

Elaboration of Bentonite layered double hydroxide composite (LDH-bentonite) to enhance adsorption of sulfide in water

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Abstract— Low cost bentonite clay was chemically modified using magnesium chloride in order to enhance its sulfide removal capacity. The incorporation of Co and Al into bentonite (MB). Batch adsorption experiments were conducted to study and optimize various operational parameters such as adsorbent dose, contact time, pH, and initial sulfide concentration. It was observed that the MB works effectively over wide range of pH and showed a maximum sulfide removal capacity of 2.26mgg⁻¹ at an initial fluoride concentration of 5mg L⁻¹, which is much better than the unmodified bentonite. Desorption study of MB suggest that almost all the loaded fluoride was desorbed (~97%) using 1M NaOH solution however maximum sulfide removal decreases from 95.47 to 73 (%) after regeneration. From the experimental results, it may be inferred that chemical modification enhances the sulfide removal efficiency of bentonite and it works as an effective

Key-Words:

Bentonite, LDH, Modification, Adsorption, Sulfide.

I. INTRODUCTION

Rapid development in social and economic area has created a serious environment contamination of soil and groundwater. The discharge of toxic material and contaminant has increased significantly during the last few years. Sulfides presence in waste waters is a nuisance to human health and environment due to their toxicity and unpleasant odor (rotten eggs), making it inconvenient to consumption even at small concentrations [1].

Chemical methods for removing sulfur (S) compounds caused product quality reducing and side effects on aquatic environments.

Among the available methods for removing these pollutants, adsorption is still one of the most preferred methods, especially for effluents with moderate and low pollutant concentration [2].

In the last decades clays especially minerals ones have gain an important interest in waste water treatment, due to their abundance, affordable price, and their various chemical and physical properties (cation exchange, porosity, high specific surface, and thermal and mechanical stability... etc).

Bentonite is a clay mineral belongs to smectite group; these latter have the highest surface area and exchange capacity among them all, allowing them to be used in water treatment industry as adsorbent.

Their negative surface charge adsorb only cations, which require a modification to adsorb anions as well, there are three major modification reaction: organic (using surfactants ex: CTAB) [3], inorganic (using metallic salts as precursors) [4], and inorganic-organic (using the combination of both).

Recently, LDH based nanocomposites such as those hybridized with carbon materials, polymers or anions have been increasingly studied for the removal of various toxic pollutants from waste water. These nanocomposites exhibit mixed qualities of the parental materials which resulted in improvement in surface area, higher anion

exchange ability and excellent selectivity. The superior characteristics of LDH nanocomposites resulted in significant improvement in adsorption properties [4].

In this work we synthesize an anionic bentonite from metallic salts and sodium hydroxide to form LDH to adsorb sulfides from synthetic waters, effect of adsorbent and adsorbate dosage, pH, time contact are also studied.

II. MATERIALS AND METHODS

Bentonite was purchased from ENOF (Entreprise nationale des non ferreux) in powder form with 73 μ m size, Cobalt (II) nitrate hexahydrate [Co(NO₃)₂·6H₂O] was purchased from Sigma Aldrich CO. (USA). These chemicals and all other chemical reagents (NaOH and H₂SO₄) used were of high purity and were used without any purification. De-ionized water was used for preparation of sulfides at 1000 mg/l concentration and as a stock solution for all dilution purposes

II.1. Activation of bentonite:

In a reactor of 500cm³ equipped with a refrigerant, an agitator, and a thermometer, dry bentonite is introduced, followed by sulfuric acid solution. The mixture is then heated to about

100 ° C, temperature is kept constant throughout the process of activation, by the water bath. The attack is further maintained under constant agitation. The contact time is determined from the moment the temperature of the suspension reaches 100 ° C. Activated bentonite is subsequently filtered, then washed with distilled water until all traces of acid have disappeared. The washing is finished when the filtrate no longer gives reaction of the sulphates with barium chloride. Once the bentonite is free of sulphate ions, it is then dried at 105 to 110 ° C, crushed and finally sieved.

