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Valorisation of Copper Scrap Wire: RSM-Assisted Fabrication of Cu-Mg Zeolite Nanoparticles, Detailed Characterization, and Efficient SDS Surfactant Adsorption in Wastewater

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Abstract

In recent years, E-Waste has amassed as a result of the widespread use of electronic equipment for industry innovation. E-Waste recycling technologies have a low impact on the environment, are nontoxic, and are being promoted in the field of nanotechnology. This approach maintains environmental sustainability. This work aimed to synthesize Copper-Magnesium zeolite nanoparticles from discarded copper scrap wires. Acid leaching is a mechanism for synthesizing metal nanoparticles, which are then replaced by chemical components. The function of the Copper-Magnesium zeolite nanoparticles was studied utilizing FTIR, SEM, and XRD methods. In the current study, "Cu-MgZeNPs" was employed for treating aqueous solution to degrade SDS using Adsorption approach under varied parameters of time, pH, Initial SDS concentration, adsorbent dose, and temperature. Adsorption tests were carried out in a batch mode. The experimental findings revealed that the adsorption rate rose with time and adsorbent dose, but reduced with increasing SDS content and temperature. The highest rate of adsorption was reported at a pH of 7. The batch trials found a 30-minute equilibrium time. Kinetic and thermodynamic investigations were also conducted on the experimental data and adsorption studies are made using Response Surface methodology.

Keywords: E- Waste, Copper Scrap, Zeolites, Leaching, Adsorption, Surfactant Removal.

Introduction

Surfactant, also known as surface-active agent or "surfactant," is a heterogeneous and long chain molecule that contains both hydrophilic and hydrophobic moieties. Surfactant hydrophobic groups are often composed of long chain hydrocarbon residues. The hydrophilic group is either ionic or extremely polar. Anionic, cationic, non-ionic, amphoteric, zwitterionic, and gemini surfactants are some of the several varieties.

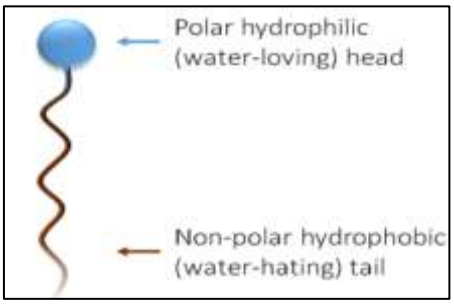


Fig. 1. Structure of Surfactant

Anionic surfactants are widely used in household cleaning products and personal care goods, as well as contemporary processes such as dry cleaning, electroplating, grease, emulsion polymerization, and paper production. Anionic surfactants are the most common kind of surfactant used in detergent formulations, accounting for approximately 59% of all surfactants in 1989. Surfactants are among the most extensively dispersed xenobiotics that can infiltrate wastewater and the aquatic environment (1,2,3). Detergents have an impact on aquatic habitats by generating foaming, reducing oxygen generation, promoting eutrophication, and posing a risk to drinkable water (4). Surfactants modify the quality of water once they have penetrated it (5). Due of these carcinogenic causes, eliminating surfactants from water flows has been a top concern for many studies.

Adsorption contacting systems for industrial and municipal wastewater treatment have gained popularity in recent years (6). Because of the high level of purification possible, an adsorption procedure is frequently utilized at the conclusion of a treatment sequence for pollution management. Because funds are an essential consideration in numerous poor nations, various approaches have been undertaken to investigate the viability of different economic adsorbing sorbents derived from E-waste.

According to the World Health Organization, improving access to clean drinking water, sanitation and hygiene, and wastewater management might avert 9% of the worldwide disease burden (7). Excessive detergent concentrations in natural waterways may result in decreased oxygen levels, a change in water colour, increased turbidity and sedimentation, and a decrease in biological activity (8).

1.1 Sodium Dodecyl Sulphate (SDS):

The majority of detergent compositions use anionic surfactants. According to Scott and Jones (2000), branch alkyl benzene sulphonate (ABS) is the most common type of anionic surfactants since the 1960s. The majority of detergent compositions use anionic surfactants. According to Scott and Jones (2000), branch alkyl benzene sulphonate (ABS) is the most common type of anionic surfactants since the 1960s. However, the branched alkyl chain of ABS was shown to be resistant to biodegradation and produced issues in receiving water bodies. This pushed manufacturers to conduct more research into altering surfactants to make them more biodegradable. Linear Alkyl Benzene Sulphate (LAS) was one of the products presented as being more biodegradable and having less of an environmental impact. Other commonly used are Alkyl Sulphate (AS) and Alkyl Ether Sulphate (AES). The surfactant used in this study is **Sodium dodecyl Sulphate (SDS)**. SDS belongs to the AS group and the structure is depicted as shown in Figure 1.2.

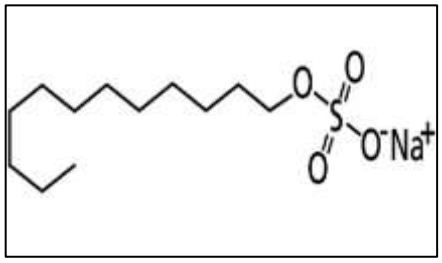


Fig. 2. Sodium dodecyl Sulphate

SDS is frequently used in bubble baths, shampoos, toothpastes, and detergents. This may be obtained at the greatest purity level. Furthermore, SDS is frequently utilized as a reference surfactant for evaluating different surfactants in a variety of chemical and physicochemical investigations and measurements. SDS may increase chemical absorption through the skin, gastric mucosa, and other mucous membranes. Importantly, it is also employed in transdermal, nasal, and ocular drug delivery systems to improve the intestinal absorption of poorly absorbed medicines, and it is currently commonly used in electrophoretic biochemical research.

SDS is found in the environment primarily through its presence in complicated residential and industrial effluents, as well as direct release from some applications (e.g., oil dispersants and insecticides). According to reports, SDS is toxic and has an impact on the life of mammals and aquatic species such as fish, as well as microorganisms such as yeasts and bacteria. Textile and organic synthesis manufacturing plants and enterprises may discharge up to 2.5 to 10 g/L of AS (9). Reducing SDS exposure can protect both present and future generations' health. Membrane filtration, coagulation and electrocoagulation, adsorption, and oxidation are some examples of removal processes. In this study, we utilized adsorption, which is a common approach for removing SDS from water and wastewater with an efficacy of 98-99%.

1.2 Cu Nanoparticles

Nanoscale copper particles are copper metal particles that are smaller than a micrometre. They are extremely reactive due to their vast surface area. In the presence of oxygen and water, it is even faster than in the bulk material. They rapidly oxidize, releasing free copper ions. They are often employed in medical and laboratory settings, and they have also been researched for the cleanup of industrial areas polluted with chlorinated organic chemicals. This feature restricts its application to inert conditions (11,12,13). Copper nanoparticles are not harmful. They, like MgO and NiO NPs, are super paramagnets. There are two main types of forms. Because of their superparamagnetic properties, they are widely used in biological imaging, heavy electrical, catalysis, and important chemical processes.

1.3 OBJECTIVES OF THE PRESENT WORK

- This study intended for synthesis of copper nanoparticles from the electronic waste (EWaste) by implementing the acid leaching technique.
- The source of E-waste for the present study is unused copper windings around the electric motor.
- The extracted copper is then supported by addition of magnesium nitrate and zeolite. This component is nonlethal and biodegradable.
- The functional modification of Cu-MgZe nanoparticles by composting with commercial adsorbents such as Cu-MgZe nanoparticle for the removal of SDS in solution.
- The characterization of the Cu-MgZe nanoparticles synthesis and adsorbents are studied using various analytical techniques such as FTIR, SEM and XRD techniques.
- This investigation deals with sorbent adsorbing studies on the effect of time, pH, sorbent quantity, initial concentration and temperature.
- Study of kinetics, isotherms and thermodynamics will be carried out to find the adsorption rate, adsorption capacity and feasibility.
- Adsorption studies are made using Response Surface Methodology.

