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Energy Dispersive Spectroscopy (EDX) and Morphological Analysis of Synthesized MgO Nanoparticles

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ABSTRACT

Magnesium oxide (MgO) nanoparticles were synthesized by simple co-precipitation method. This research provides a thorough characterization of synthesized magnesium oxide (MgO) nanoparticles using Energy Dispersive Spectroscopy (EDX) and morphological analysis (SEM). EDX analysis provides vital information about their elemental composition and level of purity. Interestingly, the morphological investigation revealed the creation of MgO nano-flakes, demonstrating a unique structural property of the nanoparticles.

Overall, the EDX analysis and morphological analysis offer a complete characterization of the produced MgO nanoparticles. The elemental composition shows that it has a good stoichiometric ratio of Mg and O i.e 1:2 and highly pure and surface morphological properties shows high potential. This study establishes a foundation for future scholars to use MgO nanoparticles in various fields. These results also paved the path for specialized applications in disciplines such as catalysis, sensing, and biomedical engineering.

Keywords:

Magnesium oxide nanoparticles

Co-precipitation method

Energy Dispersive Spectroscopy (EDX)

Scanning Electron Microscopy (SEM)

1. Introduction

Energy Dispersive X-ray Spectroscopy (EDX) and Scanning Electron Microscopy (SEM) are powerful analytical techniques frequently used in materials science and other scientific areas. SEM provides high-resolution investigation of material surface morphology, allows the examination of topography, texture, and shape. SEM can be used to investigate material cross-sections, allowing for depth profiling and understanding sample internal structure. SEM has tremendous magnification capabilities, making it possible to observe structures at the nanoscale.

The elemental composition of materials is analyzed using EDX. It detects distinctive X-rays generated when electrons in the sample are moved, revealing the elements present.

When combined with SEM, EDX allows for spatially resolved element analysis.

EDX gives quantitative data on elemental composition, allowing researchers to estimate the concentration of elements in a sample.

SEM and EDX are essential tools for thorough material characterization, assisting researchers in understanding their physical structure, surface characteristics, and elemental makeup. These techniques are commonly used in quality control processes in industries such as metallurgy, electronics, and nanotechnology to ensure product consistency and reliability. SEM and EDX are used in scientific research to create novel materials, investigate biological specimens, and progress nanotechnology. SEM and EDX are used in forensic science to analyze trace evidence such as gunshot residues and paint fragments.

Nowadays, there has been a significant increase in the usage of different kinds of nanoparticles with the rapid developments in nanotechnology. Nanoparticles are particles ranging in size 1-100 nm. In recent years, researchers have been interested in the fabrication of nano-structured metal oxide materials because of their potential applications in advanced technologies[1]. Discoveries over the last decades have revealed that materials manufactured in the form of very small particles vary dramatically in their physical and chemical properties, usually to the extent that a totally new phenomenon is established[2]. Metal oxides are extremely important materials for technology used in electrical and optical systems[3]. Due to its high melting point and low heat capacity, magnesium oxide (MgO) is a perfect choice for shielding[4]. MgO recently has been demonstrated to have high bactericidal activity in aqueous conditions due to superoxide production[5]. Magnesium hydroxide is a harmless, anti-corrosive, thermoplastic, and non-hazardous flammable substance that dehydrates endothermically while suppressing odors during a fire[6]. To synthesize MgO nanoparticles various fabrication techniques are applied, such as chemical vapour deposition(CVD)[6], plasma enhanced chemical vapour deposition (PECVD)[7], pulsed laser deposition(PLD)[8], laser ablation[9], molecular beam epitaxy (MBE) sputtering method[10], hydrothermal method[11], sol-gel method[11, 12], co-precipitation method [13]and thermal decomposition of hydroxide or carbonate[14, 15]. Among the techniques mentioned above, template free synthesis or co-precipitation method is a viable approach for large-scale production of MgO nanoparticles[8]. A new and cost-effective approach for synthesizing nano-sized MgO nanoparticles with narrow dimensions. To tackle difficulties such low reactivity and catalytic action, greater dispersion and surface area are required[16]. Moreover, oxide shape and particle size fluctuate according to conditions such as pH, calcination rate, and temperature[17]. Ultrasonic waves can produce nano-sized metal oxides[18]. From previous studies[19], it was found that using extreme chemical and physical conditions can accelerate the synthesis process and produce smaller crystals with more uniform size distributions than traditional methods for manufacturing nanoscale MgO[16].

