

***STUDIES ON INTAKE EFFICIENCY OF ARSENIC (V)
BY CHEMICALLY MODIFIED SILICA GEL***

A Dissertation

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FOR THE DEGREE OF

MASTER OF SCIENCE

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Under Academic Autonomy

**NATIONAL INSTITUTE OF TECHNOLOGY,
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CERTIFICATE

This is to certify that the dissertation entitled “*Studies on Intake Efficiency of Arsenic(V) by Chemically Modified Silica Gel*” submitted by Subhraseema Das of the Department of Chemistry, National Institute of Technology, Rourkela for the degree of Master of Science in Chemistry is based on the result obtained in the bona fide project work carried out by her under my guidance and supervision.

I further certify that to the best of my knowledge Subhraseema Das bears a good moral character.

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Subhraseema Das

ABSTRACT

A laboratory study was conducted to investigate the ability of modified silica gel for the removal of arsenate from synthetic arsenate solution. The silica gel was modified by the standard procedure and was characterized by SEM, FTIR, Chemical analysis and XRD. To know the practical applicability, a detailed removal study of arsenate ion was undertaken. The removal of arsenate was 99.87% under neutral condition and using 0.3 gm of adsorbent in 100 ml of arsenate solution having initial concentration of 1 mg/L, 5 mg/L, 10 mg/L, 50 mg/L respectively. The percentage removal increases with increase in pH till it reaches 5.0 then decreases. The process was rapid and equilibrium reaches within first 45 minutes.

INTRODUCTION

Silica gel is a granular and highly porous form of silica. The chemical compound silicon dioxide is basically known as silica (latin for silex) has been known for its hardness since time immemorial. Silica basically occurs in nature as sand or quartz. Silica is the most abundant mineral in the earth crust. Silica is used primarily in the production of window glass, drinking glasses and beverage bottles. The majority of optical fibers for telecommunications are also made from silica. It is a primary raw material for many white ware ceramics such as earthenware, stoneware and porcelain as well as industrial Portland cement.

Silica is a common additive in the production of foods, where it is used primarily as a flow agent in powdered foods, or to absorb water in hygroscopic applications. It is the primary component of diatomaceous earth which has many uses ranging from filtration to insect control. It is also the primary component of rice husk ash which is used, for example, in filtration and cement manufacturing.

In the recent era, arsenic is creating potentially serious problems to the environment and other living organisms. Arsenic contamination is also caused by human activities such as mining wastes, agricultural chemicals, ceramic manufacturing industries, petroleum refining sewage sludge and fly coal ash [1].

Objectives:

The present work was carried out with the following objectives:

- Synthesis and characterization of modified silica gel.
- Studies on the removal efficiency of arsenate from water by this material.

EXPERIMENTAL*Chemical modification of silica gel*

The chemical modification of silica gel was carried out by following a reported standard procedure [2].

Synthesis of chelating material Si-DDS-o-HB

Fifty grams of the above 3-chloropropylsilica gel (Si-Cl) were immersed in 200 cm³ of methanol and reacted with 2.64 g (10 mmol) of 4,4'-diaminodiphenylsulfone (DDS) under reflux condition, in the presence of for nearly 4 h. The product (Si-DDS) so formed was filtered, washed thoroughly with methanol and dried *in vacuo* at 60 – 70°C. In the subsequent step, 40 g of the product Si-DDS was made to react with *o*-hydroxybenzaldehyde(*o*-HB) (5 cm³, 0.6 mmol) to yield the respective Schiff base, which was the desired chelating material Si-DDS-*o*-HB. The color of the chelating material was yellowish brown. The material was filtered off, washed with methanol, and dried *in vacuo* at 60 – 70°C.

Preparation of As (V) solution

Stock solution of As (V) for sorption studies was prepared from AR grade of sodium arsenate by dissolving in double distilled water. Solutions of various concentrations such as 1ppm, 5ppm, 10ppm and 50ppm were prepared of the stock solution. Adsorption on the modified

silica gel was carried out in a 100 ml conical flask at room temperature by taking appropriate concentration of the arsenic solution. . A number of parameters such as adsorbent dose, concentration of arsenate, pH, affecting the removal of arsenate ion have been varied widely in order to optimize the removal process.

