

Synthesis and Characterization of Polymer Nano-Composite for Heavy Metal Removal

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Abstract:-- A ternary Nano-composite using kaolin clay- activated carbon (bagasse) – chitosan was prepared via sol-gel method. The raw kaolin clay was modified by using aluminium nitrate and it was characterized using Fourier Transform Infrared Spectroscopy (FT-IR). Activated carbon was synthesized from bagasse by KOH activation. The synthesized activated carbon was characterized by Fourier Transform Infrared Spectroscopy (FT-IR) and Scanning Electron Microscope (SEM). The particle size, surface area and porosity of the activated carbon were also measured. The Nano-composite prepared was characterized by using Fourier transform infrared analyzer (FT-IR) and Scanning electron microscope (SEM). The particle size, surface area and porosity of the Nano-composite were measured. The surface area of the ternary nanocomposite from Brunauer-Emmett-Teller analyzer (BET) was found to be 1957m²/g. Batch wise adsorption tests were conducted for different concentrations of the nickel effluent, for different residence time with constant amount of the prepared Nano-composite. The nanocomposite exhibited excellent performance of nickel removal from the aqueous solution. The Atomic Absorption Spectroscopy results revealed that upto 99% adsorption efficiency was achieved in 200ppm of nickel effluent for 0.5g of adsorbent. The Atomic Absorption Spectroscopy analysis was conducted for 200,400,600,800 and 1080 ppm of nickel effluent with 0.5g of the adsorbent for a contact time of three days and seven days. After three days the concentration of the nickel effluent was found to be reduced and at the seventh day the concentration of nickel effluent was increased when compared to the concentration of the nickel at the end of three days. This explains the phenomenon of desorption. The cause of desorption is due to the increase in temperature. Hence the optimum contact time for this adsorption process was found to be three days.

Keywords:-- Chitosan, Clay, Activated carbon, Nano composite, Nickel effluent, Batch adsorption.

INTRODUCTION

Industrial waste water may contain a large number of heavy metals in it. Nickel, one among them is a non-biodegradable toxic heavy metal ion present in waste water. The main source of nickel pollution is in the water drives from the processes such as galvanization, smelting, mining, dyeing, batteries manufacturing and metal finishing. Trace amount of nickel are beneficial to human body as an activator of some enzyme systems, but if it is beyond the scope of normal levels it causes different types of diseases such as lung cancer, renal edema and gastro intestinal disorder. For this reason, the WSH (Water Sanitation and Hygiene) under the WHO (World Health Organization) established the toxic limits of permissible concentrations of nickel at a level of Ni(II) and insoluble compounds of 1.0mg/m³, nickel carbonyl of 0.05-0.12 mg/m³, and nickel sulphide of 1.0 mg/m³. The conventional methods for removal of Ni(II) from water include chemical oxidation or reduction, chemical precipitation, ion exchange, membrane separation, filtration, electrochemical treatment and adsorption, etc. As an economical and efficient method, adsorption technique has been widely used to remove heavy metal ions from water.

A Nano composite is a multiphase solid material with one of phases having dimensions less than 100 nanometers in one, two or three dimensions or structures having nanoscale repeat distances between the different phases that makeup the material. Nowadays a considerable interest is developed in polymer nanocomposites. Varieties of nanoparticles can be incorporated into the polymer matrix to form polymer nanocomposites.

Here we have selected our composite with components kaolin clay- activated carbon from bagasse- chitosan. These are going to be the components for the polymer nanocomposite using which Nickel is adsorbed.

RAW MATERIALS

Kaolin clay, Aluminium nitrate, Bagasse, Potassium hydroxide, Chitosan powder, Acetic acid, Ethanol, Tetraethylorthosilicate, Hydrochloric acid and Distilled water.

Experimental Procedure

Modification of kaolin clay

Kaolin clay was modified using Aluminium nitrate solution Al(NO₃)₃.

Blending

5 ppm of aluminium nitrate solution was prepared by dissolving 5 g of $Al(NO_3)_3$ in water and made up to 100ml in a standard flask.

Stirring

12 g of kaolin clay was weighed, dissolved in aluminium nitrate solution and stirred in magnetic stirrer for 24 hours at room temperature.

Drying

The solution was allowed to settle, filtered and washed several times using distilled water. The residue (clay) was then dried in a hot air oven for 10 hours at 100°C and it was powdered.

Preparation of activated carbon from bagasse

Raw bagasse was collected from local sugarcane juice vendors.

