

Synthesis of Delafossite Materials Towards Gas Sensing Applications

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ABSTRACT

Delafossite oxides (CuMO_2) are emerging as versatile materials due to their thermal stability, p-type semiconductivity, and adjustable optical features, making them useful for transparent electronics, catalysis, and sensing. This study reports the synthesis of Cu-Al-MgO_2 via a sol-gel combustion method, producing porous nanostructures with uniform elemental distribution. Structural analyses confirmed rhombohedral crystallinity and nanoscale grain size, while microscopy revealed extensive porosity favorable for adsorption. Gas sensing evaluations showed strong p-type response, rapid recovery, and stability at moderate temperatures. Magnesium incorporation enhanced oxygen vacancy formation, charge carrier mobility, and adsorption kinetics, significantly boosting sensing efficiency compared with single-cation delafossites.

Keywords: Cu, MgO_2 , Al, Nanofillers, Solution Cast Technique, SEM and UV etc.,

1. INTRODUCTION

A sensor is a device that detects changes in its surroundings—such as heat, pressure, movement, light, or gases—and converts them into signals, most often electrical, that can be processed or stored. In simple terms, sensors function like the sensory organs of machines, giving them the ability to interpret and react to the world, much as humans rely on sight, hearing, and touch. Among the many types, gas sensors are particularly important because they identify the presence or concentration of gases in the environment. Since many gases are invisible, odorless, and harmful, these devices provide critical protection where human senses cannot. Once a gas is detected, the sensor transforms this data into a measurable signal, which can then be analyzed for safety, quality, or operational control. Different technologies are used in gas detection, including semiconductor sensors, electrochemical sensors, infrared sensors, catalytic bead sensors, and photoionization detectors. Each type has specific strengths, whether in sensitivity, selectivity, or response time. Their applications span across safety systems, environmental monitoring, medical devices, industrial processes, and smart technologies. Compact design, low energy use, and affordability make gas sensors essential not only for industry but also for modern homes, wearable devices, and Internet of Things (IoT) solutions [1-3].

Delafossite compounds (ABO_2 type) represent a unique class of layered oxides with remarkable structural and functional versatility. Their crystal structure, composed of alternating linear O–A–O bonds and sheets of edge-sharing BO_6 octahedra, produces strong anisotropy and diverse physical properties. What sets them apart is their wide spectrum of electrical behaviors. For instance, CuAlO_2 and CuCrO_2 act as rare p-type transparent

conducting oxides, combining optical transparency with electrical conductivity—an uncommon trait among oxides.[4-5] On the opposite end, PdCoO_2 exhibits metallic conductivity that rivals pure metals, making it one of the most conductive oxides known. Beyond electrical properties, delafossites are valued for their stability and adaptability. Their structural flexibility allows substitution at both A and B sites, enabling researchers to fine-tune optical, magnetic, and catalytic performance. Transition-metal-based members such as CuFeO_2 display magnetism and multiferroic characteristics, offering potential in spintronics and multifunctional devices. Environmentally, many delafossites are composed of earth-abundant, non-toxic elements, making them sustainable materials for green technologies. Due to this rare combination of conductivity, stability, and tunability, delafossites are being explored in transparent electronics, energy storage, photocatalysis, and gas sensing, highlighting their growing significance in modern materials science [6-7].

2. SYNTHESIS OF THE MATERIAL

The synthesis of delafossite compounds such as CuAlO_2 and CuFeO_2 is commonly achieved through the sol–gel auto-combustion method, chosen for its simplicity, cost-effectiveness, and ability to produce fine, homogeneous powders. The process begins with dissolving stoichiometric amounts of copper(II) nitrate trihydrate and the desired B-site nitrate salt—either aluminum or iron—in a solvent mixture of ethanol and deionized water. Citric acid is then introduced as a chelating agent to stabilize the cations and prevent premature precipitation, while also serving as fuel for combustion. The pH is adjusted to around 7 using aqueous ammonia, allowing controlled gel formation. Upon heating, the gel undergoes auto-combustion, generating a porous, lightweight powder composed of nanometer-scale particles. This precursor is further calcined at 800 °C for several hours to eliminate residual organics, enhance crystallinity, and ensure phase-pure delafossite. The resulting materials exhibit excellent structural stability and functional properties for sensing application [8-10].

