Synthesis & Characterization of Activated Carbon with Fe_3O_4-Nano Composites (AC-Fe_3O_4-NCs) by Hydrothermal Process

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Abstract: ***The Activated carbon with Fe_3O_4 mixed nanocomposites were successfully prepared by using hydrothermal process. The resulting nanocomposite has been characterized by using XRD, SEM and FTIR analytical techniques. The X-Ray Diffraction (XRD) analysis studies clearly suggest the formation of activated carbon with Fe_3O_4 nanocomposite. The SEM analysis indicates the formation of process and spherical shape morphology in mixed composite nano materials. The FTIR spectroscopy studies clearly suggest the functional group present in the Fe_3O_4 nanocomposite. The synthesized AC-Fe_3O_4-NC has been used for the adsorption studies for removal of dyes and metals from industrial effluents.***

Keywords: Hydrothermal, Adsorption, AC-Fe_3O_4-NC

1. Introduction

Discharge of hazardous waste water without further treatment can seriously damage the environment. Sunlight and oxygen are very important requirements of aquatic life, but the colored discharged effluents inhibit penetration of sunlight and oxygen. It adversely effects on life. [1] We have to remove dyes from waste water of dyeing and finishing operation in textile industry.

Nanotechnology is a new scientific field being developed since 1980's. Nanotechnology is the manipulation of individual atoms and molecules to create materials and devices with vastly different properties. The physical and chemical properties of metal nanoparticles are mainly determined by its size, shape, composition, crystallinity and structure. [2, 3] Nanoparticles are a special group of materials with unique features and extensive applications in diverse fields. [4]

Nanoparticles are traditionally synthesized by wet chemical techniques where the chemical used are quite often toxic and flammable. A conventional method to prepare the iron oxide nanoparticles are coprecipitation method. [5-7] A number of approaches are available for the synthesis of iron oxide nanoparticles such as top-down method, bottom-up method, sonochemical process, hydrodynamic cavitation, radiolysis, microwave and laser ablation method. [8-10]

The magnetic nanoparticles have many uses such as magnetic drug target, magnetic resonance imaging for clinical diagnosis, recording material and catalyst, environment, etc. [11-13] Iron oxide nanoparticles play a major role in many areas of chemistry, physics and material science.

The application of magnetic technology to solve environmental problems has received considerable attention in recent years. Many of papers have been published demonstrating that magnetic Fe_3O_4 can be used for water purification, such as to adsorb arsenite, arsenate, chromium, cadmium and nickel. [14, 15]

Nanocorbon materials with large surface area, microporous character and chemical nature of their surface have made them potential adsorbents for the removal of dyes from industrial waste water. [16, 17]

2. Experimental Preparation of AC-Fe_3O_4-NC by Hydrothermal Process

2.1 Preparation of Activated Carbon

The Amorphophallus Paeoniifolius waste collected from Agricultural land in and around, Erode District, Tamil Nadu. They were cut into small pieces and dried for 20 days. Finally, it was taken in a steel vessel and heated in muffle furnace. The temperature was raised gradually up to 500^{o} C and kept it for half an hour. The carbonized material was ground well and sieved to different particle size. It was stored in a plastic container for further studies. In this study particle size of 0.15 to 0.25mm was used.

2.2 Preparation of Fe_3O_4/AC-NCs:

The Fe_3O_4/AC-NCs were prepared by a hydrothermal method. In typical experiments, 50 mg of AC were suspended in 50 mL of de-ionized water to form stable black color solutions. Subsequently, 50 mm of $FeCl_2 \cdot 4H_2O$ and 100 mm 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 $180^{o}C$ for 12 hours. After completed 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 as-prepared Fe_3O_4/AC NCs sample was dried in vacuum oven at $70^{o}C$ for overnight.

