SYNTHESIS DELAFOSSITE FOR REMOVAL OF PHOSPHATE

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Keywords:

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Introduction:

The high amount of phosphate in water bodies creates an undesirable problem known as Eutrophication reducing dissolved oxygen in water bodies leading to death of aquatic life disturbing ecological balance. The effluent discharged from phosphate manufacturing industries or fertilizer industries without removal of phosphate reduces the quality of water in water bodies. [1] Therefore, it is important to develop technologies which are capable of removing phosphate from water. The delafossite structures are feasible and economical solution for the removal of phosphate. In this study, delafossite is synthesised by coprecipitation method at low temperature at constant pH 11. The synthesized delafossite was characterized by X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), and Fourier Transform Infrared Spectroscopy (FT-IR) analysis. All these analyses confirmed the presence of delafossite structure. Further, experiments were carried out for removal of phosphate with respect to varying dosage of delafossite, varying pH and contact time. Results of these experiments showed that 0.4g was the optimum dosage for phosphate reduction. With variation in pH, maximum removal efficiency was observed at pH 1.4 and contact time 180 min. The efficiency of removal was found to be 94.26%.

Objectives:

1. To synthesize CuFeO2 delafossite by low temperature synthesis method (co-precipitation method).
2. To characterize the delafossite using X-Ray Diffraction (XRD), Scanning electron microscope (SEM), and Fourier-transform infrared spectroscopy analysis (FT-IR).
3. To study the efficiency of removal of phosphate in synthetic water for varying dosage, pH and contact time of delafossite.

Methodology:

Chemicals used are cupric sulphate (〖C 〗 _U SO_4.5H_2 O SDFCL chemicals 99% assay), ferrous sulphate (〖Fe〗 _2 SO_4.7H_2 O SDFCL chemicals 99%

assay), Sodium hydroxide (NaOH SDFCL chemicals 97% assay), potassium dihydrogen phosphate (avra).

Instruments used during synthesis are Magnetic stirrer with hot plate (Remi make), pH meter (Systronics - µ pH system 362). XRD (Japan smartlabSE), SEM (jeol JSM-IT500LA), FTIR (spectrum 2 FT-IR/ SP 10), tube rotator (M/s PSM scientific systems Hubballi), spectrophotometer (shimandzo M/s PSM scientific systems Hubballi) are used during water treatment.

Method : Synthesis of Delafossite

The Delafossite were synthesized by the co-precipitation method at pH 11. Then 9.8218g copper sulphate is dissolved in 250 ml of distilled water in the beaker. Then solution is heated to temperature 700C. While stirring continuously 12.437g ferric sulphate is added. pH 11 is maintained using NaOH and stir continuously at 700C. Afterwords the solution containing the precipitates is filled in conventional laboratory bottle and stored at 700C for 24h. After this process, this solution is filtered and washed with distilled water and then kept in oven for drying at 500C for 24 h. After this process delafossite is obtained. Then characterization of sample is done by XRD. After characterization of Delafossite, degradation of phosphate by using synthesized Delafossite and evaluating the efficiency of degradation of phosphate.[2]

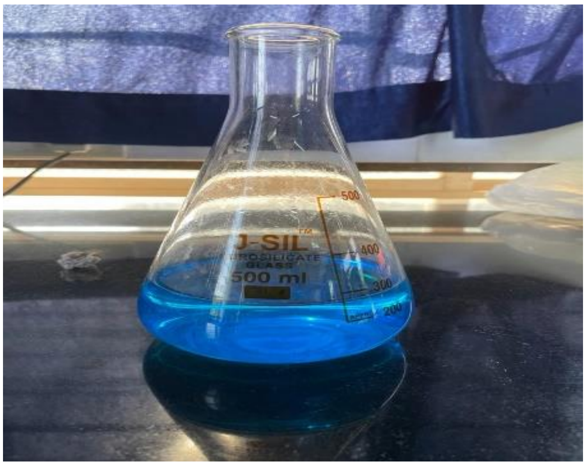


Figure 1: Solution of Copper Sulphate and 250 ml Distilled Water

Figure 2: Magnetic stirring

Figure 3: Filtration process delafossite

Figure 4: Dried sample of

Batch studies

All the experiments are carried out with one control and samples in duplicate. Initial concentration of the solution is 65mg/L and volume is 50mL. The first set of experiment is done with varying dosage of 0.1g, 0.2g, 0.3g, 0.35g, 0.4g for 30min at natural pH (6.1). In the second set of experiments, the experimentation is carried out varying contact time from 0 to 180min with time interval 30min.The third set of experimentation is done with varying pH at 1.4, 6.1 and 9.8. The thorough mixing of solution during the experiment is done using tube rotator by end-to-end rotation. After that the solution is filtered and analysed for phosphate concentration.