RESULTS AND DISCUSSION

The IR spectra of the silica gel and the chelating material are presented in the fig.-1 & 2 respectively which clearly indicates the formation of chelating materials with the organic components. The chemical analysis of the chelating materials confirms the formation of the materials.

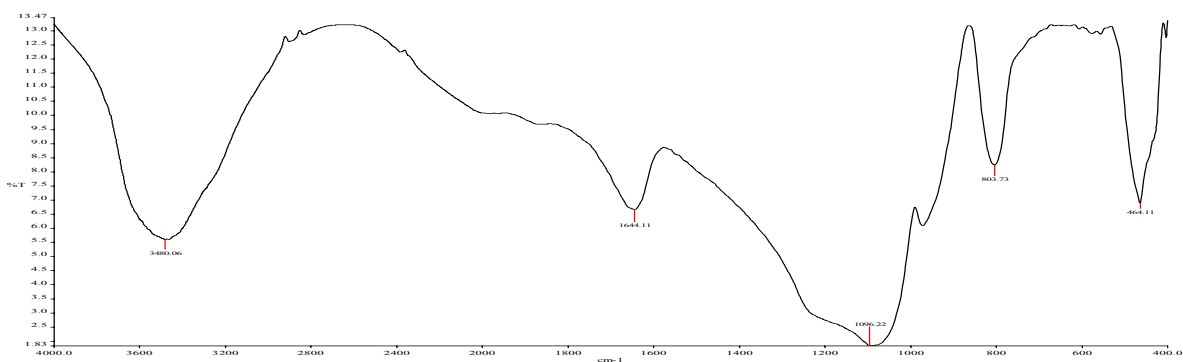


Fig-1:IR spectrum of silica gel

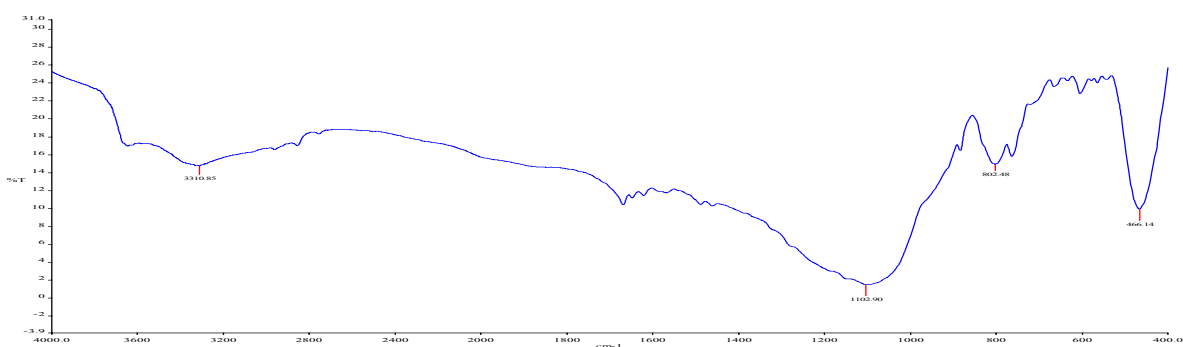


Fig-2 IR spectrum of chelating material

Removal study of arsenate by batch mode experiments

Effect of adsorbent dose

The effect of variation of adsorbent dose on percentage removal of arsenate from aqueous solution with modified silica gel used in this study is graphically shown in Fig. 3. It is evident from the figure that the removal of arsenate increased and reaches the maximum at 0.3 g in 100mL of synthetic solution of initial arsenate concentration, 1mg/L, 5 mg/L, 10 mg/L, and 50 mg/L respectively. However it is observed that after dosage of 0.30g/100mL, there was no significant change in percentage removal of arsenate. This may be due to the overlapping of active sites at higher dosage. So, there wasn't any appreciable increase in the effective surface area resulting due to the conglomeration of exchanger particles. So, 0.30 mg/100mL was considered as optimum dose and was used for further study.

