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Synthesis and Characterization of Barium Oxide Nanoparticles

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Abstract: *Nano meter sized oxide draws much attention because of their unusual physical and chemical properties. The metal oxide nanoparticles have wide range of applications in the field of electronics, fuel cells, batteries, agriculture etc. The research has been focused on the preparation and characterization of Barium oxide nanoparticles. It was carried out using anhydrous $BaCl_2$ powder and ammonia with distilled water as a precipitant agent by Thermo-chemical method. The synthesized nanoparticles were characterized by XRD, FTIR spectroscopy. The XRD pattern showed that the synthesized BaO nanoparticles were of tetragonal structure. They were crystalline in nature with average crystalline size of 29nm.The FTIR study confirmed that the functional groups appeared at 1610 and 856 cm^{-1} in BaO nanoparticles were due to the BaO stretching and N-H bending modes.*

Keywords: *Barium oxide nanoparticles, Thermo-chemical method, XRD, FTIR.*

I. Introduction

Nanophase materials are newly developed materials with grain size at the nanometer range(10^{-9}m) i.e., in the order of 1-100 nm. The particle size in a nano material is 1 nm. They are called as nanomaterials.A nanometer is one billionth of a meter. For comparison, thickness of a single human hair is about 80,000nm, a red blood cell is approximately 7000nm wide and a water molecule is almost 0.3nm across.

Scientists and engineers are nowadays interested in nanoscale which is from 1nm to 100nm. At nanoscale, the properties of materials are very different from those at a larger scale. Therefore, the nano-world is in between quantum world and macro world. Nanoscience is concerned with the study of phenomena and manipulation of materials at nanometer scales. Nanotechnology is the design, characterization, production and application of structures, devices and system controlling shape and size at the nanometer scale.

II. Materials used for synthesis

2.1 Barium chloride

Barium chloride is an inorganic compound with the formula $BaCl_2$. It is one of the most common water soluble salts of barium. Like other barium salts, it is toxic and imparts a yellow-green coloration to a flame. It is also hygroscopic. The physical properties of Barium Chloride

- Molar mass - 244.26 g/mol.
- Density - 3.0979 g/cm^3.
- Melting point - 960°C.
- Apperance - white.

2.2 Ammonia

Ammonia is a colourlessgas with a characteristic pungent smell. It is lighter than air, its density being 0.589 times that of air. Ammonia is a compound of nitrogen and hydrogen with the formula NH_3. Ammonia solution, also known as ammonium hydroxide, ammonia water, ammoniacal liquor, ammonia liquor, aqua ammonia, aqueous ammonia, or simply ammonia, is a solution of ammonia in water. It can be denoted by the symbols NH_3(aq). The ammonia molecule has a trigonal pyramidal shape as predicted by the valence shell electron pair repulsion theory (VSEPR theory) with an experimentally determined bond angle of 106.7°.

The important properties of ammonium hydroxide has its values of molar mass, density, melting point, boiling point and appearance are 35.04 g/mol, 0.91 g/cm3 (25 %), 0.88 g/cm3 (32 %), −57.5 °C (−71.5 °F; 215.7 K) (25%), −91.5 °C (32%), 37.7 °C (99.9 °F; 310.8 K) (25%), 24.7 °C (32%) and Colourless liquid.

It is lighter than air, its density being 0.589 times that of air. It is easily liquefied due to the strong hydrogen bonding between molecules; the liquid boils at −33.3 °C (−27.94 °F), and freezes at −77.7 °C (−107.86 °F) to white crystals. Ammonia may be conveniently deodorized by reacting it with either sodium bicarbonate or acetic acid. Both of these reactions form an odourless ammonium salt.

III. Synthesis of barium oxide nanoparticles

The research has been focused on the preparation and characterization of bariumoxide nanoparticles synthesized by thermo-chemical method. To synthesis barium oxide nanoparticles,20ml ammonia was gradually added (approximately 30 mins) to 2g anhydrous $bacl_2$ powder under vigorous stirring (550-700rpm) to produce the solution containing ba^{+2}. Because of the exothermic reaction of $bacl_2$ with ammonia, ammonia should be slowly added to the precursor. The solution temperature was set at rt 33˚c, 55˚c and 75˚c and the ph was set in the range of 10. Distilled water was subsequently added as a precipitant agent while vigorous stirring of solution continued until white precipitate formed. The resulting precipitate was filtered and washed twice with deionized water. The white precipitate is dried at 100˚c for 1 ½hrs at oven.to produce bao nanoparticles dried precipitate is calcinated at 500˚c for 2hrs at furnace.

