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Simultaneous Removal of Copper and Fluoride from Wastewater by Adsorption Using Chicken Eggshell

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***Abstract:** In recent years, agricultural wastes and biomass have been extensively investigated as low cost adsorbents in heavy metal removal owing to the facts that they are relatively cheap and exhibit high adsorption capacities. Even though promising results are reported in literature, information on the simultaneous removal of co-existing pollutants is still very scarce and limited. Since industrial effluents contain various pollutants, there is a need to develop biosorbents and system that are able to remove more than one pollutant at one time. In this research, chicken eggshell was investigated for its ability to remove copper and fluoride simultaneously from aqueous solution. The optimization study showed that the highest removal percentage for copper and fluoride could be achieved at the process conditions as such: adsorbent dosage of 2.5 g, temperature of 40°C, pH 6 and stirring speed of 350 rpm. Simulatenous removal of both copper and fluoride ions from mixed solution was possible, however, with a reduction of approximately 26 – 35 % in fluoride removal but insignificant drop in copper removal percentage compared to single pollutant solution. Scanning Electron Microscopy (SEM) analysis revealed deposition of flake-like copper and fluoride crystals on the surface of the chicken eggshell powder thus evidenced its adsorption ability of copper and fluoride ions from aqueous solution.*

***Keywords:** Scanning Electron Microscopy (SEM), copper, fluoride ions*

I. INTRODUCTION

Heavy metals have been employed in various processes and industries such as electroplating, galvanizing, textile and pigment manufacturing, tanneries and paint production [1]. Rapid industrialization has witnessed excessive release of heavy metals into the environment in recent years, with remarkable adverse effects on humans and other living organisms. These toxic heavy metals that present in effluent waste are non-degradable, persistence and will be accumulated throughout the food chain [2,3]. Copper, Lead, Cadmium, chromium and Mercury are among the toxic metals that pose potential danger to humanhealth and environment [4]. For instance, excessive copper in soil induces stress and causes injury to plants, leading to plant growth retardation and leaf chlorosis [5]. Severe chronic and cumulative over fluoride exposure on the other hand, can lead to incurable cripping of skeletal fluorosis [6]. Thus, the removal of heavy metals and halide ions from industrial wastewater or effluents is of primary importance nowadays.

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Different physicochemical strategies, for example, extraction, particle trade, synthetic precipitation, film filtration, adsoprtion and electrodialysis have been widely considered for the expulsion of substantial metals from fluid arrangements [1,4]. As per Buasri et al. [7], adsorption is an alluring technique because of its straightforwardness, comfort and high evacuation productivity. In like manner adsorption forms, actuated carbon and manufactured tars are generally used to increase high expulsion proficiency. In any case, because of their high creation cost, water disinfecting by utilizing these two adsorbents is somewhat costly. Actually, numerous analysts called attention to that the vast majority of the customary techniques included generally mind-boggling expense and low possibility for use in little scale businesses [4,8] .Therefore, various mechanical inorganic squanders, for example, powder, or characteristic inorganic materials, for example, earth and zeolite, and additionally living or non-living biomass have been investigated as shabby adsorbents fit for supplanting the outstanding, yet increasingly costly ones [9].

The utilization of non-living biomaterials as metal restricting mixes has raised incredible consideration as they require least consideration and upkeep, with demonstrated adequacy in the expulsion of follow metals from the earth [4]. Sameer et al. [8] and Fawzi et al. [10] revealed that horticultural squanders, for example, creature bones and quills could be utilized for substantial metal expulsion from wastewater. The nearness of different practical gatherings in these waste materials prompted their novel capacities to tie metals and thusly draw in and sequester metal particles. What's more, dried leaves, sawdust, wheat grain and natural product strips have additionally been found to expel copper from water effectively in different explores [5]. In any case, the work of untreated agrarian results or squanders as adsorbents can likewise bring serious issues, for example, high synthetic oxygen request (COD), natural oxygen request (BOD) and aggregate natural carbon (TOC), because of the filtering of natural substances which incorporate lignin, tannin, gelatin and cellulose into the arrangement [2,8].

This marvel has along these lines prodded enthusiasm for other regular materials that display comparable viability in substantial metal expulsion, while meeting the ease and normal plenitude qualities.

