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Comparative Characteristic Study of Agricultural Waste Activated Carbon and AC/FE_3O_4–Nano Particles

S.Sivaprakash[1]*, P.Satheeshkumar[1], Dr.S.K.Krishna[2]

[1]Research and Development center, Bharathiar University,Coimbatore, Tamilnadu, India,
[2]Dr.S.K.Krishna, Department of Chemistry, Chikkaiah Naicker College, Erode, Tamilnadu, India

Abstract : Removal of dyes and other impurities in industrial polluted water at low cost using adsorption process. Large number of adsorbent materials used in this field based on the adsorption capacity and cost. In this paper deals about the preparation of activated carbon and activated carbon magnetic nano composite from cajanus cajan stem as a agricultural waste and X-ray Diffraction study, FTIR study of cajanus cajan stem activated carbon as well as activated carbon Fe_3O_4magnetic nano composite. From the obtained result of the XRD and FTIR we can confirm that the materials are pure with specific characteristics.
Keywords : activated carbon; adsorption; nanoparticle; pores; cracks.

1. Introduction

In olden days peoples are used charcoal for filtration process. Egyptians used carbonized wood as a medical adsorbent and purifying agent. Activated carbon from agricultural waste material was introduced industrially in the first part of this century, and used in sugar refining. In the dying industries activated carbon from black ash was found very effective in decolorizing liquids[1]. The treatment of industrial effluents is a challenging topic in environmental science, as control of water pollution has become of increasing importance in recent years. Synthetic dyes are widely used in a number of industrial processes, such as the textile industry, paper printing. Although dyes not particularly hazardous, it can cause some harmful effects like increasing heart beat rate, shock, Heinz body formation, cyanosis, jaundice, quadriplegia, and tissue necrosis in humans[2]. Recently, textile, printing, and other related industries are facing problems of treatment and disposal of dye wastewater. Many countries discharge the effluent to surface water without any treatment because of technological and economical limitations[3]. There are currently numerous treatment processes for effluent discharged from industrial processes containing dyes, the important and economic method is adsorption process[4]. The use of nanoparticles for separation and treatment of waste water is new methodology that is faster and simpler. Nanoparticles have been widely studied because of structural and functional elements have various applications[5]. Among the treatment methods, adsorption on commercial activated carbon is a very effective removal technique which produces effluents containing dissolved organic compounds. However, the expensive price of the commercial activated carbon had encouraged many researchers to investigate the use of cheap and efficient alternative substitutes to remove dyes from wastewater[3].The magnetic nanoparticles have many uses such as magnetic drug target, magnetic resonance imaging forclinical diagnosis, recording material and catalyst, environment, etc[5,6] .Iron oxides nanoparticles play a major role in many areas of chemistry, physics and materials science. Fe_3O_4 (magnetite) is one of the magnetic nanoparticles. There are many various ways to prepare Fe_3O_4 nanoparticles, which have been reported in other papers. Further more, the presence of magnetic iron oxide (Fe_3O_4) leads to chemical stability, low toxicity, and excellent re-cyclability of adsorbent and these have caused to use this method widely for removal of toxic ions and organic contaminants from water and wastewater[7]. Use of the magnetic particle in the nano scale have attracted by many authors. Extremely fine size of nano-particles yields favorable characteristics with a reduction in size, more atoms located on the surface of a particle results to a remarkable increase in surface area of nanopowders[8]. In this study, Fe_3O_4/Cajanus cajan stem activated carbon magnetic nano particles were prepared by a Hydrothermal method. The resulting Fe_3O_4/AC nano particles were characterized by X-ray diffraction study (XRD) and Fourier Transformation Infrared Spectroscopy (FTIR)[9]. In this study nanoparticles of Fe_3O_4 supported on cajanuscajan stem activated carbon (AC)[9,10]. The present research investigates the obtained activated carbon and Fe_3O_4/AC magnetic nanoparticles are confirmed with related characteristic study and it will be used as a cheap and effective adsorbent[11].