II.2. Preparation of LDH-Bent:

About 0.03 mol of divalent cation salt, Co(NO₃)₂·6H₂O, was added to 0.01 mol of trivalent cation salt, Al(NO₃)₃·H₂O put in 500ml round bottom flask. 60 ml of water was added to dissolve the mixture after which it was stirred vigorously at 600 rpm in an oil bath at 60 °C for 15 mins. Subsequently 1 M NaOH was added drop by drop to stabilize the pH at 10 ± 0.5 while maintaining the stirring speed and the temperature at 60 °C. Thereafter, the temperature was raised to 90 °C and stirring increased to 1000 rpm while the reaction was refluxed for 24 h. The suspension obtained was centrifuged and washed twice with deionized water and then followed by two washings using ethanol to remove impurities. The densely-obtained slurry was dried in a vacuum oven at 90 °C for 24 h. The B-CoAl was synthesized via a modification made to the procedure outlined above prior to the coprecipitation reaction stage. At that point, 500 mg of the bentonite in 60 ml of deionized water was ultrasonicated at 60 rpm for 30 min. The mixture was then added to the dissolved mixture of the divalent and trivalent salts after which remaining steps were implemented to produce The 500 mg bentonite used was in a total mixture of 12.98 g which was 3.852% by weight bentonite.

II.3. Batch adsorption experiments:

Batch mode adsorption tests were carried out to explore the effect of pH, contact time, initial sulfide concentration via equilibrium and kinetics studies. Exactly, 5 mg of each adsorbent containing 40 ml of sulfide solution (20–100) mg/l in 50 ml plastic tubes was agitated for 5–240 min at 275 rpm and at different temperatures (25, 35, 45 °C). The initial pH of the solution was adjusted to desired values using 0.1 mol/l H₂SO₄ and NaOH solutions. After agitation, the mixture was centrifuged at 4000 rpm for 10 min to separate the spent adsorbent from the residual sulfide in

solution. The final concentration of the pollutant in the supernatant was quantified by a spectrophotometer set at a wavelength of 530 nm

III. RESULTS AND DISCUSSION

A. Effect of contact time:

The effect of contact time on fluoride removal efficiency of sulfides was studied. It was observed that the sulfide removal gradually increases with time and reaches equilibrium in 10–12 h after which, no significant increase in the fluoride removal was observed. Therefore, 12 h duration was considered as the optimum equilibrium contact time for further adsorption studies.

B. Effect of adsorbent dose:

It was observed (**fig1**) that, sulfide removal efficiency increased from 2.9% to 95.47% with increase in adsorbent dose of 0.2–6 g L⁻¹ of MB, while it was almost negligible in case of bare bentonite. This increase in the sulfide removal efficiency of MB with increase in adsorbent dose was obviously due to the enhancement in the number of activesites available for adsorption of sulfide ions. In the present study, the sulfide concentration below 1.5mgL⁻¹ was achieved at an adsorbent dose of 3 g. L⁻¹ .

C. Effect of adsorbate dose:

It was observed (**fig 2**) that with increase in the initial sulfide concentration, percent removal of sulfide decreases, while the sulfide adsorption capacity increases. This decrease in the percent of sulfide removal is obviously due to the availability of more sulfide ions in solution at higher fluoride concentration, which also indicates that the fluoride binding capacity of MB was almost exhausted. However, at low sulfide concentration,

the ratio of surface active sites to total sulfide is high and therefore the interaction of sulfide

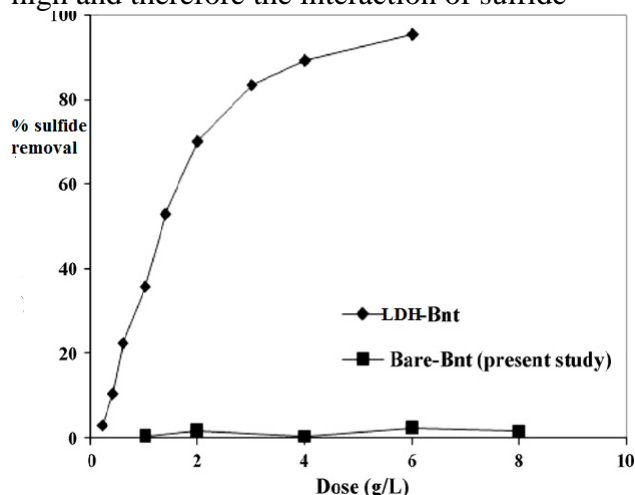


Figure1: Effect of adsorbent dosage on pourcentage of sulfide removal for raw bentonite and modified bentonite

