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A Comparative Study on The Efficacy of Modified Egg Shell for Removing Heavy Metals and Dye from Water

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ABSTRACT

This study explores the potential of discarded eggshells as sustainable and cost-effective biosorbents for removing heavy metals and dyes from water. The influence of chemical and thermal activation on their adsorption properties was thoroughly examined. Raw eggshells, mainly composed of calcium, carbon, and oxygen forming carboxylic and carbonate functional groups, were chemically activated with 5% and 10% orthophosphoric acid and then heat-treated at 550 °C for 2 hours. The materials were characterized using FT-IR, SEM, UV–Vis spectroscopy, and AAS. FT-IR confirmed the retention of functional groups after activation, while SEM images showed that acid treatment produced a more porous and irregular surface, enhancing the material's adsorption capacity. The modified eggshells showed remarkable improvement in removing contaminants. After 60 minutes of contact time, removal efficiencies reached 98.76% and 98.51% for Cr, 100% for Cd, and 96.23% and 96.17% for Pb using 5% and 10% activated samples, respectively. Methylene blue removal also increased from 27% for the raw powder to 89% for the 10% activated eggshell. Overall, phosphoric acid activation significantly enhanced the surface reactivity and porosity of eggshells, demonstrating their strong potential as eco-friendly biosorbents for wastewater purification.

Keywords: Adsorption, Eggshells, Acid activation, Heavy metals, Dye.

1. Introduction

Textile processing facilities release highly colored water effluents with dye contents ranging from 10 to 200 mgL^{-1}.The waste water's colors are predicted to pose major eco-toxic risks, and as a result adds to the possible risk of bioaccumulation and the human food chain [1].Lead, cadmium, zinc, nickel, chromium, cobalt, copper, and mercury are among the more than sixty metals in this category(heavy metals). In order to guarantee that our communities and future generations will have access to clean water, heavy metal removal from water is urgently needed. Conventional methods (filtration, coagulation, oxidation, ion exchange, and chemical precipitation) can be used to eliminate heavy metals from water. Additionally, biological techniques including phytoremediation, bioremediation, and phyto-filtration are being employed. The main drawback of these procedures is that they are thought to be very costly methods and are not very effective in temperate locations. Because of their higher operating costs, increased sludge production, and problems with final disposal, the aforementioned procedures are therefore not very effective [2]. Finding more affordable, ecologically friendly, and technically efficient technologies is necessary given the numerous problems with the current treatment techniques. The process of adsorption is one such method that has garnered a lot of interest recently and has been the focus of several studies [3].

The utilization of eggshells as an adsorbent media in the process of environmental restoration is the main focus of this work. C, Ca, and O make up the modified eggshell's constitution. It is a porous material that extracts molecules from a gas or liquid, the majority of which are organic [4]. People use it most frequently as a cleansing agent because of how successfully it achieves this [5].Its low acid/base reactivity, thermo-stability, variable pore geometry, and wide porous surface area [6], it has been demonstrated that the oldest adsorbent, modified eggshell, is efficient at eliminating a variety of inorganic and organic [7] contaminants from gaseous environments or those dissolved in aqueous medium [8-9]. Polymers, wood, coconut shell, peat, pitch, petroleum, and bituminous coal are the most often utilized precursors to make modified eggshell [10-17]. Developing a workable, reasonably priced method to deal with metal contamination in aqueous solutions is the aim of this study. It was looked at how well an eggshell biosorbents system removed heavy metal ion and dye. The selection of metals and dyes were based on their toxicity and prevalence in the majority of industrial effluents. To create adsorbents having the qualities of both, different eggshell proportions were used to prepare the eggshells. Batch tests were conducted in order to assess the eggshells' adsorption capability under various operating circumstances.

2. Materials & methodology

2.1 Materials lists

To make eggshell powder, chicken eggshells were gathered from the RUET cafeteria, canteens, homes, and eateries. Distilled water was used for washing, orthophosphoric acid (H_3PO_4) for surface activation of the adsorbent, and sodium hydroxide (NaOH) for pH adjustment. Standard solutions of chromium, cadmium, lead, and dye were obtained from laboratory stocks or purchased for experimental use.