2. Experimental

2.1 Materials

Agricultural waste cajanus cajan stem was collected from fallow lands in and around Erode District, Tamil Nadu, India and washed with tap water followed by washing with distilled water[12]. The material was cut into pieces of 2-4 cm size sun dried for one week. The dried mass was used for the preparation of adsorbent as per the following procedure[13].

2.2 Preparation of Activated Carbon by Physical method

A dried sample of cajanus cajan stem placed in a muffle furnace and heated at 800^0C for two hours. This was allowed to cool and washed with distilled water to a pH of 7, oven dried at 105^0C for four hours and grounded. It was sieved with a 53µ mesh to obtain a fine powdered cajanus cajan stem activated carbon and it was kept in an air tight container and used for various experiments[14].

2.3 Synthesis of Nano composites by hydrothermal method

Hydrothermal synthesis is a typical solution-based approach, which is usually employed under high temperature and pressure. Unlike the thermal decomposition method, which can only use an organic compound as a solvent, hydrothermal synthesis can occur in a water-based system and at a lower reaction temperature (160–220^0C) in a relatively environment friendly approach. It is an effective and convenient process in preparing nono composite materials[15]. The Fe_3O_4/ACMNCS were prepared by a hydrothermal method. In typical experiment 50 mg of cajanus cajan stem AC were suspended in 50ml of di-ionized water to form stable black color solutions. Subsequently, 30ml of $FeCl_2 \cdot 4H_2O$ and 80ml of $FeCl_3 \cdot 6H_2O$ were dissolved in to the above solution and pH value was adjusted 10-11 by adding 30 % of ammonium hydroxide solution (NH_4OH).After that, the final solution was transferred into the 75 ml Teflon-lined stainless steel autoclave were placed in an oven at 180^0C for 12 hours. After hydrothermal reaction, the autoclave was cooled down to room temperature and black color precipitate was washed with double distilled water and ethanol several times. Finally, the prepared Fe_3O_4/ cajanus cajan stem AC MNCS sample was dried in vacuum oven at 70^0C for overnight[16].

2.4 Characterization

Solid state chemists use primarily the Powder X-ray Diffraction techniques which are the most important characterization tools used in solid state chemistry and material science. The size, shape, lattice parameter determination and phase fraction analysis of the unit cell for any compound can be determined easily by XRD. The information of translational symmetry-size and shape of the unit cell are obtained from peak positions of diffraction pattern[17].

Fourier Transform Infrared Spectroscopy (FTIR) study was carried out to identify the functional groups present in the adsorbents in the 4000-400 cm range. The adsorption capacity of adsorbent depends upon porosity as well as chemical reactivity of functional groups at the adsorbent surface[18].

