours and equilibrium was observed for the two metal ions studied. Generally, we deduced that the membrane sorption capacity increase with increasing contact time.

Contact time is a critical factor in adsorption, where removal rate is initially rapid due to numerous available active sites, then gradually slows as sites become occupied finally reaching equilibrium where maximum adsorption is achieved. Longer contact time generally increases adsorption capacity until the adsorbent becomes saturated (Mahmoud 2023). Therefore effect of contact time on (Na-Alg/MOL-g-PAAm) sorption capacity is summarized as follows. Rapid initial rate: The adsorption rate is fast initially because of a high concentration of active surface sites and high concentration gradient between the sorbate and the sorbent. Equilibrium approach: As the sites becomes occupied, the rate of removal slows down and eventually reaches the equilibrium (highest metal ions uptake), that is a state where the adsorption and desorption rates are equal. Optimal time: determines the ideal contact time that is essential in order to maximize the waste water remediation efficiency at a minimized cost (Eka and Desy, 2021).

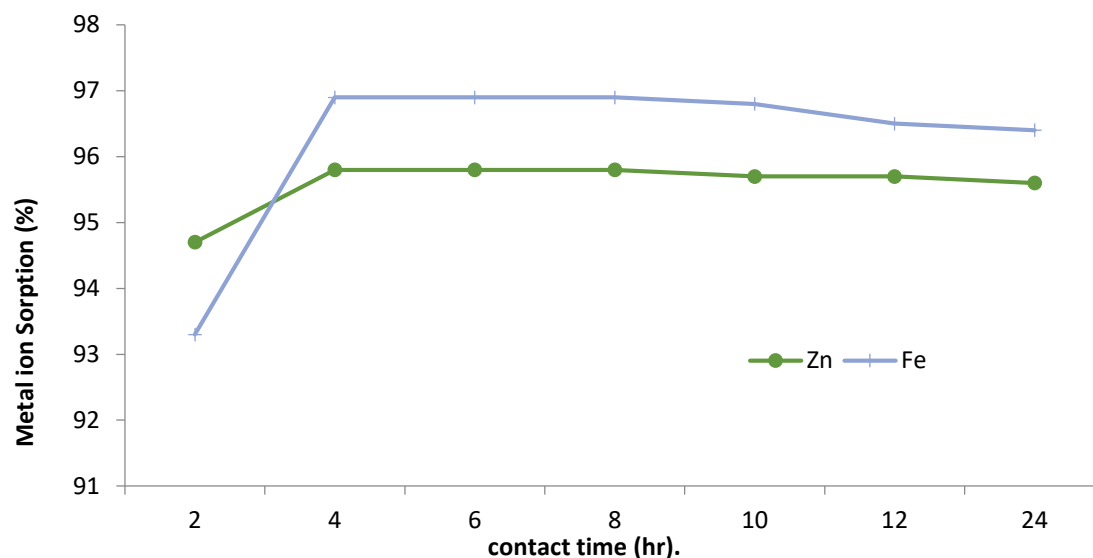


Figure 6: effect of contact time on sorption capacity of the membrane

3.1 Adsorption Isotherm Analysis of Iron II Ions

The equilibrium adsorption of iron(II) ions, Fe^{2+} , was evaluated using the Langmuir and Freundlich isotherm models. The analysis relates the equilibrium concentration, C_e , to the amount adsorbed per unit mass of adsorbent, q_e . Numerical estimates below are provisional because the original plots and regression outputs were not supplied.

3.2 Langmuir isotherm analysis

The Langmuir model assumes a homogeneous surface with a finite number of equivalent adsorption sites, monolayer coverage and no interaction between adsorbed species. These are model assumptions rather than properties demonstrated by the graph alone.

The linearized Langmuir equation is:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_{max}} + \frac{C_e}{q_{max}} \quad (1)$$

Here, q_{max} is the maximum monolayer adsorption capacity and K_L is the Langmuir affinity constant.

For a fitted line with slope m_L and intercept b_L :

$$m_L = \frac{1}{q_{max}} \quad ; \quad b_L = \frac{1}{K_L q_{max}} \quad (2)$$

$$q_{max} = \frac{1}{m_L} \quad ; \quad K_L = \frac{m_L}{b_L} \quad (3)$$

The reported approximate slope of 0.007 to 0.008 corresponds to $q_{\max}$ of approximately 125 to 142.86 mg/g, provided that C_e and q_e are expressed in mg/L and mg/g, respectively. The final value should be calculated from the fitted regression equation.