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2.2 Equipments

Laboratory apparatus included: crucible (high temperature heating), desiccator (moisture-free storage), digital balance (precise mass measurement), measuring cylinder (liquid volume measurement), burette (titration), pipette (liquid transfer), sealer (sample preservation), knife (sample preparation), blender (sample homogenization), centrifuge (density-based separation), vortex mixer (rapid mixing), rotating shaker (uniform liquid mixing), magnetic stirrer with magnet (solution stirring), conical flask (mixing and heating), microwave oven (rapid heating/drying), and water bath (temperature controlled incubation)

2.3 Eggshell Powder Preparation

To get rid of any dirt impurities, the eggshells were rinsed many times with double-distilled water after being carefully cleaned with tap water. Once the eggshells reached a constant weight, they were then dried for a whole day in an oven dryer. It was then ground using a grinder and mortar. It was then stored for subsequent use at room temperature in an airtight container after being filtered using a metal sieve of oko mesh 0.01 to 0.05 to a geometrical particle size of 50 to 150 µm.

Fig.1 Preparation of Eggshell Powder

2.4 Preparation of Modified Eggshell

2.4.1Chemical Activation Process

Using the H_3PO_4 (Ortho phosphoric acid, 85% wt in water) activated agent; the eggshell powder was chemically activated. After the pH of the suspension was stabilized, water (80ml) and eggshell powder (20 g) were transferred to a glass beaker that was set over a magnetic stirrer (600 rpm) and constantly swirled at 50ºC, 4 and 8 milliliters of orthophosphoric acid were added at a rate of one milliliter per minute. The solution was stirred continuously for two hours to ensure a full reaction between the sample and the activated agent. Following a 72-hour soak of the samples, the reaction was maintained at ambient temperature. The liquid and solid phases of samples extracted from the reaction mixture were separated using filter paper. After being regularly cleaned with distilled water, the solid was allowed to dry at 110ºC for the entire night. Then a mortar was used to grind it into a fine powder.

2.4.2 Physical Activation Process

Finely ground chemically activated powder was heated to 550ºCfor two hours in a muffle furnace. The material that had been chemically activated was heated to 550ºC for 2 hours in a muffle furnace. A pestle and mortar was then used to grind the powder. To get rid of any inorganic residual material, 1 M Conc. HCL was used to rinse the crushed powder. After that, until the pH achieved 6-7, after being repeatedly cleaned with distilled water, it was left to dry overnight at 110ºC. Eventually, the modified eggshell gets obtained and kept in airtight container for additional study.

(a) (b)

Fig 2 Modified Eggshell Preparation: a) 5% acid activated b) 10% acid activated

2.5 Procedures for the Elimination of Heavy Metals

2.5.1 Adsorbate Solution's formation

1mL solution(stock) containing 1000 ppm of chromium , cadmium , lead and 0.002 g of methylene blue was further diluted with deionized water of 100 mL for preparation of 10 ppm standard solutions of heavy metals and 20 ppm standard solution of methylene blue. The formula used –

$$S_1V_1 = S_2V_2 \quad \text{....................} \quad (i)$$

Where S_1 and V_1 represent the initial concentration and volume of a solution, respectively, and S_2 and V_2 represent the final concentration and volume after dilution.

2.5.2 Batch adsorption studies

2.5.2.1 Effect of Adsorbents on Cr^{2+}, Cd^{2+}, Pb^{2+} Adsorption for Eggshell Powder

After adding 25 millilitres of adsorbate with the ideal pH 7 to an Erlenmeyer flask with the lead concentration 10 ppm, each sample obtained 0.1g, 0.5g, 1g of eggshell powder and the sonicate machine was adjusted for 60 minutes at 25ºC. The metal concentration was obtained after subsequently centrifuging the treated solution and filtering it with filter paper.

