Extraction of Nanosized α-Fe_2O_3 Particles from Red Mud

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Abstract:

Red mud (RM) is a waste generated in Bayer's process after extraction of alumina from bauxite. In this work nanosized α-Fe_2O_3 has been extracted from it by simple wet chemical method. Extracted iron oxide was investigated by XRD technique. BET surface area and bulk density is found to be 199 m^2g^{-1} & 3.0 g cm^{-3} respectively. Particle size was calculated using these values of BET surface area and bulk density and same has been found to be 10 nm. Morphology of the extracted iron oxide was tested using SEM studies. About 20% of the total iron oxide present in the raw sample could be extracted in the form of nanosized α-Fe_2O_3. The extracted iron oxide considered as a promising material with great technological importance in the area of humidity sensor, catalyst, pigments, ferro fluids, recording systems etc.

Keywords:

α-Fe_2O_3, Red mud, Surface area, Nanomaterial

Introduction:

The synthesis of oxide nanoparticle attract more and more attention because of these nanoparticles exhibit good electrical, optical and magnetic properties that are different from their bulk counter parts[1]. Iron oxide has attracted great attention over the past decades due to its potential applications in catalysis[2-4], gas sensors[5], high density magnetic recording system[6], ferrofluid[7], magnetic resonance imaging[8], and in biomedical field[9-.

During the production of alumina from bauxite by treating it with caustic soda, iron oxide along with other impurities is filtered off as red mud (RM) in Bayer's process[11]. Under normal conditions when one ton of alumina is produced, nearly a ton of RM is generated as a waste product. The red colour of the RM is due to the presence of iron oxide. During the middle of the 20th century it was established that RM is a pollutant and has detrimental effect on marine life. Now a days it is dumped in holding ponds

for which large area of land is required which causes pollution to surrounding environment. Despite of this RM is a good source of inorganic chemicals like aluminium, titanium, vanadium, etc. General attempts made to recover inorganic chemicals were summerised by Thakur and Sant[12-13] in their review. Ramsay and Lowe were first to recognize RM as a source of inorganic chemicals[14]. Many attempts have been made to recover inorganic chemicals from RM[15-19] but no one has reported recovery of nanosized α-Fe_2O_3 from RM. We, for the first time, reporting extraction of nanosized α-Fe_2O_3 from RM.

Experimental procedure:

All chemicals used in this work were of AR grade. RM was procured from HINDILCO, Belgaum (India). In the beginning RM in the form of lumps were dried at 110°C in hot air oven for four hours and then powdered. To the 10 g RM powder, 100 ml of 5N HCl was added slowly and carefully, in 250 ml glass beaker. The mixture was stirred for half an hour with the help of glass rod. Above mixture was allowed to stand for 1 h. After 1 h the upper layer of HCl was removed in another glass beaker by decantation. To the remaining RM again 100 ml of HCl was added and stirred for 10 minutes. This mixture was then kept for digestion for eight days with intermittent stirring. After this the mixture was filtered by using simple filter paper to get yellowish-reddish colored filtrate (Volume approximately 90 ml). To this solution approximately 80-100 ml 1:1 ammonia solution was added drop wise from burette slowly and carefully to get reddish-brown colored precipitate of iron hydroxide. Then it was filtered and washed using distilled water (200 ml). The precipitate of iron hydroxide was dried at 110 °C and finally heated at 500°C for three hours to obtain red colored powders of iron oxide.

The crystallinity and phase identification of the powders were determined by powder XRD using Philips PW-1700 diffractometer with Ni filtered CuKα radiation. Surface area measurement was done using nitrogen gas adsorption multipoint Brunquer-Emmett-Teller (BET) method and Micromeritics ASAP 2010 model, assuming cross sectional area of 0.162

nm^2 for nitrogen molecule. Powder density was measured using pycnometer with xylene as liquid medium. The diameter of the primary particle was calculated from superficial area using following equation :

$D_{BET} = 6/S_{BET}.\rho$ ... (1)

where S_{BET} is the superficial area (m^2g^{-1}) measured by BET analyses; ρ, the density of powders (g cm^{-3}); and D_{BET} the diameter of the produced particle. Scanning electron microscopy (SEM) micrograph was recorded on JOEL (Japan), JXA-840A electron probe analyser instrument after coating the sample with gold for evaluation of particle morphology.

Results and discussion:

Composition of RM from different places is nearly the same and variation found is minimal. It consists of Fe_2O_3 – 33 %, Al_2O_3 – 23 %, TiO_2 – 12 %, SiO_2 – 8 %, Na_2O – 6 %, CaO – 2% with other minor constituents. Ten days of air digestion and subsequent addition of 1:1 ammonia solution produce brown coloured precipitate of iron hydroxide which on drying at 110^{0}C for 10 Hrs and heating at 500 ^{0}C for 3 Hrs gave pure α- Fe_2O_3. About 20 % of the total iron oxide was obtained from the raw material (I. e.RM) in pure form. It was then Characterised by XRD technique which shows crystalline, single phase material (Fe_2O_3) has been formed. XRD pattern of Fe_2O_3 is shown in Fig. 1. BET surface area was found to be 199 m^2g^{-1}. Powder density was found to be 3.0 g cm^{-3}. Particle size calculated from BET surface area and powder density was found to be 10 nm. SEM micrograph (Fig.2) of α-Fe_2O_3 shows particle morphology of the powders.