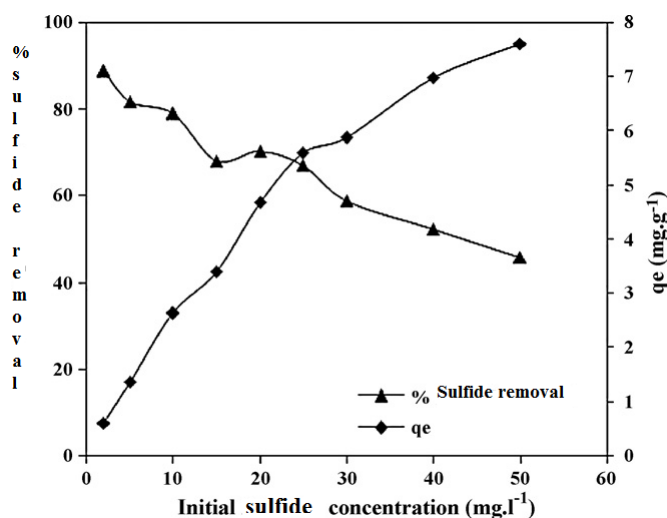


Figure 2 : Effect of sulfide initial concentration on adsorption pourcentage removal

with the active sites on adsorbent surface was sufficient for efficient sulfides removal

D. Effect of pH :

The pH of medium is one of the crucial parameters that can influence the fluoride removal efficiency

significantly, and helps in understanding the fluoride uptake mechanism of the adsorbent. The effect of pH on the sulfide removal efficiency of MB was investigated over the pH range of 3–11. It is evident from the results that MB showed significantly high sulfide removal efficiency over a wide range of pH 3–10. Decline of about 17% in the sulfide removal at pH > 10 was observed which may be due to the abundance of OH⁻ on the adsorbent surface which may affect the sulfide uptake due to extreme competition with OH⁻ (fig 3).

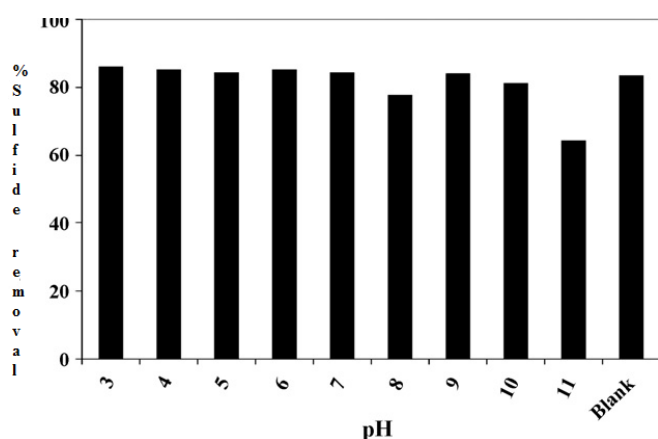


Figure 3 : Effect of pH uptake on sulfide adsorption on raw bentonite and modified one

IV. CONCLUSION

The chemical modification of bentonite clay with magnesium oxide results in significant enhancement in its fluoride removal efficiency. It was found that modified bentonite showed significant fluoride removal efficiency over wide range of pH (3–10). Maximum fluoride adsorption capacity was found to be 2.26 mg g⁻¹ at an initial fluoride concentration of 5.52 mg L⁻¹ favorable.

Negligible leaching of metals was observed from the adsorbent and it is possible to regenerate MB with strong alkali (NaOH) treatment.

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