Fig 3.1 Barium Oxide

3.1 Barium oxide nanoparticles:

Barium oxide is a chemical compound. Its chemical formula is BaO. It contains barium and oxide ions.
Barium oxide, BaO, is a white hygroscopic non-flammable compound. It has a cubic structure and is used in cathode ray tubes, crown glass, and catalysts.It is prepared by heating barium carbonate with coke, carbon black or tar or by thermal decomposition of Barium nitrate.It is also a source of pure oxygen through heat fluctuation.
The properties of BaO nanoparticles are

- Molar mass -153.326 g/mol
- Density -5.72 g/cm3
- Melting point-1,923˚C
- Crystal structure- Tetragonal

IV. Structural Studies

There are number of analytical techniques used to characterize the materials. One of the methods, X-ray powder diffraction (XRD), is an instrumental technique that is used to identify the minerals, as well as other crystalline materials. XRD provides the researcher a fast and reliable tool for routine mineral identification. XRD is particularly useful for identifying fine grained minerals and mixtures or intergrowths of minerals that may not lend themselves to analysis by other techniques.

4.1 X-Ray Diffraction

The XRD measurements were carried out at room temperature using PANALYTICAL XPERT-PRO with monochromatic beam of Cu Kα radiation (1.5406 A). An accelerating voltage of Kv and a current of 30 mA with a scan rate of 0.01° s-1 were used. The XRD patterns were recorded in the 2θ range of 10 - 80° versus intensity. The values of 2θ, d- spacing, relative intensity and FWHM were obtained from the XRD pattern. Identification of phases was carried out by comparing the diffraction pattern obtained from XRD with standard JCPDS database. The lattice parameters and cell volume were calculated using UNIT CELL software.

4.1.1 Results and Discussion

The XRD patterns recorded for the barium oxide specimens are shown in tables 4.1 & 4.2. In this pattern the peaks occur at 2θ =19.6544 , 26.9162, 33.6190, 43.0288, 44.8540, 55.9910, 60.9634 ,and 68.2371,with the hkl values (1 2 0), (1 0 1), (1 1 0), (1 1 2), (1 0 3), (2 1 1) (1 1 4) and (2 2 2) respectively, it resembles the presence of body centered structure of BaO nanoparticles with the lattice parameter a=4.209A˚ c=6.508A˚.The obtained values were well coincidence with the (JCPDS File No:89-8425). All the diffraction lines observed have been found to be in good agreement with standard lines as given in the above file. Thus, the

comparison confirms the presence of BaO phases in the present specimens with tetragonal crystal structures. The 'd' values for all the lines have been calculated and the patterns have been indexed. Consequently, the cell parameters and the cell volumes of the compounds have been computed by using the software referred as "unit cell". A comparison of all the parameters is presented in table 4.3. The table shows that the computed values of all the parameters in close agreement with those in the standard JCPDS files.

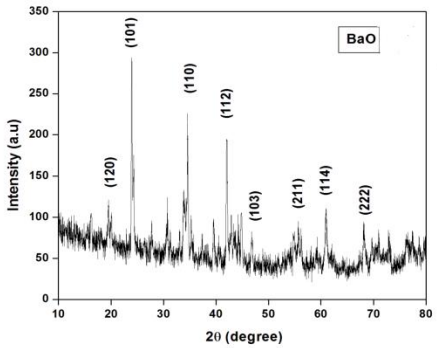

Fig 4.1 XRD graph for the BaO specimen

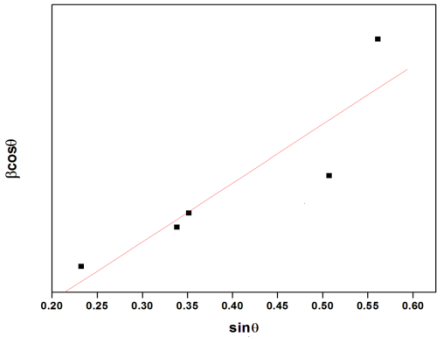

Fig 4.2 plot of βcosθ as a function of sinθ for the BaO specimen

Table 4.1. XRD Parameter of BaO Nanoparticles

S.no	2θ	Measured d-spacing (A˚)	Standard d-spacing (A˚)	FWHM (degree)	(h k l)	Relative Intensity %
1	26.9162	3.7177	3.7208	0.1673	(1 0 1)	100
2	19.6544	4.5131	4.5169	0.8029	(1 2 0)	30.38
3	33.6190	2.5889	2.5911	0.2007	(1 1 0)	72.86
4	43.0288	2.1480	2.1498	0.2676	(1 1 2)	58.76
5	44.8540	2.0190	2.0207	0.2007	(1 0 3)	12.51
6	55.9910	1.6410	1.6423	1.6059	(2 1 1)	11.41
7	60.0625	1.5198	1.000	0.2676	(1 1 4)	35.22
8	68.2371	1.3733	1.3744	0.4015	(2 2 2)	17.17

