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Research Article

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Photo-synthesis of nanosized α–Fe_2O_3

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ABSTRACT

Nanoparticles α – Fe_2O_3 were successfully synthesized by photo method using aspartic acid as gelatin agent the precursor compound was calcined at a temperature of 700 C º. around (70 nm) . the surface morphology , size of the synthesized powders were structurally characterized by Uv–Visible , FT-IR, AFM , XRD and SEM. spectral techniques particle sized was found around 70 nm . the Uv – Visible spectrum was noticed the absorption and also the band gap is around 2.46ev. The photosynthesized α – Fe_2O_3powder was simple and also can be extended to prepare nanosized particles of other interesting materials .

Keywords: Nanosized α – Fe_2O_3, photo method, nanosynthesized.

INTRODUCTION

Nanoparticles refer to materials whose size in at least one dimension is between 2 – 99 nm , nano- structured materials have generated a lot of interest over the last two decades due to their attractive properties and wide range of application [(1)] , the need to control the size range and morphology and hence the properties of these nano materials have led chemists to develop various routes for synthesis of materials with morphology suitable for application in these various fields . the most utilized transition metal oxid of technological importance are the iron oxide [(2)] . iron oxide exists in nature in many forms in which hematite (α – Fe_2O_3) , maghemite (γ- Fe_2O_3) , mahnetite (Fe_3O_4) and akaganeite (β - Fe_2O_3)are the most common [(3 , 4)] . hematite is the most stable iron oxide under ambient condition and has significant scientific and technological importance [(5)]. Moreover due to stability and interesting band gap of 2.2 ev it is also used as a photo catalyst . methods that have been employed for hematite synthesis such as thermolysis , precipitation /co-precipitation , microemulsion , sol –gel , solid state reaction and other techniques have been reported [(6,7)] .

[(8)] S.D.AL – gawi synthesized CuO by pulse laser ablation (Nd: yAG , λ = 1064 nm)

And found the average diameter of 51 nm .

EXPERIMENTAL SECTION

Iron oxide nanoparticles were prepared by photo method. placed at the pyrex tube amounts of $Fe(NO_3)_3$.$9H_2O$ powder dissolved in distilled water aspartic acid as the complexing agent was then added to the metal nitrate solution with a molar ratio of 2 :1 was irradiated using (Uv source (15 W) for 12 hr. the resulting solution was evaporated to dryness , finally the residual precursor was calcined in air at different temperature:

(400, 500, 600, 700, 800, 900 Cº) for 2 hr. and the obtained α – Fe_2O_3 nanoparticales .

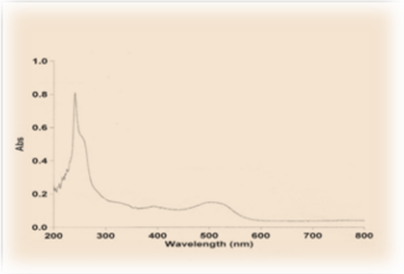
Characterizations :-

The phase identification and crystalline structure analysis were determined by X-Ray diffraction (XRD) with a Cu radiation (λ= 0.1500 nm) operated at 40 Kv and 30 mA with scan 10 ° - 70 ° . (2 Θ) at scan rate of 5 ° min^{-1} . the surface area (S_{BET}) of the nanoparticles was calculated from the nitrogen adsorption isotherms obtained at 77 Kº by using a novA2000 e apparatus . the light absorption spectrum of the MO solution was detected by Uv –Visible spectrophotometer (Uv-1800)type shimadzo at 200 – 800 nm .

FTIR spectra samples were compacted with KBr and analyzed in transmission mode in a perkin Elmer spectrum GX spectrophotometers . the recording FTIR spectrum in the wave number region of 400- 4000 cm^{-1}. the Uv – Visible absorption spectra of colloidal solution of NPs so synthesized with double beam spectrophotometer Uv-1800 with energy gap Eg (2.46ev) the average particle size and amount of aggregation of NPs were characterized with AFM CSPM

RESULTS AND DISCUSSION

NPs acquired Uv –Visible spectroscopic studies : the Uv –Visible spectra of α – Fe_2O_{3f} rom photo method and calcined at 700 Cº for 2 hr. were revealed in figure (1) the absorption peak in figure (1) correspond to α – Fe_2O_3 sample calcined at a temperature of 700 C ºshowing the absorption in the wave length of 502 nm . this can be assigned to the intrinsic band gap absorption of α – Fe_2O_3 due to the e – transitions from the valence band to the conduction band the band gap (Eg) of α – Fe_2O_3 NPs is 2.4ev .

