"INNOVATIVE SYNTHESIS OF NANOCRYSTALLINE FERRITE BY COMBINING COMBUSTION AND SOL-GEL PROCESSES"

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ABSTRACT

A major step forward in nanomaterial manufacturing has been achieved with the novel synthesis of nanocrystalline ferrite by combining combustion and sol-gel methods. The integration process starts with the mixing of fuel components and metal precursors into a sol, which is then heated to start a combustion reaction. The ensuing sol-gel stages enhance this fast synthesis and provide precise control over particle size, shape, and crystallinity. Offering scalable and reproducible production that is appropriate for varied applications, the new methodology solves problems connected to current synthesis methods.The sol-gel combustion procedure was employed to produce nanosize (d) powders containing MgNiCoFe2O4 formulations. Calcinating the puffy, porous brown powder in its combusted form took four hours at a temperature of 400^0C. To verify the existence of ferrite bonds, infrared spectroscopy was employed. The size and shape of the nanocrystalline's particles were studied using infrared characterization and X-ray diffraction (XRD). On average, the powders' particle sizes were about 25 nm.

Keywords: Nanocrystalline, Morphology, Particle, Ferrite, Crystallinity

I. INTRODUCTION

The groundbreaking integration of combustion and sol-gel techniques in the production of nanocrystalline ferrite is a major achievement in the realm of nanomaterials. This innovative technology combines two separate processes to produce nanocrystalline ferrite, which opens up new avenues for improved material qualities and a wide range of potential uses. There has been a lot of interest in ferrites in many technical fields, such as electronics, medicine, and telecommunications, because of their unusual magnetic and electrical properties. Scalability, homogeneity, and control of particle size are common problems with traditional synthesis processes. These issues are not only resolved by combining combustion with sol-gel techniques, but a new paradigm for fabricating nanocrystalline ferrite with customized characteristics is also introduced. Metal oxide powders are produced by the fast and exothermic combustion process, which entails lighting metal nitrates or precursors. Despite its low cost and ease of use, this approach often produces agglomeration and does not allow for precise control over particle size. However, if you want complete control over the nanomaterial's composition, shape, and crystallinity, the sol-gel technique is a great option. Researchers hope to overcome the shortcomings of each strategy while capitalizing on the strengths of the combined method. Making a sol using fuel and metal precursors (usually metal nitrates) is the first step in combining combustion with sol-gel processes. The next step involves heating the sol to a point where a combustion reaction may begin. This highly reactive procedure speeds up the production of nanoparticles and metal oxides. The sol-gel process then kicks in, enabling the modification of particle shape and size. The sol-gel procedure allows for the controllable formation of crystallites and the construction of a well definednanocrystalline structure during the gelation and aging processes.

The flexibility to modify the nanocrystalline ferrite's chemical and physical characteristics is a huge benefit of this novel production method. An effective and quick way to start the synthesis is with the combustion stage, and then you may regulate the structural parameters with precision in the sol-gel process. To maximize the material's performance in different uses, this fine-tuning capability is essential. For example, in order to achieve certain magnetic characteristics like saturation magnetization and coercivity, it is crucial to manage the particle size and crystal structure in magnetic applications. Also, the novel synthesis approach provides a way out of the problems with standard approaches that are related to reproducibility and scalability. The combustion-sol-gel method has promise for use in industry due to its adaptability to large-scale manufacturing. There is a growing need for nanocrystalline ferrite in new technology, and this synthesis technique has the makings of a mass-producible solution thanks to its ease of use and promising results. The novel synthesis of nanocrystalline ferrite not only addresses the technical problems, but also paves the way for the exploration of new applications and the formation of multidisciplinary cooperation. A novel class of multifunctional nanomaterials with purpose-built characteristics may be created by fusing combustion with sol-gel techniques. Composites made from materials that include nanocrystalline ferrite might have improved mechanical, thermal, or electrical characteristics; this could lead to developments in areas like materials science, nanotechnology, and more.