Pretreatment of bagasse:

The bagasse collected was first cut manually into smaller pieces and it was left to be sun dried for 4 days. It was then dried in a Hot air oven at 100oC for about 10 hours. The dry bagasse obtained was then powdered and sieved (mesh no - 12+14)

Activation of bagasse:

Blending

About 40g of powdered bagasse was activated using 10g of KOH. The mixture was dissolved in 500ml of pure distilled water.

Stirring

The solution consisting of bagasse and KOH was stirred and heated in a magnetic stirrer at 85oC for 5 hours.

Incineration

The above mixture was taken in a silica crucible and kept in a muffle furnace at 500oC for an hour.

Drying

The product obtained was washed with distilled water, filtered and then dried.

Thus, the activated carbon from bagasse was obtained.

Preparation of chitosan solution

Blending

0.5g of chitosan was dissolved in 10% acetic acid.

Stirring

The above chitosan solution was then stirred for an hour in a magnetic stirrer.

3.2.4 Synthesis of Composites

Nanocomposites was prepared by Sol gel method.

Blending:

Precursor solution was prepared by adding 30 ml of ethanol, 1 ml of distilled water, 0.5 ml of HCl, into 46.5 ml of tetraethylorthosilicate (TEOS).

Stirring

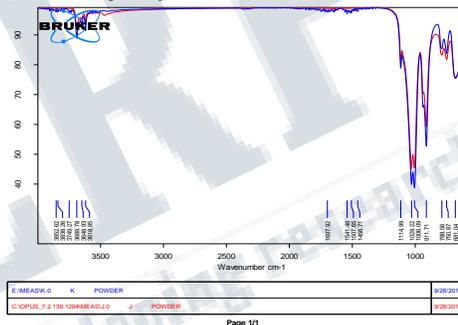
The solution was stirred for 10 minutes in magnetic stirrer.1 gram of modified clay, 0.5grams of activated carbon, chitosan solution was added dropwise into the precursor solution. The solution was stirred in a magnetic stirrer for 10 hours and allowed to settle for a day.

Drying

The composite was dried in hot air oven for 5 hours at 70°C.

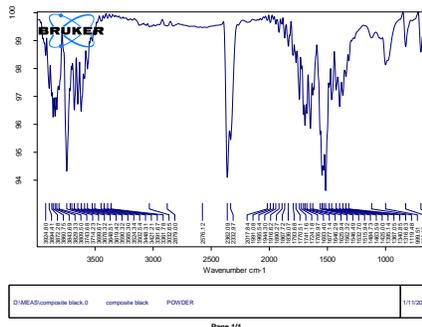
RESULTS AND DISCUSSION

Characterization of clay (FTIR)



The FTIR analysis of kaolin clay revealed the presence of various functional group. The broad region 3852-3615 cm^{-1} was due to the presence of O-H group. The adsorption band corresponds to 1697-1456 cm^{-1} was due to C=O group. The broad band located at 1456-1114 cm^{-1} was assigned to N-O group. The peak 911-681 cm^{-1} was attributed to C-H group. From the above results, we conclude the presence of the desired functional groups.

Characterization of activated carbon FTIR



The presence of functional group in the activated carbon can be analyzed by using FTIR. The sharp peak in the region

3924-3200 cm^{-1} depicted the presence of O-H group which indicated the carbon activation. The broad region from 3200-2879 cm^{-1} indicated the presence of C-H. The peak observed from 2362-2017 cm^{-1} corresponds to the presence of $\text{C} \equiv \text{C}$. The adsorption peak corresponds to 1919-1625 cm^{-1} was due to C=O group. The sharp peak in the region 1562-1425 cm^{-1} was assigned to aromatic ring stretch.

Scanning Electron Microscope(SEM)

Particle size of the synthesized activated carbon was found to be 106.3nm using the Scanning Electron Microscope. The activated carbon is in nano scale. The adsorption capacity and surface area tends to increase as the particle size decreases.