Chicken eggshells are squander materials from incubation facilities, homes and drive-thru food ventures which are bounteous and promptly accessible in bounty worlwide.

Its transfer will add to natural contamination without appropriate administration. Chicken eggshells contain calcium and follow measures of other small scale components (boron, copper, press, manganese, molybdenum, sulfur, magnesium, silicon and zinc) [6]. The nearness of cellulosic structure and amino acids proposed it as a decent biosorbent [6] with calcium carbonate which is in charge of metal adsorption [11]. A few investigations as of late focusing on the assessment of the likelihood of overwhelming metal expulsion, especially copper, fluoride, iron and chromium from fluid arrangement had shed a few lights on the utilization of disposed of chicken eggshell in wastewater treatment [3,4,6,12]. In Malaysia, chicken eggshells are disposed of from nourishment industry or family unit as city strong waste and are arranged in landfills. Despite the fact that these waste materials are accessible in bounty, its capability to be additionally abused for overwhelming metal expulsion application is still less being examined.

Possibility contemplates for vast scale applications have exhibited that biosorptive procedure utilizing non-living biomass are in actuality more pertinent than the bioaccumulative procedures that are using living microorganisms. This is on the grounds that the last require a supplement supply and confused bioreactor framework [13]. All things considered, biosorption utilizing horticulture side-effects or squanders is in its formative stages and further enhancement in both execution and expenses ought to be centered around in future. In contrast to research facility arrangements, modern effluents contain different contaminations including those of intrigue. In this manner, there is a need to examine the concurrent evacuation of many existing together contaminations. It is attractive to create "broadly useful" biosorbents that can evacuate an assortment of poisons at any given moment. The abuse of chicken eggshell squanders stayed at its earliest stages in Malaysia up to now. Thusly, the fundamental goal of this paper is to explore the likelihood of using these losses in synchronous substantial metal and halide particles expulsion from fluid arrangement, particularly for copper and fluoride. Concurrent evacuation of more than one toxin at any given moment would introduce a progressively practical methodology in biosorption framework for wastewater treatment forms which contain different sorts of contamination at once.

II. METHODOLOGY

Materials

Chicken eggshells were gathered from the eateries. The specimen was washed with refined water a few times to evacuate earth and contaminants, trailed by drying in a tourist oven at 110 °C for no less than 12 hours. The chicken eggshells were then granulated or smashed utilizing pestle and mortar, sieved well in portion of 150 µm. The powdered example was put away in an impenetrable compartment preceding adsorption forms.

Chemicals

Pentahydrate copper (II) sulphate was attained from R&M Chemicals (Malaysia). SPADNS 2 (sodium2 – (parasulfophenylazo) – 1, 8 – dihydroxy – 3, 6 – naphthalene disulfonate) reagent, sodium fluoride, sodium hydroxide, and hydrochloric acid originated from Merck Sdn. Bhd. (Malaysia).

Bio-sorption Process

The adsorption procedure was performed dependent on the strategies revealed by Rohaizar et al. [3] with slight adjustments. Powdered chicken eggshell was blended with copper (II) sulfate arrangement, fluoride arrangement, and blend of copper and fluoride arrangement. The example and arrangement were blended at a steady mixing velocity of 150 – 350 rpm for 180 minutes. The rest of the grouping of copper (Cu2+) and fluoride (F-) after adsorption process was estimated utilizing UV-Vis spectrophotometer at the wavelength of 606 nm for copper and 624 nm for fluoride [14]. Every single trial keep running in this exploration were performed in triplicate and the last fixation was introduced as the mean esteem.

Effect of Adsorbent Dosage and temperature

1 g of chicken eggshell powder was mixed with 200 mL of mixed solution of copper and fluoride solution at the temperature of 30 °C for 180 minutes, under stirring speed of 200 rpm. The adsorption process was repeated with adsorbent dosage of 1.0, 1.5, 2.0, 2.5, 3.0, 3.5 g. The final solution was filtered and final concentration of Cu2+ and F- was measured. As for the temperature test, the agitation speed was maintained at 200 rpm for 180 minutes. The adsorption process was repeated for temperature 30 °C, 40 °C, 50 °C and 60 °C. The final solution was filtered and final concentration of Cu^{2+} and F^- was measured.