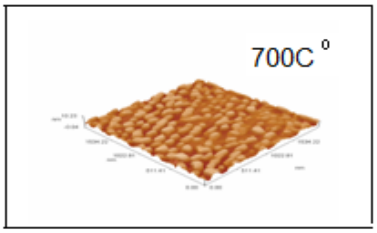


Fig(1) :UV-Visible absorption spectrum of synthesized α – Fe_2O_3 nanoparticles

Table (1) and figure (2) shows the AFM images and the corresponding size distributions of the α – Fe_2O_3nanoparticales it´ s clear from figure that average diameter of (70 nm) are observed the 3 – dimensional (3 D) AFM image of material nanoparticle in which irregular and randomly distributed and can be seen with a maximum value of 10.00 nm exhibits morphology with a root – mean – square (RMS) roughness of 1.19 nm .

Table (1) : Effect temperature and particle size of nano α – Fe_2O_3

Temp. Cº	Particle size (nm)
400	82.77
500	80.88
600	74.83
700	70.32
800	97.45
900	96.50



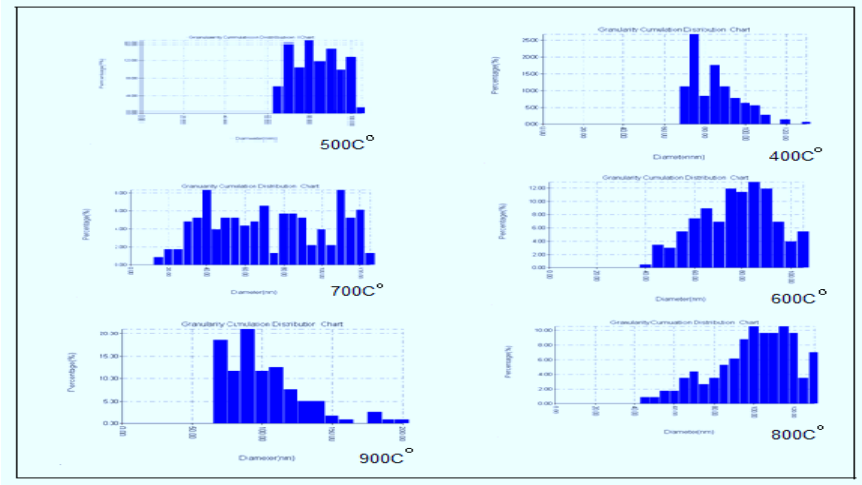


Fig.(2) AFM image of as prepared α – Fe_2O_3 nanoparticles at different temperature

Fig. (3) shows the FTIR spectrum of α – Fe_2O_3 powder acquired from photomethod and calcined at 700 C° for 2 hr. according to figure (3) the absorption peak in the range of 3863 – 3568 cm^{-1} was observed this peak corresponds to the stretching vibration intermolecular hydrogen bond (O – H) existing between the adsorbed water molecular and indicates the lower amount of hydroxyl group , the two peak at 538.14 – 484 cm -1 emexging in IR spectrum shows the presence of Fe- O vibrations made of hematite . no peak at 2900 cm -1 indicating the C – H streatching band which means all organic compounds are removed from the samples after calcinations at 700 C°.the characteristic peak at 538.14 cm-1 indicating the formation of stretching mode of α – Fe_2O_3 this specifies the occurrence of α – Fe_2O_3 NPs in calcined compounds .[3] Samira Bagheri and etc. found strong band at 586 cm^{-1} of calcined 600 C° compound shows the presence of stretching and bending vibrations of the intercalated M – O species .[9] S.Karthikeyeni and etc. were synthesized iron oxide NPs biologically using potato as starch template and found the size ranges from 29 – 40 nm and investigating the XRD using half diffraction angle of 2 ɵ.. well indexed to the pure hexagonal phase of hematite , the crystallite size was found to range between 19 and 33 nm . the XRD structural analysis of the as prepared sample shows pure hematite phase of iron oxide.

