Synthesis and Characterization of nano ZrO_2 and MoO_3-ZrO_2 mixed oxide Nano Particles

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Abstract- **Nano MoO_3-ZrO_2 mixed oxides were prepared using wet chemical method by mixing equimolar solutions of Ammonium molybdate(0.1M) and Zirconium oxychloride(0.1M) in 1M aqueous Sodium hydroxide and it refluxed at elevated temperature. The prepared nano MoO_3-ZrO_2 mixed metal oxide nanoparticles were characterized by XRD, SEM, EDAX studies.. From XRD studies the size of the nano ZrO_2 and MoO_3-ZrO_2 mixed oxide nanoparticles are in the nm size by Debye scherr's formula. SEM morphological studies of nano mixed MoO_3-ZrO_2 revealed the particle size with crystal and granular structure. EDAX analysis indicates the presence of Mo, Zr and O elements.**

Keywords: MoO_3-ZrO_2 , XRD,SEM,EDAX .

INTRODUCTION

Nanotechnology is an important field of modern research dealing with synthesis, strategy and manipulation of particle's structure ranging from approximately 1 to 100 nm in size. Within this size range all the properties (chemical, physical and biological) changes in fundamental ways of both individual atoms/molecules and their corresponding bulk. Novel applications of nanoparticles and nanomaterials are growing rapidly on various fronts due to their completely new or enhanced properties based on size, their distribution and morphology. It is swiftly gaining renovation in a large number of fields such as health care, cosmetics, biomedical, food and feed, drug-gene delivery, environment, health, mechanics, optics, chemical industries, electronics, space industries, energy science, catalysis, light emitters, single electron transistors, nonlinear optical devices and photo-electrochemical applications.[1].

Metal oxides play a very important role in many areas of chemistry, physics and materials science[2]. The metal elements are able to form a large diversity of oxide compounds[3]. These can adopt a vast number of structural geometries with an electronic structure that can exhibit metallic, semiconductor or insulator character. In technological applications, oxides are used in the fabrication of microelectronic circuits, sensors, piezoelectric devices, fuel cells, coatings for the passivation of surfaces against corrosion, and as catalysts. In the emerging field of nanotechnology, a goal is to make nano structures or nano arrays with special properties with respect to those of bulk or single particle species[4].

(ZrO_2) sometimes known as zirconia (not to be confused with zircon), is a white crystalline oxide of zirconium. Its most naturally occurring form, with a monoclinic crystalline structure, is the mineral baddeleyite. A dopant stabilized cubic structured zirconia, cubic zirconia, is synthesized in various colours for use as a gemstone and a simulant.[5]. Molybdenum does not occur naturally as a free metal on Earth; it is found only in various oxidation states in minerals.The free element, a silvery metal with a gray cast, has the sixth-highest melting point of any element. It readily forms hard, stable carbides in alloys, and for this reason most of world production of the element (about 80%) is used in steel alloys, including high-strength alloys and supper alloys.[6]

2.EXPERIMENTAL METHODS

2.1 PREPARATION OF ZrO_2 NANO METAL OXIDE

Zirconia nanoparticles were synthesized by using Zirconium oxychloride and sodium hydroxide as precursors. All the reagents were of analytical grade and used without further purification. The entire process was carried out in deionised water for its inherent advantages of being simple and environment friendly. In a typical preparation, solution of 0.1M Zirconium oxychloride was prepared in 100ml of deionised water and then aqueous solution of (100ml, 1M) Sodium hydroxide was added dropwise to this solution making a final volume of 100ml. This mixture was stirred well for 1hour and refluxed at 70-80ºC which resulted in the formation of white powder of zirconia nanoparticles.The precipitate was separated from the reaction mixture,washed several times with deionised water to remove the impurities.The precipitate was dried at room temperature.

2.2 PREPARATION OF MoO3-ZrO2 MIXED OXIDE

MoO_3-ZrO_2 mixed oxide was prepared at room temperature by wet chemical method. 100ml of 0.1M solution of Zirconium oxychloride, 100 ml of 0.1M solution of Ammonium molybdate and 100ml of 1M solution of sodium hydroxide were prepared by deionised water. Zirconium oxychloride and Ammonium molybdate solutions were mixed. Sodium hydroxide solution(100ml,1M) was added dropwise to the above mixture. The resulting solution was stirred for 1hour and this solution was refluxed for 2-3 hours at 70-80ºc which resulted in the formation of white powder of mixed oxide nanoparticles Sodium hydroxide is used as a capping agent.The precipitate was filtered and the filterate was washed several times with distilled water to remove the impurities. The precipitate was dried at room temperature

3. CHARACTERIZATION

The computer controlled XRD system JEOL IDX 8030 was used to record the X-ray diffraction of samples. EDAX and SEM measurements were carried by JEOL JSM-6700F field emission scanning electron microscope.

4. RESULT AND DISCUSSION

The characterization results of the synthesized mixed metal oxide nanoparticles are described below by various techniques. The results obtained are discussed in detail as follows.

