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Research Article

Synthesis and Characterization of Metal Oxide Nanoparticles via Chemical Co-Precipitation

Dhonde Prerana Dnyandev[1*], Tanaji N. Bansode[2]

[1,2]Dept. of Chemistry, BNN College Bhiwandi, Dist-Thane, India

**Corresponding Author: preranadhonde1717@gmail.com*

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Abstract—The purpose of this research is to investigate the possible uses of synthesized and characterized metal oxide nanoparticles in a range of industries, including electronics, catalysis, and environmental remediation. The remarkable qualities of metal oxide nanoparticles, such as their large surface area, catalytic activity, and adjustable electrical and optical properties, make them very intriguing. Because of these qualities, they are perfect for many technical and industrial uses. This research used a chemical co-precipitation method to create oxide nanoparticles of zinc, copper, iron, and cobalt. Synthesized nanoparticles show potential for application in environmental cleanup, sensor development, and other high-tech fields due to their customized features.

Keywords— Characterization, Nanoparticles, Synthesis, Application, Metals

1. Introduction

The distinctive physicochemical characteristics of metal oxide nanoparticles, which vary substantially from those of their bulk equivalents, have recently attracted a great deal of interest. Catalysis, environmental remediation, sensors, and energy storage are just a few of the many promising fields that could benefit greatly from these nanoscale materials due to their extraordinary surface area, improved catalytic activity, and adjustable electrical and optical properties. The remarkable versatility and ease of manufacture of zinc oxide (ZnO), titanium dioxide (TiO_2), and iron oxide ($Fe_2O_ก$) make them stand out among the other metal oxides.

It is essential to precisely manage the particle size, shape, and crystalline structure during the manufacture of metal oxide nanoparticles in order to tune their properties for specific applications. To get this degree of control, several technologies have been developed, including as sol-gel, hydrothermal, and co-precipitation procedures. Because different synthesis methods produce nanoparticles with different levels of homogeneity, crystallinity, and scalability, selecting the right one is critical for the nanoparticles' usefulness.

Understanding the chemical, structural, and morphological characteristics of these nanoparticles requires their characterization, which is of equal importance. It is usual practice to evaluate the chemical composition, surface morphology, size, shape, and crystallinity of synthesized nanoparticles using imaging microscopy, scanning electron microscopy, transmission electron microscopy, and Fourier-transform infrared spectroscopy. These characterizations are critical for optimizing the performance of the nanoparticles in diverse technological applications and for understanding their behavior in varied settings.

Selected metal oxide nanoparticles, including ZnO, TiO_k, and Fe_kO_t, were synthesized and characterized in this study utilizing optimal production methods. Through a comprehensive analysis of their chemical and structural characteristics, the research endeavors to bring attention to the possible uses of these nanoparticles in domains such advanced materials science, environmental preservation, and catalysis. This study's results lay the groundwork for more investigation into the potential of metal oxide nanoparticles as a tool in future scientific and industrial breakthroughs, adding to the expanding area of nanotechnology.

1.1 Chemical Co-Precipitation of Metal Oxide Nanoparticles

The simplicity, low cost, and capacity to create homogeneous and highly crystalline nanoparticles make chemical co-precipitation a popular approach for manufacturing metal oxide nanoparticles. This technique controls the growth and prevents nanoparticle aggregation by precipitating several metal ions from a solution at once by changing the pH with a precipitating agent, usually with the help of a capping or stabilizing agent.

The metal ions needed for the co-precipitation process are provided by dissolved metal salts in water, such as chlorides, nitrates, or sulfates. Sodium hydroxide (NaOH) or ammonia (NH_3), two strong bases, are often used as precipitating agents to create metal hydroxides. When subjected to heat or allowed to age, these metal hydroxides break down into nanoparticles of metal oxide.

Producing nanoparticles with excellent purity and uniform size distribution are two of the many benefits of the co-precipitation process. Another is the ability to adjust the composition of mixed metal oxides by changing the ratio of metal precursors. The fact that this approach can be used to synthesize nanoparticles on a massive scale makes it ideal for use in industrial settings. Some difficulties with the approach include making sure the end result is homogeneous and managing the size and shape of the nanoparticles. The reaction circumstances, such as temperature, pH, reactant concentration, and stabilizing agent type and amount, might affect these parameters.

When optimized, chemical co-precipitation can yield nanoparticles with suitable properties for many uses, such as sensors, environmental remediation, and catalysis, making it a very practical and flexible approach for manufacturing metal oxide nanoparticles.

