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Synthesis, characterization and electrochemical studies of nanosized Barium Cerate

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ABSTRACT

Nano sized bimetallic oxide materials have been extensively studied worldwide because of their unique properties such as electrical conductivity, magnetic property and superior mechanical properties. The exercise objects the synthesis, characterization and studies like thermal and electrochemical study of the barium cerate ($BaCeO_3$). The facial approach to preparing well dispersed nanocrystals of ($BaCeO_3$) was prepared by oxalate precursor method. Barium oxalate and cerium oxalate precursors were prepared by direct dispersion of barium and cerium salt in to oxalic acid solution separately. These precursors are undertaken for self-propagating combustion reaction under the influence of polyvinyl alcohol (PVA) fuel in the weight ration 1:1:5 to form $BaCeO_3$ as required product. X-Ray diffraction (XRD) tool which is used to study the structural confirmation of prepared bimetallic oxide nanomaterials sample. The presence of a 100% peak (110) along with other reflections in the pattern confirms the sample. Morphological study of the sample was carried out by scanning electron microscope (SEM) tool. Bonding nature of the sample waswell studied by Fourier transfer infrared (FT-IR) instrumentation. Metal confirmation in the prepared sample was identified by EDX analysis. Absorption variation was well analyzed by UV-Vis spectroscopy. Maximum absorption band at 425 nm signifies the sample phase. Raman spectroscopic (RS) study was undertaken to view its structural organization. Dynamic light scattering (DLS) study was implemented to know the size of the sample. Cyclic voltammetry (CV) and thermal gravimetric analysis (TGA) studies are also experimented to know the electrolytic and thermal behavior of the barium cerate sample. Complete decomposition of the sample takes place at 779.41°C records thermal stability.

1. Introduction

Bimetallic oxide materials can be formed by combining monometallic oxides with two different metals using thermal treatment. [1-2]. Up gradation of properties and applications may possible in bimetallic oxide materials in comparison with its monometallic oxide materials. Many researchers have extensively studied perovskite ($AB0_3$-type) materials because of their special properties. [3-4Materials that utilize $BaCeO_3$ pervoskite have gained prestige due to their multidimensional and potential technological significance in materials science. [5-6]. $BaCeO_3$ materials possess high proton conductivity in humid atmospheres and have potential for energy-related applications [7]. It is considered as a candidate material for use in solid-oxide fuel cells and other solid-state ionic devices, due to its high proton conductivity in reduced-temperature operating conditions. [8]. Barium cerate proton-conducting perovskites have been examined over decades for various applications such as, fuel cells, proton separation membranes, etc

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[9]. Furthermore, barium cerate can be also applied as a functional material for semiconductor gas sensors [10] and solid solutions can be used for capacitor applications [11]. One of the most basic methods of yielding barium cerate is solid-phase synthesis. The main disadvantages of this method are low homogeneity, the complexity of size control for material particles high sintering temperatures, and long sintering time. The solid-state reaction-based mixed oxide method of ceramic synthesis necessitates the use of repeated and prolonged mechanical grinding and calcination steps. The driving force for sintering of low-density ceramic powders into the high density ceramics are used for fuel cell application. These ceramic powders have small particle size having high surface energies makes relatively high densities in their pre-sintered state. Recent research reviews that, perovskite type $BaCeO_3$ceramic materials have received considerable attention due to their electrical properties, photo catalytic activity, and high temperature conductivity [12-14]. Literature review reporting the different synthesis methods for the synthesis of nanosized $BaCeO_3$ materials with meaningful procedures [15-16]. Self-propagating high temperature synthesis takes advantage of the self-substituting merit from highly exothermic reaction and has the potential of energy and time savings. Extension investigation work in this manner represents the synthesis of nanosized $BaCeO_3$materialintegrates the science and technology of barium cerate nanomaterials. Special attention is extrapolated by the assistance of microwave irradiation. The fluency of this method finds its importance due its simplicity and towards environmental eco-friendly system [17-19]. Homogeneous mixing of reactant precursors with fuel gives the desired homogeneous product at low temperature with better sinter ability.

