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Original Research Article

SYNTHESIS, CHARACTERIZATION OF BFO AND IT'S CATALYTIC APPLICATION IN OXIDATION OF PRIMARY ALCOHOL

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Abstract: Bismuth ferrite ($BiFeO_3$/BFO) was prepared using wet chemical route. Precursors were bismuth nitrate and iron nitrate and citric acid as chelating agent. Solid mass was given thermal treatment .The prepared samples were characterized by XRD, SEM and BET method. BFO has several applications in physics and device applications. Applications of BFO as a highly efficient heterogeneous and recoverable catalyst for organic reactions were remain unattended. So applications of BFO for oxidation of primary alcohols are taken for study.

Key words: Bismuth Ferrite, primary alcohol, oxidation

Introduction: Applications of mixed metal oxides (MMO) have an important role in organic transformations, due to their simplicity in handling, cost effectiveness and are reusable and recyclable. Ferrites containing mixed metal oxides (perovskite ABO_3) are specially used for this. These are magnetically separable and so are easy to reuse. $BiFeO_3$ (BFO) is both magnetic and a strong ferroelectric at room temperature. Synthesis of BFO has many alternatives with respect to its application. BFO are prepared by sol gel method having fixed calcination temperatures. [1] The uniform multiferroic BFO nanoparticles have been prepared by a simple glycol-based sol–gel route at low temperature. Enhanced photocatalytic activities with H_2O_2 addition may prove novel in water treatment.[2] Barium can be doped with BFO using sol–gel process.[3] Nanoparticles assembled BFO microrods were successfully prepared via a polymer-directed solvothermal route. [4] Titania thin films on BFO substrates were deposited by pulsed laser deposition. These are used for photochemical reduction of aqueous silver cations from solution.[5] A facile aerosol-spraying approach was also developed to prepare mesoporous BFO hollow spheres with

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enhanced activity and durability in visible photocatalysis. [6] A BFO photocatalyst in the shape of uniform microspheres has been synthesized by solvothermal process assisted with chelating effect of citric acid. [7] Single-crystalline BFO nanoparticles have been synthesized through a simple chemical coprecipitation process using bismuth and iron nitrates. [8] BFO microcrystals with various morphologies (microspheres and micro/ submirocubes) were successfully synthesized by a controlled hydrothermal method. [9] These were also prepared by a sol-gel process.[10] These are also used in degradation of organic waste and hydrogen generation by water splitting, in place TiO2 as photocatalysts.[11] Microwave assisted hydrothermal method was used to synthesize crystalline BFO nanoparticles at temperature of 180°C with times ranging from 5 min to 1 h.[12] Multiferroic BFO nanoparticles were prepared by a sol–gel rapid calcination technique [13] In the present work BFO is prepared by using simple coprecipitation method using nitrate precursors and citric acid as chelating agent. It is found very robust and effective for organic reactions as catalyst.

Experiment

Catalyst preparation: All source chemicals (analytical grade) are purchased from Sigma Aldrich and used without further purification. In a typical run of synthesis, 0.040 mol/L bismuth nitrate ($Bi(NO_3)_3 \cdot 5H_2O$), iron nitrate ($Fe(NO_3)_3 \cdot 9H_2O$) with a molar ratio of 1:1 are added to form a solution using 10.0mL glycerol and 30.0mL ethanol. After 10 minutes of sonication, citric acid was added in the same molar ration drop wise to form a fluffy precipitate. This precipitate was subjected to digestion for 1 Hr at 80^0C. It is then filtered and dried at 150^0C for 2 hrs. The brown coloured lump was well grind and calcined at 450^0C for 2 hrs.

Characterizations: The sample composition is determined by EDAX. The catalyst structure is investigated by X-ray diffraction (XRD, Cu Ka radiation). (Fig 2) The grain size is calculated by using Scherrer equation based on the principal XRD peak. Surface morphology and particle size are observed through scanning electron microscopy SEM. (Fig 3) N_2 adsorption–desorption isotherms are measured using Brunauer–Emmett–Teller (BET) method. Oxidation of primary alcohol such as benzyl alcohol has been carried out to benzaldehyde in good yield.