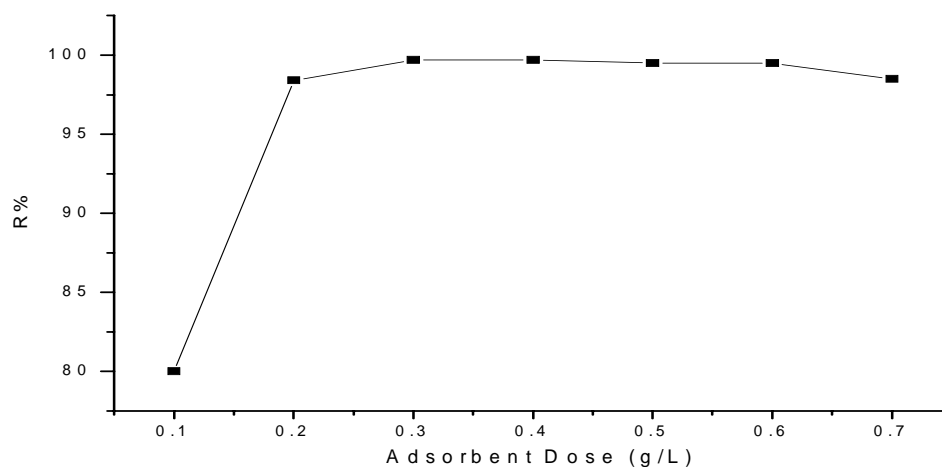


Fig.3. Removal of arsenate versus adsorbent dose

Effect of pH

Percentage removal of arsenate was studied at ambient temperature (25 ± 2 °C) and contact time of 45 minutes for initial arsenate concentration of 1mg/L, 5 mg/L, 10 mg/L, and 50 mg/L at different pH. The results are presented in Fig. 4. It is evident from the figure that the percentage removal of arsenate by modified silica gel increases till it reaches pH than decreases as it is evident from the figures-4. This may be due to the fact that with increase in pH, With increase in pH of the medium, the metal sorption of the chelating materials

increased due to the increase in coordination ability for the formation of phenoxide ion upon phenolic OH-group and due to the increase in basicity of the imine nitrogen atom[3].. The above data suggests that the optimum pH for removal of arsenate was 5.