The synthesis of magnesium oxide (MgO) nanoparticles can be carried out effectively using the sol–gel technique. The process begins with dissolving magnesium nitrate hexahydrate in distilled water to prepare a transparent precursor solution. Once the salt is fully dissolved, N-methyl-2-pyrrolidone (NMP) is carefully introduced while stirring. This addition plays a crucial role in stabilizing the solution and encouraging sol formation. The mixture is then placed on a magnetic stirrer and maintained at around 50 °C for approximately five hours, allowing a homogeneous colloidal sol to develop. After sol formation, the system is left to undergo condensation reactions, during which the fluid gradually thickens, eventually forming a gel-like substance. This gel is then transferred into an oven and dried at 100 °C to eliminate residual water and solvent, yielding a solid dried gel [11-13]. To obtain the desired oxide phase, the dried sample is subjected to calcination in a muffle furnace at about 600 °C for three to four hours. This high-temperature treatment decomposes nitrate groups and organic residues, leaving behind pure MgO. Finally, the brittle calcined mass is carefully ground using a mortar and pestle to produce fine white MgO nanopowder, typically exhibiting a cubic crystalline structure suitable for diverse applications.

3. CHARACTERIZATION

3.1. ANALYSIS OF ULTRAVIOLET (UV):

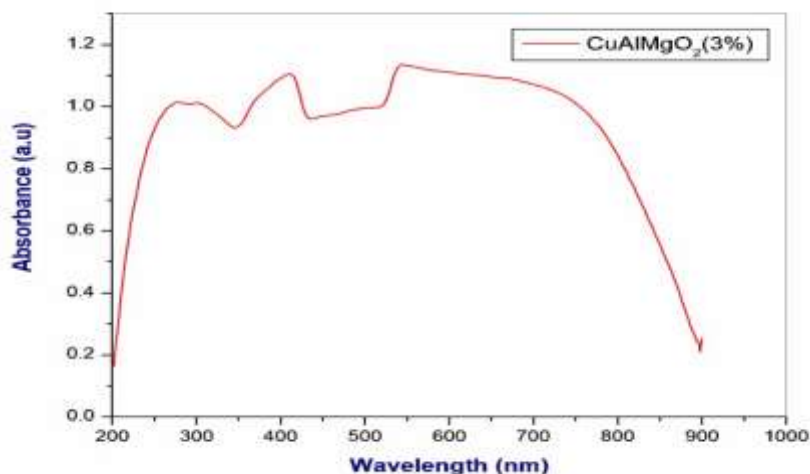


Fig.1: UV-Vis absorbance spectrum of CuAlMgO₂(3%)

The UV–Vis absorption spectrum of CuAlMgO₂ (3%), measured across the 200–900 nm range, reveals a broad absorption pattern that extends through both the ultraviolet and visible regions. A sharp absorption edge is noticeable around 250–300 nm, reflecting the material’s strong ability to absorb UV photons. Between 300–500 nm, small shoulders and spectral variations appear, which are often linked to electronic transitions caused by dopant states, structural defects, or irregularities on the material’s surface. Importantly, the spectrum continues into the visible region, which indicates that the compound possesses a relatively narrow optical band gap. This property allows it to capture visible light effectively, improving its efficiency in light-driven processes. Such optical behavior is highly beneficial for photocatalytic and optoelectronic applications, as it enhances the interaction between light and matter, ultimately boosting the material’s performance and making it a suitable candidate for advanced energy and electronic technologies [14].