Results:

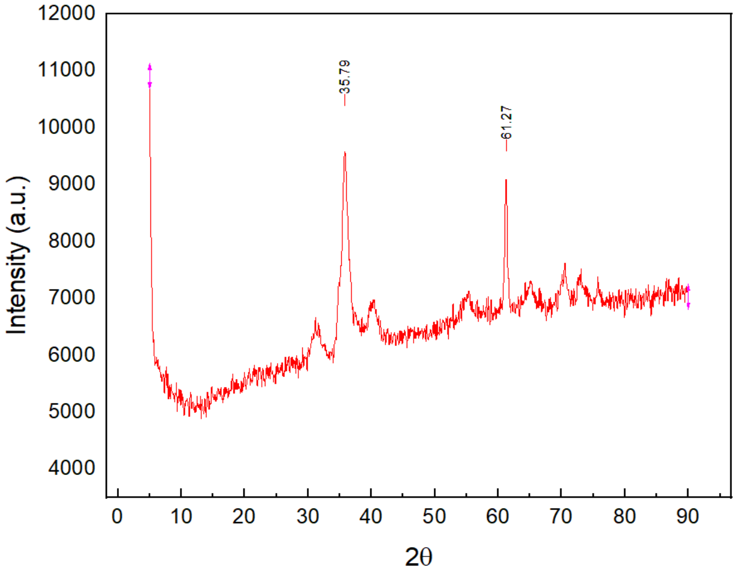


Figure 5: XRD Result of pure delafossite

The peak at pure delafossite 35.79° is matching with the JCPDS card number 85-0605, wavelength 1.54060 with the chemical formula CuFeO2. From this result of XRD patterns both before and after treatment, we can conclude that in acidic pH of phosphate solution there is an interaction with metal cations (Fe+3 and Cu+2) to form precipitate and the presence of phosphate might react with ferric ions to form insoluble ferric phosphate precipitate. This would effectively remove some phosphate from the solution.[3]

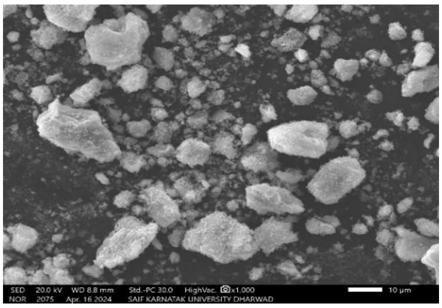


Figure 6: Showing SEM Image Of pure delafossite

SEM images of delafossite samples show their typically thin hexagonal plates Most of crystallites are well-shaped with smooth surfaces and defined edges in raw sample of delafossite. All of these results are in good agreement with XRD results of delafossite. In acidic, alkaline and natural pH the morphology of metal structure is changed compared to the pure delafossite and the number of thin hexagonal plates have reduced as reaction is taking place in reduction of phosphate however there is a need of higher magnified images to study the fate of metals during the reaction.[4]

