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Application of 4-HADTOT-HCAC Composite for Removal of Cd(II) from Contaminated Water

Dongapure A.C.[1*], Rahangdale P.K.[2] and Burghate P.D.[3]
[1]Shankarlal Agarawal College, Salekasa-441916, India
[2]Bhawabhuti College, Amgaon-441902, India
[3]Science College, Congress Nagar, Nagpur-440013, India
avinashdongapure888@gmail.com

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Abstract

The present research article reports the practical applicability of newly obtained 4-HADTOT-HCAC Composite for removal of toxic divalent Cadmium from contaminated water. Initially 4-HADTOT tercopolymer was synthesized by acid catalyzed condensation polymerization method using 4-Hydroxy Acetophenone (4-HA), Dithio Oxamide (DTO) and Trioxane(T) as starting materials. Activated carbon derived from Hibiscus cannabinus fruit shell (HCAC) was generated using known methods. The new Composite material was obtained from 4-HADTOT and HCAC and it has been abbreviated as 4-HADTOT-HCAC. The resulting Composite was characterized by SEM and FTIR spectroscopy studies. Adsorption studies for removal of Cd(II), a toxic pollutant, were conducted in the laboratory. The optimum conditions like adsorbent doses, agitation time, initial metal ion concentration and pH on adsorption of Cd(II) by 4 -HADTOT-HCAC Composite were investigated. AAS was used to determine Cd(II) concentration. At 300K temperature and pH 6, 95% of the Cd(II) was removed from metal ion solution (25 ml, 0.1 mg dm^{-3}). Thus 4-HADTOT-HCAC Composite has been proved to be the new promising excellent material for waste water treatment with special reference to removal of Cd(II).

Keywords: Tercopolymer, Activated carbon, Composite material, Metal pollutants, Cadmium toxicity, Waste water treatment.

Introduction

From the last few decades pollution of water arises due to the disposal of heavy metals from industries. It is well known that Cadmium has harmful effects on many life forms. In recent years this problem has significant concentration. Therefore, it becomes necessary to remove cadmium from wastewater by using appropriate treatment before releasing it into the environment. The acute over exposure to cadmium fumes can cause pulmonary diseases and chronic exposure can cause renal tube damage and prostate cancer[1]. Cadmium and their salts are used in electroplating, paint pigments, plastics, silver cadmium batteries[2]. The commonly used methods for removing metal ions from waste water include precipitation, lime-coagulation, reduction, electrolytic removal, ion-exchange, reverse osmosis, membrane filtration and solvent extraction[3-10]. Use of surface modified/chitosan coated bio-sorbent as low cost material for abatement of Cr (VI) has been reported in the literature[11-12].

Existing commercial sorbents including, activated carbon, activated alumina, zeolites and silica gels play important roles in adsorptive separation and purification. However, innovative technological developments are needed in the new economy and under the stringent environmental regulations. Despite of very promising features of the newly developed Composite structured sorbent materials, basically exploring and systematic investigations are needed on both synthesis methods and adsorption characteristic studies[13].

Now days, polymer doped activated Carbon Composites have been studied increasingly as an alternative adsorbent in water treatment. Saifuddin et al. have studied a polymer based Composite for the removal of contaminant from water[14-16].

The present investigation reports the synthesis of 4-HADTOT polymer, generation of new HCAC activated carbon, to develop new 4-HADTOT-HCAC Composite material, characterization followed by systematic study towards application of this new Composite material for removal of Cd(II) employing batch experiments.

Materials and Methods

Chemicals: The entire chemicals used were procured from Merck, Mumbai, India and were of analytical or chemically pure grade. Deionized/distilled water was used thought the investigation.

