Evaluation of Modified and Unmodified Eggshells for the Removal of Excess Nitrates in Drinking Water

Kgopotso Makofane[1], Tholiso Lebelo[1], and Mabel Mphahlele-Makgwane[1]

***Abstract*— Water crisis in rural communities persists due to limited access to safe drinking water, leading residents to rely on nitrate-contaminated sources such as rivers and unprotected wells. Nitrate pollution mainly results from agricultural fertilizers, septic systems, poor sanitation, and natural nitrogen leaching. This study investigated the use of waste-derived eggshells as a low-cost, eco-friendly adsorbent for nitrate removal from drinking water. Eggshells were calcined to enhance their adsorption performance, and batch experiments were conducted to assess the effects of contact time, adsorbent dosage, concentration, and pH. Characterization using FTIR and SEM confirmed structural improvements after calcination. Optimal removal occurred at 90 minutes, with a dosage of 0.1 g, a concentration of 30 mg/L, and a pH of 12. Calcined eggshells (CES) achieved a superior nitrate removal efficiency of 98.92% and an adsorption capacity of 16.32 mg/g compared to raw eggshells (RES). CES effectively reduced nitrate levels in borehole water to meet SANS 241-1:2015 drinking water standards, demonstrating their potential as a sustainable and affordable adsorbent for improving water quality in rural areas like Ga-Makanye.**

***Keywords*—Adsorption, Calcination, Eggshells, Nitrate Removal**

I. INTRODUCTION

Access to clean and safe drinking water is essential for human health. However, approximately 2.2 billion people globally still lack reliable access to potable water [1]. Increasing urbanization and population growth have intensified global water demand, projected to rise by 55%, and contributed to widespread water stress [2]. Water scarcity and pollution are closely linked, as pollution reduces the availability of freshwater and increases treatment costs. Water pollution, defined as any changes in water quality that affect human and ecological health, mainly originates from agricultural runoff, wastewater discharges, and other effluents containing nitrates and phosphates.

Nitrate (NO_3^-), a form of nitrogen, is vital for biological processes but becomes problematic when it accumulates in water sources. Overapplication of nitrogen-based fertilizers in agriculture often leads to leaching of unabsorbed nitrates into groundwater [3].

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In addition to natural sources such as atmospheric deposition, major contributors to nitrate contamination result largely from anthropogenic activities, including fertilizer misapplication, septic tank leakage, wastewater discharge, and poor sanitation practices [4].

Excess nitrates in surface and groundwater cause eutrophication, biodiversity loss, and serious health effects such as methemoglobinemia (blue-baby syndrome) in infants, gastrointestinal disorders, and thyroid or stomach cancers in adults [5]. To mitigate these risks, various treatment methods, including reverse osmosis, ion exchange, oxidation, ultrafiltration and electrodialysis, have been developed to remove excess nitrates in water; however, adsorption stands out as a reliable method as it is effective, affordable and environmentally friendly [6].

Agricultural waste-based biosorbents such as coconut shells, rice husks, sugarcane bagasse, and eggshells have shown high potential for nitrate removal. Eggshells, composed mainly of approximately 98% calcium carbonate ($CaCO_3$), possess a highly porous structure with functional groups that are suitable for adsorbing contaminants like heavy metals, surfactants, and pharmaceuticals [7]. In a study conducted by Abatan et al. (2020), an FTIR spectrum indicated that Cr(VI) and Cd(II) ions were adsorbed through interactions with the hydrogen and oxygen atoms of the O–H and R–COOH functional groups.

The utilization of agricultural waste materials, such as eggshells, in adsorption-based treatment technologies promotes waste reduction by repurposing shells rather than disposing of them. This practice helps reduce the burden on landfills, aligning with the United Nations Sustainable Development Goal (SDG) 12, which promotes responsible consumption and production to mitigate environmental degradation. This study also supports SDG 6, which advocates clean water and sanitation, as well as SDG 3, which emphasizes good health and well-being [8].