MATERIALS

3.1 Materials Required: Copper scrap wires were gathered from the mechanical workshop in the regional location. Magnesium Nitrate, Zeolite, Nitric Acid, Acridine orange, NaOH, Glacial Acetic Acid, Sodium dodecyl sulphate, Toluene.

3.2 Glassware: 1 lit beaker, 500ml Volumetric flask, Measuring jar, Pipette, conical flasks etc.,

3.3 Equipment's: pH meter, UV Spectrophotometer and Glass Ware etc.

3.4 Estimation of SDS:

***3.4.1 Preparation of SDS**:*

1000 ppm stock solution of SDS was taken by mixing 0.1g of SDS tiny granules in water and diluting to the mark in a 100ml volumetric flask with distilled water.

3.4.2 Preparation of 18% HNO_3:

18ml of HNO_3 & 82ml of distilled water in 100ml conical flask for the preparation of preparation of 18% HNO_3 .

3.5 Preparation of Cu-MgZe nanoparticles using copper scrap wires:

Unused Copper wires which are used around the electric motors were collected from the regional work shop. The collected wires were then reduced to very much smaller size in order to increase the surface to volume ratio (s/v) so it would increase the yield of acid leaching (14). Then the small pieces of wire are taken into a glassware and they are used for the further process.

3.6.1 Synthesis of Cu-MgZeNPs:

The procedure is explained by the following steps:

Step 1- Firstly,100gm of small pieces of copper wires is weighed with the help of measuring balance. and taken into a medium size glassware.

Step 2- Now, 25ml of 18% HNO_3 prepared solution is added into the glassware and said to leave for 2hrs at room temperature and Ph 3 for the acid leaching to take place effectively.

Step 3- After 2hrs it is observed that the leaching process is said to be completed and copper nitrate solution is obtained.

Step 4- The above steps are repeated until 100 ml of copper nitrate has been collected.

Step 5- Then to the obtained copper nitrate solution 10gm of magnesium nitrate and 3gm of zeolite powder are added.

Step 6-The obtained solution pH is maintained at 8 by slowly adding drops of NaOH.

Step 7-Then the slurry is stirred with the help of magnetic stirrer at 80°C by maintaining constant pH for 2hrs.

Step 8- Then the obtained mix is cooled, filtered and dried overnight at 110°C.

3.6.2. Flow diagram for Production of Cu-MgZeNPs:

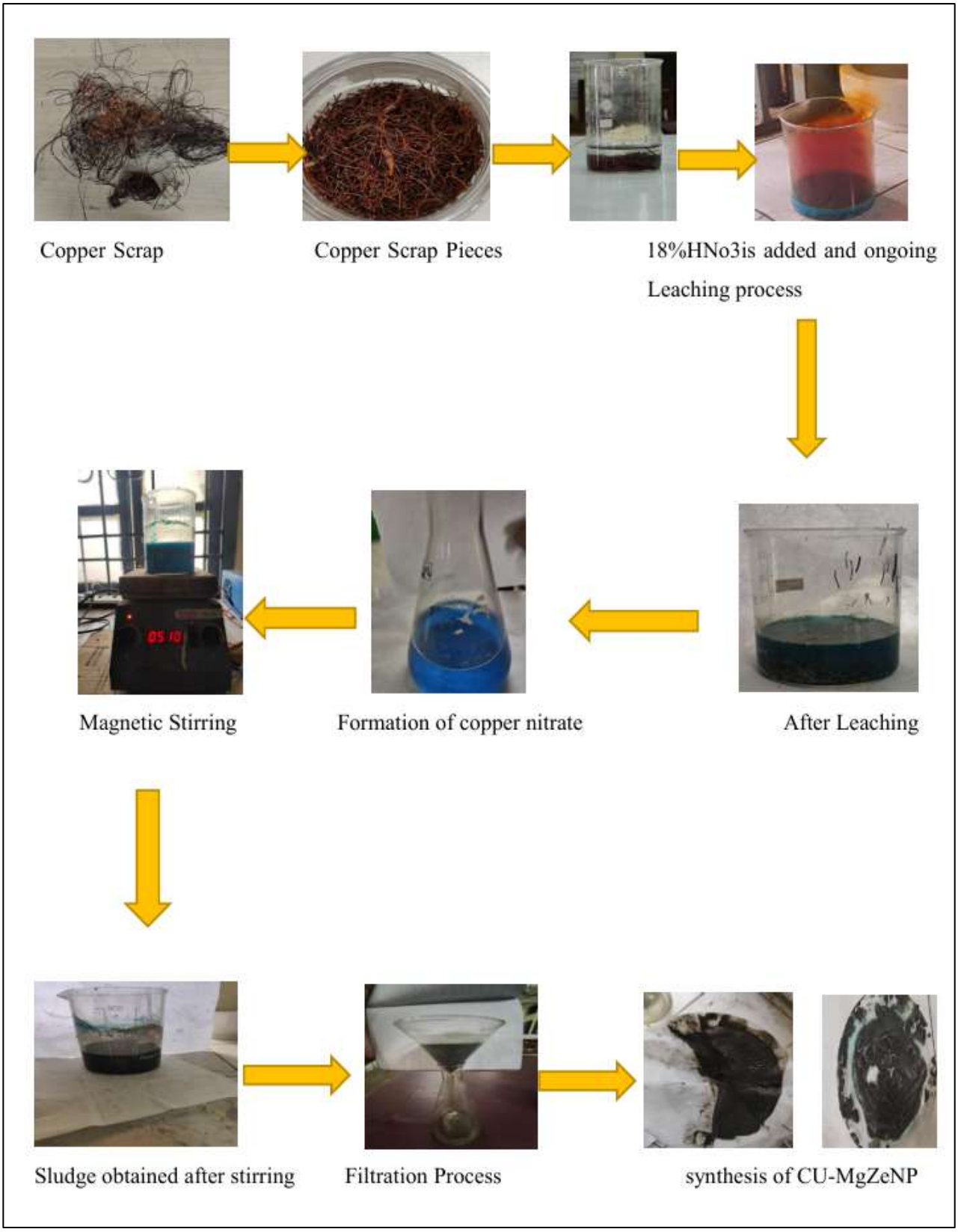


Fig. 3. Detailed Flow diagram for synthesizing Cu-MgZeNPs

3.6.3 Determination of SDS:

SDS is determined by colour using UV Spectroscopy.10ml Sample of Prepared SDS solution is taken to which 100µl of Acridine Orange and Glacial acetic acid are added. To which 5ml of Toluene is added as a solvent and then the mixture is shake for a minute and then introduced into the separating funnel. Initially the mixture is of colourless after giving a sufficient amount of time for separation in the funnel yellow colour extract layer is seen. Then the absorbance is directly measured from U-V spectroscopy at 467nm.

3.7. Characterization of CU-MgZeNP adsorbent:

3.7.1 Scanning Electron Microscope (SEM):

The SEM (Scanning Electron Microscope) picture in Figure 4 clearly shows the tiny structures and various sizes of Cu-MgZeNPs. It offers excellent resolution and steady probe currents for optimal imaging.