II. REVIEW OF LITERATURE

Waheed, Ibrahim et al., (2019) The sol-gel auto combustion process is used to generate magnesium ferrite ($MgFe_2O_4$) nanoparticles, which are then calcinated at temperatures of 200, 450, and 900 degrees Celsius. The capping agent consisted of urea, while the sources of metal were magnesium nitrate ($Mg(NO_3)_2.6H_2O$) and iron nitrate ($Fe(NO_3)_3.9H_2O$). Both X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FT-IR) characteristics reveal distinct phases of the creation of the cubic magnesium-ferrite structure. The tetrahedral and octahedral sites of the magnesium-ferrite structure are highlighted in the infrared spectra of the metal-oxygen vibration at the frequencies of (703-636) and (574-433) cm^{-1}. Images obtained from a scanning electron microscope (SEM) revealed a microstructure that was entirely composed of crystalline crystals, exhibiting polyhedral forms and a relatively tiny number of globular microscopic particles. According to the Debye-Scherrer relation, the crystallite size of magnesium ferrite was a range of 29 nanometers. This was determined by calculation.

Bhagwat, Vilas et al., (2019) In order to effectively synthesis spinel-structured cobalt ferrite ($CoFe_2O_4$) nanoparticles, the sol-gel auto combustion method was utilized, and three distinct fuels (ethylene glycol, glycine, and urea) were utilized in the process. In the X-ray diffraction patterns, it was noticed that all of the samples exhibited pure phase development of cobalt ferrite with a cubic spinel structure. Through the use of XRD data, many structural characteristics, including the average crystallite size, the lattice parameter, and others, were determined. There was a decrease in the value of the average particle size, which was 15 nm, when urea was used as the fuel. There was no difference in the morphology of the sponge-like spherical form found regardless of the fuels. It was discovered that the grain size, on average, fell somewhere in the range of 65–86 nm. The superparamagnetic behavior of the samples was validated by the M-H plots, which revealed an increase in saturation magnetization, coercivity, and other magnetic parameters. Based on the findings of the experiments, it has been discovered that when urea is used as a fuel, it is possible to get a smaller particle size. On the other hand, when ethylene glycol is used as a fuel, the magnetic characteristics become enhanced.

Niu, Bo et al., (2017) Decreasing the size of the particles is anticipated to enhance the characteristics and expand the range of uses for high-entropy alloys. Thus, in this investigation, a new sol-gel autocombustion method was employed for the synthesis of high-entropy alloys. The nanocrystallineCoCrCuNiAl high-entropy alloys had an average grain size of 14 nm, characterized by a uniform dispersion and outstanding magnetic behavior resembling superparamagnetism. We demonstrate that the metal nitrates first generate high-entropy oxides (Co,Cu,Mg,Ni,Zn)O, which are subsequently reduced to CoCrCuNiAl high-entropy alloys by the reducing gases. The primary reaction pathways responsible for this transformation include the chelation between citric acid and the metal ions, as well as the in situ chemical processes. We show that the sol-gel autocombustion process is an effective method for producing solid solution alloys, bypassing the limitation of a large mixing entropy.

Šutka, Andriset al., (2013) The study primarily focuses on the production of monophasic spinel cadmium ferrite (CdFe2O4) using the sol-gel combustion method. An investigation was conducted to examine the impact of several reducing agents, such as citric acid, glycine, and urea, on the degree of phase purity in CdFe2O4. The X-ray diffraction technique was employed to determine the phase composition, while the scanning electron microscopy (SEM) was used to assess the particle size. Analysis has verified that the synthesis of CdFe2O4 powder at a temperature of 900°C, using urea as a reducing agent, resulted in the development of a monophasic powder with an average particle size of 0.3m. The practical application of synthesized CdFe2O4 particles as gas sensors was showcased.

Zhang, Linfeng& Wu, Yuanxin (2013)As catalysts for the oxidative degradation of methyl orange (MO), the MnFe2O4 spinel ferrite nanoparticles were produced from metal nitrates by the sol-gel technique. These nanoparticles have sensitive magnetic response qualities and a high specific surface area. X-ray powder diffraction (XRD), scanning electron microscopy (SEM), BET surface area analysis, H2-Temperature programmed reduction (H2-TPR), X-ray photoelectron spectra (XPS), and vibration sample magnetometer (VSM) were the techniques that were utilized in order to study the nanoparticles and determine their specific characteristics. With regard to the catalytic activity, the findings of the experiments demonstrated that the spinel ferrite nanoparticles made of MnFe2O 4 possessed an exceptionally high MO degradation activity. As a result of its sensitive magnetic response qualities and strong catalytic activity, it is anticipated that this particular type of MnFe2O4 spinel ferrite nanoparticles will have the potential to be utilized in the field of water treatment.