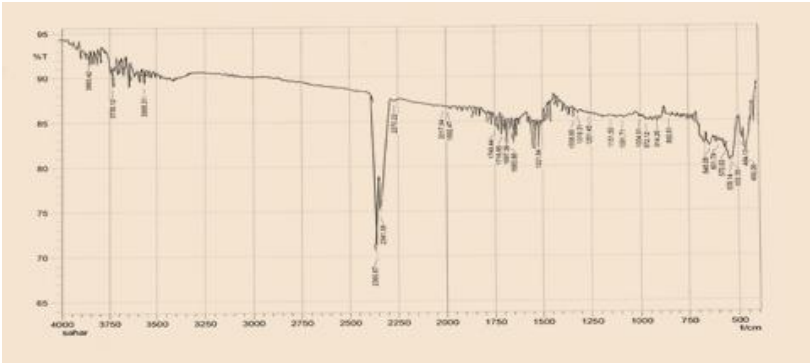


Fig.(3) FTIR spectra of the α – Fe_2O_3 NPs

XRD

Fig (4) shows the X-ray diffractograms of α – Fe_2O_3 samples synthesized by photo method and calcined at 700 C° in muffle furnace , the XRD peaks in the wide angle rang of 2θfrom 10° to 70° with Cu radiation (voltage 40 Kv and current 30 mA , λ = 1.540 A°) . speed 5 ° / min. it can be seen from figure (4) nine characteristic peaks were observed for α – Fe_2O_3 NPs (2θ= 24.2° , 33.2° , 35.6° , 41.1° , 49.5° , 54.1° , 57.4° , 62.4° and 64.0°)can be attributed to the(012 , 104, 110 , 113, 024 , 116 , 018 , 214 and 300)crystalline structures corresponds to pure α – Fe_2O_3 NPs . it was found that the ratio of the integrated intensities of (I / II)is higher than unity which correspond well to the standard XRD pattern of hematite . the diffraction peak of the synthesized α – Fe_2O_3 are in good agreement with those reported in literatures [9,10] .

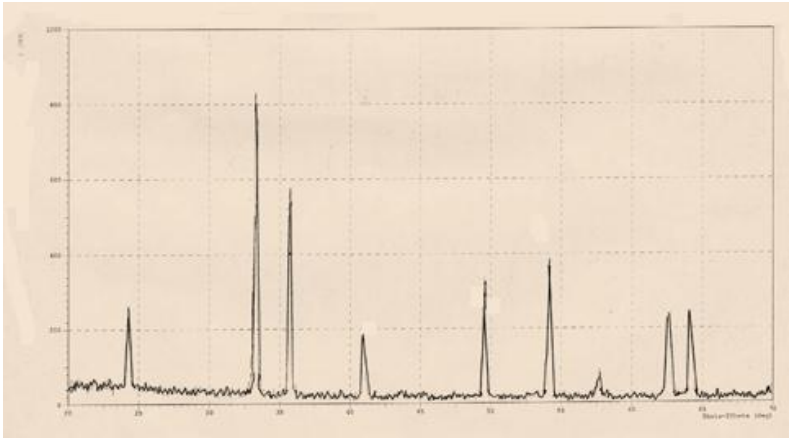


Fig .(4) XRD spectrum of α – Fe_2O_3 NPs at 700 C°

CONCLUSION

In the present work the nanosized α – Fe_2O_3 (hematite) particles were synthesized by photomethod using aspartic acid as gelatin media successfully . in AFM analysis the particle size of produced α – Fe_2O_3 was approximately 70 nm . the UV-Visible spectrum of the NPs shows absorbance at 502 nm with band gap energy of 2.4ev indicating the formation of Fe_2O_3 from the FTIR spectrum of Fe_2O_3NPs the peak 538.14 cm^{-1} corresponds to the vibrational mode of Fe – O .

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