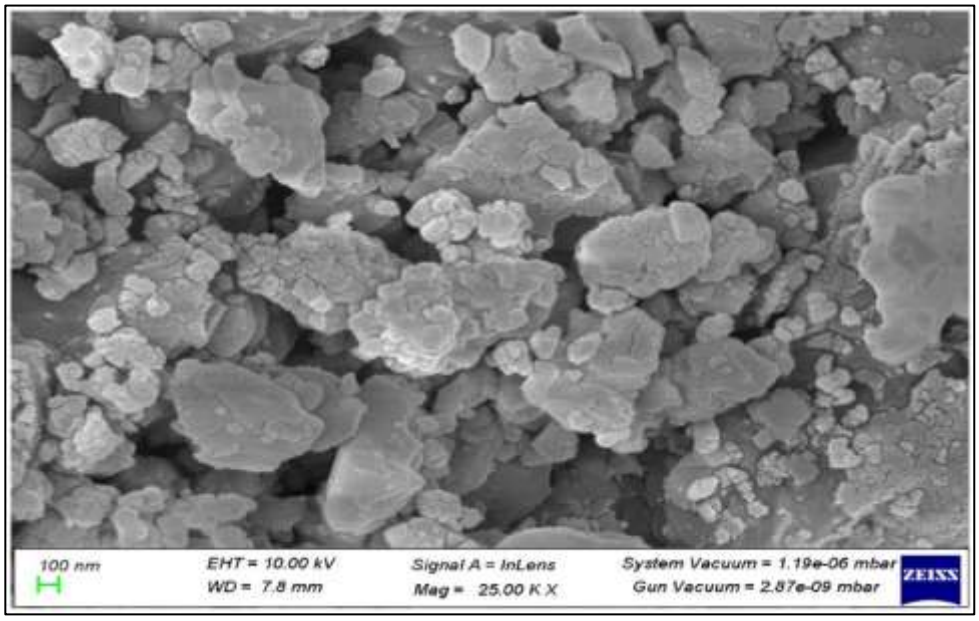


Fig. 4. SEM Image of Cu-MgZeNPs

3.7.2 X-Ray Diffraction (XRD):

The peak positions in Figure 5 show the creation of a spherical crystal structure. The peaks at 2q values of Cu-MgZeNPs are 32.21°, 39.03°, and 48.27° with intensities of 17.91, 59.33, and 19.62, respectively.

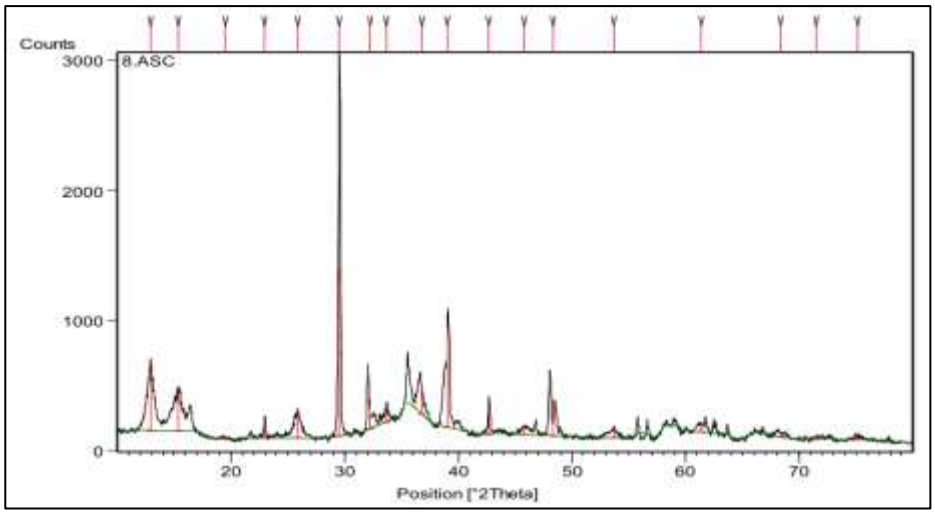


Fig. 5. XRD Image of Cu-MgZeNPs

3.RESULTS AND DISCUSSION

The experimental data is obtained batch by batch to report the influence of varied factors such as time, sorbent quantity, pH, initial concentration, and temperature on the degradation of SDS from a laboratory-prepared SDS solution including Cu-MgZeNPs as an adsorbent. Kinetics, adsorption isotherms, and thermodynamics were all investigated.

3.1 Characterization studies of Cu-MgZeNPs by FTIR:

The results revealed a large number of peaks, which provided information on the complicated structural material. Figure 6 shows the FTIR spectrums of Cu-MgZeNPs before and after analysis. The peaks at various places correspond to distinct functional categories. The spectra obtained for Cu-MgZeNPs after adsorption exhibited appearance of peaks at 427cm-1, 1403cm-1, and 2856cm-1, which correspond to -OH groups stretching in alcohols. The spectral peaks at 1632.10 cm-1 correspond to C=C stretching, and the chemical class is alkene. Other peaks were identified at 1543cm-1, with a medium look that can be attributed to C-O stretching and a compound class of tertiary alcohol.

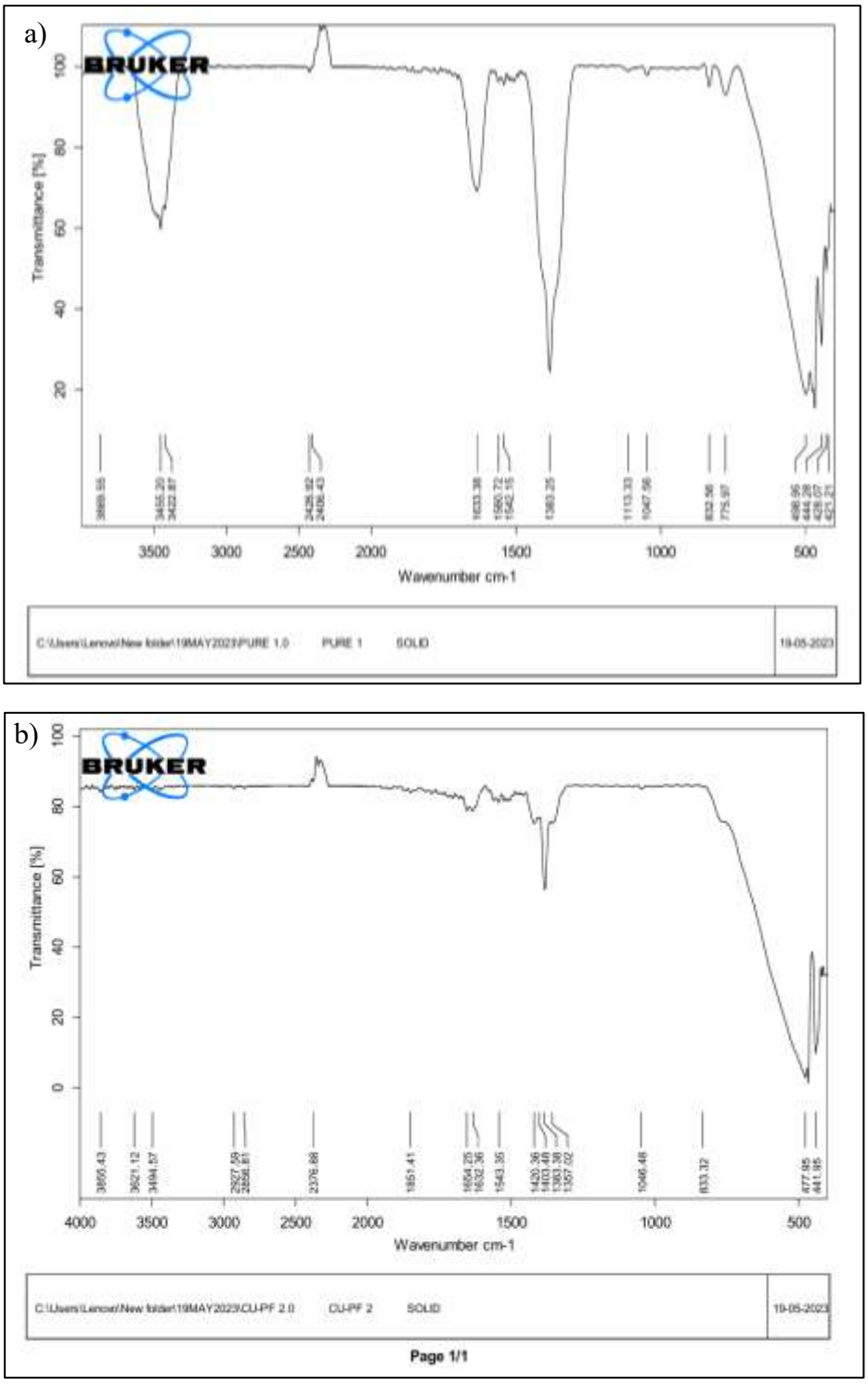


Fig. 6. a) FTIR (Before) Image of Cu-MgZeNPs;
b) FTIR (Before) Image of Cu-MgZeNPs;