The present experimental attempt is reporting that, synthesis of nanosized barium cerate by self-propagating combustion route using polyvinyl-alcohol (PVA) as a fuel with microwaves irradiation. Microwave derived $BaCeO_3$ bimetallic oxide material was well characterized by characterization tools. Electrochemical and thermal behavior of the sample was studied by instrumentation exercise. The novelty in the experimentation is reaches the facile, hybrid, and energy efficient synthetic route within very less time. Electrochemical study gives the useful technical information for various applications.

2. Experimental

2.1 Materials and methods

The chemicals used were of the analytical reagent (AR) grade (purity 99.99%) and are perchaged from Merck (Mumbai, India). Purified solvents are used in the present experimentation for preparation of solutions. Self-propagating combustion method was used for the synthesis of barium cerate nanomaterials using PVA as a fuel for the combustion reaction.

2.2 Synthesis of barium and cerium oxalate precursors

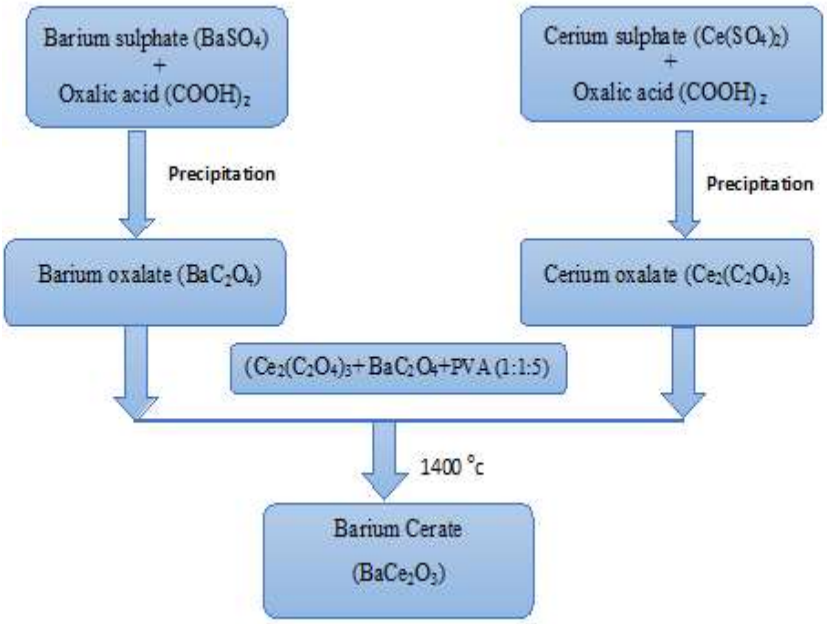
A measured quantity of barium sulphate were dissolved in minimum quantity of double distilled water and mixed with the 0.1N oxalic acid solution in equimolar ratio. Stir this solution in magnetic stirrer thoroughly for complete conversion of barium sulphate into its barium oxalate. As formed barium oxalate precipitate is washed with distilled water until it was free from sulphate ions and oxalic acid. The precipitate product was dried by passing hot air with minimum pressure for its final product [20] and is as shown in figure 1(a). Similar procedure is used for the synthesis of cerium oxalate using cerium chloride salt and the final precursor product is shown in figure 1(b).

2.3 Synthesis of $BaCeO_3$

Prepared barium oxalate and cerium oxalate precursors were grinded in a pestle and mortar with PVA in the weight ratio 1:1:5. The mixture was transferred into a china dish and was ignited for completion of preliminary combustion products. Further, it is transferred to a silica crucible and ignited in a microwave oven for its final phase product. It is observed that, initially PVA melted, then frothed and finally ignited at 1400℃ for 8 hours for final $BaCeO_3$ product. On cooling to room temperature no trace of carbon impurities was observed in the final residue. The ash colored final product of barium cerate [21]is as shown in figure 1(c) and its synthetic scheme is shown in scheme-1.The possible chemical reaction involved in the synthesis process is as given below:

$$Ba^{2+} + (COOH)_2 \rightarrow (COOBa)_2$$
$$Ce^{2+} + (COOH)_2 \rightarrow (COOCe)_2$$
$$BaO + CeO_2 \rightarrow BaCeO_3$$