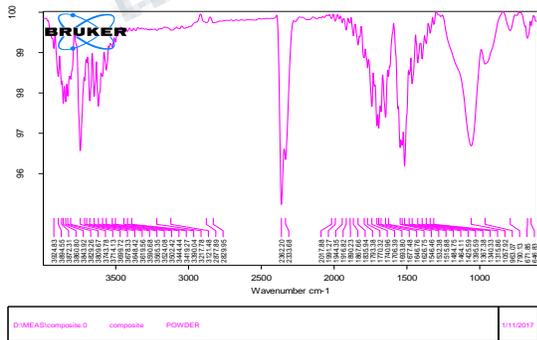
Porosity of the activated carbon was measured using Brunauer – Emmett – Teller analyzer and it was found to be 0.6 cm^3/g . Higher, the porosity higher the presence of void space and this indicates the availability of space for the particles to get adsorbed. Surface area of the activated carbon was found to be 1218 m^2/g from the Brunauer – Emmett – Teller analyzer. Since the surface area is larger the adsorption rate and efficiency also increases. This also indicates the presence of more number of activated sites for high surface reaction.

Hence high values of porosity and surface area ensures effective chance of adsorption.

Characterization of composites

FTIR

The FTIR analysis of synthesized composite revealed the presence of various functional group. The broad peak from 3924-3590 cm^{-1} indicated the presence of O-H. The sharp peak in the region 3565-2829 cm^{-1} depicted the presence of C-H group. The adsorption peak corresponds to 2362-2333 cm^{-1} was due to $\text{C}=\text{C}$ group. The broad region from 2333-2017 cm^{-1} indicated the presence of $\text{C} \equiv \text{C}$. The peak 1991-1626 cm^{-1} is attributed to C=O group. The sharp peak in the region 1484-1057 cm^{-1} was contributed by N-O group. The peak observed from 983-643 cm^{-1} corresponds to the presence of C-H.



Scanning Electron Microscope- SEM

Particle size of the synthesized ternary nanocomposite was found to be 0.1mm using the Scanning Electron Microscope. The adsorption capacity and surface area tends to increase as the particle size decreases.

- Porosity of the synthesized nanocomposite was measured using Brunauer – Emmett – Teller analyzer and it was found to be 0.97 cm^3/g . Higher the porosity higher the presence of void space and this indicates the availability of space for the particles to get adsorbed.

- Surface area of the nanocomposite was found to be 1957 m^2/g from the Brunauer – Emmett – Teller analyzer. Since the surface area is larger the adsorption rate and efficiency also increases. This also indicates the presence of more number of activated sites and high surface reaction.

Hence high values of porosity and surface area ensures effective chance of adsorption.

Treatment of nickel effluent

The synthesized composite was utilized for the adsorption of heavy metal nickel from industrial effluent.

Nickel effluent was obtained from the electroplating industry. The effluent was analyzed by Inductively Coupled Plasma Mass Spectrometry (ICPMS). The concentration of effluent was found to be 1080 ppm.

Batch adsorption:

Nickel effluent was diluted to different concentration 200ppm, 400ppm, 600ppm, 800ppm.

| Concentration of the samples (ppm) | Concentration of the samples after 3 days (ppm) | Concentration of the samples after 7 days (ppm) | Efficiency |
|------------------------------------|---|---|------------|
| 200 | 2.8 | 9.68 | 98.6 |
| 400 | 23.2 | 29.7 | 94.2 |
| 600 | 50.6 | 61.4 | 91.56 |
| 800 | 78.9 | 90.1 | 90.13 |
| 1080 | 150 | 254.6 | 86.1 |

Adsorption test

About 0.5grams of composite was weighed and it was transferred to each 20 ml of samples and stirred for two hours in magnetic stirrer and centrifuged. Atomic Absorption Spectroscopic analysis was performed on each sample at the end of third and seventh day. The readings were tabulated and plotted below.

CONCLUSION

To summarize we have successfully synthesized polymer nanocomposite (kaolin clay- activated carbon- chitosan) via sol -gel method and characterized them. The SEM analysis clearly exhibit the vast arrangements of pore size showing that Bagasse could be an excellent waste material for the preparation of activated carbon. The presence of various functional groups were identified using FTIR. The BET analyzer was used to find the surface area of both the activated carbon as well as the composite. Analysis revealed that the composite prepared was found to be efficient adsorbent. Batch tests were conducted to find the adsorbing nature of the composite towards Ni ions in aqueous medium. The batch test was conducted with 200, 400, 600, 800 and 1080 ppm of the nickel effluent using a constant amount of the composite of 0.5 grams. The reduction of nickel concentration was found at different times using AAS (Atomic absorption spectroscopy). From a series of tests the optimum adsorption time was found to be 3 days. The concentration of Ni(II) ion was increased when the contact time was increased to 7 days this explains the action of desorption. Hence, we conclude that the nanocomposite prepared was efficient in removing the nickel ion from industrial effluent which has an adverse effect on the respiratory system of the human beings.

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