4.1. X-RAY DIFFRACTION ANALYSIS

The XRD pattern of the ZrO_2 nanoparticles are shown in the (Fig:1). It shows one diffraction peak at 2θ values of 27.4163. The values of (β) observed for zirconia is 0.1632 . The peaks are identified to cubic phase of Zirconia respectively[7]. Based on the Scherrer's equation, the amorphous size of the nanoparticles are calculated as 49nm.

The XRD pattern of nano MoO_3-ZrO_2 mixed oxide nanoparticles are shown in (Fig:2).The main diffraction angles are observed at 2θ values of 27.4827 and 28.8040. The values of (β) observed for MoO_3-ZrO_2 mixed oxides are 0.4015 and 0.8029. The broad diffraction peaks suggest a well-amorphous nanomaterial and were identified to originate from (211) and (100) planes[8].Based on the Scherrer's equation, the average amorphous size of the nanoparticles are calculated as 16nm.

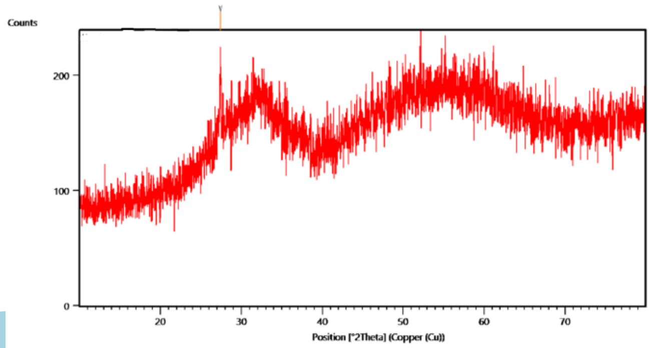

Fig:1 XRD pattern of ZrO_2 nanoparticle

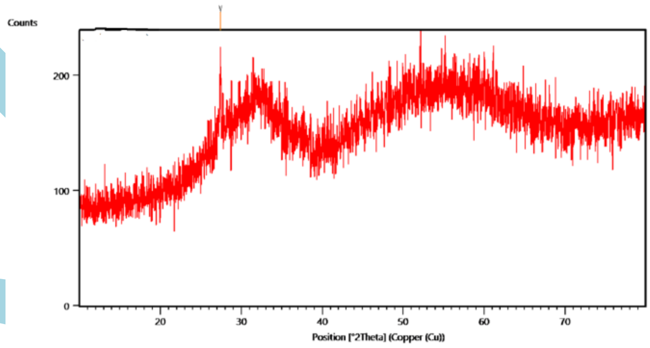

Fig:2 XRD pattern of MoO_3- ZrO_2 nanoparticle

4.2. SEM ANALYSIS

Scanning Electron Microscopy was employed to analyse the morphology and the growth feature of the nanoparticle. The representative morphology of ZrO2 oxide are displayed in (Fig:3). The representative morphology of MoO3-ZrO2 mixed oxides are displayed in (Fig:4). The SEM image reveals that the particles are of crystal and granular nanosized in nature as depicted in (Fig:3). From SEM it can be seen that the crystallite size of the particle is found to be nm. The SEM images of ZrO2 reveals that the particles are of spongy nanosized in nature as depicted in (Fig:3). The SEM images of MoO3 -ZrO2 reveals that the particles are of stone sized in nature as depicted in (Fig:4). From SEM it can be seen that the crystallite size of nanoparticles is found to be nm.