3. Result and Discussion

3.1.1 X-ray Diffraction Analysis of Activated Carbon

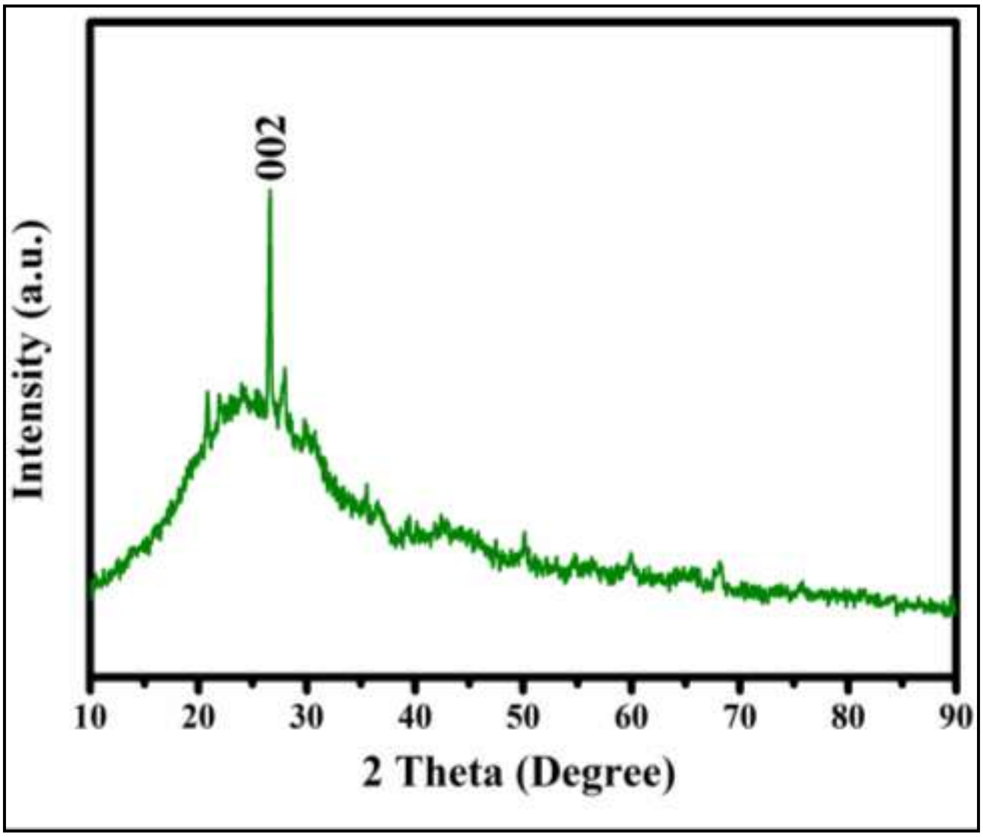

Fig.1X-ray Diffraction Analysis of Activated Carbon

The X-ray diffraction (XRD) measurements of pure cajanus cajan stem AC were carried out at room temperature using a PAN analytical (X-Pert-Pro) diffractometer with a Cu $K\alpha_1$ radiation (λ= 1.5406 Å) over a scanning interval (2θ) from 10 to 90°[18].

The (002) peak of the sample in the observed diffraction profile(Fig.1) is almost at around 26^o reveals to amorphous nature of carbon[19].

3.1.2 X-ray Diffraction Analysis of Fe_3O_4/Activated Carbon Nano composite

The powder XRD pattern for the as-prepared magnetite (Fe_3O_4) nanoparticles was recorded by a Rich Secifer, X-ray diffractometer using monochromatic nickel filtered CuK (= 1.5416 Å) radiation[20]. The crystal structure and the phase purity of the synthesized cajanus cajan stem activated carbon magnetic (Fe_3O_4) nanoparticles were examined[21]. Fig.2 displays the typical XRD pattern of the cajanus cajan stem activated carbon magnetic (Fe_3O_4) nano particles samples[22]. The stronger peaks reveal the high purity, good crystallinity and the peak broadening indicates the formation of cajanus cajan stem activated carbon Fe_3O_4 nanoparticles[23]. For cajanus cajan stem activated carbon Fe_3O_4 magnetic nanoparticles shows various peaks corresponding to planes (220), (311), (400), (422), (511) and (440) are observed[24]. The crystal structure is found to be face centered cubic with lattice constant a = 8.4272 Å and this matches well with JCPDS (89-3854) data (a=8.393(Å) [25, 26, 27]. The peak value also shows that the average particle size is 30 nm were calculated from scherrer formula[28].

