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Synthesis & Characteristic Study of Agricultural Waste Activated Carbon/Fe_3o_4–Nano Particles

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Abstract

Adsorption is an important surface operation in industrial polluted water treatment for removal of dyes and other impurities. Among many types of agricultural waste adsorbent used, because of their large adsorption capacity and its low cost. In this study we have to prepare the activated carbon from cajanuscajan stem as a agricultural waste by physical method and synthesis of Fe_3O_4/Activated carbon nano particles by hydro thermal method. The Fe_3O_4/Activated carbon nano particles are studied by X-ray Diffraction (XRD), Fourier Transform Infrared (FTIR) spectroscopy and Scanning Electron Microscopy (SEM) respectively. From this study we have to confirm that the obtained material is a nano particle and it is used as a excellent adsorbent.
Keywords- activated carbon; adsorption; nanoparticle; pores; cracks.

I. INTRODUCTION

Ancient Hindus in India used charcoal for drinking water filtration. 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 the 20th century, and used in sugar refining. In the US 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, etc. 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 a 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 important magnetic nanoparticle. There are many various ways to prepare Fe_3O_4 nanoparticles, which have been reported in other papers. Furthermore, 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 particles 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/Cajanuscajan 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), Fourier Transformation Infrared Spectroscopy (FTIR) and Scanning Electronic Microscopy (SEM) [9]. In this study nanoparticles of Fe_3O_4 supported on cajanuscajan stem activated carbon (AC) [9,10]. The present research investigates the obtained Fe_3O_4/AC magnetic nanoparticles are confirmed as a nano composites and it will be used as a cheap and effective adsorbent [11].

II. EXPERIMENTAL

A. *Materials*

Agricultural waste cajanuscajan 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].

B. *Preparation of Activated Carbon by Physical method*

A dried sample of cajanuscajan 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 cajanuscajan stem activated carbon and it was kept in an air tight container and used for various experiments [14].

C. *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 °C) 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 hydrothermal method. In typical experiment 50 mg of cajanuscajan stem AC were suspended in 50ml of di-ionized water to form stable black color solutions. Subsequently, 30ml of FeCl2·4H2O 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/ cajanuscajan stem AC MNCS sample was dried in vacuum oven at 70^0C for overnight [16].

D. *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].

Scanning Electronic Microscopy (SEM) have a variety of applications in a number of scientific and industry-related fields, especially where characterizations of solid materials is beneficial.In addition to topographical, morphological and compositional information, a Scanning Electron Microscope can detect and analyze surface fractures, provide information in microstructures, examine surface contaminations, reveal spatial variations in chemical compositions, provide qualitative chemical analyses and identify crystalline structures [19].