Fig. 1: Optical images of a) Barium oxalate b) Cerium oxalate and c) Barium cerate



Scheme 1. Synthesis of $BaCeO_3$ nanomaterials

2.4 Characterization Techniques

The powder X-ray diffraction patterns were recorded on a JEOL JDX-8P diffractometer using CuKα radiation (1.54 Å) at 30 kV. The Fourier transform infrared (FT-IR) spectra of the samples were recorded on a Perkin-Elmer FT-IR (Model No. 1000) in the range 4000-400 cm^{-1} at a resolution of 4 cm^{-1}. JEOL JSM-6380 LA scanning electron microscope with energy dispersive micro analysis of X-Ray (EDAX) is used to study particle morphology with metal confirmation of the sample. The absorption behavior of the sample was carried out by UV visible spectrophotometric measurements using Elico spectrophotometer Technos Micro Raman spectrophotometer was used for Raman spectral measurements. Metllor Toledo star instrument was used for the thermal characterization. Cyclic Voltammetry can be performed with a general purpose potentiostat like the Germany Interface 1010B.

3. Result and discussion

3.1 Infrared studies

To assess the molecular bonding nature of the synthesized material, the FT-IR spectrum of the barium cerate sample was scanned. FT-IR measurements were carried out using KBr method at room temperature. Figures 2 shows FT-IR spectrum of microwave-assisted derived $BaCeO_3$nanopowder. The wide absorption bands that appeared at 3,480 cm^{-1} are attributed to the stretching vibration of water O-H bond (moisture). [22].Peak at 1626.cm^{-1} may be due to carbonyl group. The peak found around 1500-1550 cm^{-1} showed a stretch for C-H bond, peak around 1450-1500 cm^{-1} showed the bond stretch and also due to some overtones. It is observed from the figure that, the

vibrational bands are reflecting below 1000 cm^{-1} are due to metal-oxygen (M-O) bond and peak at 550 cm^{-1} shows the presence of metal-metal (Ba-Ce) vibrational mode [23]. This spectral data confirms the formation barium cerate sample and these results are well supported by XRD analysis.

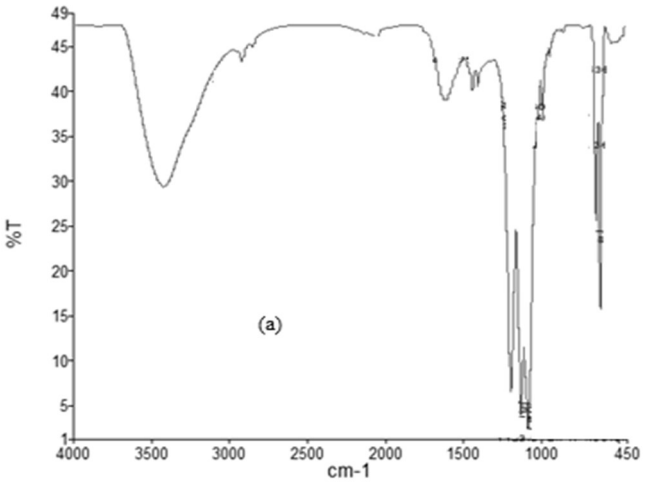


Fig. 2. FT-IR spectra of $BaCeO_3$nanomaterials

3.2 UV-Vis studies

The most widely used technique for sample analysis is UV-visible study, which identifies potential absorption. For the successful bimetallic oxide synthesis process, this stands a testimony for robust evidence. The reaction progress between barium oxide and cerium oxide was monitored by UV spectral study in aqueous product solution. Figure 3 shows the UV spectrum of a synthesized barium cerate nanomaterials sample. This figure indicates the presence of single surface plasmon absorption band at 425 nm and is the only maximum absorption band in the spectrum. Reduction of metal ions and formation of stable bimetallic oxide occurred rapidly in short span of time.