Effect of pH

Chicken eggshell powder was mixed with 200 mL of mixed solution of copper and fluoride solution at optimum adsorbent dosage and temperature obtained previously. The adsorption process was repeated for pH ranged 3 – 8. pH of the solution was adjusted using 0.1 M NaOH and 0.1 M HCl. The agitation speed was maintained at 200 rpm for 180 minutes. The final solution was filtered and final concentration of Cu^{2+} and F^- was measured.

Effect of Agitation Speed

Chicken eggshell powder was mixed with 200 mL of mixed solution of copper and fluoride solution at optimum adsorbent dosage, temperature and solution pH obtained previously. The adsorption process was repeated with agitation speed 150 – 350 rpm for 180 minutes. The final solution was filtered and final concentration of Cu^{2+} and F^- was measured.

Scanning Electron Microscopy (SEM)

SEM pictures of the chicken eggshells were acquired utilizing JEOL JSM 6400 LV display checking electron magnifying lens at 15 kV. This was done to evaluate the auxiliary and morphology changes of the egg shell.

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III. RESULT AND DISCUSSION

Preliminary study had been conducted prior to the experiments in determining the best particle size for the chicken eggshell powder to be utilized. Three molecule sizes had been tried (150 μm, 425 μm and 600 μm) and it was discovered that the adsorption of both Cu2+ and F-particles from watery arrangement was exceedingly subject to the molecule size of the adsorbent, whereby little molecule measure displayed bigger surface territory and in this way the adsorption limit expanded. Consistent with the reports by Hossain et al. [15] and Krishna and Swamy [16], it was observed that the removal efficiency for both Cu^{2+} and F^- decreased as the adsorbent particle size increased, with the highest removal percentage recorded at 150 μm showing 84.46 % removal for Cu^{2+} and 55.36 % removal for F^-, in single pollutant aqueous solution (Figure 1). Krishna and Swamy [16] expounded that at a settled adsorbent measurement, the expansion in molecule estimate diminished metal take-up chiefly because of more noteworthy availability to the pores on adsorbents utilized and more noteworthy surface region accessible for mass adsorption per unit mass of the adsorbent. Based on the results shown in Figure 1, chicken eggshell with the particle size of 150 μm was selected for the subsequent experimental works.

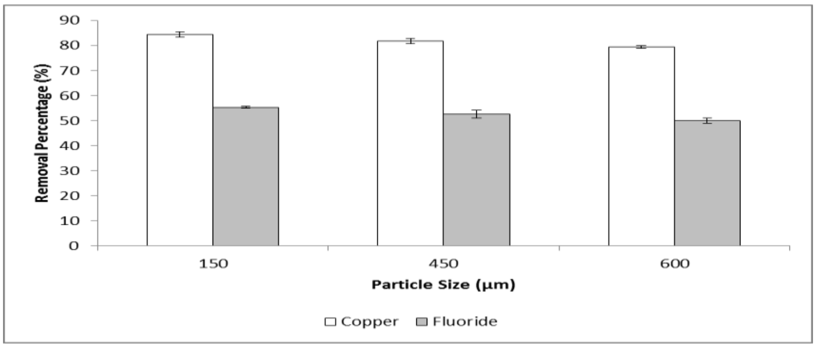

Fig. 1 The effect of chicken eggshell particle size on the removal percentage of Cu^{2+} and F^- in the mixed pollutant solution (Experimental conditions: pH of 5, adsorbent dosage at 1 g/ 200 ml, agitation speed at 200 rpm, contact time of 180 minutes and temperature at 30 °C).