According to WHO and EU guidelines, nitrate levels in drinking water should not exceed 50 mg/L [9], while the U.S. EPA and South African National Standards (SANS 241) limit nitrate-nitrogen to 10 mg/L and ≤11 mg/L, respectively. This study, therefore, aims to utilize waste-derived eggshells as an environmentally friendly, cost-effective adsorbent to reduce nitrate concentration in contaminated water to meet the established standards and protect environmental and human health.

II. MATERIALS AND METHODS

A. Sampling

Eggshells were collected in bulk from the University Cafeteria. Adsorption conditions were optimized using synthetic nitrate solutions prepared in the CDM laboratory

using Silver Nitrate ($AgNO_3$) salt. The water to be tested for nitrate removal was collected from a borehole in the Ga-Makanye community in Limpopo Province, South Africa, an area characterized by extensive use of septic tanks due to the absence of a centralized sewer system. This was done to assess the performance of the material under the optimized conditions for real-world applicability.

B. Preparation of Eggshell Biosorbent

The collected eggshells were washed three times with deionized water to remove residual suspended solids and organic matter. The clean eggshells were dried at 100 ^{0} C in a Carbolite Gero furnace for 24 hours to remove excess moisture. After drying, the eggshells were crushed using a blender and sieved to a particle size of 500 µm with a universal laboratory test sieve to obtain a powder with finer particles. Smaller particle sizes increase the surface area of the eggshells for adsorption.

C. Modification of Eggshells via Calcination

A mass of 100 g of the eggshell powder was placed in crucibles and heated at 900 °C for 1 hour using the Carbolite furnace to allow the decomposition of $CaCO_3$ into CaO. Following calcination, the powder was allowed to cool in a desiccator to prevent it from absorbing moisture from the surroundings. The eggshell powder was stored in labelled airtight containers to avoid contamination and to maintain stability.

D. Preparation of Synthetic Nitrate Solutions

Nitrate stock solutions (NO^-_{3aq}) of 1000 mg/L were prepared by dissolving 2.74 g of Silver Nitrate ($AgNO_3$) in 1000 ml distilled water in volumetric flasks. The solution was mixed on a magnetic stirrer to ensure the solute dissolved completely. The solution was then covered by a stopper and stored in a refrigerator to protect it from direct sunlight. This helps prevent decomposition and contamination and reduces the chances of chemical reactions occurring.

E. Preliminary Evaluation: Comparing the Performance of Both Modified and Unmodified Eggshells

Before proceeding with the main batch experiments, the efficiency of both the RES and calcined eggshells CES was tested using the synthetic nitrate solution under optimized conditions (contact time: 120 minutes; adsorbent dosage: 0.5 g; initial NO3- concentration: 100 mg/L; pH:6 and mixing speed: 150 rpm) obtained from a study conducted by AI-Agili (2023). This was done to confirm whether CES performs better than RES.

F. Batch Adsorption Experiments for Nitrate Removal by Modified Eggshells

Batch adsorption experiments were conducted to evaluate the nitrate removal efficiency under varying operational conditions. The effects of contact time, biosorbent dosage, initial nitrate concentration, and initial pH were investigated using a synthetic nitrate solution in six 100 mL beakers. The solutions were stirred at 200 rpm on a jar test stirrer. Contact time was varied from 0-180 minutes with a fixed dosage of 1 g and a concentration of 10 mg/L. After stirring, the solutions were allowed to settle, and the supernatant was obtained by filtering the water using a 0.45 µm membrane filter. The eggshell biosorbent was varied with different dosages of 0.05, 0.075, 0.1, 0.5, 1, and 1.5 g, and a fixed concentration of 10 mg/L. The solutions were stirred at an optimal contact time on the jar test stirrer. Initial nitrate concentrations were tested with 10, 30, 60, 90, 120, and 150 mg/L of the synthetic nitrate solution using the optimal biosorbent dosage. And finally, pH was adjusted to 2, 4, 6, 8, 10, and 12 using 0.1 M HCl and 0.1 M NaOH for the optimum nitrate concentration. The solutions were stirred at an optimum contact time with an optimum dosage. All experiments were carried out in triplicate at room temperature.

