Synthesis and Characterization of Magnesium Oxide (MgO) Nanoparticles by Co-precipitation Method

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***Abstract* - Metals are able to form a large number of oxides. These metal oxides play an important role in many areas of chemistry, physics and material science. An important metal oxide known as magnesium oxide (MgO) having good reactivity is widely used in producing electronics, catalyst, ceramics, oil, paint *etc.* Magnesium oxide Nano-particle is non-toxic and need a small amount, so it is suitable for the development of flame-retardant fiber additives. In addition, nano magnesium oxide added in fuel can inhibit corrosion. In this communication Magnesium oxide nanoparticles were successfully synthesized by co-precipitation technique at room temperature using magnesium nitrate and sodium hydroxide as a precursor. The morphological investigation of MgO nanoparticles was done by various analytical techniques Scanning Electron Microscope (SEM-EDX) for morphological studies. X-ray Diffraction (XRD) indicates the crystallinity and crystal size of MgO nanoparticle. Fourier Transform Infrared (FTIR) spectroscopy is used for analysing the functional groups of the sample.**

***Index Terms* - MgO, Nano-particle, Spectroscopy, X-ray Diffraction (XRD)**

INTRODUCTION

Nano size materials have different properties compared with bulk materials. Nanoparticles are particles between 1 and 100 nanometers in size and it are having attracted a great attention in recent years because of their unique electronic, physical, magnetic, chemical and optical properties compare with bulk materials [1, 2]. Most of the researchers are working with metal oxide nanoparticles because of their unique properties such as hydrophobic, photo catalytic, stability and etc. Hence, they are used in many applications named as coatings, catalysts, anti-bacterial, medical sciences, sensors, semiconductors, capacitors and batteries [3]. In past decade, the unique properties of nanomaterials have created interest to the researchers to develop simpler and inexpensive techniques to synthesis nanostructures for technologically importance. Metal oxides nanomaterial with high surface area and porosity have attracted considerable interest for scientific research due to their potential application such as functional components for nano electronics, optoelectronics and sensing devices.[4] Magnesium oxide is an inorganic compound having thermal stability, high surface reactivity, very good heat resistance, high chemical and alkali resistance [5, 6]. Magnesium is II A group element with atomic number 12 and Oxygen is VIA group element with atomic number 8. The compound MgO is having boiling and melting points as 3600ºC and 2852ºC [7].

Magnesium oxide, often called periclase [8] (from Greek word periklao, peri “around”, klao “to cut”), is white hygroscopic solid mineral. Its empirical formula is MgO and its lattice consist of Mg^{2+} ions and O^{2-} ions, together bonded by ionic bond (Figure 1). Magnesium oxide is generally produced by the calcination of magnesium hydroxide $Mg(OH)_2$ or magnesium carbonate $MgCO_3$. Thermal treatment, used when calcination process occurs, affects the surface area and pore size and also the final reactivity of formed magnesium oxide. Used temperature can be divided into three groups, 700 ˚C to 1000 ˚C, where caustic calcined magnesium oxide is formed, 1000 ˚C to 1500 ˚C, where lower chemical activity magnesium oxide is formed and calcination over 1500 _C, where reduced chemical activity type of refractory magnesium oxid is formed, that is mostly used for electrical and refractory applications [9].