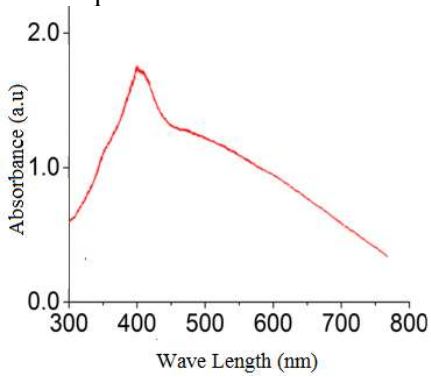


Fig .3. UV-Vis spectrum of $BaCeO_3$

3.3 TGA studies

The TGA curves of the sample $BaCeO_3$ are conducted and resulted trace is shown in the figure 4.The thermogram shows three step weight loss of the sample at different temperature. In the first region weight loss at temperature up to 41.73ºC to 194 ºC may be due to removal of physical and chemical linked water moiety. The second step feasible weight loss from 194.84ºC to359.28 ºC is observed indicates the stability of the compound. In the third region of temperature the thermal effect at 779.41 ºC was caused by the complete decomposition of the sample [24].This slow step decomposition followed leads to ash formation of sample.

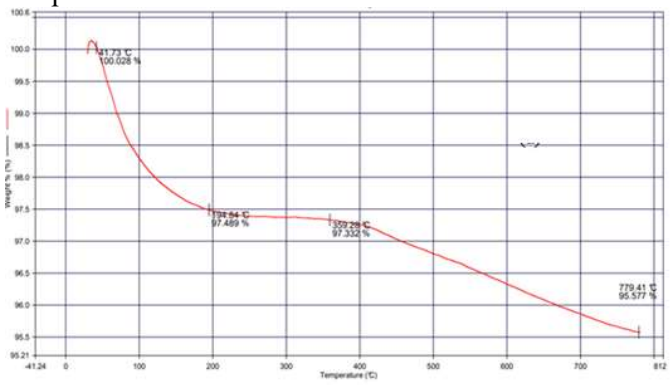


Fig. 4. TGA studies of $BaCeO_3$nanomaterials

3.4 SEM studies

Figures5 shows the SEM image of $BaCeO_3$powder sample prepared by microwave assistance. It indicates that most of the particles are in the nano range, and the sample powder has an uneven morphology and is heterogeneously distributed. Most of the particles are polydispersity and forms compacting cluster. It also shows the formation of larger grains surrounded by small interconnected grains. The sample image indicating that, the crystalline nature and an irregular morphology with some open porosity. These results are supported XRD results.

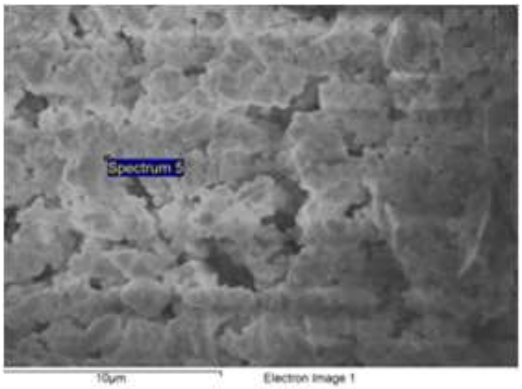


Fig 5. SEM studies of a) $BaCeO_3$

3.5 EDX studies

The EDX tool is used to hack the chemical composition of the prepared sample, which is used to reach the phase formation. Figure 6 presents the EDAX spectra for $BaCeO_3$ powder and inset table lists the chemical composition data for the samples obtained by EDX analysis. From the data, it was found that the elements were present as per the requirement. It is observed in the pattern that, the presence of Ba, Ce and O atom signals at respective binding energy confirms the formation of $BaCeO_3$ sample. The purity of the prepared samples cannot be indicated by any other elemental reflections other than those observed above [25].