Synthesis of 4-HADTOT polymer: The polymer 4-HADTOT was synthesized by the polymerization reaction of 4-hydroxyacetophenone (0.6mol) and Dithio Oxamide (DTO) (0.3mol) with Trioxane (0.1mol) using hydrochloric acid as the reaction medium at 124±2°C in an oil bath for 5 hrs under refluxed condition with occasional shaking. The reaction mixture was then cooled and the separated polymer out was washed with warm water followed by extraction with diethyl ether. The dried sample was then purified by dissolving in 10% aqueous NaOH and regenerated using 1: 1 (v/v) HCl/water with constant and rapid stirring to avoid lump formation. The process of re-precipitation was repeated twice. The polymer 4-HADTOT thus obtained was filtered, washed with hot water, dried in air, powered and kept in vacuum desicator over silica gel. The yield of the polymer was found to be 92 %.

Generation of Activated Carbon from the *Hibiscus cannabinus* fruit shell (HCAC): The fruit shell of *Hibiscus cannabinus* species was collected from the agriculture waste[1]. Then fruit shell was cut into little part, and then washed with water to clean from the sand particles and to avoid release of any colour of fruit shell into aqueous solution, cut fruit shell applied with formaldehyde then again cleaned numerous times with de-ionized water. Then it was sun dried for 6 days. After completely drying, the cut fruit shell of *Hibiscus cannabinus* material was subjected to pyrolysis for carbonization using muffle furnace at 800-9000^{0}C for about 7 to 8 hr so that it was converted into a char. Then microwave activation of char was done using microwave oven. Set at 360 W for 30 min the microwave equipment input power. The activated carbon was obtained grounded and sieved in 120-200 mm particle size. Then it was washed with double distilled water and dried at 105°C for 3 hr.

Synthesis of 4-HADTOT-HCAC Composite: 4-HADTOT polymer was dispersed in N-N Dimethylformamide using mechanical stirrer for 75 min at room temperature. Then HCAC was added and stirring was continued for 6 hrs. The temperature of the system was raised to 65°C and kept overnight so that the solvent was evaporated completely. Equimolar polymer-activated carbon composition was taken in the Composite. The solid 4-HADTOT-HCAC Composite was characterized by FTIR, SEM and XRD Studies. The morphological properties were investigated from SEM picture and thermal properties from TG analysis.

Preparation of stock solution: Stock solution of Cd(II) was prepared by dissolving required quantity of Cadmium Sulphate salt in the distilled water. This solution was diluted to proper proportions to obtain various standard solutions ranging their concentrations 10-100mgl^{-1}. Adjustment of pH was done using 0.5N HCl and 0.5N NaOH solution.

Batch Experiment: This experiment studies were performed with varied parameters such as pH, Cd(II) initial concentration solution and effect of adsorbent doses. The systems were agitated on rotary shaker at 200 rpm, filtered through Whatmman no.42 filter paper and filtrate was analyzed for Cd(II) concentration using Atomic Absorption Spectrophotometer (AAS). From experimental data, the applicability of Langmuir model was judged.

Results and Discussion

Characterization of 4-HADTOT-terpolymer: FTIR Analysis: Figure-1 and Figure-2 represents FTIR spectrum of 4-HADTOT polymer with its characteristic bands. Band at 1547–1498 cm^{-1} is due to aromatic ring stretching. Appearance of band at 3130-3070 cm^{-1} is due to aromatic C-H stretching and 1304-932 cm^{-1} is due to aromatic C-H bending (in the plane). Bands at 770-735 cm^{-1} and 860-800 cm^{-1} is indicative of ortho and para substitution in aromatic ring. Very strong absorption band 3744cm^{-1} proves the presence of aromatic secondary amino linkage. Presence of ketonic group is evident by appearance of bands at 1615 cm^{-1}. Comparatively weak absorption band at 3618 cm^{-1} indicates the presence of CH_3 group (C-H stretching).