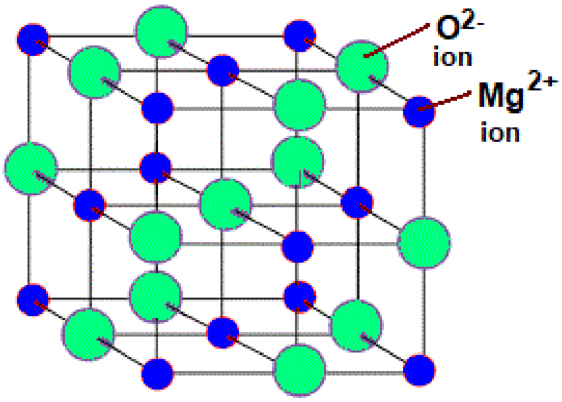

Figure 1. Structure of magnesium oxide crystal

Physical properties (see [10]) make magnesium oxide a good candidate for various applications. It is colorless to brown or black (based on the presence of iron or other foreign element). Considering the surface structure, it is visible that MgO has simplest oxide structure, called Rock-Salt structure. Its density is around 3.579 g/cm^3 and hardness around 5 on Mohs scale. Thermal conductivity value of sintered magnesium oxide is defined at T = 100˚C as 36 W/(mK). Due to refractory properties, the melting and also boiling points of magnesium oxide are very high (melting point: 2800˚C, boiling point: 3600˚C). Value of electrical resistance is depended on the purity of magnesium oxide. For high purity magnesia, the values of electrical resistivity can reach 1016 Wm. Specific resistance is mostly depended on chemical purity, but for higher values of temperature, i.e., 2000˚C and more, the purity of magnesia does not have any influence on values of electrical resistivity. The dielectric constant of magnesium oxide is in the range from 3.2 to 9.8 at 25˚C and under frequency 1 MHz. Chemical properties and surface composition of magnesium oxide are also influenced by the calcination procedure [11,12] (used temperature and used medium, i.e., air or vacuum) and also by the source of the precursor. Based on the various result, physical adsorption of water only occurs if MgO contains surface defects, such as high quantity of pores [13].

Applications of magnesium oxide includes various industry sectors. For their refractory properties, it is a valuable fireproofing ingredient in construction materials. Also, in applications where corrosion [14] is not acceptable such as nuclear, chemical or superalloy industries. It has a usage in medical applications [15], where MgO is used for relief of heartburn and sour stomach, as an antacid, magnesium supplement, and as a short-term laxative. Other applications include insulators [16], fertilizers [17], water treatment [18], protective coating [19], etc. Currently, there are trends to use nanoscale fillers [20]. In general, the nanotechnology is the production of functional structures in the range of 0.1–100 nm by various physical or chemical methods [21]. This fact also applies to magnesium oxide. The sol-gel technique [22] or hydrothermal technique [23] could be used for the production of nanoscale magnesium oxide. For an electrical application, e.g., in high voltage insulation, the MgO represents a prospective filler. Especially, due to wide band gap (7.8 eV) and high-volume resistivity (1017 Wm). It is the highest value of volume resistivity from commonly used nanoscale oxides [24]. Also, owing to its very large band gap, excellent thermo dynamical stability, low dielectric constant and refractive index, it has been used as a transition layer for growing various thin film materials [25, 26]. Generally, MgO nanostructures were synthesized by dehydration of Mg(OH)2 or by decomposition of various magnesium salts using co-precipitation method, thermal evaporation [27].

These oxide materials can be prepared by different synthesis methods such as solution combustion, Co-precipitation, Sol-Gel, hydrothermal, Solvothermal, Microwave Assisted Sol-Gel, green synthesis in these methods Co-precipitation is a one of the best method to synthesis nanoparticles without agglomeration in the yield, size can be easily controlled [28]. The prepared nanoparticles are characterized by X-ray diffraction (XRD), UV-Visible Spectroscopy. SEM-EDX and FTIR Spectroscopy. The Interpretation or analysis of the data are discussed in this chapter and confirmed that the particles are synthesized are of the MgO nanoparticles by Co-Precepitation method.

3.2 Experimental Detail

The chemical reaction occurred during the synthesis is shown here and also block diagram of all over process is shown below:

$Mg(NO_3)_2 .6H_2O + 2 NaOH \rightarrow Mg(OH)_2 + 2NaNO_3$

Δ

$Mg(OH)_2 \rightarrow 2 MgO + H_2O$

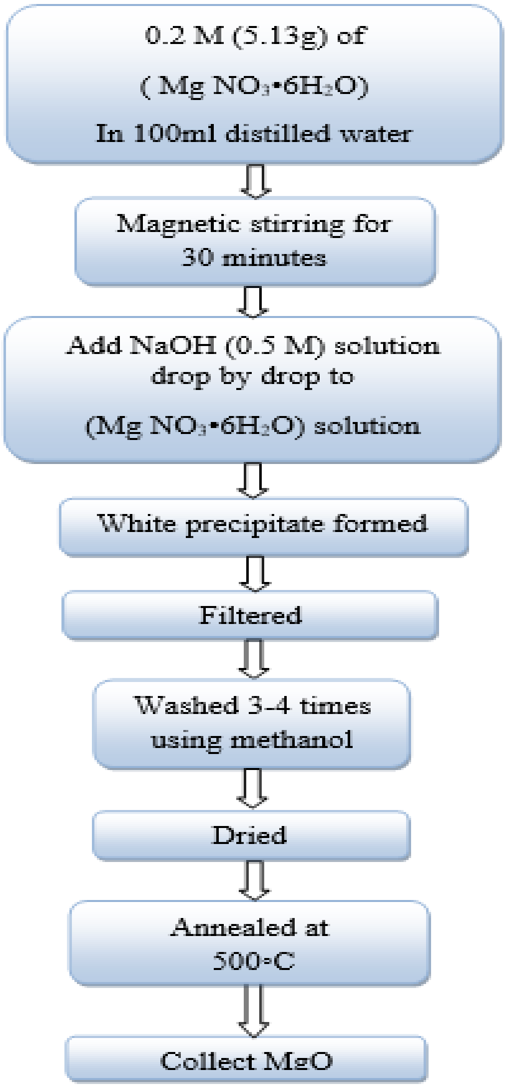

Figure 2: MgO nanoparticle synthesis by Co-precipitation method

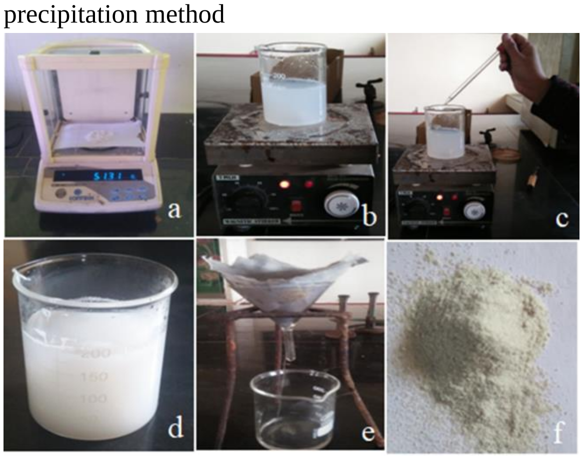

Figure 3: Experimental images of the synthesis process where (a) Weighing , (b) Stirring, (c) Dropping, (d) White Precipitate, (e) Filtering, (f) MgO Nano Particles

The synthesis of MgO nanoparticles is divided into various steps, such as mixing, stirring, filtering, drying, and grinding. Initially $Mg(No_3)6H_2O$ of weight 5.13 g taken into beaker to form 0.2 M solution in distilled water. Then 2.041 g (0.5 M) of NaOH dissolved in 100 ml distilled water. Magnesium nitrate solution stirred for half an hour using magnetic stirrer for constant stirring. Then 0.5 M sodium hydroxide solution was added drop wise by dropper to the prepared Magnesium nitrate ($MgNo_3.6H_2O$) Solution while Stirring it continuously. After 30 minutes milky white color precipitate of Magnesium Hydroxide appeared in beaker. The PH of the solution was 12.5 as measured by the pH paper. Precipitate were filtered by filter paper and washed with methanol three to four times to remove ionic impurities and then dried in air, then after drying white powder sample were annealed in air for two hours at 300 ◦C and 500 ◦C. The dried powder is then crush and make it very fine powder by using mortal pestle.

RESULT AND DISCUSSION

SEM-EDAX Analysis:

The figure 4 presents the scanning electron microscopic (SEM) image for the characterization of MgO NPs, where the scale bar is 200 nm and the nano particles formed are agglomerated forming cluster. Average particle size of synthesized nanoparticles is 14nm.