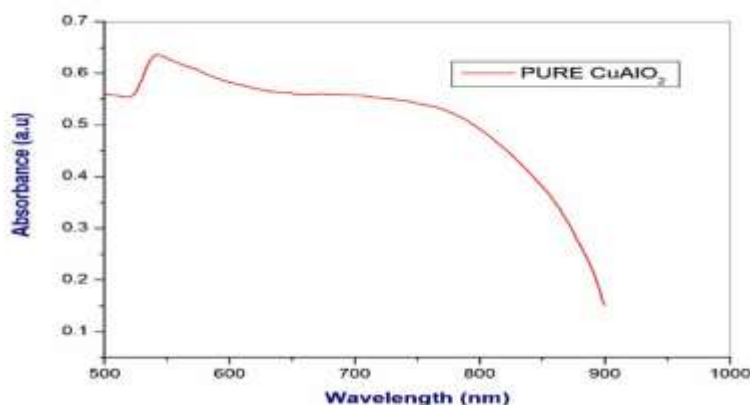


Fig.2: UV-Vis absorbance spectrum of pure CuAlO₂

The UV–Vis absorbance spectrum of pure CuAlO₂, measured within the wavelength range of 500–900 nm, exhibits a relatively broad absorption response in the visible region. A distinct shoulder peak is observed around

~550–600 nm, followed by a gradual decrease in absorbance as the wavelength increases toward the near-infrared region. The absorption intensity remains moderate, with a maximum absorbance of approximately 0.65 a.u. This spectral behavior suggests that pure CuAlO_2 can interact effectively with visible light, although its absorption is weaker compared to the UV range typically associated with semiconducting oxides. The extended tailing of the absorption edge into the near-infrared region may be related to defect states or sub-bandgap transitions. Such optical characteristics indicate potential utility of CuAlO_2 in visible-light-driven photocatalysis and optoelectronic applications, where moderate light absorption and defect-mediated transitions can play an important role [15].

3.2. ANALYSIS OF SCANNING ELECTRON MICROSCOPE:

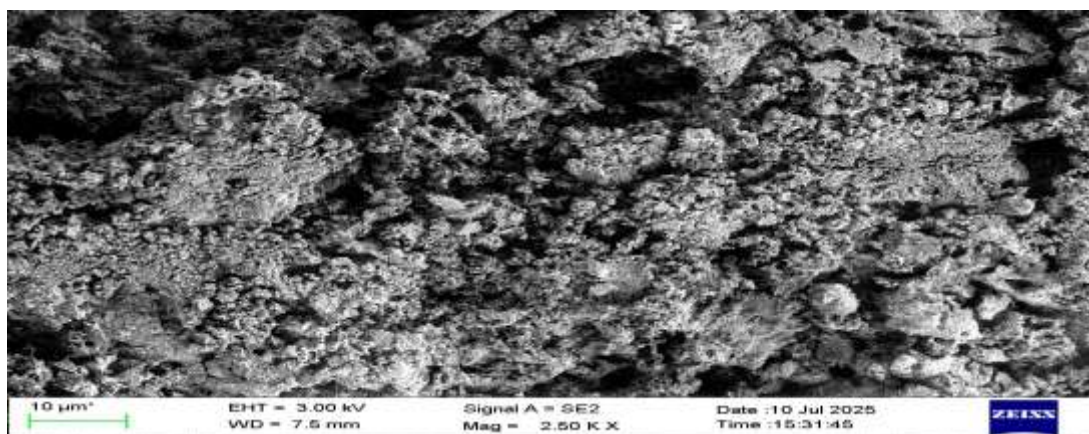


Fig.3: SEM image of the synthesized material at 2.50 KX magnification

At a magnification of 2.50 KX, the SEM micrograph reveals a distinctly irregular and porous surface texture. The material exhibits a highly agglomerated particle arrangement, with individual grains poorly distinguished due to the tendency of particles to cluster into compact aggregates during synthesis. The porous framework is clearly visible through the presence of voids and inter-particle channels, characteristics that enhance the material's potential for high-surface area applications such as gas sensing and catalytic reactions. The surface roughness further suggests uneven grain development, pointing toward heterogeneous growth dynamics and partial crystallization at this stage of formation.