3.2 Thermo gravimetric analysis:

Thermogravimetric analysis was performed on Mg-promoted Cu nano-adsorbent, and TGA curves are shown in Fig. 7. The TGA curves of Mg-promoted Cu nano-adsorbent showed three distinct weight loss temperature areas. The first weight loss up to roughly 200°C suggested

water desorption from the Mg-promoted Cu nano-adsorbent. The second weight drop was detected at roughly 200°C, with a tail at 450°C. This weight loss was caused by the breakdown of Cu and Mg nitrate precursors on the surface of the zeolite support. A third weight loss was seen in the higher temperature range above 550 °C, which might be attributable to the degradation of the hydroxyl group in the zeolite support.

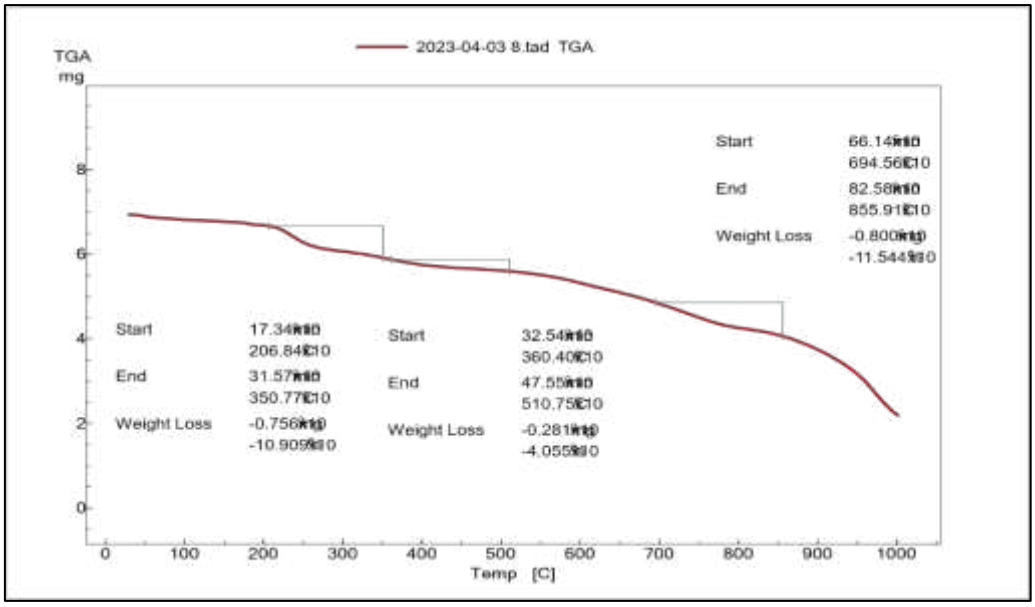


Fig. 7. TGA Image of Cu-MgZeNPs

3.3 Experimental Parameters:

3.3.1 Effect of Time:

Figure 8 depicts time course profiles for the elimination of SDS solution over 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 55, and 60 minutes. The results acquired from the% Removal of SDS onto utilized Cu-MgZeNPs revealed that a contact period of 30 minutes was necessary to attain significant constant time, with no due difference with further increase. For future tests of adsorption with different parameters using CU-MgZeNP as an adsorbing agent, an equilibrium time of 30 minutes was used.

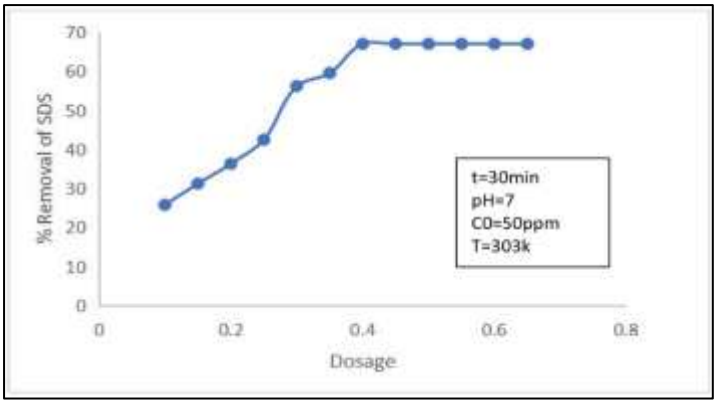


Fig. 8. Effect of Time

3.3.2 Effect of Adsorbent Dosage:

To investigate the effects of adsorbent dose on SDS elimination. The adsorbent dose varies from 0.1 to 0.6g while the other parameters remain constant. The contact period with Cu-MgZeNPs was 30 minutes. Figure 9 indicates that the increase in SDS removal with adsorbent dose from 0.1 to 0.65g and this justified removal is because of high availability of surface area and accessible sorption sites.

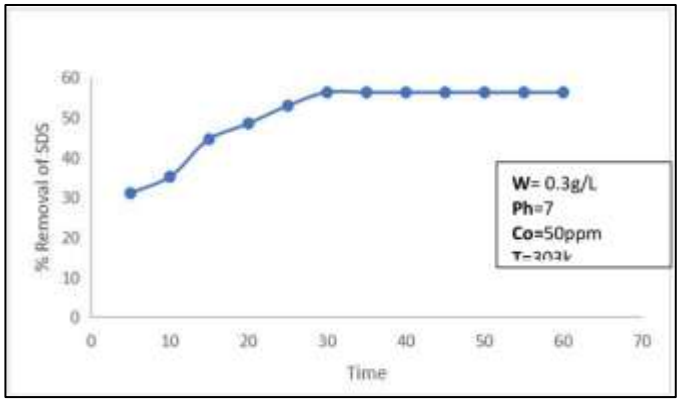


Fig. 9. Effect of Adsorbent Dosage

3.3.3 Effect of pH:

Figure 10 shows the effect of initial pH on the % Removal of SDS using CU-MgZeNPs. The percentage of SDS adsorbing had rosen with an increase of pH from 5 to 9 and there onwards the values decreased with increase in pH up to 12. Hence optimum pH was taken as 9. At lower pH both the hydrogen ions as well as SDS compete together resulting in less adsorption.

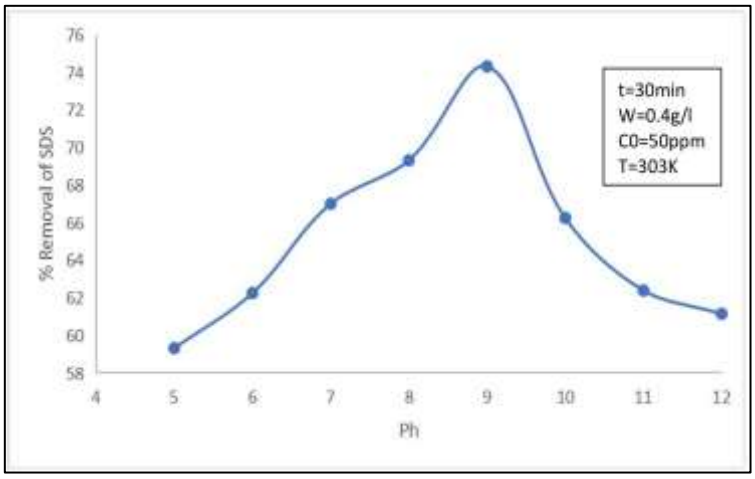


Fig. 10. Effect of pH

3.3.4 Effect of Initial concentration:

Fig. 11 evidently explains that the adsorption capacity rose as the starting concentration of SDS increased, however the % removal of SDS. At lower concentrations, all SDS in the solution could interact with the binding sites, thus therefore concentration is fixed at 10 ppm for further experimentation.