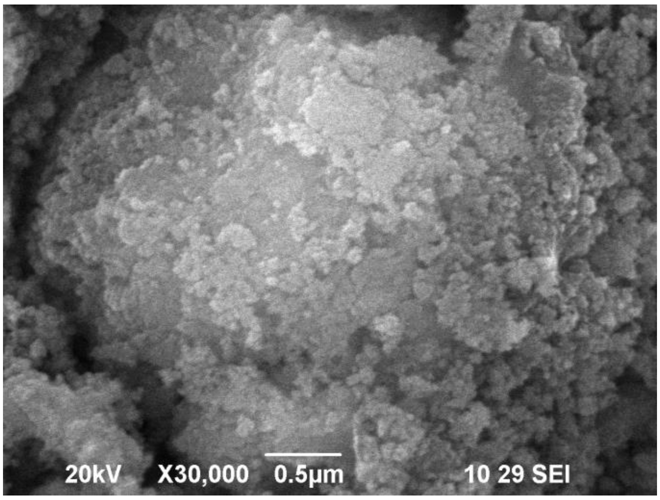

Fig:3 SEM image of nano ZrO_2 oxide

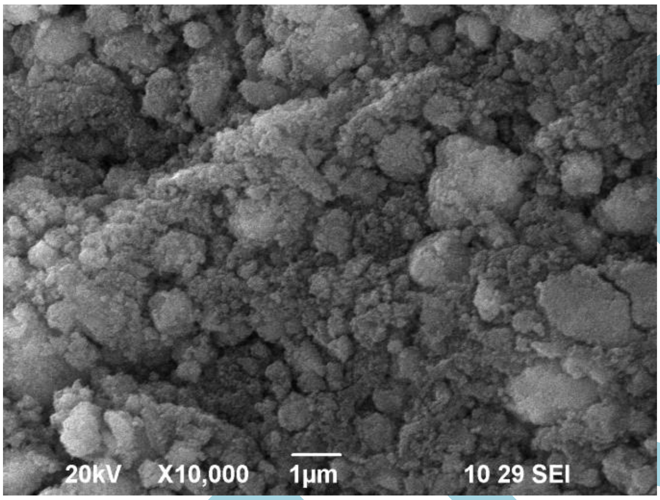

Fig:4 SEM image of nano MoO_3-ZrO_2 oxide

4.3. ENREGY DISPERSIVE X-RAY ANALYSIS

The elemental composition of the synthesized nanoparticles are carried out by EDAX spectroscopy. The EDAX spectrum (Fig:5) of the ZrO_2 oxide revealed that our samples contain Zr and O as constituent components.ZrO_2 oxide is found to have maximum value of atomic percentage in Zr and minimum value of O which confirmed the formation of nano ZrO_2 oxide.

The elemental composition of the synthesized MoO_3- ZrO_2 mixed oxides are carried out by EDAX spectroscopy. The EDAX spectrum (Fig:6) of the MoO_3-ZrO_2 mixed oxide revealed that our samples contain Mo, Zr and O as constituent components. MoO_3-ZrO_2 nanoparticles are found to have maximum value of atomic percentage in Zr,Mo and minimum value of O which confirmed the formation of nano MoO_3-ZrO_2 oxide.

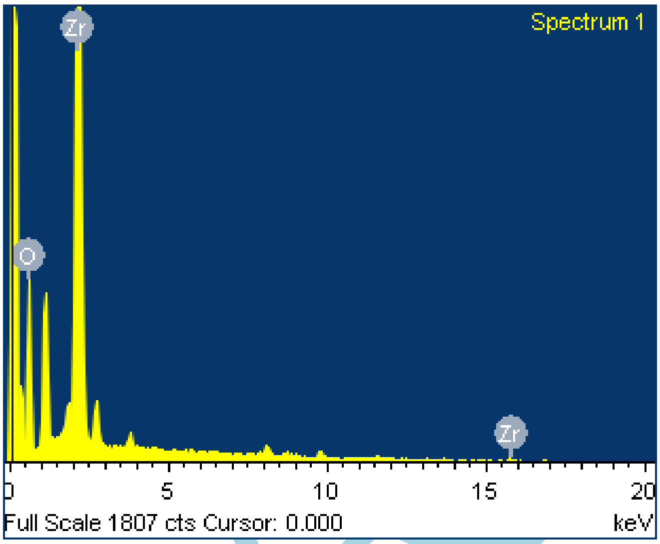

Fig:5 EDAX Spectrum of nano ZrO_2 mixed oxide

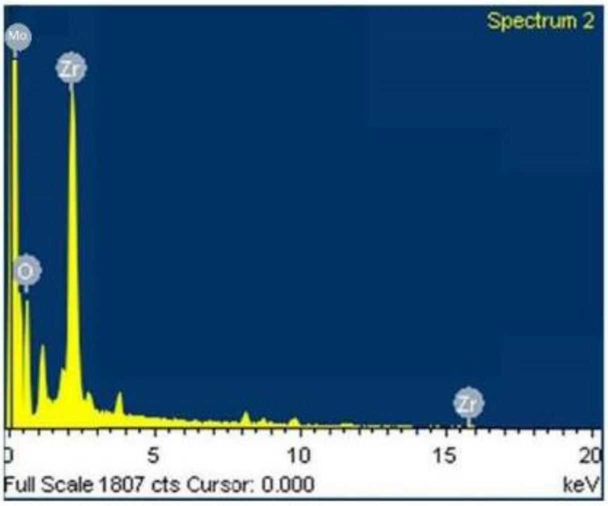

Fig:6 EDAX Spectrum of nano MoO_3-ZrO_2 mixed oxide

CONCLUSION

Nano MoO_3-ZrO_2 mixed oxides are synthesized by wet chemical method. The mixed metal oxide nanoparticles are characterized by XRD,SEM and EDAX. XRD behaviour also suggested the mixed oxide nanoparticles are in the nano scale range.The surface morphology of the synthesized mixed oxide nanoparticles exhibited different structures. The crystallite size of the systhesized nano ZrO_2 and MoO_3-ZrO_2 *oxides are* determined in the *nm range.EDAX spectroscopy confirmed the presence of Zr,Mo and O in the nano* ZrO_2 and MoO_3-ZrO_2 nano oxides. Thus the mixed oxides can be used as a potential photocatalyst, electrode material and for further medicinal applications.

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