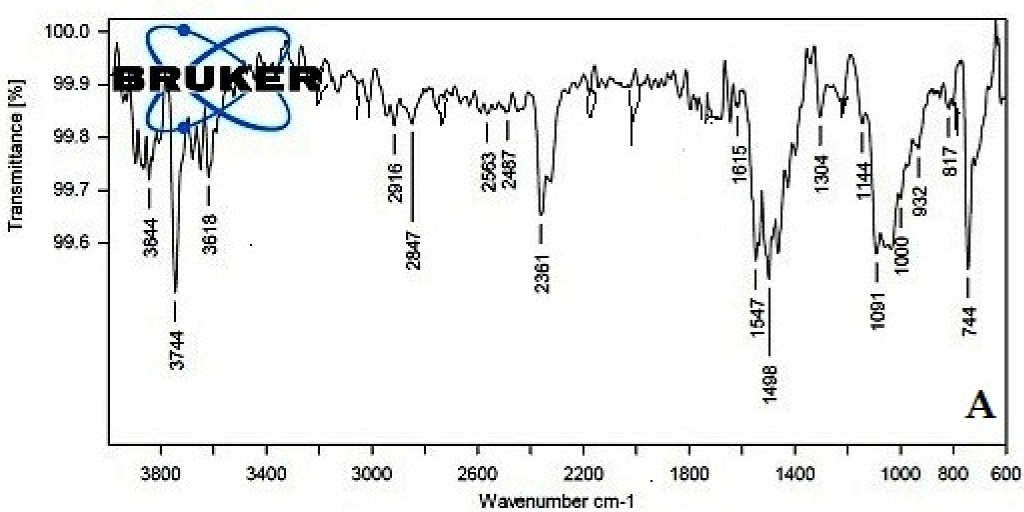

Figure-1
FTIR Spectra of 4-HADTOT-terpolymer

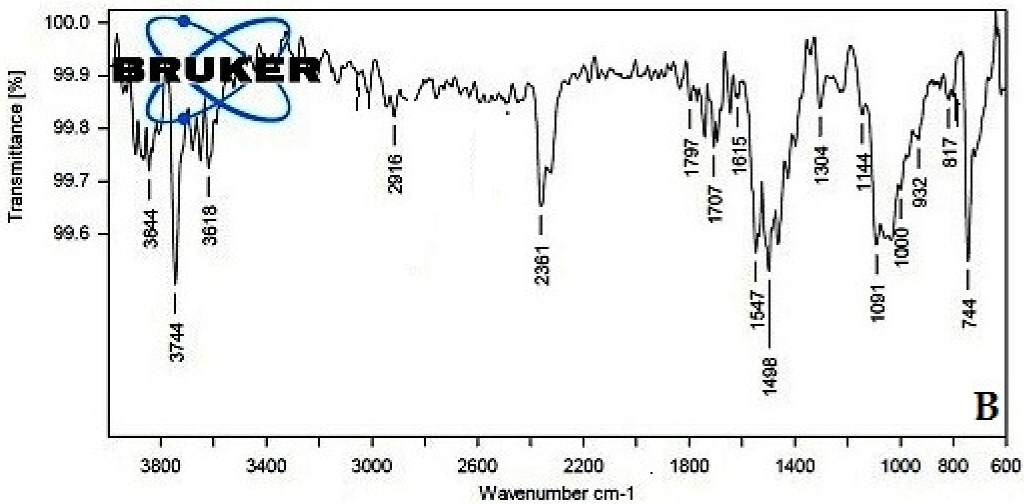

Figure-2
4-HADTOT-HCAC Composite

On the basis of physicochemical and spectral evidences the most probable structure has been proposed for 4-*HADTOT*-terpolymer which is presented in Figure-3.