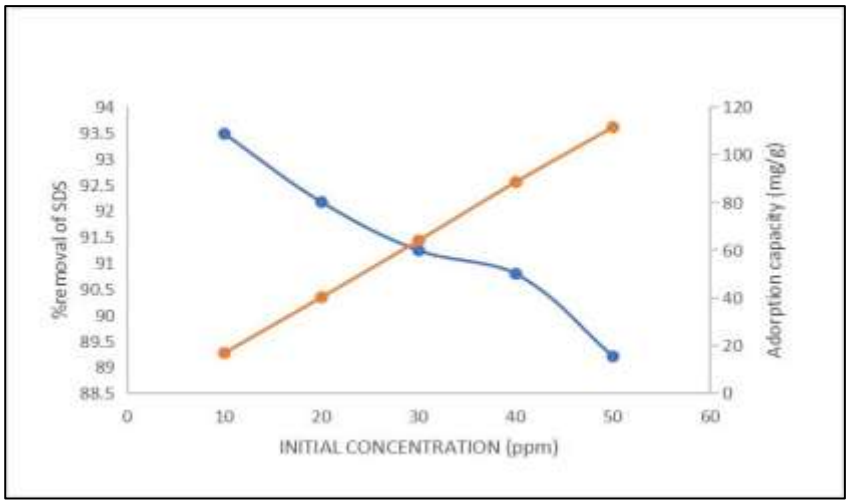


Fig. 11. Effect of Initial Concentration of SDS

3.3.5 Effect of Temperature:

Batch studies were conducted at five constant temperatures: 308, 313, 318, 323, and 328K, to study the influence of temperature. Figure 12 shows that as temperature increased, the percentage of removal increased from 86.45% to 93.48% and 7 for an initial concentration of 10ppm. This suggests that the adsorption mechanism is endothermic in nature.

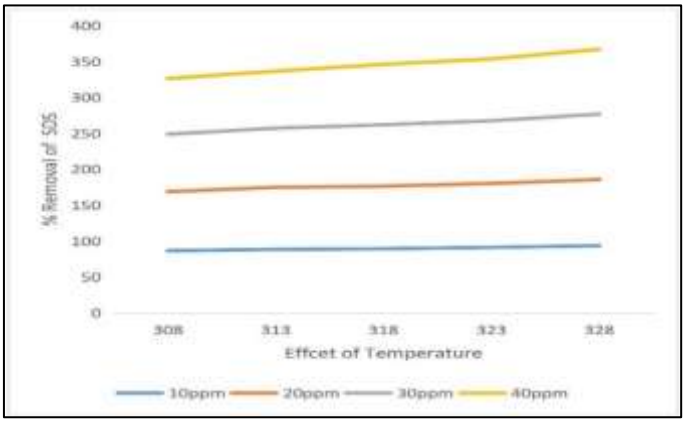


Fig. 12. Effect of Temperature

3.4 Adsorption kinetics:

3.4.1 Pseudo-first order model:

3.4.1 Pseudo-first order kinetic model-

$$\ln(q_e - q) = -K_1 t + \ln q_e \qquad (1)$$

A plot is drawn between $\ln(q_e\text{-}q)$ and t, which is used to examine the pseudo-first order rate constant k_1 and q_e.

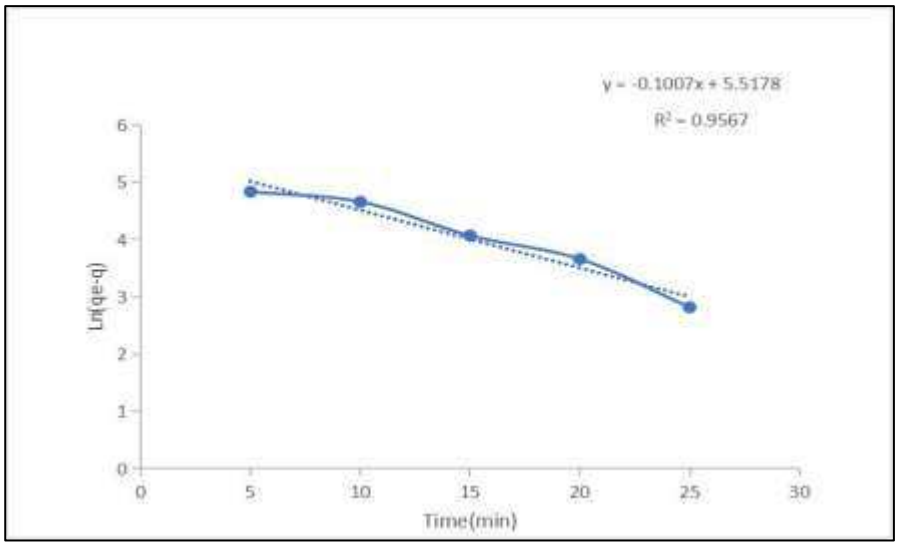


Fig. 13. Pseudo-First Order Kinetics

From Figure 13, Slope=-K_1=-0.2597

K_1=0.1007 min^{-1}

Intercept=lnq_e=5.5178

q_e=249.0865 mg/g

R^2 =0.9567

Therefore, we can conclude that pseudo-first order kinetic model is **not an appropriate fit** for this data.

3.4.2 Pseudo-second order kinetic model-

$$\frac{t}{q} = \left(\frac{1}{q_e}\right) t + \left(\frac{1}{K_2} . \frac{1}{q_e^2}\right) \qquad (2)$$

A plot is drawn between t/q and t, which is used to examine the pseudo-first order rate constant k_1 and q_e.

From Figure 14, Slope=$\frac{1}{q_e}$=0.003

q_e= 333.3333 mg/g

Intercept=$\frac{1}{K_2}$ q_e^2=0.0216

K_2=0.0514 g/(mg.min)

R^2 =0.9771

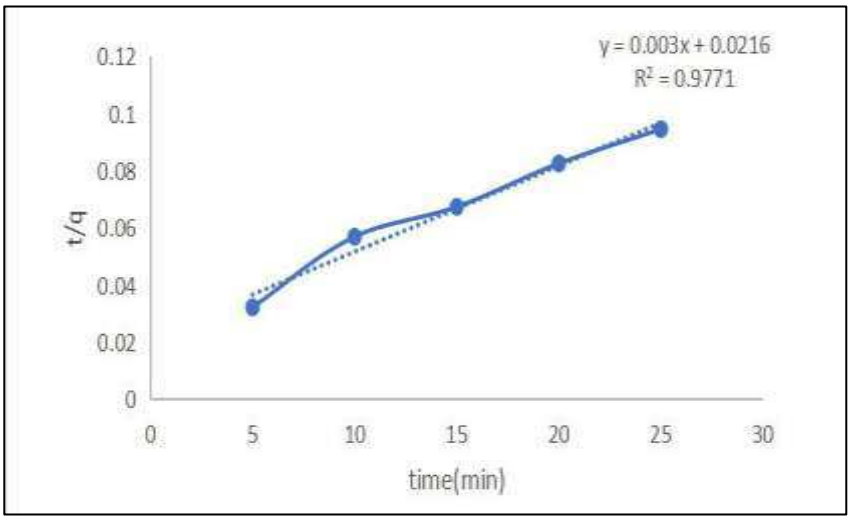


Fig. 14. Pseudo-Second Order Kinetics

Therefore, we can conclude that pseudo-second order kinetic model **is an appropriate model** for the obtained experimental data.