1.2 Review of Related Studies

Rami, Jitesh et al., (2024). This research used a chemical co-precipitation method to create oxide nanoparticles of zinc, copper, iron, and cobalt. This allows for the mass manufacture of nanoparticles made of metal oxides. By employing X-ray diffraction (XRD), researchers were able to examine the produced metal oxide nanoparticles' structural characteristics. The XRD analysis shows that the nanoparticles of metal oxide are crystalline. The morphology of the four metal oxide nanoparticles that were chosen for investigation was further investigated using scanning electron microscopy (SEM). Nanoparticles of CuO, CO2O3, Fe2O3, and ZnO, as synthesized and interspersed, were observed to have a spherical shape in the scanning electron micrograph.

Hui, Beh & Salimi, M. Nabil. (2020). The nanoparticle of iron oxide, maghemite (γ-Fe2O3), has attracted a lot of attention and is widely utilized in the field of biomedicine. Research into optimizing the co-precipitation process for producing γ-Fe2O3 nanoparticles was conducted. To generate dried dark brown precipitated γ-Fe2O3 powder, the following steps were used: dissolving iron (II) chloride and iron (III) chloride in distilled water, centrifugation, drying, and grinding. Using Response Surface Methodology (RSM) and Central Composite Design (CCD), the impact of varying processing temperatures (30 to 70°C), pH values (10 to 12), and stirring rates (300 to 700 rpm) on the size of the γ-Fe2O3 crystallites was examined. Results from an analysis of variance (ANOVA) showed that the parameter stirring rate had the greatest impact on crystallite size, with a determination coefficient (R^2) of 0.9890. Using the Design of Expert program (DOE), the optimal processing conditions that resulted in the shortest crystallite size of 7.3657 nm were 50 °C, pH 11.40, and 550 rpm. Various analytical methods, including Fourier Transform Infrared (FTIR), X-Ray Diffractometer (XRD), and Scanning Electron Microscope (SEM), were used to assess the γ-Fe2O3 powder samples' characterization. The FTIR spectra revealed the presence of carbon dioxide (CO2), hydroxyl (OH-) groups, and iron oxide (Fe-O) groups. The samples were found to contain γ-Fe2O3 because of the distinctive peak that appeared at $2\theta = 35.4°$. In SEM analysis, the γ-Fe2O3 particles seemed to have a typically spherical form.

Besenhard, Maximilian et al., (2020). Because of the ease of the experimental procedures, the low cost of the precursors, and the relative lack of environmental impact, co-precipitation has become the method of choice for synthesizing magnetic iron oxide nanoparticles (IONPs). Unfortunately, the processes involved in particle production are still poorly understood, making it difficult to optimize co-precipitation syntheses. Reasons for this include the lack of time to characterize early precipitates and the lightning-fast particle production (in seconds). One workaround for this shortcoming is a flow chemistry method that "freezes" transitory reaction states locally by employing steady-state operation. For the first time, this enabled a thorough examination of the initial phases of co-precipitation syntheses using in-situ synchrotron X-ray diffraction and small-angle X-ray scattering. Based on these research, it was found that the most magnetic phase of iron oxide occurs after 5 seconds of mixing the ferrous/ferric chloride precursor with the NaOH base solution. Particle size changes occur simultaneously, and co-precipitation and agglomeration also take place. Columnized IONPs needed to be de-agglomerated because their size prevented them from becoming colloidal upon further stabiliser addition. The correct amount of citric acid solution was added, and within minutes, IONP solutions with a neutral pH were obtained, demonstrating colloidal stability. With the help of new knowledge about particle production and an innovative stabilization method that doesn't include ultrasonication or washing, a multistage flow reactor was designed to constantly synthesize and stabilize IONPs with a residence time of less than 5 minutes. The reactor was able to consistently produce stable IONP solutions with a particle concentration of approximately 1.5 mg/ml with minimal materials expense, thanks to its resistance to fouling and its ability to mix quickly (<50 ms) and accurately (> 500 ml/h) regulate temperature.

Fadli, Ahmad et al., (2019). Nanoparticles of magnetite (Fe 3 O 4) are a recent development that has caught the interest of researchers in the field of biomedicine. Due to its low toxicity and excellent biocompatibility, magnetite has potential as a medication carrier in cancer treatment. The objective of this research was to identify the magnetite particle properties that were affected by the co-precipitation method's temperature and stirring rate. To begin, in a beaker of glass, a mixture of 10% NH 4 OH and 2:1 mole ratios of FeCl 3 and FeCl 2 were heated to 40–80°C and stirred at 300–500 rpm. Filter paper was then used to separate the precipitate, which was then dried in an air oven set at 100°C. Utilizing XRD and SEM, the characteristics of the produced magnetite powder were

ascertained. The XRD pattern shows that magnetite, with a crystallite diameter between 10 and 12 nm, formed at all temperatures tested. The clumping of magnetite particles ranging in size from 2.7 to 3.3 μm is revealed by the scanning electron microscopy data. However, a more uniform agglomeration of particles will result from increasing the temperature and stirring rate of agitation. The crystallinity level of magnetite can be enhanced by raising both the temperature and the stirring rate.