G. Characterization of Nitrate Water Samples

pH, EC, and TDS were measured using the HACH HQ40d multiparameter probe. Nitrate concentrations in the treated samples were measured using the Jenway 7205 UV-Vis spectrophotometer at 220nm.

H. Use of Optimized Conditions for Nitrate Removal from Ga-Makanye Borehole Water

The optimum conditions obtained from the batch adsorption experiments using modified eggshells were applied to nitrate removal in Ga-Makanye borehole water.

I. Calculating Percentage (%) Removal and Adsorption Capacity (q_e)

Nitrate removal efficiency (%) was calculated using equation 1 below:

$$\% \text{ Removal} = \frac{(Ci - Ct)}{Ci} \times 100 \quad \text{(1)}$$

where C_i, C_t, and C_e represent the nitrate concentrations (mg/L) at initial time, at time t, and at equilibrium, respectively. While the adsorption capacity of nitrate per gram of eggshell adsorbent (mg/g) was calculated using equation 2 below:

$$q_e = \frac{(Ci - Ce)V}{W} \quad \text{(2)}$$

where q_t and q_e denote the quantity of the adsorbed nitrate (mg/g) at a specific time *t* and at equilibrium, respectively. C_i, C_t, and C_e correspond to the nitrate concentrations (mg/L) at the initial stage, at time *t*, and at equilibrium. *V* denotes the solution volume (mL), while *W* indicates the weight of the adsorbent (g).

J. Characterization of Eggshells Before and After Treatment

The structural and surface properties of the RES, CES, and sludge obtained after nitrate adsorption were analyzed using Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscope (SEM).

Characterization using FTIR

FTIR spectra were obtained with an Alpha-Bruker spectrometer (Bruker, Germany) in the range of 4000–400 cm^{-1} at a 4 cm^{-1} resolution.

Characterization using SEM and SEM-EDS

SEM analysis was performed using a Zeiss Ultra Plus microscope to observe surface morphology, texture, and structural changes of the eggshell samples. Imaging was conducted at 3.00 kV and 7.00 K X magnification. Elemental composition was determined using Energy Dispersive X-ray Spectroscopy (SEM-EDS).

III. RESULTS AND DISCUSSIONS

A. Preliminary Evaluation: Comparing the Performance of both Modified and Unmodified Eggshells

Table I presents the comparison of nitrate removal efficiencies between raw eggshells and calcined eggshells using the synthetic nitrate solution under optimized adsorption conditions obtained from Al-Agili et al. (2023).

TABLE I: THE DIFFERENCE IN THE REMOVAL EFFICIENCY OF NITRATE BY BOTH RAW AND MODIFIED EGGSHELLS USING THE ADOPTED OPTIMIZED CONDITIONS

Raw eggshells (RES)	Calcined eggshells (CES)
70%	72%

To maintain consistency and comparability, the adsorption conditions optimized by Al-Agili in her study "Removal of Nitrate from Aqueous Solution by Bio-Calcium from Iraqi Eggshells" were adopted. Under these conditions, this study achieved 70% removal efficiency for raw eggshells, which is slightly lower than the 72% for calcined eggshells. Although these removal efficiencies are lower than those reported by Al-Agili et al. (2023), the adoption of her optimized parameters provided a solid benchmark, allowing this research to continue focusing on full batch adsorption experiments for calcined eggshells.

B. Physicochemical Characteristics of the Ga-Makanye Borehole Water

Table II presents the physicochemical characteristics of Ga-Makanye borehole water.