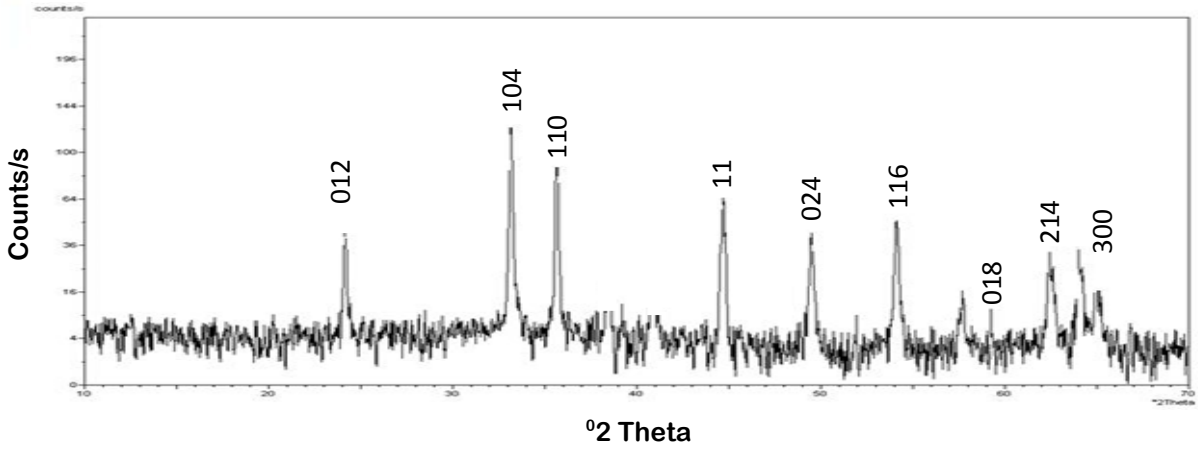

Fig. 1 : XRD pattern of as extracted α-Fe_2O_3

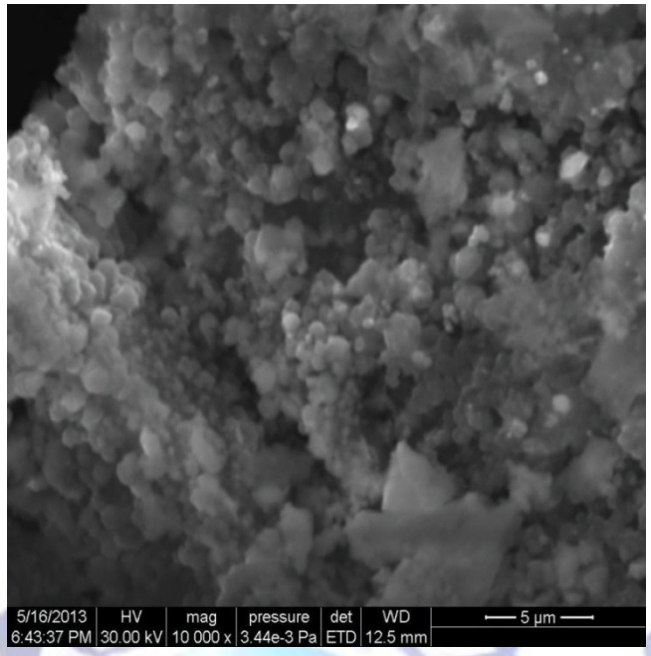

Fig. 2 : SEM Image of α-Fe_2O_3

Conclusion:

Ten days of digeston and subsequent addition of ammonium hydroxide gave brown coloured precipitate which on drying at 110 ^{0}C produced fine powders of ferric oxide with nanosized particle diameter and high surface area of about 200 gm^{-2}. Hence it can be concluded that it is possible to extract nanosized α-Fe_2O_3 from red mud though efficiency of extraction is low of about 20%.

References:

Black C T, Murray C B, Sandstorm R L, Sun S, Science, 290 (2000) 1137.

Desai R D & Peer Mohammed, J. Ind. Chem. Soc., Ind. News Edn., 8 (1945) 9.

Hosseiniam A, Reraei H, Mahjoub A R, World Aceademy of Science, Engineering & Technology, 52 (2011).

Huang D, Cao y, & Jiao H, J. Phys. Chem. B., 110 (2006) 13920.

Kazazafura K & Feges J., Ruduarsko, Met Zbornik, 3 (1964) 225.

Khalil M N, Saad E E, Wansh M M S, 3rd International Conference on Chemistry and Chemical Engineering, IPCBEE Vol. 38, IACSIT Press, Singapore (2012).

Li P, Miser D, Rabaei S, Yadav R & Hageligol M R, Appl. Catal. B, Environ, 43 (2003) 151.

Mehta S M & Patel S R, J. Am. Chem. Soc., 73 (1951) 226.
Mitsubishi Shipbuilding and Eng. Co. Ltd., Fr. Pat. 1353118 (1964).

Nakray I, Hung. Pat. 115639 (1936).

Raj K, Moskowitz B, Casciari R, J. Mag. Mater., 149 (1995) 174.

Ramsay J H & Lowe F R, Br. Pat. 9705 (1915).

Shekhah O, Ranke W, Schule A, Kolios G, Schlogl R & Angew, Chem. Int. Ed. Engl., 42 (2003) 151.

Thakur R S, Das S N, Red Mud – Analysis & Utilisation, 1st Edn., Wiley Eastern Ltd., New Delhi (1994).

Thakur R S, Sant B R, Chemical Era, XI, No. 12 (1976) 1.

Thakur R S, Sant B R, J. Sci. Ind. Res., 42 (1983) 456.

Tifenauer L X, Tschirky A, Kuhne G, Andres R, Magn. Reson. Imaging, 14 (1996) 391.

Xu H, Wang X & Zhang L, Powder Technology, 185 (2008) 176.
Zeng H, Li J, Liu J P, Wang L, Sun S H, Nature, 420 (2002) 395.