Effect of Adsorbent Dosage

The impact of adsorbent measurement was examined by changing adsorbent dose from 1 – 3.5 g adsorbent/200 ml arrangement. It was discovered that the adsorption limit, Qe of copper diminished as the adsorbent measurements expanded. The most noteworthy adsorption limit 4.744 mg/g was accomplished when 1 g of adsorbent was utilized. Moreover; comparative perception was acquired for the expulsion of fluoride. As the adsorbent measurement was differed, the most elevated adsorption limit at 1.107 mg/g was achieved when 1 g of adsorbent was used. Along these lines, there was a drop in the Qe for both copper and fluoride with the expansion of adsorbent measurements. As indicated by Bhaumik et al. [6], this marvel may be contributed by the arrangement of totals between the chicken eggshell powder particles at high adsorbent doses, decreasing the successful surface region for adsorption. Then again, the connection between the expulsion level of Cu2+ and F-with adsorbent dose is introduced in Figure 2. The copper expulsion rate as high as 96.27 % was accomplished with 2.5 g of adsorbent and 60.92 % of fluoride was expelled from the equivalent blended fluid arrangement.

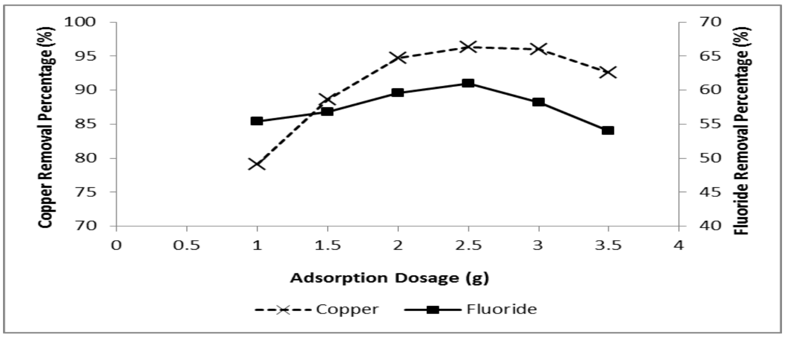

Fig. 2 The effect of adsorbent dosage on the removal percentage of Cu^{2+} and F^- in the mixed pollutant solution (Experimental conditions: pH of 5, agitation speed at 200 rpm, contact time of 180 minutes and temperature at 30 °C).

As the adsorbent dosage was increased from 1 to 2.5 g, it resulted in a positive incline in the removal of both pollutants. This pattern was attributed by increase in surface area for adsorption when larger amount of adsorbent was dosed into the solution. It resulted in positive correlation between the efficiency of adsorption with the number of active binding sites available. This was confirmed by numerous researchers using different types of adsorbent such as Delaila et al. [17], Oladunni et al. [18], Nasution et al. [19] and Zubairu et al. [20].

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Nevertheless, beyond the dosage of 2.5 g, a gradual decrease in copper removal percentage while a sharp reduction in fluoride removal percentage was observed. As explained by Delaila et al. [17] and Solomon et al. [21], overcrowding of adsorbent particles could occur following higher dosage of adsorbent in the solution. Apart from overlapping of the adsorption sites [19], over dosing of adsorbent could also contribute to the screening effect of the dense outer layer of the particles [22], shielding the active binding sites from metal and fluoride ions. Therefore, chicken eggshell dosage at 2.5 g was identified as the optimum amount of adsorbent required for the removal of Cu^{2+} and F^- in this research.

Effect of Temperature

Figure 3 demonstrates the impact of temperature on the evacuation level of Cu2+ and F-from the blended fluid arrangement. As the temperature was increased from 30 °C to 60°C, the highest removal of copper was achieved when the solution was at 40 °C with 95.91 % and the lowest percentage removal was recorded at 60 °C with 92.83 %. Similar removal pattern was obtained for fluoride. It was obvious that from Figure 3, the highest percentage removal of fluoride was achieved at the temperature of 40 °C with 64.98 % while the lowest percentage removal was also at 60 °C with only 53.98 % percentage removal.