2.5.2.2 Effect of Adsorbents on Cr^{2+}, Cd^{2+}, Pb^{2+} Adsorption for 5% and 10 % acid activated eggshell

After adding 25 millilitres of adsorbate with the ideal pH 7 to an Erlenmeyer flask with the lead concentration 10 ppm, each sample obtained 0.1g, 0.5g, 1g of modified eggshell and the sonicator machine setting was adjusted for 60 minutes at 25ºC. The metal concentration was obtained after subsequently centrifuging the treated solution and filtering it with filter paper.

Adsorption Percentage:

$$q = \frac{(Co - Ce)V}{W} * 100 \text{ .... (ii)}$$

2.6 Methodology for Dye Removal

2.6.1 Experimental Methodology for Dye Removal using eggshell powder

1g of eggshell powder was mixed with 20ml solution containing 10 mg/L methylene blue. Allowed the mixture in the sonicate machine setting was adjusted at 25ºC for 30 minutes, providing time for adsorption and potential degradation reactions to occur. After that, the eggshell powder was separated from the solution, typically through filtration and centrifugation. Then, the treated solution was analyzed for a decrease in methylene blue concentration, which indicates degradation or adsorption.

2.6.2 Experimental Methodology for Dye Removal using Modified eggshell

1g of eggshell powder was mixed with 20ml solution containing 10 mg/L methylene blue. Allowed the mixture in the sonicate machine setting was adjusted at 25ºC for 30 minutes, providing time for adsorption and potential degradation reactions to occur. After that, the modified eggshell is separated from the solution, typically through filtration and centrifugation. Then, the treated solution is analyzed for a decrease in methylene blue concentration, which indicates degradation or adsorption.

(a)

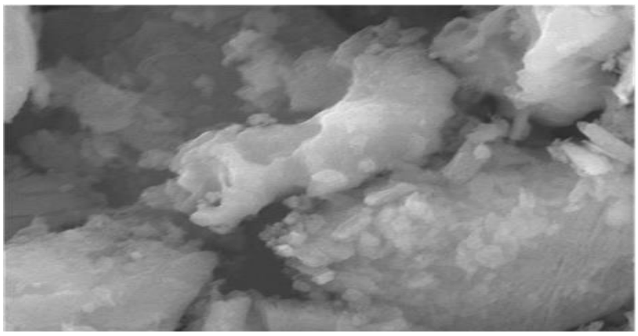
(b)

Fig 3 Sonicate machine(a) and Batch Study for dye elimination(b)

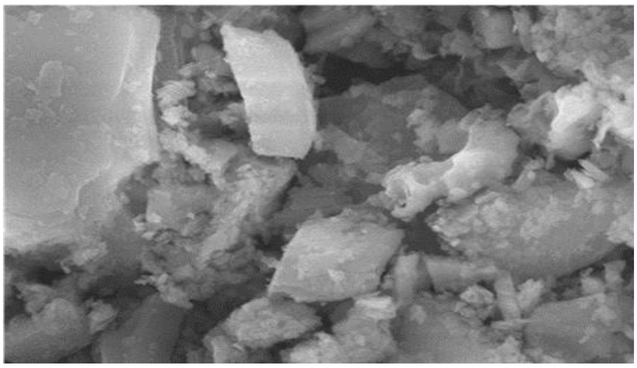
3. Results and discussions

3.1 SEM –EDX Test

Eggshells have unique, tightly spaced structures on their surface, as seen in Figure 4. The ununiform shape of the modified eggshell from the egg shell is revealed by the morphological perspective. Investigated modified eggshell led to an irregular carbon structure. Images shows that lamp shadows been formed and micro particles are clearly showed. Also particles are coagulated with each other, they can be found also in nano size.



