PREFACE

Among the various types of materials, metal oxides and mixed metal oxides are known for their different kinds of applications in human life. Metal oxides play a very important role in various areas of chemistry, physics, biology and materials science because of their interesting properties. The metal elements are able to form a large diversity of oxide compounds by employing different synthesis techniques. These can adopt an enormous number of structural geometries with an electronic structure that can exhibit metallic, semiconductor or insulator character. When two or more metal oxides are mixed together either by physical or by chemical methods to fabricate mixed metal oxide, a novel set of physical and chemical properties may be obtained that would be completely different from that of the individual constituents. The distinct properties of nanomaterials arise from quantum size effects, quantum tunneling effects and surface effects. The confinement of carriers like electrons or holes in low dimensional systems or nanostructured materials could lead to a remarkable change in their physical and chemical properties due to the appearance of quantum size effects. The confinement or quantum size result becomes important when at least one dimension of the material is comparable to the de Broglie wavelength of the particle. Physical properties of the materials at nanoscale size are predicted in expressions of quantum mechanics by Schrödinger wave equation which gives a quantitative understanding of different properties of nanostructured materials or low dimensional systems. Unique properties of nanostructured materials mostly, metal oxide and mixed metal oxide nanostructures and their applications in different fields of science and technology. It has been found that these nanostructured materials are of great significance due to its tendency to fabricate unique shape, morphology and size dependent physical and chemical properties. However, expensive preparation methods, tedious procedures, complicated steps and agglomeration problems of the nanostructure's synthesis intricate task the synthesis of controlled shape and size and therefore their possible applications in different fields. An extensively controlled preparation of desired shape, morphology and size depending upon its applications of the nanostructured materials with high purity is still a large challenge for the scientific community. The worldwide researchers have been still trying to develop a simple and efficient way to synthesize highly controlled shape and size of the nanoparticles. In this context,a simple and low cost and environment friendly for the large-scale production of metal oxide and mixed metal oxide nanostructured materials and studies on their optical, catalytic and antifungal properties have not been in detail addressed in the literature. The influence of calcination temperature on the morphology, crystallinity, optical band gap, specific surface area, electric, dielectric and particle size of the nanoparticles has been investigated. The present aims of the thesis to study (a) crystal structure (b) microstructure (c) elemental analysis (d) particle size (e) electrical and dielectric behavior(f) photo catalytic properties of the following Bi based mixed metal oxide compound prepared by semi-wet route:

1 $BFeO_3$

2 $Bi_2Fe_4O_9$

3 $Bi_{12}TiO_{20}$

4 $Bi_{25}FeO_{40}$

The results of these investigations are reported in this thesis. The present thesis has been divided in to six chapters.

Chapter I:

This chapter contains a brief introduction of the subject, describing briefly the technical investigations carried out in the field of perovskite oxides and sillenite. This includes the effect of photocatalytic property and electric, dielectric properties.

Chapter II :

describes the experimental techniques used for preparation and characterization of these perovskite oxide ceramics. The semi-wet route used for preparation of these materials has been described with the help of a flow chart. DTA/TGA has been used to characterize the materials that exhibit a weight change due to decomposition or dehydration. Thermal analysis, DTA/TGA curves of the powder precursor of materials at a heating rate of 10 0C/min in static air from room temperature to 1000 0C. Powder X-ray diffraction and scanning electron microscopy have been used for study of crystal structure and microstructure of these materials respectively. Methods for density and porosity measurements have been described. Energy Dispersive X-ray spectroscopy (EDX) technique has been used for elemental analysis of the materials. Transmission Electron Microscopy (TEM) has been used for determination of particle size in the sample

Dielectric characteristictics of all the samples has been exhibited as a function of temperature (300-500 K) in the frequency range 100Hz-1MHz with the help of PSM 1735 Newton's 4th limited LCR Meter.

Chapter III

Bismuth ferrite nanostructure, $BiFeO_3$, (BFO) belonging to perovskite family, was synthesized by a chemical route sintering temperature at 800 °C for 6 h. The crystalline phase of the materials was investigated by X-ray diffraction (XRD). The morphology and particle size of the BFO samples were determined by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) analysis, respectively. The SEM images displayed the bimodal structure with the average grain size was found to be 0.6-3.7 μm. TEM observation confirmed the nanocrystalline nature of the particle having the size in the range of 50±20 nm. We have been studied the charge storage capability and impedance of the BFO using cyclic voltammetry and electrochemical impedance spectroscopy, respectively. The prepared material demonstrates pseudocapacitive nature with a charge transfer resistance of 33Ω. The synthesized material showed that the optical band gap value (E_g) of $BiFeO_3$ varies between 2.2 eV.

Chapter IV

$Bi_2Fe_4O_9$crystalline ceramic was successfully synthesized trough soft chemical route at lower sintering temperature for analysis of photocatalytic behavior with respect to adsorption of Congo Red(CR)in the presence of UV-VIS irradiation. The single phase formation of $Bi_2Fe_4O_9$ceramic was confirmed by powder X-ray diffraction studies and particle size observed by TEM analysis was found to be 148 ± 5 nm which reveals the crystalline nature of the materials. Additionally, photocatalytic activity of $Bi_2Fe_4O_9$ceramic was evaluated by the degradation of Congo Redin presence of visible light.The optical band gap (E_g) of the synthesized materials was found to be 1.8 eV. It exhibited greater photocatalytic activity than other synthesized materials like as $BiFeO_3$ as well as TiO_2 due to smaller band gap (1.8 eV). Furthermore, process variables such as pH.

Chapter V

$Bi_{25}FeO_{40}$ (BFO) polycrystalline ceramic was synthesized by soft chemical route at lower sintering temperature for investigation of photocatalytic activity with respect to adsorption of methylene blue in the presence of UV-VIS irradiation. The single phase formation of BFO ceramic was confirmed by powder X-ray diffraction studies and particle size observed by TEM measurement was found to be 25 ± 5 nm reveals the nanocrystalline nature of the materials. Additionally, photocatalytic activity of $Bi_{25}FeO_{40}$ was evaluated by the degradation of Methylene blue (MB) under visible light displayed higher photocatalytic activity than other synthesized materials like as $BiFeO_3$ as well as TiO_2 due to smaller band gap (1.8 eV). Furthermore, process variables such as pH, process time, and $Bi_{25}FeO_{40}$ dose were optimized and found to be 10, 30 min, and 0.8 mg/L, respectively. Kinetic study reveals that the adsorption process of BFO ceramic obey pseudo second order kinetics.

Chapter VI

Bismuth titanate nanostructure, $Bi_{12}TiO_{20}$,(BTO) belonging to sillenite family was synthesized by chemical route at sintering temperature 800 °C for 6 h. The crystalline phase of the materials was investigated by X-ray diffraction (XRD). The morphology and particle size of the BTO sample was determined by scanning electron microscopy (SEM) and transmission electron microscopy (TEM) analysis, respectively. The SEM images displayed the bimodal structure and average grain size was found to be in the range of 0.6- 3.7μm. TEM observation confirmed the nanocrystalline nature of the particle having size of 50±20nm. The value of dielectric constant and dielectric loss for $Bi_{12}TiO_{20}$ceramic was found to be $6.1 \times 10^3$and 2.45, respectively at 468 K and 100 Hz.