At 40 °C, chicken eggshell powder exhibited the highest adsorption affinity for copper and fluoride ions, where this phenomenon indicated that the elevation of solution temperature to around 40 °C could provide suitable driving force to raise the mobility of copper and fluoride ions thereby increased the interaction with adsorbent binding sites which enhance the removal efficiency. In numerous adsorption forms, expanding temperature was known to upgrade the dispersion rate of adsorbate atoms over the outside limit layer and inner pores of the adsorbent particles [23,24]. Moreover, higher temperature could prompt swelling impact inside the interior structure of the eggshell, which therefore empowering Cu2+ and F-particles to infiltrate further into the eggshell structure. However, the adsorption of copper and fluoride on chicken eggshell powder dropped when there was elevation of temperature from 40 to 60 °C. This could be due to the fact that temperature beyond 40 °C might denature the structure of chicken eggshell and lead to failure of its capability to adsorb the ions. Another plausible explanation given by Sujana et al. [25 and Bhaumik et al. [6] was that the rise in temperature escalated the escaping tendency of molecules from adsorbent interface and therefore diminished the extent of adsorption (i.e. decrease in adsorption capacity). An increase in thermal energy of the adsorbate (Cu^{2+} and F^- ions) resulting from higher temperature that favoured desorption process was also possible for the phenomenon.

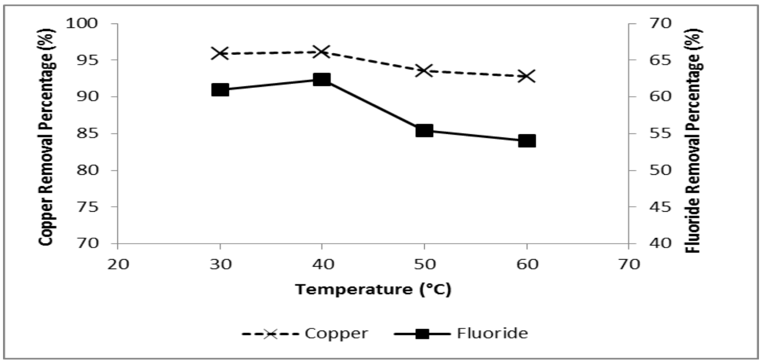

Fig. 3 The effect of temperature on the removal percentage of Cu2+ and F- in the mixed pollutant solution (Experimental conditions: pH of 5, adsorbent dosage of 2.5 g, agitation speed at 200 rpm and contact time of 180 minutes)

Effect of pH

The surface charges of adsorbent materials were significantly affected and controlled by the pH of the arrangement [26]. In this examination, pH of the blended arrangement of copper and fluoride was changed by including hydrochloric corrosive or sodium hydroxide into the arrangement, contingent upon the estimation of wanted pH. Inside the pH scope of 3 – 8, the most noteworthy evacuation for both Cu2+ and F-particles from the arrangement was accomplished at pH 6, with the level of 97.10 % and 65.09 %, individually. The change in proficiency of expulsion because of the impact of pH is appeared in Figure 4. In view of Figure 4, it unmistakably demonstrated that the adsorption limits of copper and fluoride particles onto chicken eggshell powder expanded essentially as the pH was expanded from 3 to 6, yet diminished radically past pH 6. As clarified by Kanyal and Bhatt [27], at low pH esteems, copper adsorption was bring down because of the opposition among hydrogen and copper particles onto the adsorption locales, which confined the official of metal cations onto the surfaces. Likewise, this was enhanced by the discoveries of Rohaizar et al. [3] that the impact of pH could be contributed by particle trade component of adsorption in which the carbonate bunches on the chicken eggshells displayed cation-trade properties.

As the pH expanded, the carbonate bunches in the chicken eggshells would be uncovered, expanding the negative charges on the surface of adsorbent. Thusly, the metal cations (copper) would be pulled in and advanced adsorption process onto the surface of chicken eggshells to happen. Concerning the expulsion of fluoride particles, the outcome in this examination was practically identical with the investigation revealed by Bhaumik et al. [6].

The evacuation of fluoride utilizing chicken eggshell powder was noted to increment with increment in pH of the fluoride arrangement considerably up to pH 6. With respect to this, Kashi et al. [28] recommended that the component of fluoride take-up rate onto the eggshell powder surface included the substitution (particle trade adsorption) of the carbonate radicals of the eggshell powder, by F-particles, framing an insoluble fluorite. Be that as it may, deprotonation of dynamic destinations on the surface of chicken eggshell powder could happen at pH over 6, which altogether decreased the measure of fluoride restricting onto the adsorbent surface and hence causing the decrease of fluoride take-up at the soluble pH go [29].