(a)



(b)

Fig 4 SEM of modified egg shell a) 5% activated b) 10% activated

3.3 Comparative Evaluation of Different Adsorbents' Performance in Removing Cr^{2+}

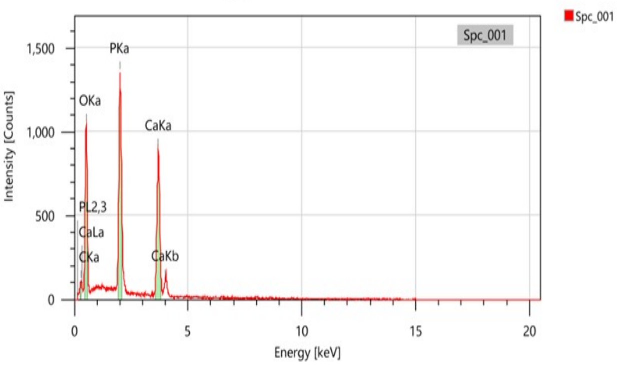
According to the comparison tests of the batch study, 5% acid-activated eggshell was more effective in removing Cr. The following was the order of efficiency for the removal of Cr:
5% modified egg shell activation > 10% acid activation Eggshell powder > Eggshell modification

3.4 Comparative Evaluation of Different Adsorbents' Performance in Removing Cd^{2+}

Both the 5% and 10% acid-activated eggshell absorbed all of the Cd compounds from the solution, and no residues were detected, according to the batch study comparative tests. The concentration result was negative, indicating that the solution had no traces of Cd ions. The following was the order of effectiveness for the elimination of Cd:
10% activated modified egg shell > 5% acid activated Modified eggshell > Eggshell powder

3.5 Comparative Evaluation of Different Adsorbents' Performance in Removing Pb^{2+}

According to the comparison tests of the batch study, 5% acid activated eggshell was more effective at removing lead. The following was the order of effectiveness for Pb removal:



Name	C	O	P	Ca	Total
Spc_001	4.82	56.83	17.44	20.92	100.00
Average	4.82	56.83	17.44	20.92	
Standard Deviation					

Atom/Mol%

Fig 5 EDX of Modified eggshelll

5% modified egg shell activation > 10% acid activation Eggshell powder > Eggshell modification.