The supplied intercept range, “0.003 to 0.00”, is incomplete and must be corrected before K_1 can be calculated reliably. A small positive intercept relative to the slope can yield a large K_1 ; the intercept alone does not establish strong affinity.

An initial steep increase followed by a plateau may indicate saturation in an untransformed q_e versus C_e plot. The linearized Langmuir plot, however, should be assessed for a straight-line relationship between C_e/q_e and C_e .

3.3 Freundlich isotherm analysis

The Freundlich model is an empirical expression used to describe adsorption on surfaces with heterogeneous adsorption energies. Its linearized equation is:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (4)$$

For a fitted line with slope m_F and intercept b_F :

$$m_F = \frac{1}{n} \quad ; \quad b_F = \log K_F \quad (5)$$

$$n = \frac{1}{m_F} \quad ; \quad K_F = 10^{b_F} \quad (6)$$

Equation (6) applies when base-10 logarithms are used. For natural logarithms, $K_F = \exp(b_F)$. The units of K_F depend on the concentration units and the fitted value of $1/n$. The reported FeF response ranges narrowly from approximately 1.689 to 1.700. If these are $\log_{10} q_e$ values, they suggest limited variation in equilibrium adsorption capacity across the concentration range examined. A base-10 intercept of 1.695 to 1.698 gives a numerical K_F value of approximately 49.55 to 49.89. A positive slope satisfying $0 < 1/n < 1$ is commonly interpreted as favourable adsorption. A slope near zero indicates weak concentration dependence, while an exactly zero slope does not yield a finite n . Consequently, a nearly flat plot does not independently demonstrate exceptionally favourable adsorption or multilayer coverage.