2.3 Characterization of Materials

The crystal structure of the products was characterized by X-Ray Diffraction (XRD) (XPERT-PRO Diffractometer). The patterns with the Cu Kα_1 radiation (λ= 1.5406 Å) were recorded in the region of 2θ range from 10 to 70°. The morphology of the composite was determined by Field emission scanning electron microscopy (FEI Quanta-250). The infrared spectrum of the sample was obtained by using a Fourier transform infrared (FTIR) spectrometer (Bruker Tensor 27, Germany).

3. Results and Discussion

3.1 X-Ray diffraction

It is a non-destructive and analytical method for identification and quantitative analysis of various crystalline forms of AC-Fe_3O_4-NC. Diffraction occurs when the waves collide with a regular structure in which the repeating distance is approximately same as the wavelength of the wave. It happens that X-Rays have wavelength 'n' the order of a few angstroms. This means that the X-rays can be easily diffracted from materials which, are crystalline and have repeating and regular atomic structures. When the required parameters met, the X-Rays that get scattered from a crystalline solid can interfere constructively, thus producing a diffracted beam of light.

In 1912, W. L. Bragg derived a relationship among several factors:

- The inter atomic spacing which is known as d-spacing and is measured in angstroms.
- The angle of diffraction which is known as the theta angle and is measured in degrees.
- The wavelength of the incident X-rays, denoted by the lambda and, in this case, equal to 1.54 angstroms.

$$n\lambda=2dSin\theta\text{------------->}1$$

Where n is an integer 1, 2, 3…. . (usually equal 1)
λ is wavelength in angstroms (1.5 armstrong for copper)
D is interatomtic spacing in angstroms, and
θ is the diffraction angle in degrees.

The XRD pattern provides structural information for the activated carbon and Fe_3O_4/AC nanocomposites as shown in Fig.1 (a&b). Figure 1 (a) shows the XRD pattern of activated carbon which was shows that broad peaks attributed at around 26° to the corresponding plane (002). Figure 1 (b) shows the XRD pattern of Fe_3O_4/AC nanocomposites reveals face-centered cubic structure (JCPDS No.89-3854) with an average grain size of 30 nm were calculated from Scherrer formula. The diffraction pattern shows peaks of the Fe_3O_4 nanocomposites at 2θ = 30.08 ^{0}, 35.43^0, 43.07^0, 56.95^0, 62.54^0 which were corresponding to the (220), (311), (400), (511) and (531) crystal planes of a pure Fe_3O_4 with cubic spinel structure. [18-24]

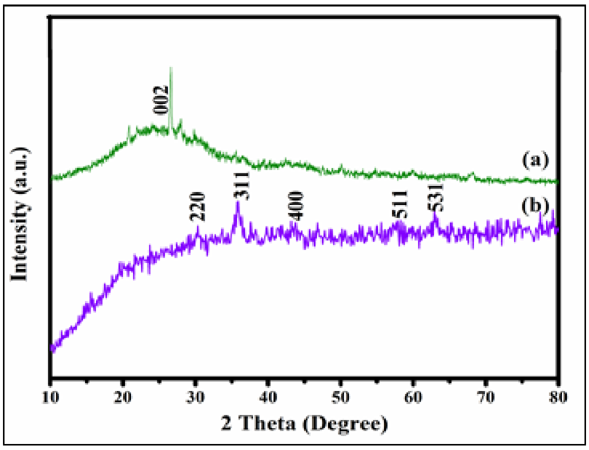

Figure 1: X-ray diffraction analysis of (a) activated carbon (AC) and (b) Fe_3O_4/AC nanocomposites synthesized by hydrothermal process at 180ºC for 12 hrs.

The average grain size of the samples was estimated with the help of scherrer equation
$$D=k\lambda/\beta Cos\theta\text{----------------------->}2$$

Where D is crystallite average size,
k is constant (usually between 0.9 to 1.0)
λ is X-Ray wavelength (1.54 Armstrong)
β is Full maxima half width,
θ is Diffraction angle.