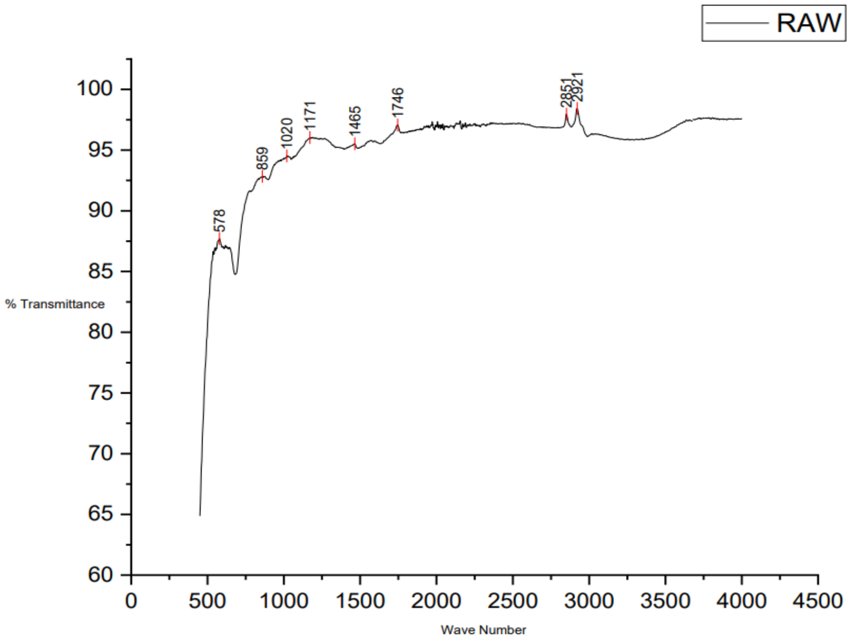
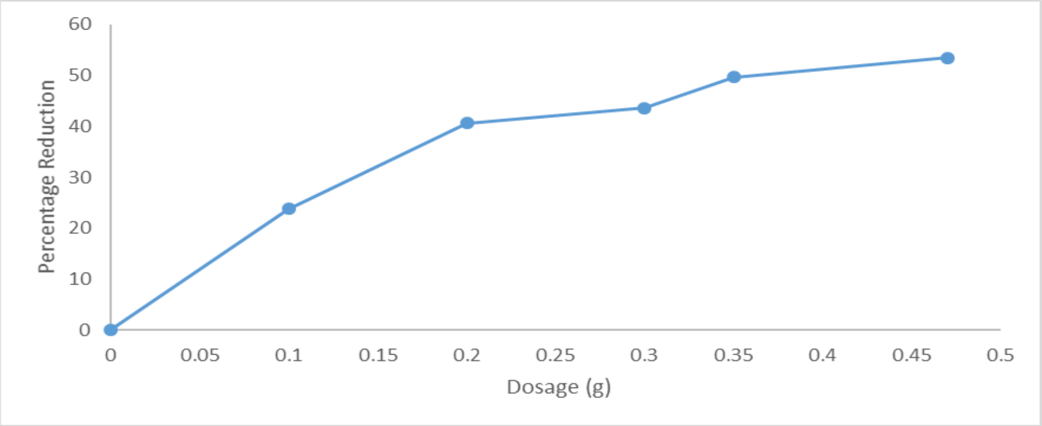


Figure 7: FTIR Results of Raw Sample

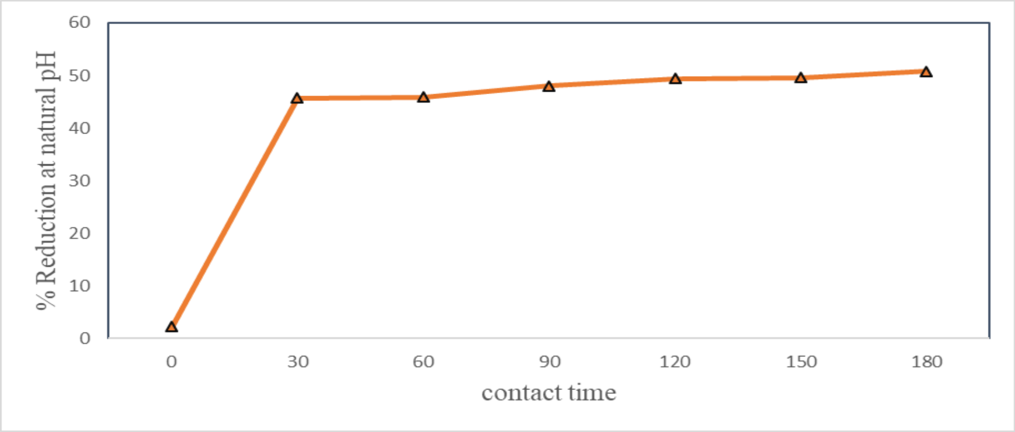
Effect of Delafossite Dosage

The graph is plotted between concentration of phosphates with respect to dosage of delafossite and the graph of percentage reduction of phosphate versus dosage of delafossite. As dosage increases the surface area of delafossite increases, thus have larger contact area with reactants hence the removal ratio of phosphate is increased with increasing amount of dosage. The maximum phosphate reduction of 53.06 % is observed at 0.4g of delafossite dosage.