TABLE II: PHYSICO-CHEMICAL CHARACTERISTICS OF THE GA-MAKANYE BOREHOLE WATER

pH	EC (µS/cm)	TDS (mg/L)	NO_3^- conc. (mg/L)
6.86	73.07	47.50	16.5

The pH of the water was slightly acidic, which remained within the acceptable range for drinking water (5-9.7) as per SANS 241 standards, Volume 1. The adsorption of anions, such as NO_3^-, works best at pH values below 7. The EC was below the guideline of 1700 µS/cm, indicating that the water has a low ionic concentration and is of good quality in terms of dissolved ions. Similarly, the TDS was lower than the recommended guideline of 1200 mg/L, confirming the water's low mineral content and its suitability for human consumption. As a result, the limited presence of competing ions minimizes competition for adsorption sites on the biosorbent surface. However, the NO_3^- concentration exceeded the SANS 241 permissible limit of 11 mg/L, indicating nitrate pollution likely associated with septic tank leakage. This poses serious health risks, such as methemoglobinemia and cancers, to the people of Ga-Makanye.

C. Batch Adsorption Experiments

Evaluation Of Nitrate Adsorption onto Modified Eggshells in Relation to Contact Time

Fig. 1 shows the change in % removal and adsorption capacity of nitrate by modified eggshells in relation to contact time, varied from 0-180 minutes. Controlled variables: 10 mg/L nitrate concentration, 1-gram adsorbent dosage, 100 mL of solution, a speed of 200 rpm, and room temperature.

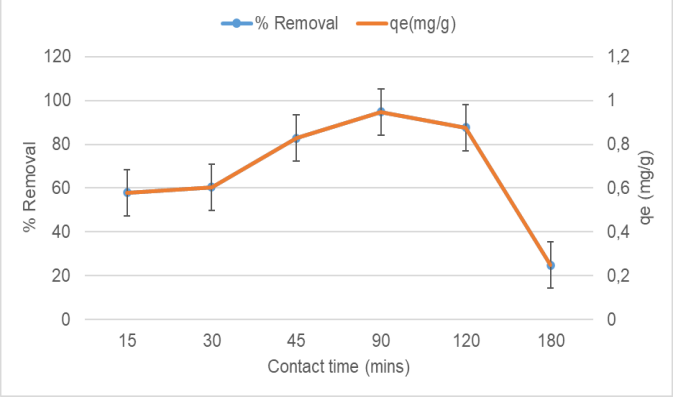

Fig 1: Change in % removal and adsorption capacity of nitrate by modified eggshells in relation to contact time.

Fig. 1 illustrates that the percentage removal of nitrate increased from the first minute of contact, and the same was observed with the adsorption capacity. The highest % removal of 94.675% was observed at 90 minutes with the highest adsorption of 0.95 mg/g. Beyond this point, both parameters declined, indicating equilibrium had been reached and that the biosorbent's active sites were saturated. However, in some cases, desorption of nitrate ions may have occurred. The release of nitrate ions back into the solution can be driven by the replacement with Ca^{2+} when the ionic strength of the solution changes due to the dissolution of $Ca(OH)_2$.

Another reason is the presence of competing ions such as chloride (Cl^-), sulfate (SO_4^{2-}), or bicarbonate (HCO_3^-), which can reduce nitrate retention, especially in multi-ion systems. This is common in real water samples. This can be concluded to say that an increase in contact time results in the desorption of nitrate ions. The ability for desorption from the biosorbent suggests the process is governed by physical adsorption (physisorption), involving weak Van der Waals interactions between the adsorbate and the adsorbent surface.

Evaluation of Nitrate Adsorption onto Modified Eggshells in Relation to Biosorbent Dosage

Fig. 2 shows the change in % removal and adsorption capacity of nitrate by modified eggshells in relation to biosorbent dosage, varied from 0.005-1.5 grams. Controlled

variables: 10 mg/L nitrate concentration, 90 minutes contact time, 100 mL of solution, a speed of 200 rpm, and room temperature.