Parthasarathi, Rathinamet al., (2012) A unique combustion approach that makes use of hexamine as a fuel and metal nitrates as oxidizers has been utilized in order to successfully produce nanocrystalline nickel ferrite. At a temperature of 750 degrees Celsius, the produced compounds were sintered. In order to characterize the ferrite powders that were produced, XRD, FTIR, EDAX, and SEM were utilized. It has been established by XRD analysis that a single-phase spinel nickel ferrite compound has been formed. FTIR spectra illustrate the stretching and bending vibrations that occur in nickel ferrite bonds, which are composed of Ni-O and Fe-O. The EDAX analysis was used to determine the elemental makeup of the chemical that was brought into existence. Through the use of SEM, the morphological characteristics of the ferrite powders were investigated.

Bilecka, Idaliaet al., (2011) When the microwave technique is used with the surfactant-free nonaqueous sol–gel method it is feasible to synthesis nanoparticles of Fe_3O_4, $CoFe_2O_4$, $MnFe_2O_4$ and $NiFe_2O_4$ that are around 5–6 nanometers in size and have a high crystallinity and good morphological uniformity. Under microwave irradiation for a period of twelve minutes, the synthesis is carried out by reacting metal acetates or acetylacetonates as precursors with benzyl alcohol at a temperature of 170 degrees Celsius. When glass substrates are submerged in the reaction solution, the outcome is the deposition of homogenous metal ferrite films. The thickness of these films may be altered by adjusting the concentration of the precursor. When prefabricated nickel nanoparticles are employed as a sort of curved substrate, the seeds are coated with ferrite nanoparticles, which then form core–shell structures. These findings broaden the scope of the microwave-assisted nonaqueous sol–gel method above and beyond the straightforward synthesis of nanoparticles to include the creation of thin films on substrates that are either flat or curved.

Khorrami, Saeedet al., (2011) An innovative mix of the combustion and chemical gelation processes, sol–gel auto-combustion is a novel approach to combustion. The sol–gel combustion technique was utilized in this study to perform the synthesis of nanosize (d) powders that included compositions of $ZnFe_2O_4$. The puffy, porous brown particles that had been combusted were calcined for four hours at temperatures ranging from 750 to 1000 degrees Celsius. The X-ray diffraction and scanning electron microscopy techniques are utilized in order to characterize these powders. Both the XRD and the SEM were utilized in order to ascertain the shape and particle size of the nanocrystalline material. It was calculated that the average particle size for calcined powders was around 17 nanometers. The surface area of zinc ferrite that is produced using this process is 36 square meters per gram.

III. MATERIAL AND METHODS

Citric acid and analytical-grade magnesium, nickel, cobalt, and iron nitrates are used to make MgNiCo ferrite. The stoichiometry is used to determine the weight of the materials. A magnetic stirrer was used to homogenize the nitrates and citrates after dissolving them in deionized water at 75^0C.

Liquid ammonia was added to the solution in order to change its pH. Throughout the procedure, a magnetic stirrer was utilized to maintain the fluid's motion. To create a xerogel, the mixture was heated to 1000C while swirling continuously. When ignited, the dried gel burnt in a manner similar to self-propagating combustion, eventually turning into a fine powder.

The powder was thoroughly ground in a crucible before being sintered in a furnace set at 400^0C for four hours. The furnace was heated at a steady rate of 50 degrees Celsius per minute until it reached 400 degrees Celsius. After four hours of this, the temperature was left unchanged, and then it was cooled gradually to room temperature.

Using the KBr pellet approach, the infrared spectra of the sintering of the as-burnt powder at 600^0C were obtained from 400 to 4000 cm-1.

IV. RESULTS AND DISCUSSION

X-Ray Diffraction

The phase formation behavior of MgxNi0.6-xCo0.2Fe2O4 (where x = 0.2, 0.4, 0.6) was investigated using XRD. An analytical metal oxide phase was missing from the burned powder. For each peak, the crystallite size was calculated using the Scherrer formula:

$$t = \frac{0.9\lambda}{\beta \cos\theta}$$

β=full width at half maximum,

θ=bragg angle for the actual peak.