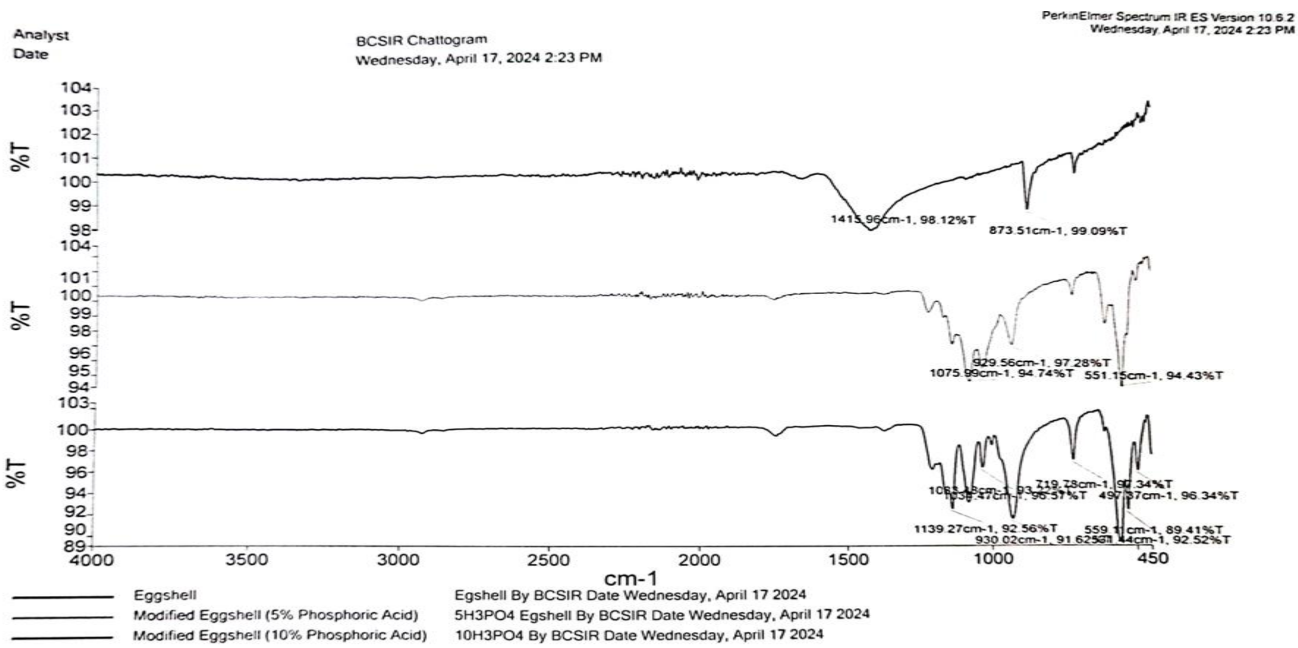


Fig 6 Comparison of FT-IR spectra of eggshell, 5% and 10% activated eggshell

Table 1 Parameters of removal performance of Cr^{2+} onto eggshell powder, 5% and 10% modified eggshell

Mass of adsorbents (g)	Initial Conc. (mg/L)	Eggshell Powder		5 % activated		10% activated	
		Final conc. (mg/L)	Elimination efficiency (%)	Final conc. (mg/L)	Elimination efficiency (%)	Final conc. (mg/L)	Elimination efficiency (%)
0.1	10	4.3732	56.26	0.1548	98.45	0.2046	97.95
0.5	10	3.9232	60.76	0.1349	98.65	0.1569	98.43
1	10	3.3609	66.39	0.1238	98.76	0.1488	98.51

Table 2 Parameters of removal performance of Cd^{2+} onto eggshell powder, 5% and 10% modified eggshell

Mass of adsorbents (g)	InitialConc. (mg/L)	Eggshell Powder		5 % activated		10% activated	
		Final conc. (mg/L)	Elimination efficiency (%)	Final conc. (mg/L)	Elimination efficiency (%)	Final conc. (mg/L)	Elimination efficiency (%)
0.1	10	0.3602	96.39	0.0125	99.87	0.003	99.97
0.5	10	0.2893	97.10	-0.2037	100	-0.3092	100
1.0	10	0.2671	97.32	-0.2144 (no traces found)	100	-0.3122 (no traces found)	100

Table 3 Parameters of removal performance of Pb^{2+} onto eggshell powder, 5% and 10% modified eggshell

Mass of adsorbents (g)	Initial Conc. (mg/L)	Eggshell Powder		5 % activated		10% activated	
		Final conc. (mg/L)	Elimination efficiency (%)	Final conc. (mg/L)	Elimination efficiency (%)	Final conc. (mg/L)	Elimination efficiency (%)
0.1	10	4.7705	52.29	0.4587	95.41	0.4698	95.30
0.5	10	4.375	56.25	0.4021	95.97	0.4295	95.70
1.0	10	4.214	57.86	0.3763	96.23	0.3828	96.17