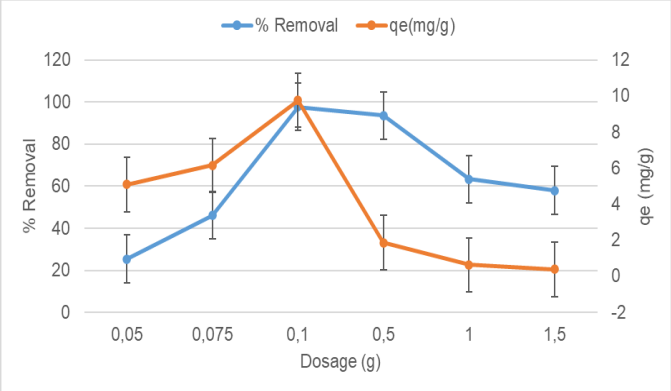

Fig. 2: Change in % removal and adsorption capacity of nitrate by modified eggshells in relation to dosage

The effect of biosorbent dosage on nitrate removal is an important parameter because it determines the amount of available active sites and influences the driving force for mass transfer from the solution to the adsorbent surface. Fig. 2 shows an increase in % nitrate removal and adsorption capacity with increasing dosage up to an optimum of 0.1 g, obtaining 93.5%, after which both declined at higher dosages. The initial increase in the two parameters with dosage can be attributed to the greater surface area and more active binding sites provided by higher biosorbent quantities. Therefore, more nitrates can be captured per unit volume of solution. However, beyond the optimum dosage, a decrease in performance was observed, likely due to system equilibrium, particle aggregation, and overlapping of adsorption sites, which reduce the effective surface area available for adsorption.

Evaluation of nitrate adsorption onto modified eggshells in relation to initial nitrate concentration

Fig. 3 shows the change in % removal and adsorption capacity of nitrate by modified eggshells in relation to initial nitrate concentration, varied from 10-150 mg/L. Controlled variables: 90 mins contact time, 0.1 dosage, 100 mL of solution, and a speed of 200 rpm at room temperature.

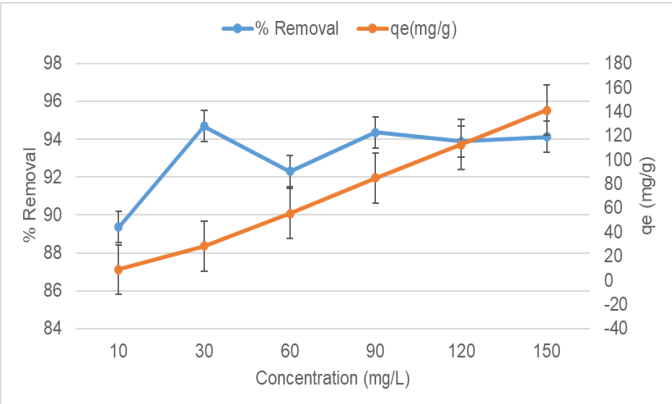

Fig. 3: Change in % removal and adsorption capacity of nitrate by modified eggshells in relation to initial nitrate concentration.

Fig. 3 illustrates the effect of initial nitrate concentration on the removal efficiency and adsorption capacity of modified eggshells. Nitrate removal increased with concentration up to 30 mg/L, where an optimum nitrate removal was achieved due to a balanced interaction between available active sites and the concentration gradient. However, as the concentration increased, there was an inverse relationship between nitrate concentration and % removal. This is because the number of nitrate ions competing for available adsorption sites had exceeded the number of active sites on the adsorbent surface, leading to a decline in the % removal efficiency. The increase in adsorption capacity with increasing concentration indicates that more nitrate ions were adsorbed per unit mass of adsorbent (mg/g), indicating a higher driving force of concentration gradient.

Evaluation of nitrate adsorption onto modified eggshells in relation to initial pH

Fig. 4 shows the change in % removal and adsorption capacity of nitrate by modified eggshells in relation to initial pH, varied from 2-12. Controlled variables: 90 mins contact time, 0.1 dosage, 30 mg/L concentration, 100 mL of solution, and a speed of 200 rpm at room temperature.