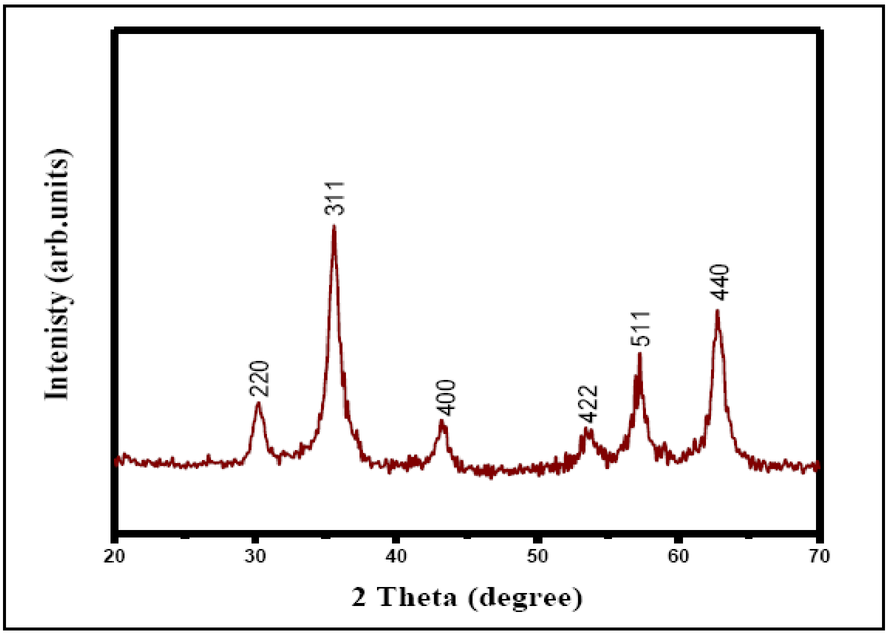

Fig.2. X-ray Diffraction Analysis of Fe_3O_4/Activated Carbon Nano composite

3.2.1 Fourier Transform Infrared (FTIR) spectroscopy of Activated Carbon

Functional groups of the activated carbon Fourier transform infrared (FTIR) transmission spectra were obtained to characterize the surface groups on the pulp, the peel, and the ACs prepared from these two precursors. Figure.3 shows the FTIR spectra of the activated carbon and the wave numbers and assignments of the main bands observed

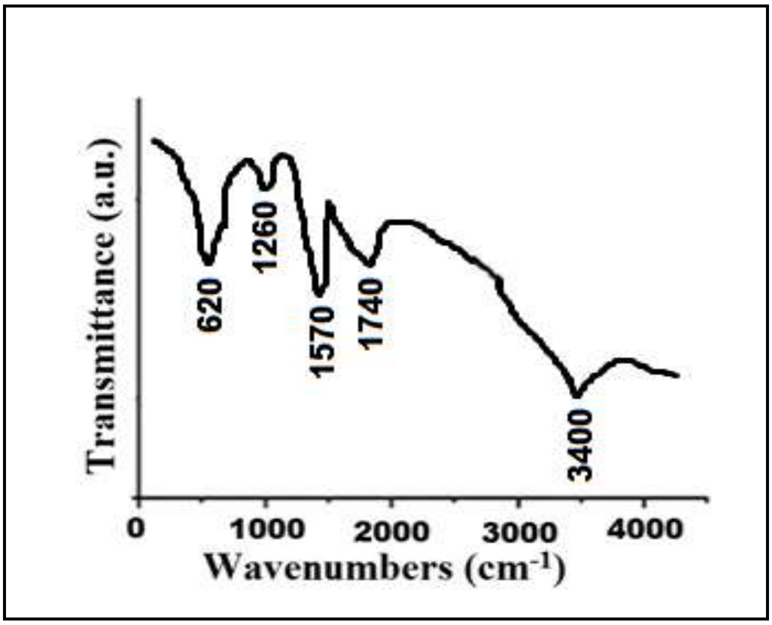

Fig.3. Fourier Transform Infrared Spectroscopy of Activated Carbon

Fourier Transform Infrared Spectroscopy (FTIR) spectra was performed to the dried sample of magnetite using a FTIR –Shimadzu 8400 spectrophotometer in wave range of 3500 - 400 cm with a resolution of 4 cm^{-1}. The dried activated carbon was placed on a silicon substrate transparent to infrared, and spectra were measured according to the transmittance method[29]. FTIR spectrum in fig.3 that very strong band around 3500-3200 cm^{-1} could be assigned to O-H and N-H stretching vibrations[30]. The spectrum shows an absorption band at 1740 cm^{-1}, which presents the stretching vibration of the carboxyl group (C = O), associated to the acid molecule, adsorbed on to the surface of the activated carbon[29], the peak at 1570 cm^{-1} is assigned to the C=C stretching vibration[31], the peak at 1260 cm^{-1}indicating C-O in carboxylic acids and phenols[32], Peak at 620 cm^{-1} may be attributed to vibrations of C-C stretching vibrations[33]. This results confirms the activated carbon having best characteristics[31].