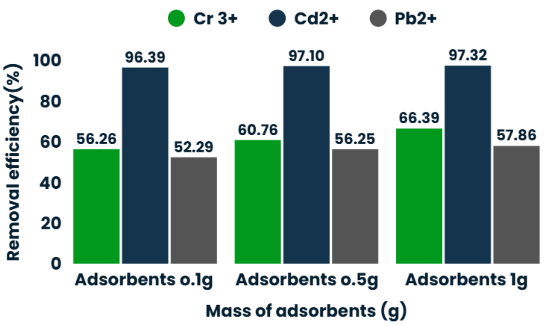


Fig 7 Removal efficiency of eggshell

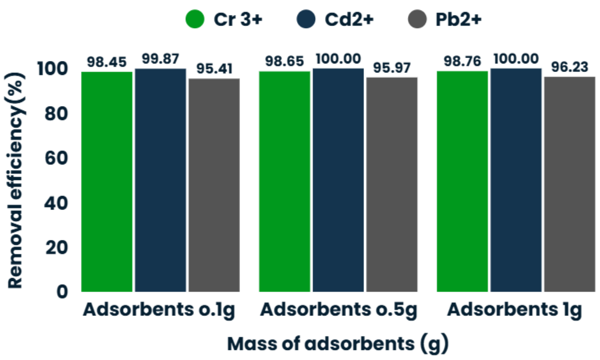


Fig 8 Removal efficiency of 5% activated eggshell

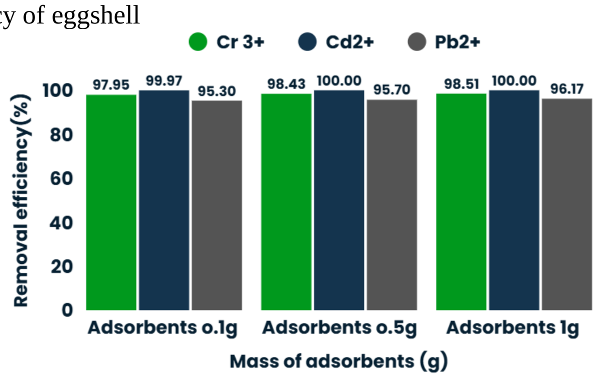


Fig 9 Removal efficiency of 10% activated eggshell

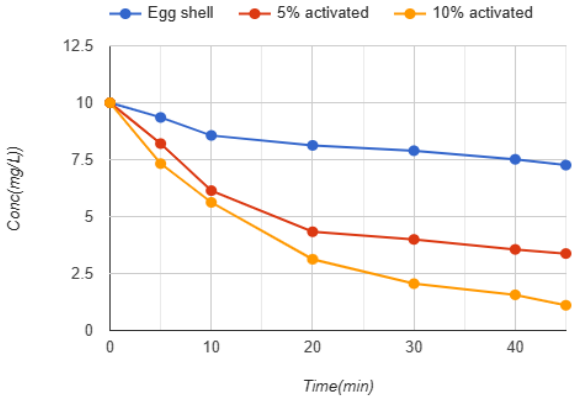


Fig 10 Dye adsorption with time

3.6 Comparative Performance test on Dye Removal of various adsorbents

According to modified eggshell data, the highest degradation efficiency for 10% activated modified eggshell, 5% activated modified eggshell, and eggshell powder is roughly 89%, 66%, and 27%, respectively. The largest amount of dye was eliminated in this case by 10% activated modified egg shell.The following was the dye removal efficiency order:

10% activated modified egg shell > 5% acid activated Modified eggshell > Eggshell powder

Table 4 Parameters of removal performance of dye onto eggshell powder, 5% and 10% modified eggshell.

Sample ID	Amount (g)	Initial conc. (mg/L)	Final conc. (mg/L)	Efficiency %
Egg Shell	1.0 g	10	7.261	27.39
5% activated	1.0 g	10	3.378	66.22
10% activated	1.0g	10	1.107	88.93

4. Conclusion

Eggshells contained carbon, calcium, and oxygen atoms, which make up carboxylic and carbonate functional groups. The modified eggshell also contains a small quantity of phosphate. According to the SEM micrograph, the surface morphology of the modified eggshell showed irregularities. The percentages of Cr adsorption for eggshell powder, 5% modified eggshell removal, and 10% modified eggshell removal were 66.39%, 98.76%, and 98.51%, respectively. Eggshell powder, 5%, and 10% modified eggshell removal percentages achieved 97%, 100%, and 100% for Cd adsorption, respectively. The percentages of Pb adsorption for eggshell powder, 5% modified eggshell removal, and 10% modified eggshell removal were 56.86%, 96.23%, and 96.17%, respectively. It was 21.51%, 62.10%, and 72.89% for dye removal. Additionally, this study's data indicates that repurposing natural agricultural wastes, such eggshells, has a lot of potential for usage as effective adsorbents in bioremediation.

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Nomenclature

Symbol	Meaning	Unit
Co	Initial concentration	(mg/L)
Ce	Final concentration	(mg/L)
W	Mass of biosorbent	(g)
V	Volume of solution	(L)