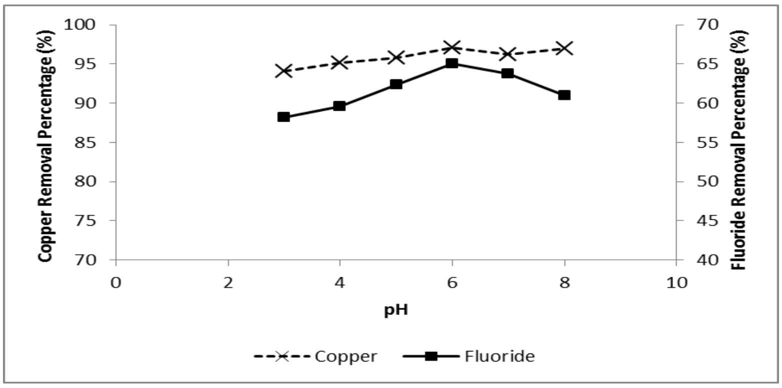

Fig. 4.The effect of pH on the removal percentage of Cu2+ and F- in the mixed pollutant solution (Experimental conditions: Adsorbent dosage of 2.5 g, agitation speed at 200 rpm, contact time of 180 minutes and temperature at 40 °C).

Effect of Agitation Speed

The impact of tumult speed on the degree of adsorption is exhibited in Figure 5. Bunch probes the impact of fomentation speed fluctuating from 150 rpm to 350 rpm on the rate expulsion of copper and fluoride particles were performed. Tumult rate assumes a vital job in the adsorption procedure. It was seen that the level of copper and fluoride expulsion expanded with expanding disturbance speed (Figure 5), likely because of legitimate contact between the metal/halide particles in arrangement and the adsorbent restricting destinations that advanced viable exchange of both copper and fluoride particles to the adsorbents locales [3]. Despite the fact that immaterial upgrade was watched for copper expulsion rate at 350 rpm when contrasted with 150 rpm, the most astounding adsorption by chicken eggshell powder was accomplished at 97.65 %. All things considered, extreme upgrade in the adsorption productivity was watched for fluoride evacuation as the expulsion rate expanded from 56.75 to 67.87 % when the disturbance speed was fluctuated from 150 to 350 rpm. These outcomes uncovered that higher tumult speed supported the adsorption procedure whereby at lower speed, there was high likelihood that adsorbents will collect at the base and brought about the internment of dynamic adsorption locales under the layered adsorbents stacking above [3].

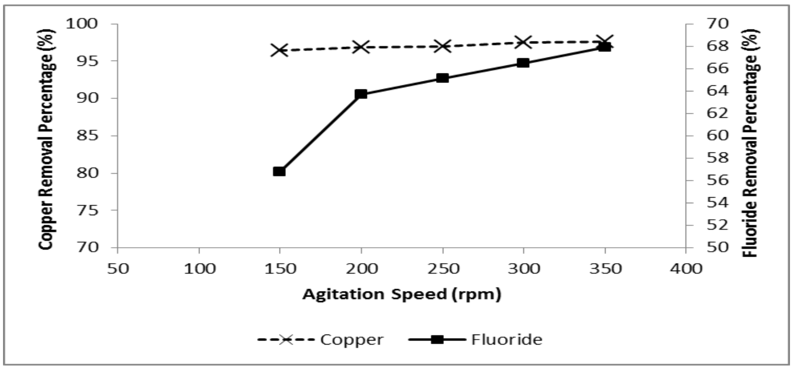

Fig. 5 The effect of agitation speed on the removal percentage of Cu2+ and F- in the mixed pollutant solution (Experimental conditions: Adsorbent dosage of 2.5 g, pH of 6, contact time of 180 minutes and temperature at 40 °C).

Comparison of Copper and Fluoride Removal in Single solution and Mixed Solution

In order to determine the possibility of utilizing chicken eggshell powder for simultaneous heavy metal and halide ions removal from aqueous solution, comparison was made between the removal percentage for copper and fluoride in both single and mixed (copper-fluoride) solutions, at the optimum process conditions. The results are presented in Table 1. Based on the comparison results, it was observed that with the presence of fluoride in copper solution (mixed solution), insignificant effect was imposed on the removal percentage of copper from the solution, as compared to single pollutant solution (copper only). Reduction in the removal efficiency was less than 2 % at all optimum process conditions. According to [30], the presence of fluoride at 1 mg/l would only increase copper solubility by 0.05 % in the solution.