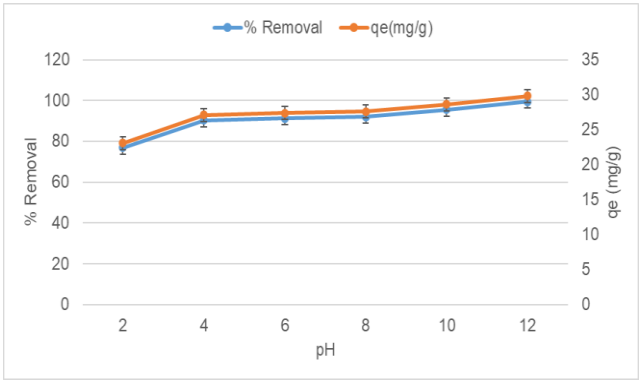

Fig. 4: Change in % removal and adsorption capacity of nitrate by modified eggshells in relation to initial pH

Fig. 4 shows the influence of initial pH on the % removal and adsorption capacity of nitrates by modified eggshells. Nitrate removal efficiency increased with increasing pH, achieving maximum values of 99.4% nitrate removal and 29.8 mg/g adsorption capacity at pH 12. This suggests that nitrate adsorption is highly pH dependent. The enhanced adsorption performance is due to the electrostatic attraction between the negatively charged nitrate ions and the positively charged active sites that form on the eggshell surface. At low pH, excess H^+ ions compete with nitrate ions for adsorption sites, reducing efficiency. As pH increases, this competition decreases, allowing more nitrate ions to be adsorbed.

As the pH increased towards alkaline conditions, the surface of the biosorbent became negatively charged due to deprotonation and the release of OH^- ions from CaO. However, the primary mechanism in alkaline conditions is not electrostatic attraction but rather chemical interaction and ion

exchange. Under these conditions, CaO reacts with water to form calcium hydroxide ($Ca(OH)_2$), which dissociates into Ca^{2+} and OH^- ions. The Ca^{2+} ions can react with nitrate to form calcium nitrate ($Ca(NO_3)_2$), enhancing nitrate removal efficiency. The increase in adsorption capacity at higher pH values also supports the conclusion that alkaline conditions enhance the chemical interaction between nitrate ions, Ca^{2+} ions, and the active sites on the modified eggshell surface.

D. Characterization of eggshells

Characterization using FTIR

Fig. 5 illustrates the FTIR spectra of raw eggshells (RES), calcined eggshells (CES), and eggshells after adsorption (post-treatment).

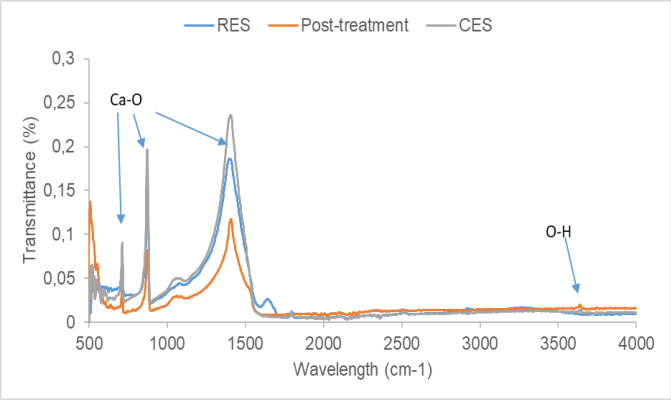

Fig. 5: FTIR spectra for RES, CES, and post-treatment eggshells

The spectra display the transmittance of functional groups at different wavenumbers, which helps to identify chemical bonds and structural changes associated with the modification and adsorption processes. The three samples have similar spectra, shown by the peaks on the graph. The sharp peaks demonstrated at 711 cm^{-1}, 875 cm^{-1}, and 1419 cm^{-1} indicate the breakdown of $CaCO_3$ into CaO as a result of calcination, as shown by the Ca-O stretches in the CES spectrum. This transformation increases the availability of active sites on the eggshell surface and the material's reactivity. The RES spectrum also shows a similar trend, indicated by the disappearing carbonate groups (CO_3^-) at 400-600 cm^{-1}. The peaks confirm that the major mineral composition of the eggshells is $CaCO_3$.