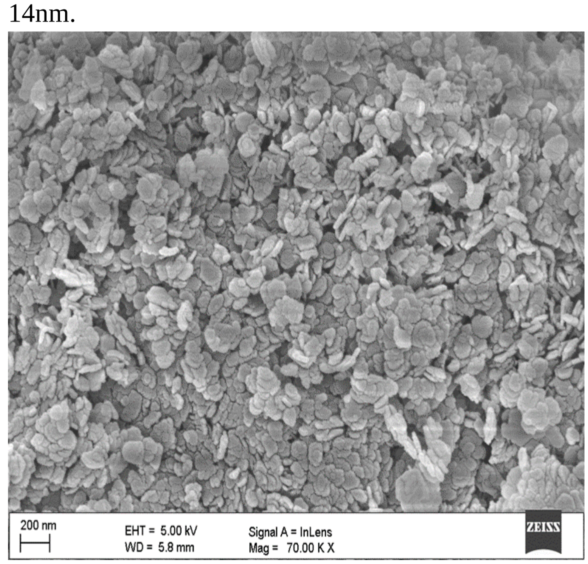

Figure 4: SEM image of MgO nanoparticles

The chemical composition of the Magnesium oxide nanoparticles was studied using EDAX and is drawn in Fig. 5. The weight percentages of Mg and O are close to the stoichiometry, this result confirms that the the MgO nanoparticles contains only Mg and O elements.

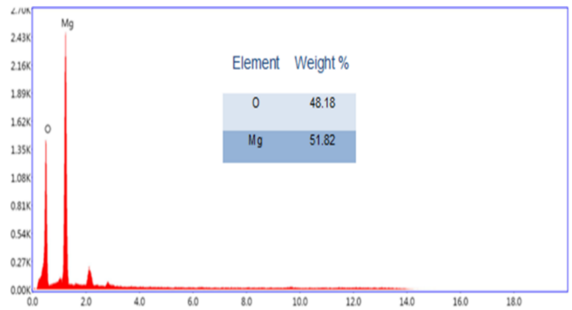

Figure 5: EDAX image of MgO nanoparticles

FTIR Analysis:

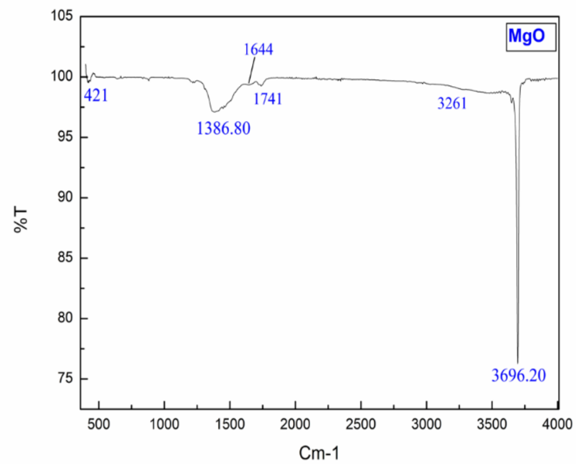

Figure 6: FTIR Spectrum of MgO nanoparticles.

Figure 6 shows the spectra of MgO nanoparticles and the analysis done in the range of 400-4000cm^{-1}. The strong infrared band near 3696 cm^{-1} was observed for the O–H bond vibrations of hydroxy group. The peaks observed at 421 cm^{-1} indicate the presence of MgO NPs. The absorption band at 1386 cm^{-1} is related to C–H bending vibrations of aromatic tertiary amine group. The peaks observed at 1741 cm^{-1} and 1644 cm^{-1} indicate the presence of Strong C=O and C=C stretching, while peak at 3261 cm^{-1}indicates the presence of weak broad O-H bond stretching. The presence of MgO nanoparticle peak is near to the earlier result given by Shikha *et al* [29].

CONCLUSION

Magnesium oxide (mgo) nanoparticles synthesis and characterization by Co-precipitation method. MgO Nanoparticles were successfully made by co-precipitation method. Co-precipitation is a one of the best method to synthesis nanoparticles without agglomeration in the yield, size can be easily controlled. The synthesized nanoparticles have been characterized by FTIR, SEM-EDX. FTIR revealed the spectra of MgO Nanoparticles. FTIR spectroscopy was applied to identify of the functional group and feature of Mg-Oxalate and Magnesium Oxide Nanoparticles. The sample is scanned at the range of the wave numbers 400-40000 cm^{-1}. The SEM images showed morphology of mgo nanoparticles and the nanoparticles formed were agglomarated forming cluster with an average particle size of 14 nm. Chemical composition of prepared nanopaticles were studied using EDX.

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