Kandpal, N.D. et al., (2014). A cost-effective co-precipitation process was used to manufacture iron oxide crystallites of nano-sized. It was determined from the X-ray diffraction pattern that the magnetic nanoparticles were spinel-structured pure Fe3O4. In order to characterize the nanoparticles, we used X-ray diffraction (XRD), Fourier-Transform infrared spectra (FT-IR), a transmission electron microscope (TEM), and vibrating sample magnetometry (VSM) to quantify their magnetization. According to XRD, the fresh nanoparticles have a cell thickness between 5.65 and 8.16 nm, while the TEM measured 20 to 22 nm for the particles. Particles with a diameter of 20-50 nm were formed by aggregating a small number of ferrite grains, according to the results. A thermogravimetric study of the nanoparticle was also performed within the temperature range of 70–700°C. The procedure utilized to create the particles in the study had a higher magnetism value than the previously made ferrite, and it may be used as a template for creating other nanoparticles with Fe (II) added to them.

Riaz, Saira et al., (2014). There has been a lot of interest in the potential of nanostructured functional materials, especially nanoparticles (NPs), to diagnose and treat diseases like cancer by releasing therapeutic medications in a controlled, precise, and efficient manner. It is possible to take advantage of the therapeutic potential of magnetic NPs by combining the inherent characteristics of their magnetic cores with the medicine they contain. For bionanotechnology to work, superparamagnetic materials must meet two magnetic criteria: a large saturation magnetic moment and a near-zero remanence. We present here the results of a modified coprecipitation-based synthesis of magnetite NPs. This research also reports on the effect of pH on the size and shape of NPs. Because of their superparamagnetic properties, the size of the NPs is crucial, and they must be compatible with living cells. To adjust the size and shape of the nanoparticles, the amount of NaOH was changed, and ferric and ferrous chlorides were utilized as precursors. Magnetite production is revealed by X-ray diffraction patterns. Using scanning electron microscopy, we can see that the manufactured nanoparticles are the right size to target the sick cell. These NPs are ideal for use as contrast agents in magnetic resonance imaging (MRI), cell labeling, magnetic separation, and hyperthermia therapies, as shown by their M-H curves, which also demonstrate their superparamagnetic nature.

2. Proposed Methodology

2.1 Chemicals

The chemicals utilized in the experiment are of analytic reagent grade and have not been previously purified. To separate nanoparticles, scientists utilize newly made chemical solutions in water. The EDTA acid and metal oxide solution were procured from the Chemistry Department of HNGU in Patan, Gujarat, India. The Physics Department of Sheth M.N. Science College in Patan, Gujarat, India, supplied the sodium hydroxide (NaOH) pellets. To make the solutions, we utilized deionized water.

2.2 Synthesis

To make copper, cobalt, iron, and zinc oxide nanoparticles, we drop 60 ml of a 0.4 M metal oxide solution into 60 ml of a 0.0125 M ethylene-diamine-tetra-acetic acid solution and mix them violently using a magnetic stirrer. In order to prevent the particles from aggregating and perhaps shuffling to nano size, EDTA was used as a stabilizer. To remove any original reactant and any contaminants, such as EDTA waste, the metal-carbonate precipitate that forms in this reaction is rinsed with alcohol after being extracted from the reaction mixture and rinsed multiple times with filtered water. A very fine powder of metal-carbonate precursor is obtained by dehydrating the wet precipitate and then carefully grinding it with an agate-mortar. The metal-carbonate precursor becomes nanoparticles of metal oxide when heated to high temperatures.

2.3 Characterization

Utilizing a Bruker, D2 Phaser X-ray Diffractometer (equipped with CuKα radiation) within the 10° to 90° angle range, the structural analysis of oxide nanoparticles derived from copper, cobalt, iron, and zinc samples was conducted. We used a scanning electron microscope (SEM, Jeol, JSM6010LA) to examine the materials' morphology. By using Scherrer's relation to the line broadening, we were able to determine the average crystallite size (t):

$$t = 0.9\lambda/\beta cos\theta \quad (1)$$

D is the average crystallographic size, β is the X-ray wavelength, and FWHM is the full width at half maximum.