A new FTIR peak observed at 3654 cm^{-1} corresponds to the O–H stretching vibrations of hydroxyl groups (OH^-) in $Ca(OH)_2$, arising from moisture and adsorbed water on the eggshell surface after treatment. The O-H stretch bands indicate hydrogen-bonding interactions between surface hydroxyl groups and adsorbed nitrate ions, confirming successful adsorption. The formation of hydroxyl groups enhances electrostatic attraction and hydrogen bonding with nitrate ions, explaining the improved nitrate removal efficiency of calcined eggshells.

Characterization using SEM

Fig. 6 (a, b, and c), and Table III below present the SEM images and the elemental composition (SEM-EDX) of the eggshell samples, respectively. SEM was employed to examine the surface morphology, particle distribution, and structural changes that occur during calcination and after adsorption. It was then coupled with SEM-EDX to identify the elemental composition of the eggshells.

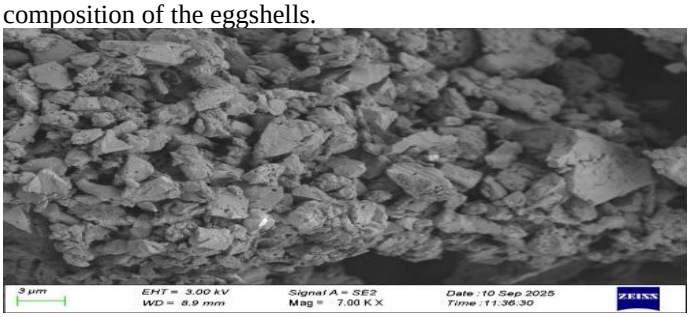

Fig. 6 (a): SEM image for RES

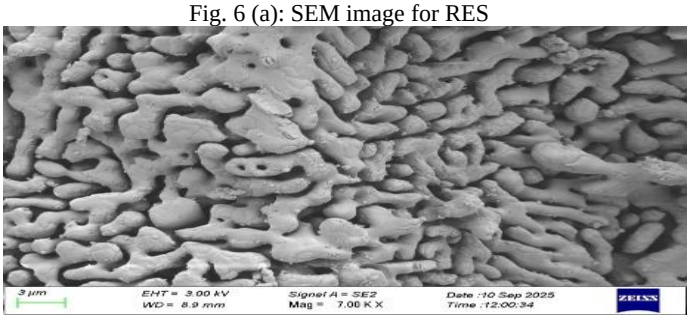

Fig. 6 (b): SEM image for CES

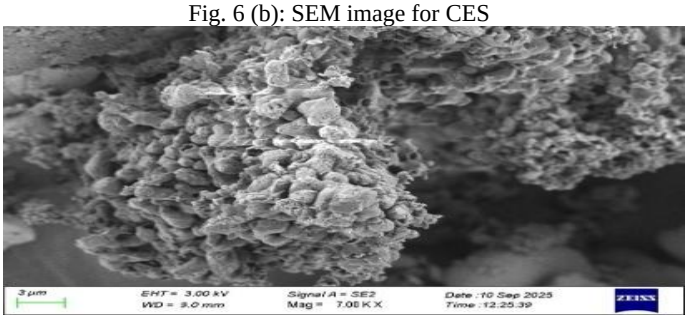

Fig. 6 (c): SEM image for eggshells after treatment

TABLE III: ELEMENTAL COMPOSITION (RES), (CES) AND EGGSHELLS AFTER TREATMENT

Element	% Composition		
	RES	CES	After treatment
Oxygen (O)	19	14	19
Calcium (Ca)	62	75	67
Gold (Au)	19	10	9
Silver (Ag)	-	-	5