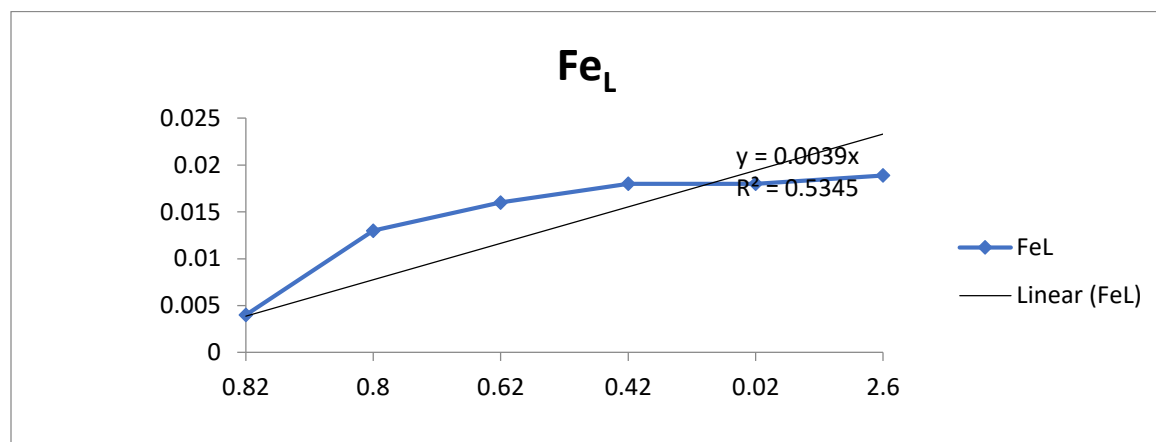


Figure 9: Langmuir adsorption plot for Fe (II) ion

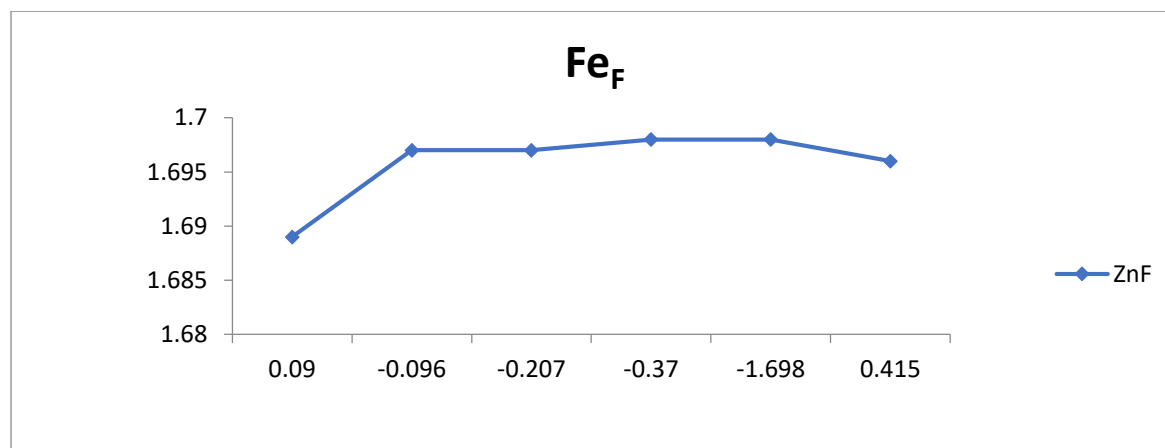


Figure 10: Freundlich adsorption plot for Fe (II) ion

Figure 9 presents the Langmuir adsorption plot for Fe^{2+} ions. In its linearized form, C_e/q_e is plotted against C_e , where C_e is the equilibrium concentration and q_e is the equilibrium adsorption capacity. The slope equals $1/q_{\text{max}}$, while the intercept equals $1/K_L q_{\text{max}}$. The reported approximate slope of 0.007 to 0.008 corresponds to a maximum adsorption capacity of approximately 125 to 142.86 mg/g, provided the stated concentration and capacity units are correct. A satisfactory linear fit would indicate consistency with the Langmuir model, which assumes monolayer adsorption on equivalent sites. However, the incomplete intercept value prevents reliable calculation of K_L , and the model fit alone does not establish chemisorption.

Figure 10 presents the Freundlich adsorption plot for Fe^{2+} ions, expressed as $\log q_e$ against $\log C_e$. Its slope represents $1/n$, and its intercept represents $\log K_F$. The reported response varies narrowly between approximately 1.689 and 1.700, indicating that adsorption capacity changes only slightly across the concentration range examined. If the fitted intercept is 1.695 to 1.698 and base-10

logarithms were used, the corresponding K_F is approximately 49.55 to 49.89. Although a positive slope satisfying $0 < 1/n < 1$ is commonly associated with favourable adsorption, a nearly zero slope requires careful interpretation. It does not independently confirm exceptionally favourable adsorption, multilayer coverage or physisorption; these conclusions require the actual regression results and additional experimental evidence.

Table 3.1: Comparative interpretation

Parameter	Langmuir model	Freundlich model
Surface description	Assumes homogeneous, equivalent adsorption sites	Represents heterogeneous adsorption energies
Capacity parameter	$q_{\max}$: finite monolayer capacity	K_F : empirical capacity-related constant
Linearized plot	C_e/q_e versus C_e	$\log q_e$ versus $\log C_e$
Slope	$1/q_{\max}$	$1/n$
Intercept	$1/(K_1 q_{\max})$	$\log K_F$
Supplied estimates	Capacity estimate depends on slope and confirmed units	Near-zero slope indicates limited concentration dependence
Fit assessment	Regression statistics and residual analysis	Regression statistics and residual analysis

The models should preferably be fitted to the original q_e data and compared using a common error measure, such as RMSE. Their intercepts cannot be compared directly because they represent different quantities.

4 SUMMARY

The reported Langmuir slope suggests a potentially substantial monolayer adsorption capacity, subject to confirmation of the regression equation and units. The nearly flat Freundlich plot indicates only a small change in adsorption capacity over the examined concentration range. The available estimates are insufficient to establish which model provides the better fit.

Fitting both models to the same dataset does not establish a dual adsorption mechanism. Langmuir behaviour should not automatically be classified as chemisorption, nor Freundlich behaviour as physisorption. Mechanistic conclusions require additional evidence, including surface characterization and adsorption energetics.

5. CONCLUSION

The study concludes that this material function as efficient biosorbent due to the rich content of functional groups like carboxyl and hydroxyl groups which facilitate metal binding through mechanisms such as ion exchange and chelation. Therefore, the research concluded finally, the membrane formed are promising green alternative to expensive conventional water treatment methods. Their abundance in tropical regions, ease of preparation (often requiring no complex treatment) and high regenerative capacity makes them especially suitable for large scale application and use in rural or developing areas facing water scarcity and pollution

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