3.2 SEM Analysis:

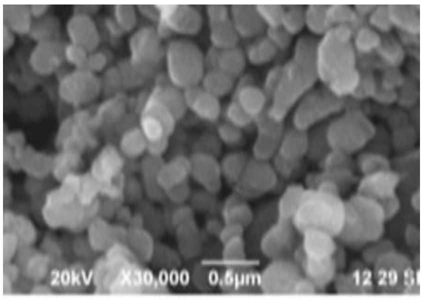

Figure 2: SEM image of AC-Fe_3O_4-NC

The morphology of the products was carried out by using Field Emission Scanning Electron Microscope operated at 20KV. SEM has been a primary tool for characterizing the surface morphology and fundamental physical properties of the adsorbent surface. It is useful for determining the particle shape and appropriate size distribution of the adsorbent. Figure 2 show the FESEM image of the AC-Fe_3O_4-NC and image showed that the AC-Fe_3O_4-NC have spherical shape [25, 26, 27, 28, 29] with average size of 5-30nm. It implies that a relatively high temperature would promote crystallization of the magnetite phase. [30] These observations are highly consistent with the above XRD results.

3.3 FTIR Analysis

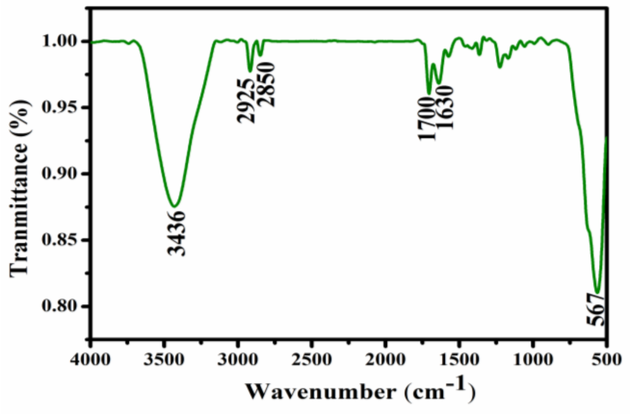

Figure 3: FTIR Spectra of AC-Fe_3O_4-NC

Fourier transform infrared (FTIR) spectrum is the spectroscopy that deals with the infrared region of the electromagnetic spectrum that is light with a longer wave length and lower frequency visible light. [31] The formation and identification of the functional group of the AC-Fe_3O_4-NC's were further supported by the FTIR analysis. The FTIR spectrum in Figure 3 shows that intense peak at 3436 cm^{-1} [32, 33] and 1630 cm^{-1} [34, 35] are due to the-OH stretching. The characteristic peaks of the c=c stretching mode can be seen at 1700 cm^{-1}. [36] The characteristic adsorption peaks of methylene groups (CH_2), which were present due to the AC-Fe_3O_4-NC was observed at 2850 cm^{-1} [35] and 2925 cm^{-1}. [37, 38] The peak at 567 cm^{-1} [38, 39] indicates that Fe_3O_4 groups.

4. Conclusions

In summary, AC-Fe_3O_4-NC are prepared with the help of hydrothermal process. The size and structure of nanocomposite is confirmed that the XRD technique. The synthesized AC-Fe_3O_4-NC particle size is calculated as 30 nm. The characteristic peak of AC-Fe_3O_4-NC at 567 cm^{-1} in FTIR absorption spectra is also noticed. SEM micrographs suggest the AC-Fe_3O_4-NC are spherical in shape. The characterisation studies of AC-Fe_3O_4-NC would be used for the fabrication and designing of waste water treatment plants for the removal of dye. Since the raw materials is freely available in large quantities for the treatment method, seems to be economical.

References

[1] Vadivelan V. V. Kumar, Equilibrium, kinetics, mechanism and process design for the sorption of methylene blue onto rice husk. J. Colloid Interface Sci., 286: 90-100, 2005.

[2] Addadi, L. and S. Weiner, 1992. Control and design principles in biological minerlization. Angewandte chemie International Edition in English, 31 (2): 153-169.