The surface of the RES appeared slightly rougher with irregular shapes and sizes. This surface has fewer visible pores and limited high-surface-area features, which explains the lower initial adsorptive reactivity. Calcination produced a relatively rougher, more fractured, and porous surface. The cracking of the eggshells was caused by thermal decomposition of $CaCO_3$ to CaO, increasing surface area and exposing new active sites. After treatment, additional surface deposits and blocked pores indicated adsorption or precipitation of the adsorbate. Elemental analysis confirmed that both RES and CES were primarily composed of $CaCO_3$, as shown by the percentage of Ca in Table III. The traces of Ag (5%) detected post-treatment suggest ion competition resulting from the

synthetic nitrate solution, and Au originates from the coating used during SEM preparation.

E. Nitrate Removal from Ga-Makanye Borehole Water using both Modified and Unmodified Eggshells

Table IV shows the performance of RES and CES in removing nitrate from Ga-Makanye borehole water.

TABLE IV: RESULTS FOR NITRATE REMOVAL IN GA-MAKANYE BOREHOLE WATER

Sample type	**Initial conc. (mg/L)**	**Final conc. (mg/L)**	**% Removal**	**Qe (mg/g)**	**SANS 241-1:2015 standards (mg/L)**
RES	16.5	2.1302	87.091	14.3598	≤11
CES	16.5	0.1775	98.924	16.3225	≤11

The results presented in Table IV show that both RES and CES effectively removed nitrate from Ga-Makanye borehole water under the optimized conditions obtained from the experiments. Raw eggshells reduced nitrate from 16.5 mg/L to 2.13 mg/L, achieving 87.09% removal and an adsorption capacity of 14.36 mg/g, while calcined eggshells achieved superior performance, lowering nitrate to 0.18 mg/L with 98.92% removal and 16.32 mg/g adsorption capacity. The marked improvement in removal efficiency and adsorption capacity by modified eggshells is attributed to structural and chemical changes caused by calcination. The removal efficiencies of the borehole water are higher compared to the synthetic samples, which is linked to differences in initial nitrate concentration and the presence of natural ions (Ca^{2+}, Mg^{2+}, HCO_3^-, CO_3^{2-}) that enhanced electrostatic interactions. When there is a greater number of ions competing for the available adsorption sites on the biosorbent surface, the active sites become easily saturated, resulting in a decline in nitrate adsorption.

In Ga-Makanye borehole water, naturally occurring ions enhanced ionic strength and surface charge interactions, promoting stronger electrostatic attraction between nitrate ions and Ca^{2+} sites, particularly on calcined eggshells ($CaO/Ca(OH)_2$). The superior performance of calcined eggshells demonstrates the effectiveness of thermal treatment in improving adsorption efficiency. Nitrate levels were reduced below 11 mg/L, meeting SANS 241-1:2015 drinking water standards.

Overall, these findings confirm that eggshells, particularly in modified form, can serve as efficient, sustainable, and low-cost adsorbents for nitrate removal from contaminated water. This will help improve water quality in rural communities like Ga-Makanye, while also reducing eggshell waste in the environment and preventing the risks of elevated nitrate levels to human health.

IV. Conclusion

This study evaluated the effectiveness of unmodified and modified eggshells for the removal of nitrate from both synthetic and real groundwater samples, using the Ga-Makanye borehole water as a case study. It also investigated the effects of contact time, biosorbent dosage, initial nitrate concentration, and pH on removal performance, and characterized the biosorbents before and after treatment. The results showed that both raw and calcined eggshells effectively removed nitrate, with calcined eggshells performing better due to increased surface area, porosity, and reactivity. Optimal removal was achieved at a 90-minute contact time and 0.1 g dosage. FTIR, SEM, and EDX analyses confirmed structural and chemical enhancements that improved nitrate binding. Treated water met SANS 241:1 (2015) standards, confirming the suitability of calcined eggshells as sustainable, low-cost biosorbents for nitrate removal and rural water quality improvement.

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