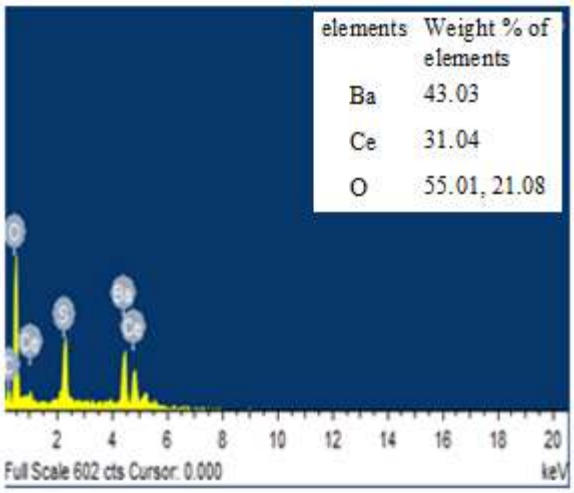


Fig. 6. EDX pattern of a) $BaCeO_3$

3.6 XRD studies

Figure 7 shows the indexed XRD pattern of combustion readied $BaCeO_3$ nonmaterial sample and obtained data is given table-1. The pattern shows the presence of Bragg reflections (110), (111), (200), (220), (310) and (222) indicates the crystalline nature of sample. Further, the pattern indicates that precursor are calcined at high temperature around 1400°C reflects an increase of peak intensities due increase in crystallinity of the oxide product. The plane is the source of the main diffraction peaks, which exhibits the features of a cubic spinal structure with only one phase. Observed diffraction peaks in the pattern are fairly in good agreement with those of standard patterns for the face centered cubic structure of JCPDS card No. 75-0431 for $BaCeO_3$ sample. It is also observed that there are no additional peak in the observed pattern suggest that, the prepared sample do not have any secondary phase formation [26-27].

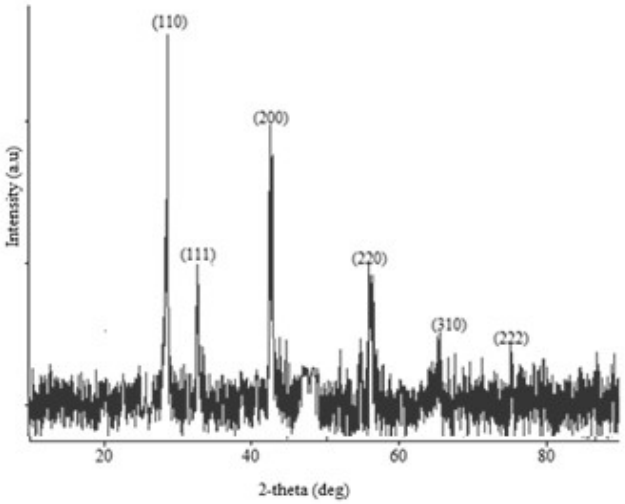


Fig. 7. XRD studies of a) $BaCeO_3$

Table 1. XRD data of $BaCeO_3$ sample

Sl.No	(hkl)	Intensity (%)	2theeta Values (Observed)
1	(110)	100	28.50
2	(011)	50	34.08
3	(200)	80	42.05
4	(220)	51	56.31
5	(310)	38	66,80
6	(222)	34	75.23

3.7 DLS studies

The particle size distribution curves obtained for prepared $BaCeO_3$ samples shown in Figures 8. From the figure it was understood that the average particle size of $BaCeO_3$ powder is found to be around the range of 60 to 80 nm. The presence of higher particle size may be due to the agglomeration of particles at high-temperature treatment.

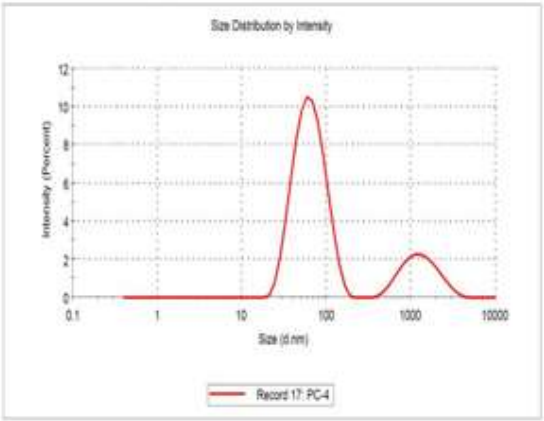


Fig. 8. DLS studies of a) $BaCeO_3$

3.8 Raman studies

Raman spectroscopic studies of bimetallic barium cerate pervoskite have extensively studied and spectral feature are well understood. This spectral technique could be used to track the local structural changes observed peaks due to local structural symmetry change induced by defects in the system. Room temperature Raman spectrum for the prepared $BaCeO_3$ sample composition is shown in figure 9. Raman modes are observed in the figure reflects the $BaCeO_3$ sample observed at 106 and 460cm^{-1}. Non observance of higher frequency mode is due large scattering supports indicates the absence of moisture. Appearance of mode 460 cm^{-1} is assigned to barium cerate means the local distorted symmetry structure. [28-29].