[3] Bazylinski, D. A., R. B. Frankel and K. O. Konhauser, 2007. Modes of biomineralization of magnetite by microbes. Geomicrobiology Journal, 24: 465-475.

[4] Matei, A., I. Cernica, O. Cadar, C. Roman and V. Schiopu, 2008. Synthesis and characterization of ZnO-polymer nanocomposites. The International Journal of Material Forming, 1: 767-770.

[5] S. J. Lee, J. R. Jeong, S. C. Shin, J. C. Kim and J. D. Kim, J. Magnetism and Magnetic Mat., 282, 147 (2004).

[6] Hou, Z. Xu, and S. Sun, Angew. Chem. Int. Ed., 46, 6329 (2007).

[7] D. Predoi, Dig. J. Nanomat. and Biostr., 2 (1), 169 (2007). .

[8] L. Rodriguez-Sanchez, M. C. Blanco and M. A. Lopez-Quintela, J. Phys. Chem. B., 104, 9683 (2000).

[9] J. J. Zhu, S. W. Liu, O. Palchik, Y. Koltypin and A. Gadanken, Langmuir., 16, 6396 (2000).

[10] Pastoriza-Santos and L. M. Liz-Marzan, Langmuir, 18, 2888 (2002).

[11] U. Hafeli, W. Schutt, J. Teller, and M. Zborowski, Scientific and Clinical Applications of Magnetic Microspheres, Plenum, NewYork, pp.324-326, (1997).

[12] vV. S. Zaitsev, D. S. Filimonov, I. A. Presnyakov, R. J. Gambino, and B. Chu, Physical and Chemical properties of Magnetite and Magnetite polymer Nanoparticles and Their Colloidal Dispersions, Journal Colloid Interface Scie, Vol.212, pp.49-57, (1999).

[13] B. A. Bolto, Magnetic particle Technology for waste water Treatment, Waste Management, Vol.10, pp.11-21, (1990).

[14] J. T. Mayo, etal., The Effect of NanoCrystalline Magnetite Size on Arsenic Removal, Journal of Science and Technology Materials, Vol.8, pp.71-75, (2008).

[15] F. Rosada, M. Otero, A. Moran, and A. I. Garcia, Adsorption of Heavy metals onto Sewage Sludge-Derived Materials, Bioresour Technology, Vol.99, pp.6332-3338, (2008).

[16] S. K. Krishna, and M. Sathya, Usage of Nanoparticle As Adsorbent In Adsorption process-A Review, International Journal of Applied Chemistry, Vol.11 (2), pp.221-227 (2015).

[17] S. K. Krishna, and. Sivaprakash, Removal of Dyes by using various Adsorbents-A Review, International Journal of Applied Chemistry, Vol.11 (2), pp.195-202 (2015).

[18] S. A. Kulkarni, P. S. Sawadh and K. K. Kokate IJAC (2012).

[19] S. Amala Jayanthi, D. Sukanya, A. Joseph Arul Pragasam and P. Sagayaraj., Der Pharma Chemica, 2013, 5 (1): 90-102.

[20] P. OU, G. OU, C. OU, Y. ZHANG, X. HOU, G. HAN, Materials Science-Poland, Vol.28, No.4, 2010.

[21] Mao Shen, Yujing Yu, Guodong Fan, Guang Chen, Ying min Jin, Wenyuah Tang and Wenping Jia, Shen et al. Nanoscale Research Letters 2014, 9: 296.

[22] Poedji Loekitowati Hariani, Muhammad Faizal, Ridwan, Marsi and Dedi Setiabudidaya, International Journal of Environmental Science and Development, Vol.4, No.3, June 2013.

[23] Eman Alzahrani, International Journal of Innovative Research in Science, Engineering and Technology, Vol.3, Issue 8, August 2014.

[24] G. Bharath, Rajesh Madhu, Shen-Ming Chen, Vediyappan Veeramani, D. Mangalraj and N.

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Ponpandian, Journal of Materials Chemistry A. (2015).