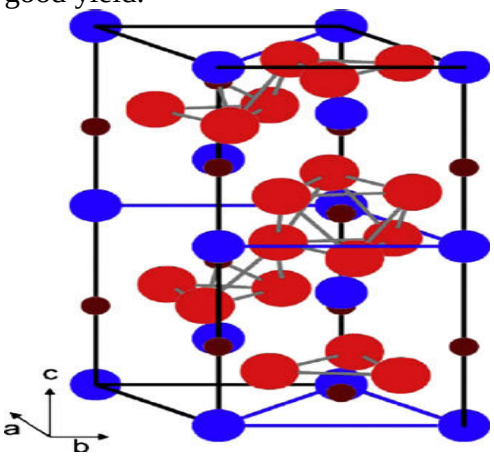

Fig 1. Structure of perovskite-type $BiFeO_3$(Blue = Bi, Red =O, Brown = Fe). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

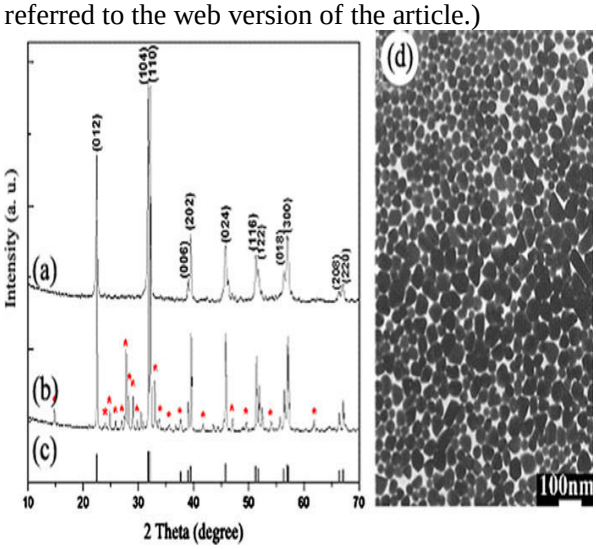

Fig. 2 XRD patterns of the samples obtained from different calcination processes:
(a) directly maintained at 450^0C for 2 hrs; (b) heated to 450^0 C with a heating rate of 5^0C/min, and held for 2 hrs. (c) Standard reflection lines from JCPDS: 86-1518. (d) TEM image of the obtained nanoparticles

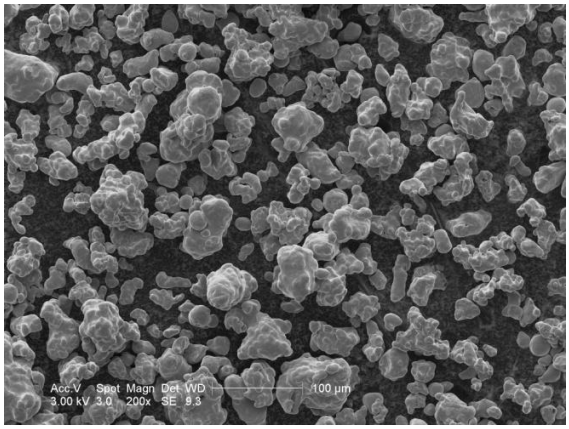

Fig 3: SEM image

Result and Discussion: From the characterization of BFO material using XRD and SEM, it is clear that nanoparticles of the crystal were obtained through this chemical route successfully. The obtained material has large surface area as well as oxidizing sites. It has carried out oxidation of primary alcohol satisfactorily.

Conclusion: From above result it can be concluded that this chemical route for the nanoparticles synthesis using citric acid is proved very easy and economical. It also adds to its catalytic properties due to large surface area.

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