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Therefore, it will not affect the bioavailability of copper which would hinder the adsorption process onto chicken eggshell powder. However, copper was found to induce approximately 26 – 35 % decrease in fluoride removal percentage in the mixed solution, in contrast to fluoride-only solution. This could be due to better affinity between chicken eggshell powder adsorption sites with copper ions in relative to fluoride ions. Also, competition between both copper and fluoride ions for the active adsorption sites could be the major reason for the reduction in removal percentage for both pollutants in the solution. Nevertheless, the possibility of utilizing chicken eggshell powder as bio-sorbent to remove both copper and fluoride ions simultaneously from aqueous solution was proven.

Table. 1 Percentage removal of copper and fluoride ions from single and mixed solution

Parameter	Copper Removal (%)			Fluoride Removal (%)		
	Copper Solution	Mixed Solution	Reduction	Fluoride Solution	Mixed Solution	Reduction
Adsorbent Dosage (2.5 g)	97.00	96.27	0.73	92.00	60.92	31.08
Temperature (40°C)	97.50	96.09	1.41	97.50	62.31	35.19
pH (6)	98.00	97.10	0.90	92.00	65.09	26.91
Agitation speed (350 rpm)	99.50	97.65	1.85	94.30	67.87	26.43

Scanning Electron Microscope (SEM)

Scanning Electron Microscopy (SEM) in Figure 6 demonstrates the morphology of powdered chicken eggshell. It affirmed the crystallinity of eggshell and the presence of pores on the surface of the eggshell. After the adsorption procedure, accomplishment in both copper and fluoride expulsion was prove by the testimony of drop like copper and fluoride precious stones on the surface of the eggshell. No conspicuous permeable structure was seen at first glance after the adsorption procedure (Figure 7).

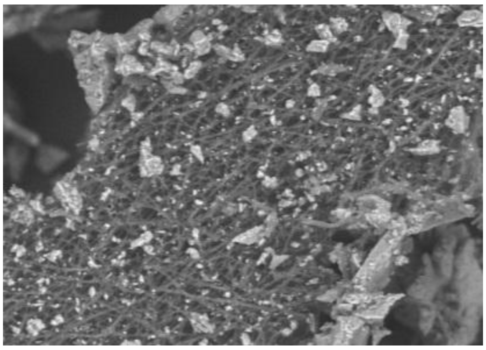

Fig. 6 SEM image of chicken eggshell powder before adsorption

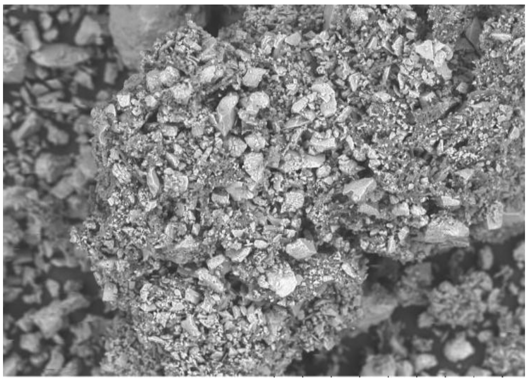

Fig. 7 SEM image of chicken eggshell powder after adsorption in mixed (copper and fluoride) solution

IV. CONCLUSION

The outcome of this investigation proposed that powdered chicken eggshell, with its minimal effort and plentiful accessibility has a potential for being utilized as an adsorbent for contaminants, for example, copper and fluoride in wastewater. Synchronous evacuation of both copper and fluoride particles in blended arrangement further confirm its capability to be abused as a multi reason bio-sorbent which serves increasingly sensible methodology in wastewater treatment which contains different sorts of toxin at once.

Concerning this, further investigation and improvement process is required to upgrade the expulsion proficiency of fluoride from fluid arrangement within the sight of copper. Physical or potentially synthetic pre-treatment of the chicken eggshell powder can be additionally investigated in improving the adsorption limits.

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