3.5 Adsorption Isotherm:

3.5.1 Freundlich isotherm:

The linear forms of the Freundlich isotherm can be expressed as shown in below.

$$\text{Log } q_e = \log K_f + {}^{1}_ \log C_e\, n) \qquad (3)$$

The adsorption coefficient (Kf) and intensity (n) were estimated as 5.24, 0.257 for sds solution. The obtained R^2 value is 0.9576 which confirms the adsorption process follows Freundlich isotherm are shown in figure 15.

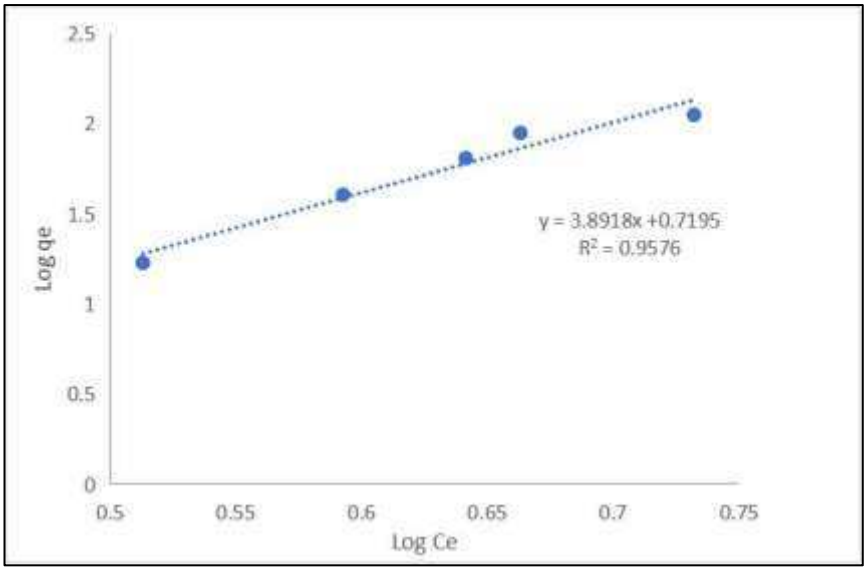


Fig. 15. Freundlich Isotherm

3.6 Thermodynamic studies:

The ΔH^o value is positive that means the adsorption is endothermic, an increase in temperature (T) result in an increase in equilibrium constant Kc .

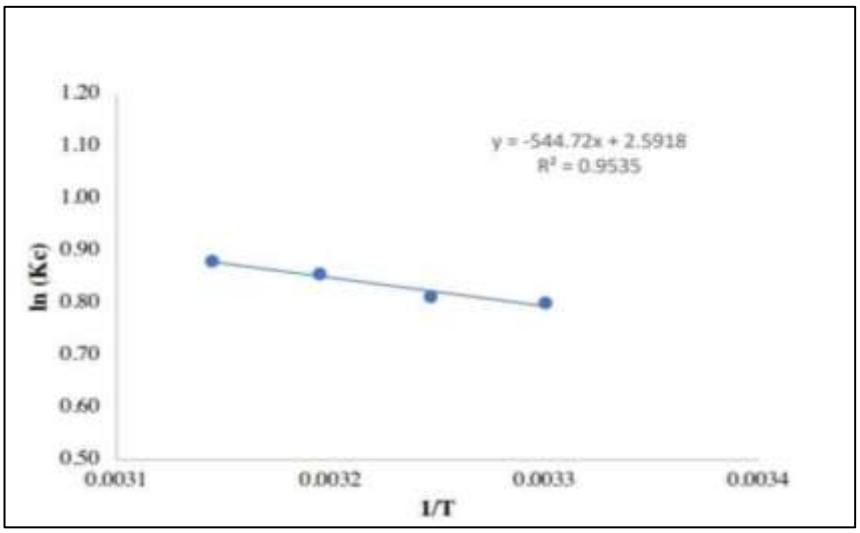


Fig. 16. Thermodynamics Plot

3.7 RESPONSE SURFACE METHODOLGY

Figure 17 demonstrated that the projected value of the model's responses corresponded to the actual values over the defined range of independent variables, with reasonably higher coefficients of determination (R^2). A subset of independent variables with reasonably high coefficients of determination (R^2).

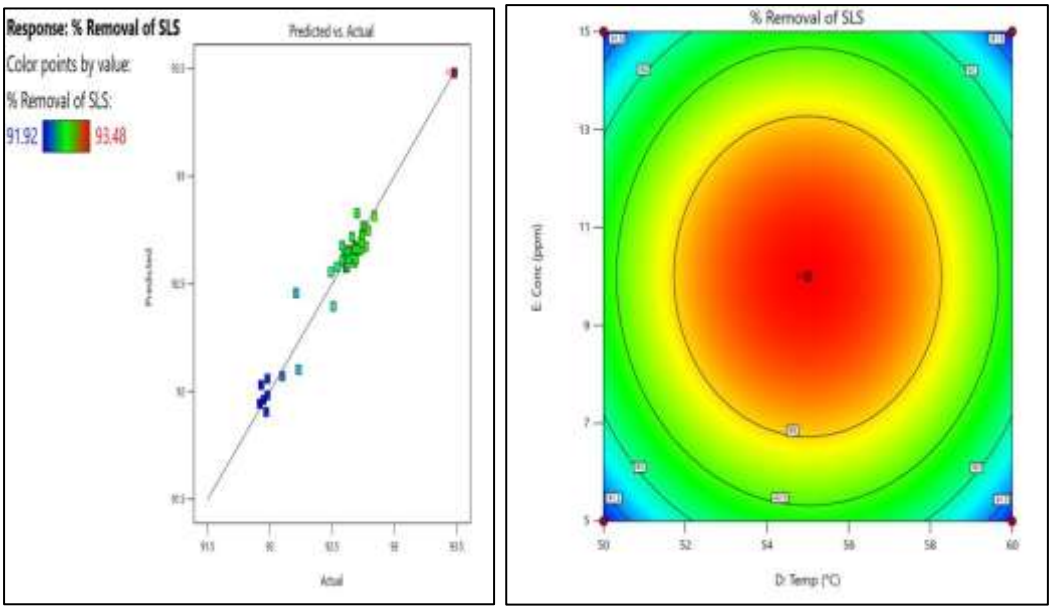


Fig. 17. Predicted value of the model's responses corresponded with the actual values

3.7.1 Effects of various parameters on the removal of SDS

Optimization

The desirability function was utilized to determine the optimum Cu-MgZeNP removal efficiency for SDS, as well as the optimal process variable settings. Figures below illustrate a desirability profile for SDS removal % vs variables. The desirability profile's desirability score recommends pH 7, dosage 0.4 g/L, and temperature 328 K as the optimal settings for each variable tested. Under these settings, SDS was removed at a 91% rate, as demonstrated experimentally. The model has an R^2 value of 0.9942 for SDS removal.