We used this formula to determine the lattice parameter:

$$\sin^2\theta = \frac{\lambda^2}{4a^2}(h^2 + k^2 + l^2)$$

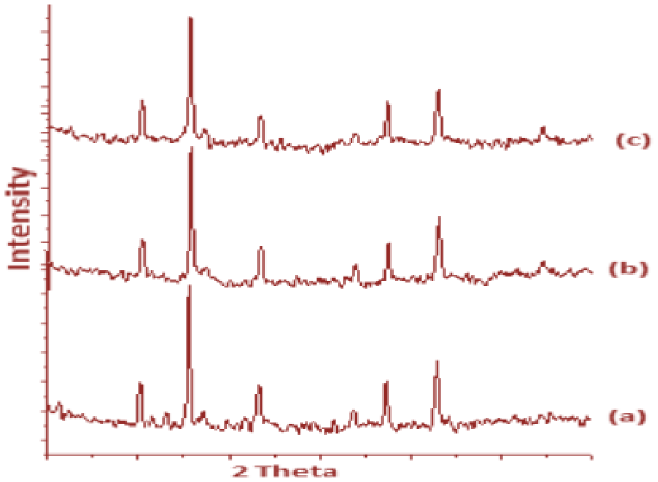

Figure 1: X-Ray Diffraction of MgNiCo ferrite (a) Mg0.2Ni0.6Co0.2Fe2O4 (b) Mg0.4Ni0.4Co0.2Fe2O4 (c) Mg0.6Ni0.2Co0.2Fe2O4

<u>Infrared Characterization</u>

When looking at the infrared curves of the powder as it was burnt, there is just one noticeable spectroscopic band at around 560 cm-1. A band like this is characteristic of AlNiZn ferrite. After burning, the infrared spectra curve lost their characteristic bands, proving that the carboxyl group and the (NO)3 ion are engaged in the combustion mechanism. So, it's reasonable to assume that the combustion process is an anionic redox reaction of the gel, where nitrate reduces citrate, an acidic acid. Nitrate ions provide an in situ oxidizing environment that accelerates the organic component's breakdown.

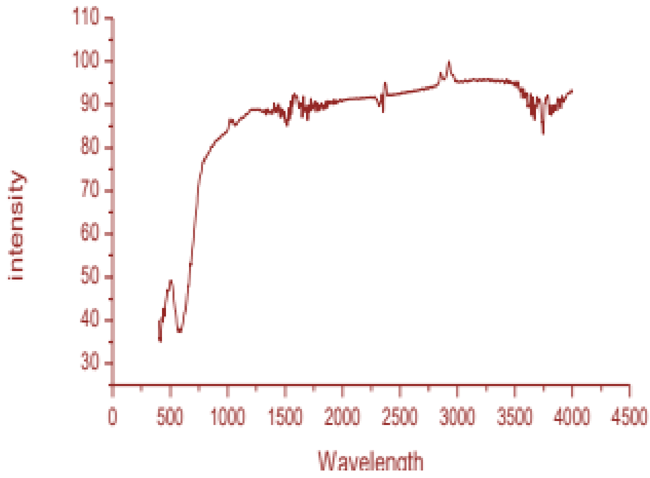

Figure 2: IR graph of MgNiCoFe2O4

V. CONCLUSION

Combustion and sol-gel technologies work together in a novel way that improves synthesis efficiency and solves problems with scalability and repeatability. With its large-scale manufacturing capability and relative ease of use, the approach might be a good fit for the increasing need for nanocrystalline ferrite in new technology and industry. The creation of new materials with customized characteristics is anticipated to be greatly aided by the further optimization and refinement of the synthesis process. This new way of synthesis has the potential to change both academic research and business processes, making its mark well beyond the lab. This approach is poised to revolutionize the world of nanomaterials due to its scalability and the ability to tailor nanocrystalline ferrite for specific uses. New opportunities and applications might be unlocked by ongoing research and development in this field, leading to technological and scientific improvements. Essentially, the groundbreaking synthesis of nanocrystalline ferrite exemplifies the efficacy of integrating several approaches to produce materials that may fulfill present-day technical demands while simultaneously igniting a fire of curiosity and creativity among the nanomaterials community.

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