[25] Poedji Loekitowati Hariani, Muhammad Faizal, Ridwan, Marsi, and Dedi Setiabudidaya, Synthesis and properties of Fe3O4 Nanoparticles by Co-precipitation method to Removal precion Dye, International Journal of Environmental Science and Development, Vol.4, No.3, June 2013.

[26] A. Mollahossini, M. Toghroli, Synthesis and identification of Fe3O4 /Clinoptilolite Magnetic Nanocomposite, Journal of Asian Scientific Research, 2015, 5 (3): 120-125.

[27] S. A. Kulkarni, P. S. Sawadh, K. K. Kokate, Synthesis and characterization of Fe3O4 Nanoparticles for Engineering Applications, International Journal of Computer Applications (IJAC).

[28] Y. T. Prabhu, K. Venkateswara Rao, B. Sivakumari, Vemula Sesha Sai Kumar, Tambur Pavani, Synthesis of Fe3O4 Nanoparticles and its antibacterial application, Int Nano Lett (2015) 5: 85-92.

[29] J. Pauline and A. Persis Amaliya, Synthesis and characterization of Highly monodispersive CoFe2O4 Magnetic Nanoparticles by Hydrothermal chemical Route, Archives of Applied Science Research, 2011, 3 (5): 213-223.

[30] A. Hasanpour, M. Niyaifar and M. Asan, Synthesis and characterization of Fe3O4 and ZnO Nanocomposites by Sol-gel Method, ICNS4, 12-14 March, (2012).

[31] White, R. Chromatography/Fourier transform infrared spectroscopy and its applications

[32] G. Bharath, Rajesh Madhu, Shen-Ming Chen, Vediyappan Veeramani, D. Mangalraj and N. Ponpandian, Solvent free mechanochemical synthesis of grapheme oxide and Fe3O4-reduced grapheme oxide nanocomposites for sensitive detection of nitrite, Journal of Materials Chemistry A. (2015).

[33] Vemula Madhavi, T. N. V. K. V. Prasad and Gajulapalle Madhavi, Synthesis and spectral characterization of Iron Based Micro and Nanoparticles, Iranica Journal of Energy and Environment 4 (4): 385-390, (2013).

[34] N. Latha and M. Gowri, Biosynthesis and characterization of Fe3O4 Nanoparticles using Caricaya papaya leaves Extract, International Journal of Science and Research (IJSR), Vol.3 Issue 11, Nov (2014).

[35] Chenglong Xia, Yan Sing, Yongzhong Jia, Duyuan Yue, Jun Ma and Xiaojie Yin, Adsorption properties of Congo red from aqueous solution on modified hectorite: Kinetic and thermodynamic studies, Desalination 265 (2011) 81-87.

[36] Muhammad Hashemifar, Ghodratolah Shamskhorramabady, HatamGodini, Nazila Nilfari, Marjan Mehrabpour and Mojtaba Davoudi, Preparation of Nano Ironoxide coated Activated sludge Granules and its Adsorption properties for Cd (II) Ions in Aqueous solutions, Research Journal of Environmental and Earth Sciences 6 (5): 259-265, (2014).

[37] Oman Zuas, Haznam Abimanyu and Widayanti Wibowo, Synthesis and characterization of nanostructured CeO2 with dyes adsorption property, Processing and Applications of Ceramics 8 (1) (2014) 39-46.

[38] M. A. Abu-Saied, E. S. Abdel-Halim, Moustafa M. G. Fouda and Salem S. Al-Deyab, Preparation and Characterization of Iminated Polyacrylonitrile for thr Removal of Methylene Blue from Aqueous solutions, Int. J. Electrochem. Sci., 8 (2013) 5121-5135.

[39] Sasikala Sundar and Shakkthivel Piraman, Nanospheres of Fe3O4 Synthesis through Sol-gel Techniques and Their structural and Magnetic characterization, Indian Journal of Applied Research, Vol.3 Issue: 7 July 2013