3.2.2 Fourier Transform Infrared (FTIR) spectroscopy of Fe_3O_4/Activated Carbon

Nano composite

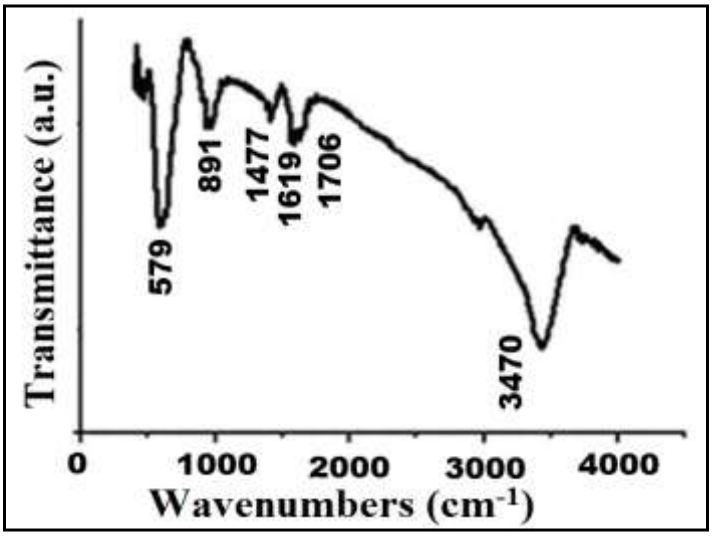

Fig.4. Fourier Transform Infrared Spectroscopy of Fe_3O_4/Activated Carbon Nano composite

Fourier Transform Infrared Spectroscopy (FTIR) spectra was performed to the dried sample of magnetite using a FTIR –Shimadzu 8400 spectrophotometer in wave range of 3500 - 400 cm with a resolution of 4 cm^{-1}. The dried sample was placed on a silicon substrate transparent to infrared, and spectra were measured according to the transmittance method[32]. FTIR spectrum in fig.4 shows that very strong band around 3500-3200 cm^{-1} could be assigned to O-H and N-H stretching vibrations[33]. The spectrum shows an absorption band at 1706 cm^{-1}, which presents the stretching vibration of the carboxyl group (C = O), associated to the acid molecule, adsorbed on to the surface of the composites[32], the peak at 1619 cm^{-1} is assigned to the carboxylate (COO-) stretching vibration[34], The $–CH_2$ deformation bending gives a band about 1477 cm^{-1}[32], Peak at 891 cm^{-1} may be attributed to vibrations of the Fe-O bond for FeO(OH)[35], and the strong peak at 579 cm^{-1} is assigned to the Fe-O bond, which confirms the presence of activated carbon magnetic nanoparticles[36].

4. Conclusion

Cajanus cajan stem activated carbon/Fe_3O_4 Magnetic nano particles were successfully synthesized usinglow-cost, renewable, eco-friendly bio templates. The activated carbon and nanoparticles were characterized using X-ray diffraction technique and Fourier Transform Infrared (FTIR) spectroscopy. From the X-raydiffraction analysis we obtain the characteristics of activated carbon (002)peak is observed diffractionis almost at around 26^o reveals to amorphous nature of carbon. The characteristics of Fe_3O_4/Activated carbon nano particles in X-ray diffraction technique various speaks corresponding to planes(220), (311), (400), (422),(511) and (440)are observed so the crystal structure is found tobe face centered cubic with lattice constant and the average particle size is 30 nm. From the results of Fourier Transform Infrared Spectroscopy (FTIR) with comparison of Cajanuscajan stem activated carbon and Cajanuscajan stem activated carbon/Fe_3O_4 Magnetic nanocomposite material the bands from MNC is 1706, 1619, 1477,891 and 579 cm^{-1} show that the different functional groups such as surface hydroxyl, carbonyl, methylene and alcohol etc were responsible for the adsorption process and it should be very effective in adsorption of dyes compare to normal activated carbon result. So the Fe_3O_4/Activated carbon nano particles are confirmed as a nano particles and it should be very effective in adsorption of dyes compare to normal activated carbon.

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