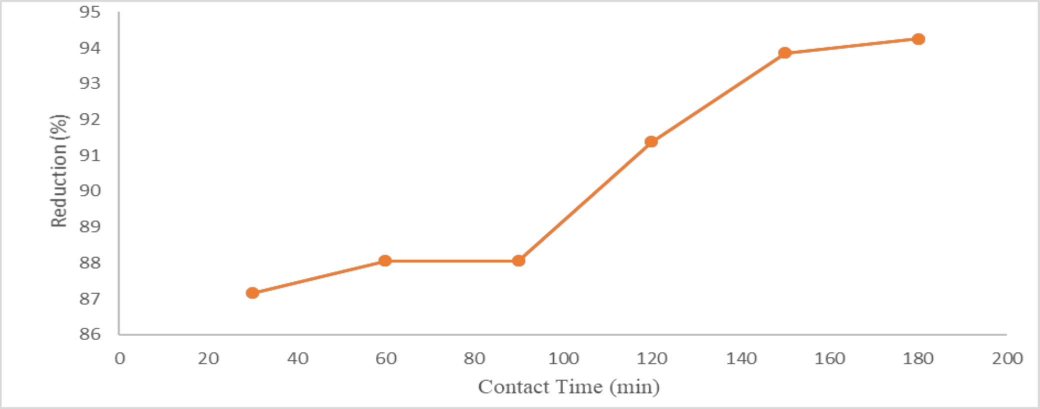
Effect of Contact Time

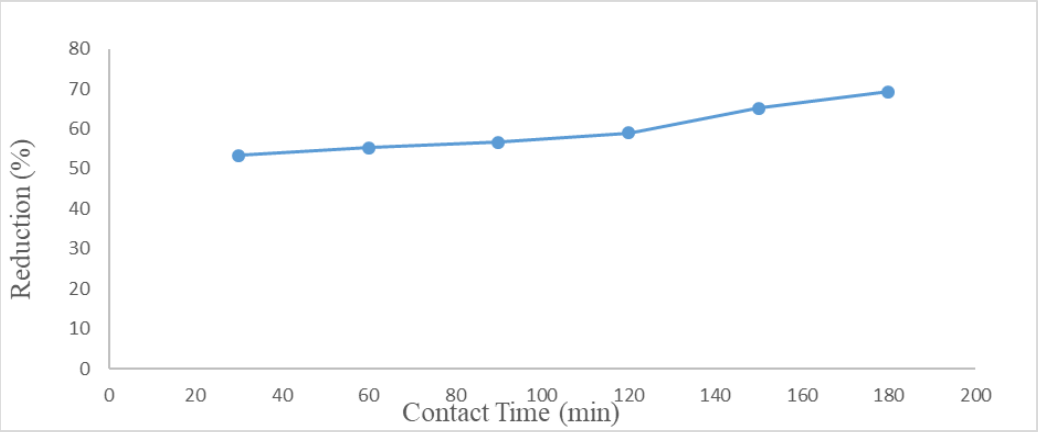
The maximum phosphate reduction is observed at 0.4g dosage, so 0.4g dosage is selected for the next experiment to determine variation of phosphate at different contact time. The procedure which is conducted to determine variation of phosphate reduction with respect to dosage, similar procedure is done here. The values of percentage phosphate reduction are noted at 30min, 60min, 90min, 120min, 150min, and 180min. The graph plotted between concentration of phosphate with respect to contact time. And the graph plotted between percentage phosphate reduction versus contact time. As the contact time increases, the phosphate reduction increases rapidly. There is rapid decrease of phosphate in first 20minutes and then the reaction reaches equilibrium at 150 minutes.



Effect of pH

The reduction at pH 1.4, 4, 6.1 and 9.8 are 94.26%, 69.2%, 50.81%, 28.91% respectively the contact time is 180min and dosage is 0.4g. It is observed that reduction in high in acidic range. The phosphate ion reduction is highest for acidic phosphate solution with 94.26% efficiency and the least reduction of 28.91% degradation is observed at alkaline phosphate solution i.e. at pH (9.8). The removal efficiency is low in alkaline media due to the formation of ferric hydroxide in Fenton reaction which slows down the degradation of phosphate.





Conclusion:

This work proposes the application of delafossite for the removal of phosphate from synthetic water. Synthesis of delafossite by co-precipitation method is feasible and cost effective.

The XRD results confirm that the synthesis method proposed is the efficient method to obtain delafossite material. In treatment part, phosphate removal experiments indicated that, the percentage reduction of phosphate increases with increase in dosage of phosphate. When using a dosage of 0.4g in synthetic phosphate solution, delafossite reached a phosphate reduction of 50.81% at pH 6.1. It did not yield good results in alkaline range i.e. at pH 9.8. When phosphate reduction experiments were conducted with respect to contact time using 0.4g of delafossite, and maximum reduction was observed to be 94.26 % at 180min at pH 1.4, On the basis of above results, delafossite could be used as potential material for phosphate removal in water.

Scope for future work:

This work can be carried out further by modifying delafossite like doping it with silver, alumina, cerium dioxide etc to degrade different pollutants like dyes, pesticides, bacteria, pharmaceutical waste and literature study shows that degradation efficiency of delafossite increases by doping it with different materials and delafossite can be modified to nano scale. Further column experiments can be carried out for practical implementation.

Reference:

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