2. Experimental Procedure

2.1 Reagents:

Magnesium sulphate heptahydrate ($MgSO_4.7H_2O$), ammonia solution (NH_4OH) and distilled water are reagents that are used for the synthesis of MgO nanoparticles.

2.2 Synthesis of Magnesium Oxide Nanoparticles

The co-precipitation approach was used to create MgO nanoparticles. In this investigation, magnesium sulphate heptahydrate ($MgSO_4.7H_2O$) was synthesized utilizing a 0.1 M solution of the chemical in 250 ml of distilled water. Then, at 100°C, 0.25-0.5 M ammonia solution (NH_4OH) was dynamically introduced into the aforementioned solution. The resulting mixture was reflux by 100°C for duration of 12 hours leading to the reaction between $MgSO_4.7H_2O$ and NH_4OH, yielding $Mg(OH)_2$. When the reaction was completed, the subsequent white product of $Mg(OH)_2$ was centrifuge with distilled water numerous times to eliminate any residual impurity[20]. The washed product was dried in air at 120°C for 1 h to obtain as-synthesized $Mg(OH)_2$ nanoparticles. The as-synthesized products were then calcinated at 400°C for 2 h in the presence of air to convert the $Mg(OH)_2$ nanoparticles to MgO nanoparticles[20]. Finally, MgO nanoparticles were collected and analyzed as indicated in Fig.1.

The reaction is summarized as follows:

$MgSO_4.7H_2O + 2NH_4OH \rightarrow Mg(OH)_2 + (NH_4)_2SO_4 + 7H_2O$

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

2.3 Characterization of Material

The detailed examinations were examined under a scanning electron microscope (SEM) that operates at 15 kV and equipped with energy dispersive analysis of X-rays (EDX), all enabled by SEM machine. These examinations included surface morphology, elemental composition, and elemental distribution mapping.

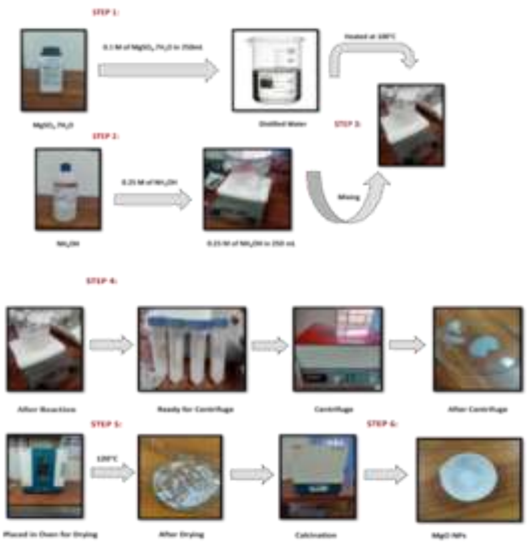


Fig.1. Synthesis of magnesium oxide nanoparticles

3. Results and Discussion

3.1 Energy Dispersive Spectroscopy (EDX) of MgO Nanoparticles

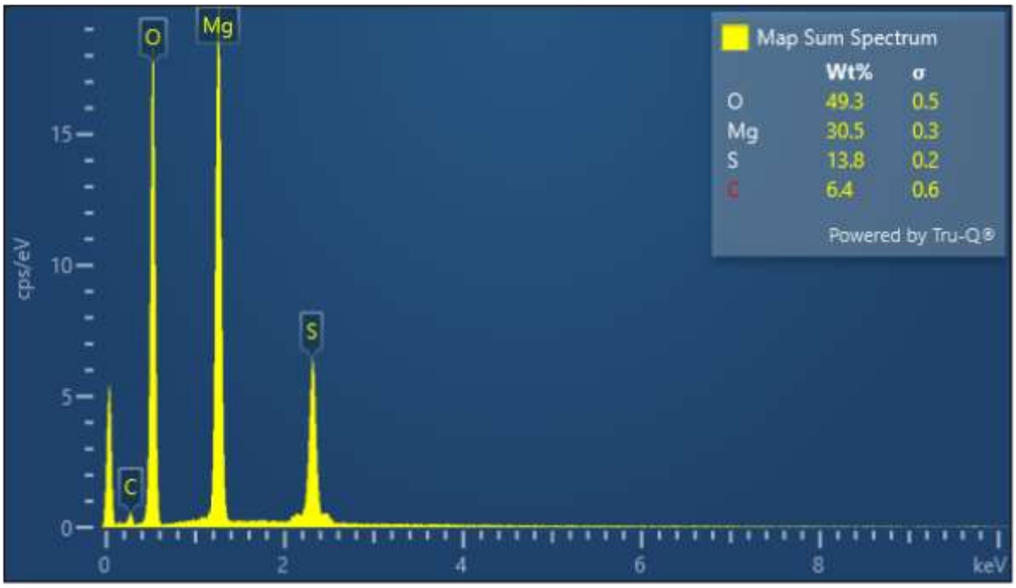
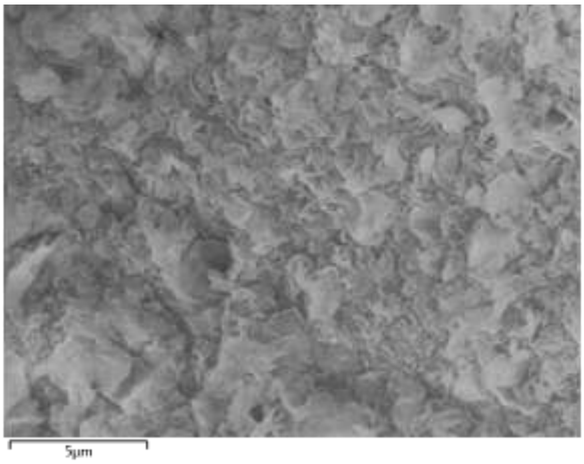