Table 4.2. Crystalline Size, Micro strain, Macro strain and Dislocation density

S.No	(h k l)	Crystalline Size (nm) ($x10^{-9}$)	Micro strain(m^{-4}) ($x10^{-3}$)	Macro strain	Dislocation density (m^{-2}) 10^{15}
1	(1 0 1)	10.4961	0.7135	0.1272	9.0770
2	(1 2 0)	40.3362	3.4492	0.1240	0.6146
3	(1 1 0)	43.3362	0.8354	0.0384	0.5324
4	(1 12)	44.7589	1.0891	0.0283	0.4991
5	(1 0 3)	27.0399	0.8089	0.0574	1.3676
6	(2 1 1)	5.8561	0.6182	0.0491	29.159
7	(1 1 4)	36.0066	1.0054	0.0778	0.7713
8	(2 2 2)	24.9839	1.4490	0.1000	1.6021

Table 4.3. Unit cell parameter for BaO

Parameters	BaO(Tetragonal)	
	Observed	Standard(JCPDS Fill No:89-8425)
a(A˚)	4.209	3.807
c(A˚)	6.508	6.621
V(A^3)	144.35	99.99

V. Spectroscopic studies

5.1 Fourier Transform Infrared (FTIR) Spectrometer

This technique is one of the most important and widely used spectroscopic techniques of analyzing quantitatively the structural units of the unknown compounds. It helps to identify the functional units, internal structure of the molecules and nature of the chemical bonds of a compound.

5.1.2 Result and discussion

Figure 5.1 shows the FTIR spectra for Barium Oxide specimen. The spectrum has two profiles 1610 $cm^{-1}$and 856 cm^{-1}, the vibration frequency at 1610 cm^{-1} is the characteristic of Ba-O stretching mode ,where as vibration frequency at 856 cm^{-1} is of characteristic of N-H Bending mode.SelvaKumar et al have reported that

the vibrational band around 1590 cm^{-1} corresponds to the characteristics of B-O stretching mode in, similarly the vibration band observed around 842cm^{-1} is associated with the distortion vibration of N-H is the characteristic of N-H Bending mode. The assignments of the observed bands in the spectra are also presented in table 5.1.

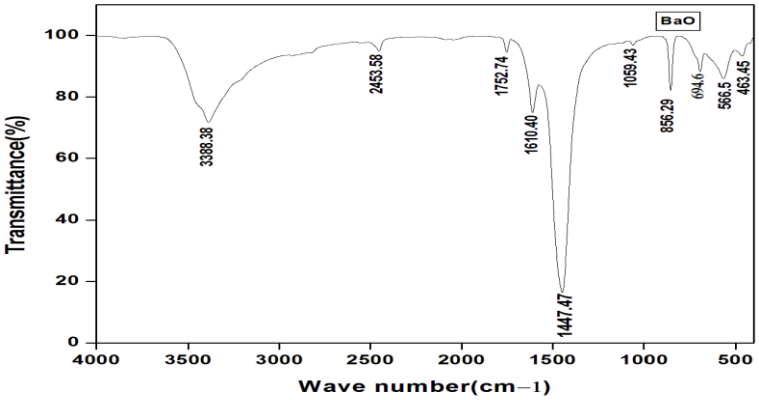

Fig 5.1 FTIR spectrum for the Barium Oxide specimen

Table 5.1 : FTIR Spectroscopy data for the Barium Oxide specimen along with the band assignments and a comparisons with literature.

S. No.	Transmittance bands (cm^{-1})		
	Selvakumar Et al(2015)	**Present Study**	**Band assignment**
1.	1590cm^{-1}	1610 cm^{-1}	Ba-O Stretching
2.	842 cm^{-1}	856cm^{-1}	N-H Bending

Table 5.2 FTIR Spectral of Barium Oxide Specimen

S.NO	WAVENUMBER (cm^{-1})	BAND ASSIGNMENT
1.	3388	O-H Stretching
2.	2453	C-N Stretching
3.	2043	C-N Stretching
4.	1752	C-O Stretching
5.	1610	Ba-O Stretching
6.	1447	C-C Stretching
7.	1059	C-N Stretching
8.	856	N-H Bending
9.	694	C-H Stretching

VI. Conclusion

- In this work the BaO nanoparticles were prepared by using thermo-chemical method.
- The synthesized nanoparticles were characterized by XRD,FT-IR spectroscopy .
- The XRD pattern showed that the synthesized BaO nanoparticles were crystallite in nature, they have tetragonal structure and the average crystallite size is 29nm.
- The FT-IR study confirmed the functional groups appeared at 1610 and 856cm-1 in BaO nanomaterials were due to the Ba-O Stretching and N-H bending modes.
- More Studies have to performed in order to shower an insight in deducing further details and discern for a better interpretation of the results obtained.
- Barium oxide nanoparticles can be used as a flame retardant in plastics, coatings, fiber and textiles and also in some alloy and catalyst applications.

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