The effects of various parameters are shown in given graphs below-

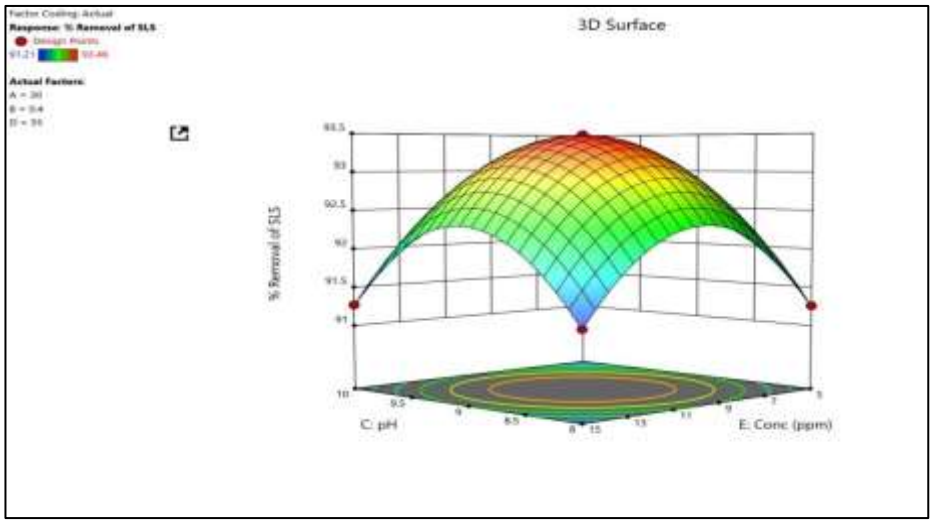


Fig. 17. Effect of pH and concentration on % removal of SDS

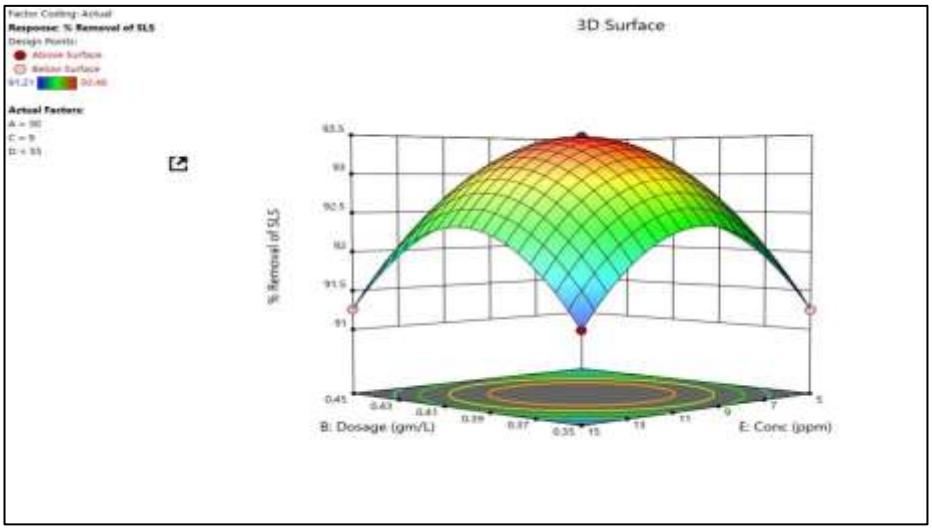


Fig. 18. Effect of pH and Temperature on % removal of SDS

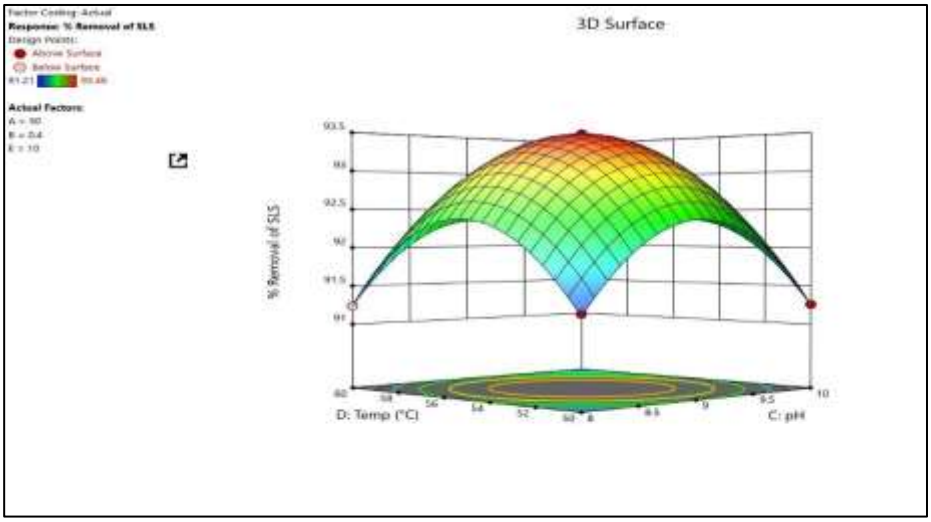


Fig. 19. Effect of dosage and concentration on % removal of SDS

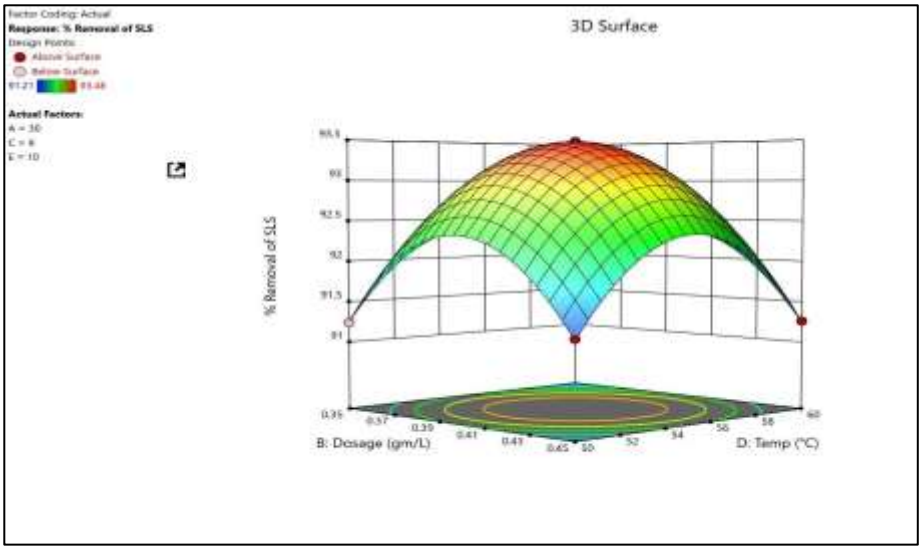


Fig. 20. Effect of dosage and temperature on % removal of SDS

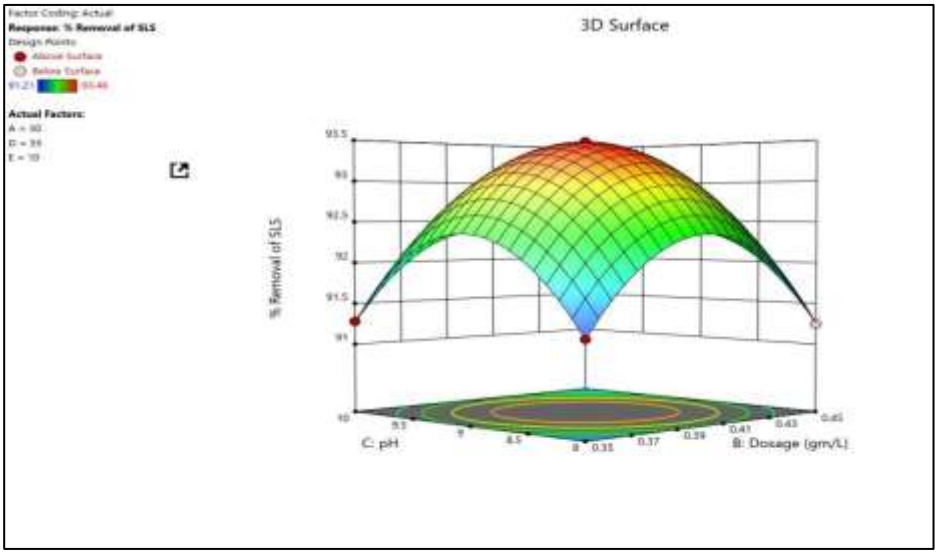


Fig. 21. Effect of dosage and pH on % removal of SDS

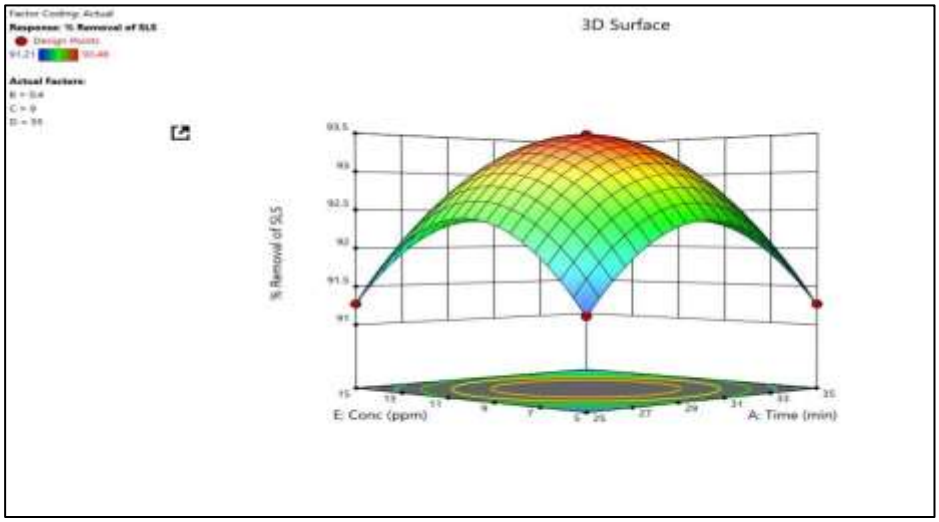


Fig. 22. Effect of conc and time on % removal of SDS

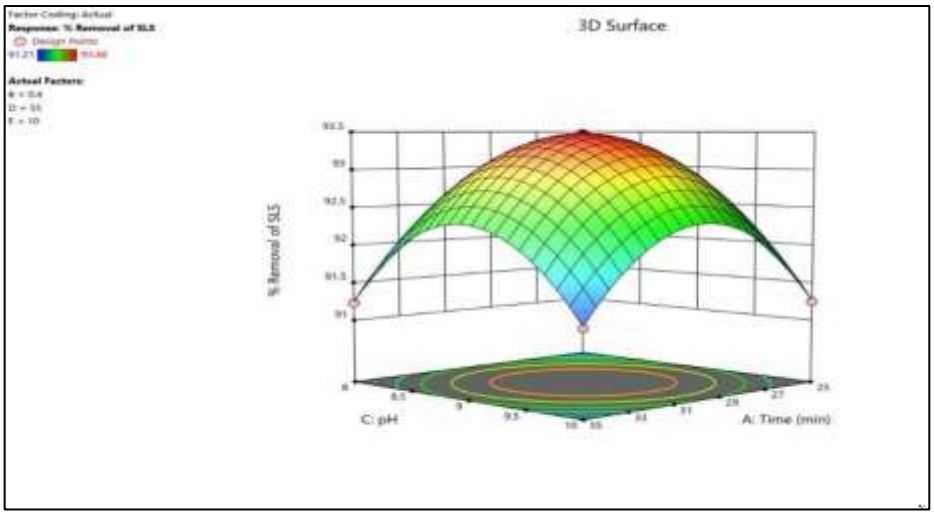


Fig. 23. Effect of time and pH on % removal of SDS

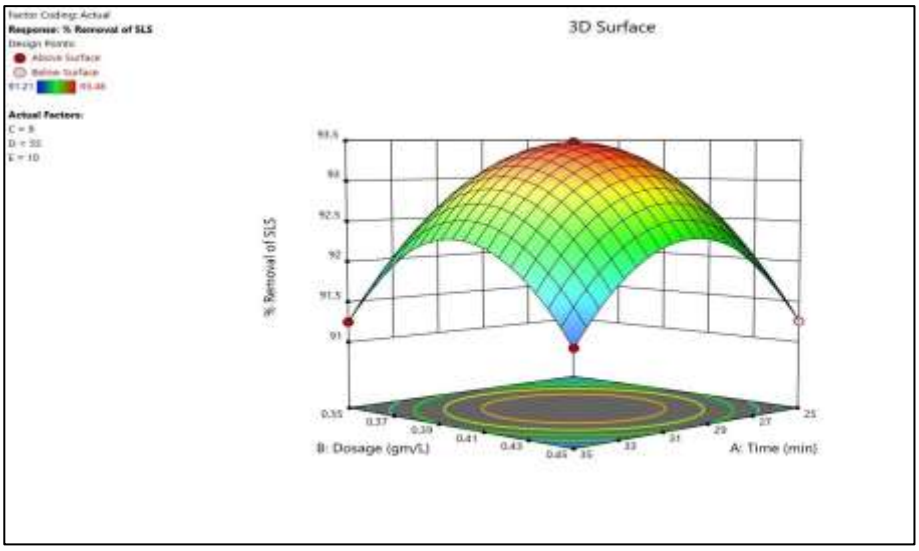


Fig. 24. Effect of time and dosage on % removal of SDS

4. CONCLUSIONS

To produce Cu-MgZeNPs, an ***acid leaching technique*** is used with copper scrap wires and hydrochloric acid to remove SDS. The Cu-MgZeNP nanoparticles were studied ***by SEM, XRD, and FTIR*** investigation.

- Cu-MgZeNPs effectively removed ***SDS (90.20%) under optimal circumstances of duration (30min), dose (0.4g/L), pH (9), starting concentration (40ppm), and temperature (328 K).*** Overall, Cu-MgZeNPs performed well.
- The adsorption kinetics of pseudo first and pseudo second order models were studied. The regression coefficient for pseudo first order kinetics of Cu-MgZeNPs is 0.9567. The regression coefficients for pseudo second order kinetics of Cu-MgZeNPs are 0.9771, respectively. ***Pseudo-second-order kinetics is the optimal modification*** for SDS elimination. The ***Equilibrium adsorption capacity qe(exp) is 333.33mg/g.***