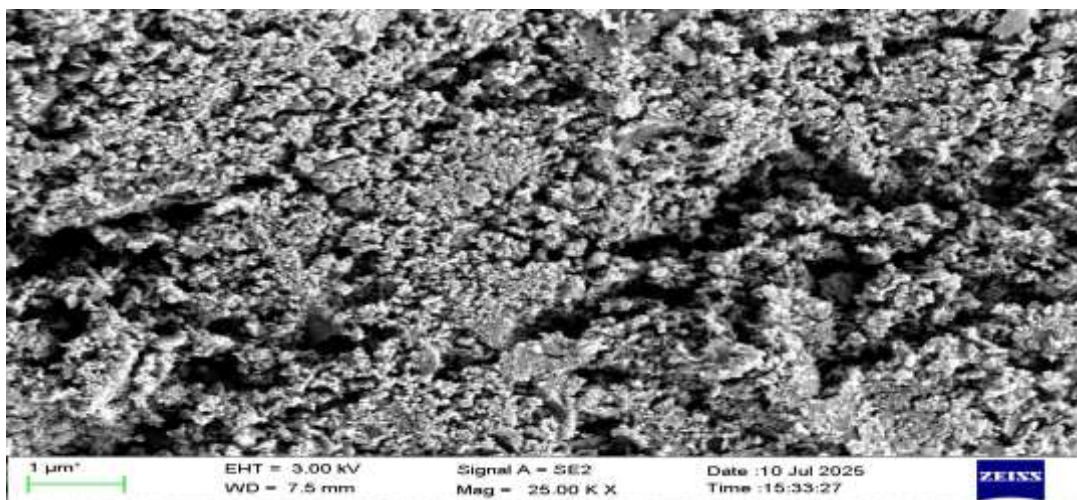


Fig.4: SEM image of the synthesized material at 25.00 KX magnification

At the highest magnification of 25.00 KX, the SEM micrograph reveals ultra-fine particle assemblies and nano-sized clusters characterized by pronounced surface irregularities. The grains appear tightly fused, forming an interconnected porous framework with clearly visible micro-pores. This level of structural refinement demonstrates that the synthesis process successfully generated a material rich in active sites. The presence of such a nano-porous architecture is particularly beneficial, as it enhances catalytic activity and sensing performance by improving gas adsorption and facilitating efficient charge transport pathways [16].