Fig.2. *EDX spectroscopy of magnesium oxide nanoparticles*

Energy dispersive X-ray spectroscopy is a most popular technique for determining and assessing the elemental composition of a very small sample of material. The EDX (or EDS) spectrum, which shows the energy versus relative counts of detected X-rays, is collected and processed to determine the qualitative and quantitative composition of elements in a sample. Elements carbon, oxygen, magnesium, sulphur are found in the EDX report of MgO nanoparticles as shown in Fig.2 [21]. The following chart shows that magnesium and oxygen are present in higher percentage as compared to other elements. Carbon and sulphur are present as an impurity. Carbon may be present due to pollution from the environment or substrate, as well as phytochemical adsorption on the surface of NPs. We observe that it has a good stoichiometric ratio of Mg and O. In MgO nanoparticles, the molecular ratio of Mg: O were determined to be 1:2 verifying the purity of the produced nanoparticles, and the ratios aligned consistently with the precursor molar ratios[21].

3.2 Morphological Analysis of MgO Nanoparticles



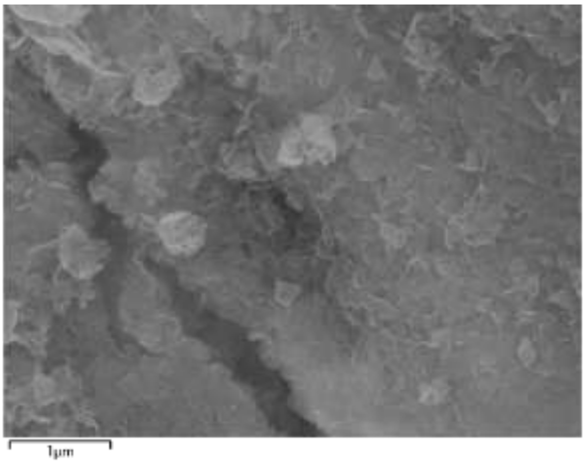


Fig.3. *SEM spectroscopy of magnesium oxide nanoparticles*

Scanning electron microscopy was utilized to analyze MgO nanoparticles structure, size, and surface morphology. SEM pictures of MgO at 5μm resolution reveal flake-like structures formed through nanoparticles aggregation. These flakes exhibit a dense and interconnected configuration, with no clearly boundaries between them. Similarly, depicts a micrograph with a resolution of 1μm as shown in Fig.3. A higher resolution of 1μm reveals fine details of aggregated nanoparticles, emphasizing the complexity and thickness of the structure[20].

4. Conclusion

In conclusion, the co-precipitation method is successfully employed for the production of MgO nanoparticles with controlled properties. EDX analysis confirms the valuable insights into the elemental composition of MgO nanoparticles, revealing the presence of carbon, oxygen, magnesium, and sulphur. Importantly, the stoichiometric ratio of magnesium and oxygen was found to be well-maintained, signifying the high quality and purity of the synthesized MgO nanoparticles. The identification of carbon as an impurity highlighted the importance of understanding and controlling environmental factors during the synthesis process.

The morphological analysis through SEM unveiled the structural characteristics of the MgO nanoparticles. The images displayed distinctive flake-like structures, formed through the aggregation of numerous nanoparticles. These structures exhibited a dense and interconnected configuration, lacking clear boundaries between individual particles. This morphology suggests a high potential for applications in various fields.

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