- The ***Freundlich isotherm models*** for SDS were shown to be the ***best fit*** for Cu-MgZeNPs experiments. The Freundlich model best fits the SDS removal data, with an adsorption coefficient of $K_f = 5.24$ ***and an*** R^2 ***value of 0.9576.***
- Cu-MgZeNPs have ***thermodynamic values of 4.53kJ/mol and 21.54J/mol.K***, respectively. The positive ΔH^0 ***value of 4.53 l*** indicates endothermic adsorption. As temperature (T) increases, so does the equilibrium constant (Kc). The ***entropy change of 21.54 mol.K*** indicates reduced randomness at the solid solution interface and no adsorbate.
- RSM analysis is conducted to optimize parameters such as time, pH, concentration, temperature, and dosage. ***Rsm analysis determines SDS removal at a rate of 91.67%.***

As a result, Copper Magnesium Zeolite Nanoparticles synthesized from copper scrap wire (Cu-MgZeNPs) operate as a suitable adsorbent because they have strong adsorbing capabilities, which were investigated in the current study, namely the adsorption of SDS surfactant removal.

CONFLICT OF INTERESTS

The authors confirm that there are no financial or personal relationships that could have influenced the findings of this study.

AUTHOR CONTRIBUTIONS

Yagna Sri Thikkada, NagaRaju Yendluri: Conceptualization, Investigation, Methodology, Data collection, Formal Analysis, Writing – Original Draft. **Shimeles Niguse Abate:** Supervision, Methodology, Validation, Writing – Review & Editing. **Pulipati King**:

Supervision, Methodology, Validation, Writing – Review & Editing. **Meena Vangalapati:** Supervision, Methodology, Resources, Writing – Review & Editing. **Akhil Arava:** Research resources, Financial support.

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