III. RESULT AND DISCUSSION

A. *X-ray Diffraction Analysis of Fe_3O_4/Activated Carbon Nano composite*

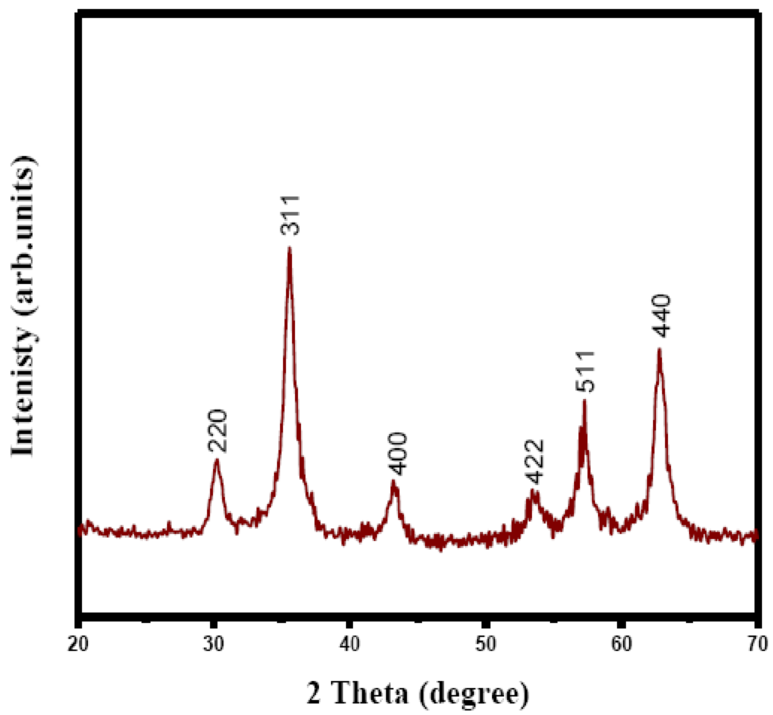

Fig.1. X-ray Diffraction Analysis of Fe_3O_4/Activated Carbon Nano composite

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 A^0) radiation [20]. The crystal structure and the phase purity of the synthesized cajanuscajan stem activated carbon magnetic (Fe_3O_4) nanoparticles were examined [21]. Fig.1 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 cajanuscajan stem activated carbon Fe_3O_4 nanoparticles [23]. For cajanuscajan 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) [25-27]. The peak value also shows that the average particle size is 30 nm were calculated from scherrer formula [28].

B. ***Fourier Transform Infrared (FTIR) spectroscopy of Fe_3O_4/Activated Carbon Nano composite***

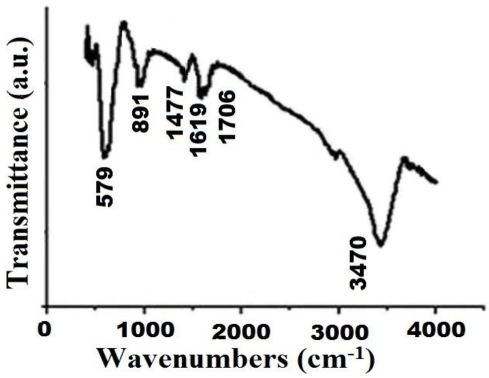

Fig.2. Fourier Transform Infrared Spectroscopy of Fe_3O_4/Activated Carbon Nano composite

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 [29]. FTIR spectrum in fig.5 shows 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 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 [29], the peak at 1619 cm^{-1} is assigned to the carboxylate (COO-) stretching vibration [31], 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) [33], and the strong peak at 579 cm^{-1} is assigned to the Fe-O bond, which confirms the presence of activated carbon magnetic nanoparticles [31].

D. ***Scanning electron microscopy (SEM) of Fe_3O_4/Activated Carbon Nano composite***

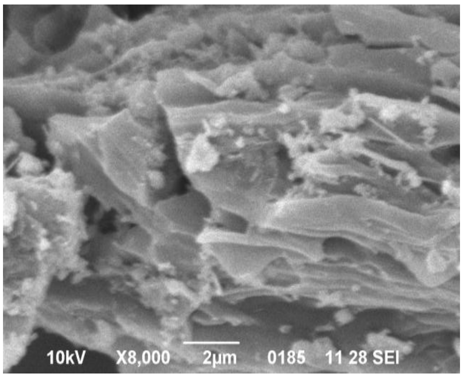

Fig.3. Scanning electron microscopy of Fe_3O_4/Activated Carbon Nano composite

Scanning electron microscopy was performed to investigate the morphology of the nano composites. It is easy to notice that the examined particles consist of a number of smaller objects. The surface of the films shows number of peaks and cracks, indicating good adhesion capacity on the thin film layer [34], the SEM of adsorbent shows irregular groove and ridges in fibrous network that is considered helpful for the accessibility of metal ions to the adsorbent surface [35], the pore size distribution of the nano composites was non-uniform due to rapid development of the pore which results in formation of too much cavities and cracks [36], The SEM image demonstrates clearly the formation of spherical shape Fe_3O_4/Activated Carbon magnetic nano composite.

IV. CONCLUSION

Cajanuscajan stem activated carbon/Fe_3O_4 Magnetic nanoparticles were successfully synthesized using low-cost, renewable, eco-friendly biotemplates. The activated carbon and nanoparticles were characterized using X-ray diffraction technique, Scanning Electron Microscopy (SEM) and Fourier Transform Infrared (FTIR) spectroscopy. From XRD analysis we obtain the characteristics of activated carbon (002) peak is observed diffraction is 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 peaks corresponding to planes (220), (311), (400), (422), (511) and (440) are observed so the crystal structure is found to be face centered cubic with lattice constant and the average particle size is 30 nm. So the Fe_3O_4/Activated carbon nano particles are confirmed as nano particles. From the results of Fourier Transform Infrared Spectroscopy (FTIR) the bands 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. The Scanning electron microscopy (SEM) result of Fe_3O_4/Activated Carbon Nano composite confirms the formation of spherical shape Fe_3O_4/Activated Carbon magnetic nano composite with large number of pores, cracks and peaks were responsible for the adsorption process and it should be very effective in adsorption of dyes compare to normal activated carbon.

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