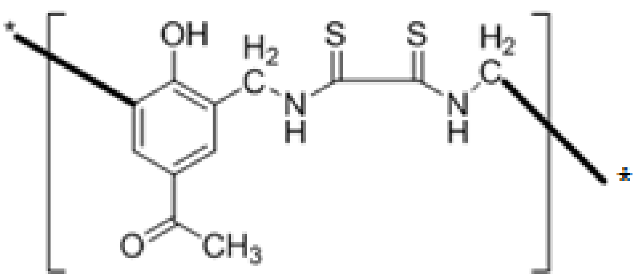

Figure-3
Structure of 4-*HADTOT*-terpolymer

Characterisation of 4-HADTOT-HCAC Composite: FTIR Studies: The FTIR spectrum represents 4-HADTOT-HCAC Composite. It can be observed that both, the FTIR for 4-HADTOT terpolymer and 4-HADTOT-HCAC Composite are almost similar. The noticeable difference is appearance of strong sharp absorption band at 1797 cm^{-1} which is indicative of C=O group (reactive oxygen functional groups). It proves the incorporation of HCAC (possessing C=O surface group) in the terpolymer matrix during Composite formation.

SEM Analysis: SEM pictures of 4-HADTOT-HCAC Composite are given in Figure-4. It is evident from the SEM pictures that the HCAC have been successfully incorporated in 4-HADTOT polymer matrix thus the synthesis of Composite is fruitful. The SEM picture also proves highly porous nature possessing rod-plate like particle structure of the Composite under investigation which is suggestive of very high surface area. Thus this Composite material may be of great use as excellent absorbent.

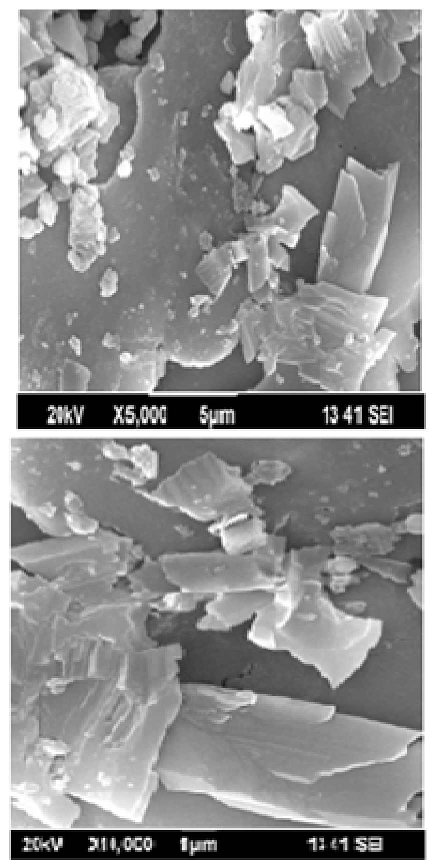

Figure-4
SEM Micrographs of 4-HCAC Composite

Effect of pH on adsorption: *Effect* of pH on Cd(II) adsorption using 4-HADTOT-HCAC Composite as an adsorbent has been studied in the pH range 1to10 and presented in Figure-5. It is noticed that solution pH plays a very important role in the adsorption of Cd(II). The percentage removal increases steadily from 44 to 80% when pH is increased from 1to 6 in Cd(II) adsorption and slowly decreases on further increases in pH.

Adsorption Studies: Batch equilibration method has been employed to study adsorptive efficacy of 4-HADTOT-HCAC Composite for removal of Cd(II) metal ion from contaminated water. The data were found to be best fitted with Frundlch adsorption isotherm. At 300K temperature and pH 6, 95% of the Cd(II) was found to be removed from its solution (25 ml, 0.1 mg dm^{-3}).

Conclusion

4-HADTOT-HCAC Composite has been found to be most effective for Cd(II) removal. At pH 6, 95% of Cd(II) was removed from aqueous solution and adsorption was found to be pH dependent.150 mg dm^{-3} absorbent dose, 300 K temperature and 6 pH were the optimum parameters during the batch experiments.

The newly obtained 4-HADTOT-HCAC Composite under present investigation can be successfully used for Cd(II) abatement from contaminated water and thus can be used for water/wastewater treatment and pollution control.

Scope for future work: There is a scope for further studies to test the practical applicability of the Composite material for removal of other metal ion /water pollutants.

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