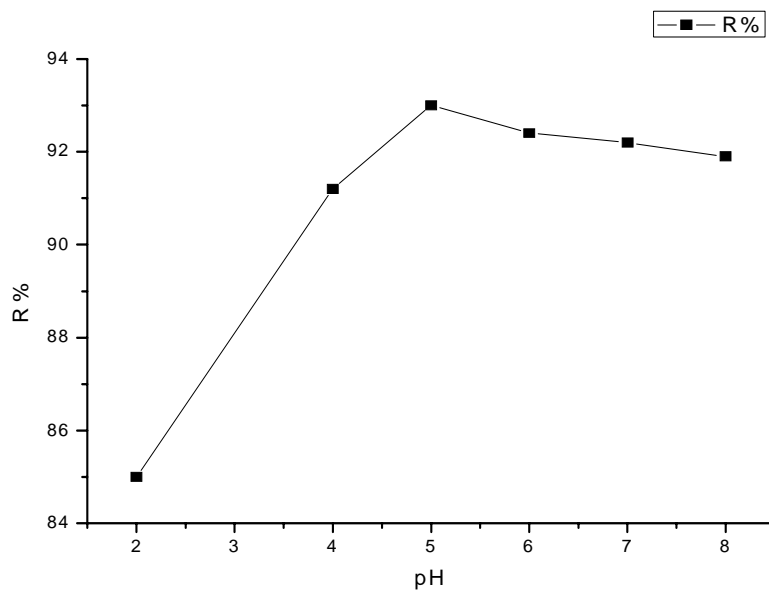


Fig-4. Effect of pH on the removal of arsenic

SEM micrographs

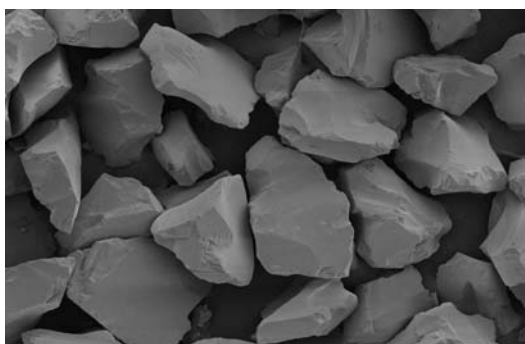


Fig-5

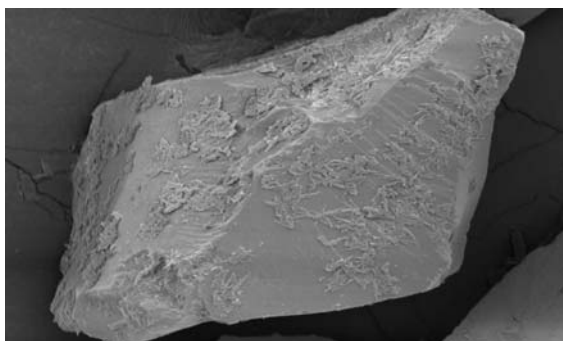


Fig-6

The SEM micrographs of the chelating material was taken before and after the adsorption processes. Fig-5 shows the SEM micrograph of the adsorbent before the process of adsorption. Fig-6 represents the SEM micrograph of the material after adsorption which clearly reveals the incorporation of arsenic ions into the chelating material matrix. The EDS spectra of the material with unloaded (Fig-7) and loaded (Fig-8) metal ion is shown below.

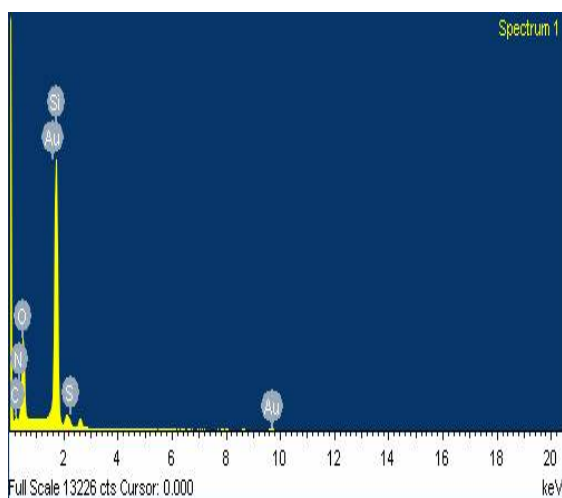


Fig-7

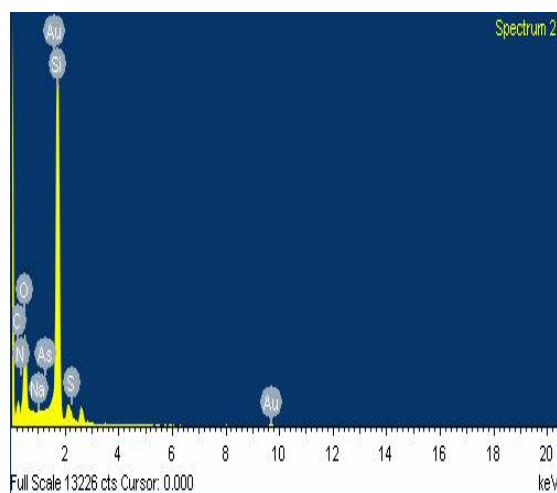


Fig-8

CONCLUSION

The chelating material (Si-DDS-o-HB) was synthesized by modifying the activated silica gel phase. The material was characterized and tested for the pre concentration of As(V) in batch techniques. The material was found to be effective in removing almost 99.87% of metal ion from the solution. The other detailed study to know the practical applicability of the above process is undergoing.

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