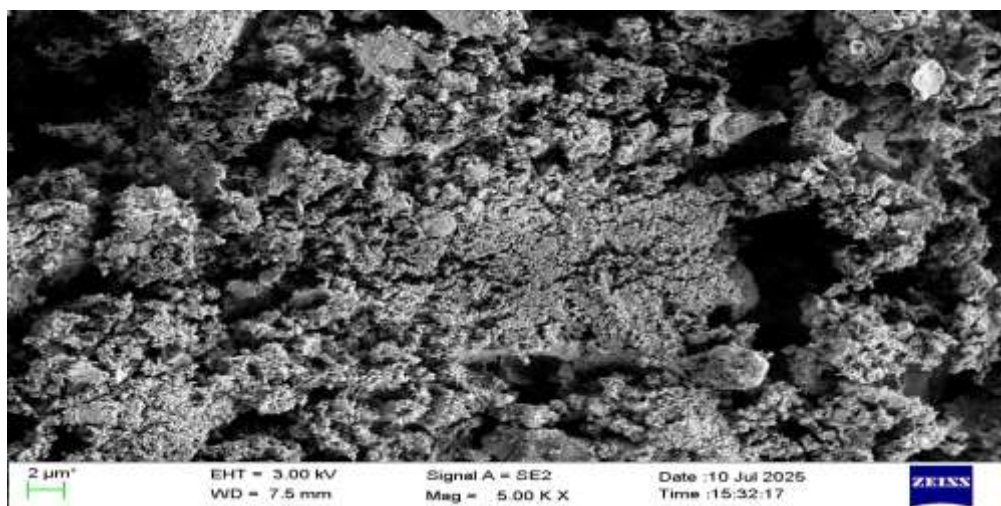


Fig.5: SEM image of the synthesized material at 5.00 KX magnification

At an increased magnification of 5.00 KX, the SEM micrograph highlights the nanostructured features of the material with greater clarity. Compared to the lower magnification image, the particles appear finer and more distinct, although agglomeration remains evident. The porous architecture is more pronounced at this scale, with nano-sized grains forming a rough, uneven surface and creating a well-connected network of voids. Such a morphology significantly enhances the surface-to-volume ratio, a property that is particularly advantageous for adsorption-driven applications and contributes to the improved gas sensitivity of the synthesized material.

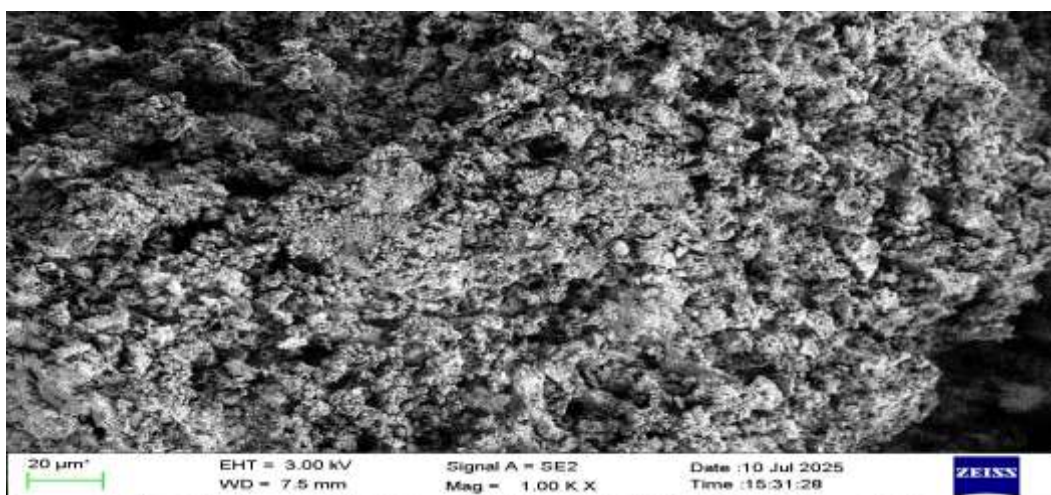


Fig.6: SEM image of the synthesized material at 1.00 KX magnification

At a magnification of 1.00 KX, the SEM image offers an overview of the material's bulk morphology. The structure is dominated by large, densely agglomerated clusters interspersed with extensive porous regions spread across the surface. This lower magnification emphasizes the overall microstructural uniformity, revealing a

sponge-like framework with notable surface roughness. Such a porous architecture is highly advantageous in functional oxide materials, as it promotes efficient gas diffusion and enhances the material's surface reactivity [17-18].

4. CONCLUSION

In this work, delafossite-type oxides were synthesized and evaluated for gas-sensing potential. The materials exhibited phase-pure crystalline structures, which are crucial for maintaining stability and reliable sensor performance. UV–Vis analysis confirmed suitable band gap values and clear absorption features, indicating strong light–matter interactions and effective charge transfer—both essential for detecting gases under visible light. SEM studies revealed a porous, well-connected microstructure with evenly distributed particles and defined grain boundaries, resulting in an increased active surface area. Such morphology supports efficient adsorption and desorption of gas molecules, enhancing sensitivity, selectivity, and rapid response characteristics. Collectively, the optical and structural findings establish these delafossite oxides as strong candidates for advanced gas-sensing applications. Their stability, adjustable band gap, and favorable surface features highlight their versatility, while further improvements through controlled synthesis and doping could significantly boost detection efficiency and expand their functional range.

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