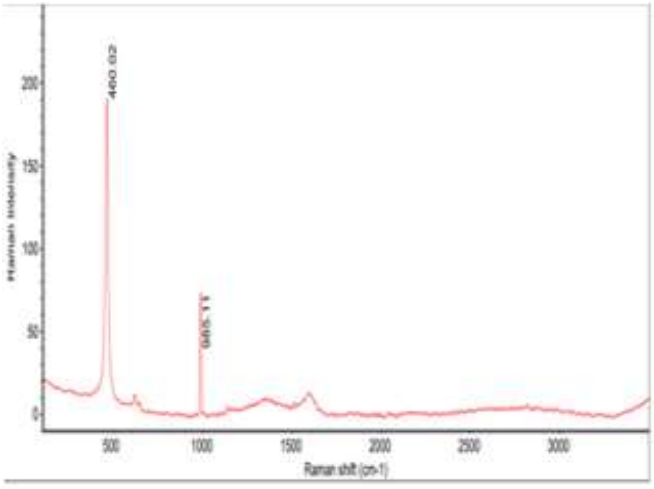


Fig. **9**. Raman studies of a) $BaCeO_3$

3.9 Cyclic voltammetry

Figure 10 shows CV spectra of as prepared $BaCeO_3$ nanomaterial. It shows the highly intense peak at 0.35V of potential indicates that sensing of mercury by compound. The obtained data indicates that, the prepared compound is good catalyst and good sense of mercury. The GCE/ $BaCeO_3$ nanocomposite was evaluated for the detection of highly toxic Hg (II) by cyclic voltammetry in 0.1 M PBS (pH=7) (PBS= phosphate buffer solution). 50 microliter Hg (II) solution was added into PBS, a peak appeared at 0.35V on the GCE/ $BaCeO_3$nanomaterial which is responsible for the detection of Hg (II) but the bare GCE did not show an appreciable peak for the addition of analytic. The cyclic voltammetry related to the electro catalytic current for Hg detection at GCE/ $BaCeO_3$was much larger with lesser over potential compared to GCE. So, one can easily find that enhanced peak current and decrease in the over potential established the barium cerate nanomaterial acts as an effective catalyst to activate the reaction for the detection of the analytic [30-31].

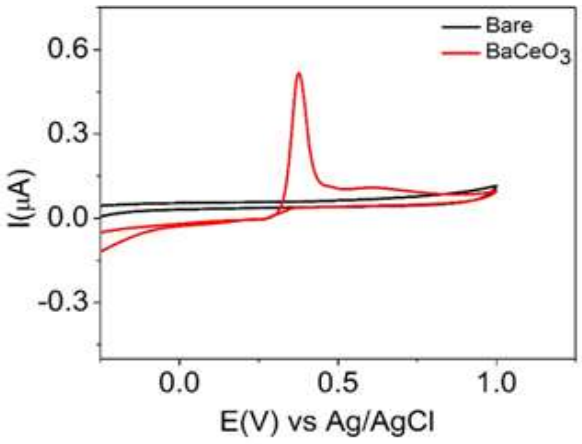


Fig.**10**. CV studies of a) $BaCeO_3$

4. Conclusions

The nanosized $BaCeO_3$ has been successfully synthesized for its phase formation by microwave-assisted route. This method offers advantages such as simplicity and relatively at required processing temperature. The obtained oxide sample exhibited desirable properties demonstrating the effectiveness of this synthesis route and hence may be used for the synthesis of other bimetallic oxide materials. Further characterization and testing could provide additional insights into their potential uses. The structural analysis of the powder sample prepared by this method has shown desired structure. It signifies the effective properties in thermal and electrochemical applications.

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