3. Result of Experimental

3.1 XRD Analysis

Figure 1(a, b, c, d) shows the results of applying Debye-Scherer's equation to the XRD configurations in order to measure the average size of the crystallites. A clear and robust peak at approximately 35° and 38° with (1 1 1) and (0 2 2), respectively, for CuO nanoparticles suggests that the sample is of high crystalline quality and has a monoclinic structure with lattice parameters; this finding is in agreement with JCPDS card number 45-0937. The presence of bars at the top of the peak positions and certain (h, k, l) values denotes that the corresponding peak is in fact traveling in the opposite direction (h, k, l). That there are no crystallinity flaws and all the peaks are very crisp suggests that the sample is very pure.

You can see the XRD pattern of CO2O3 nanoparticles in Figure 1(b). It confirms the procedure is essential to preserve the crystalline phase and prevent their agglomeration since it does not show any peaks for the originally generated sample. The presence of cubic phase (space group: Fd3m) in the

Co2O3 nanoparticles is demonstrated by peak intensities of (1 1 0), (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1), and (4 4 0). Due to contamination, the primed sample has additional, insignificant peaks. The obtained XRD peaks are in agreement with the Co2O3 standard characteristic peaks and are identical to those on JCPDS card no. 76-1802.

The XRD pattern of the Fe2O3 nanoparticles that were synthesized is shown in Figure 1(c). Approximately 35.5° was the location of the strongest peak. The JCPDS-86-0550 data indicated that the diffraction peaks occurred at various angles, which might be linked to the (0 1 2), (1 0 4), (1 1 0), (1 1 3), (0 2 4), (1 1 6), (0 1 8), (2 1 4) and (3 0 0) planes of α-Fe2O3. The absence of any further impurities or iron oxide phases in the final particles proved that they were pure α-Fe2O3. The diffraction pattern's wide width indicates that the particles are rather tiny.

Figure 1(d) shows the XRD peaks of ZnO nanoparticles in a sample. The XRD patterns, which have a lattice parameter of and indicate that the nanoparticles have a pristine hexagonal structure (space group: P63mc). The values associated with the peaks that emerged (1 0 0), (0 0 2), (1 0 1), (1 0 2), (1 1 0), (1 0 3), (1 1 2) are those of pure ZnO. This proves that the ZnO particles are crystalline since they match the typical JCPDS card no.01-79-0206.

Using Debye-Scherer's equation, we were able to determine that the average crystalline size of the four metal oxide nanoparticles was 30-50 nm.

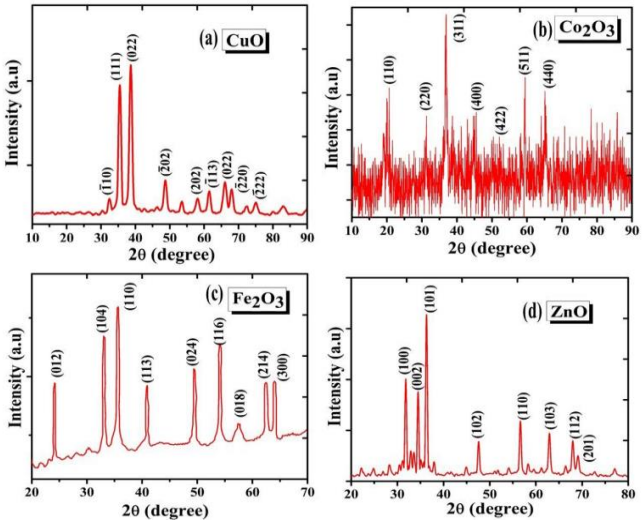

Figure.1 XRD of (a) Copper Oxide, (b) Cobalt Oxide, (c) Iron Oxide and (d) Zinc Oxide Nanoparticles

3.2 SEM Analysis

The shape of the metal oxide nanoparticles that were produced may be seen in Figure 2 (a, b, c, d). The scanning electron micrograph (SEM) depicts the metal oxide nanoparticles as having a spherical shape, with aggregated particles showing strong inter-nanoparticle connections. Scanning electron microscopy (SEM) reveals the size, homogeneity, and distribution of nanoparticles. The homogeneous dispersion of the particles is demonstrated by the SEM image. Because of the high surface area of the material, this shape is ideal for producing the electrode in cobalt oxide nanoparticles for use in supercapacitors. The sizes of the four metal oxide nanoparticles we studied ranged from 35 nm to 200 nm. The particle size that has been measured is larger than what has been reported in the literature. One possible explanation for the increased nanoparticle sizes seen in this study is the particle grouping.

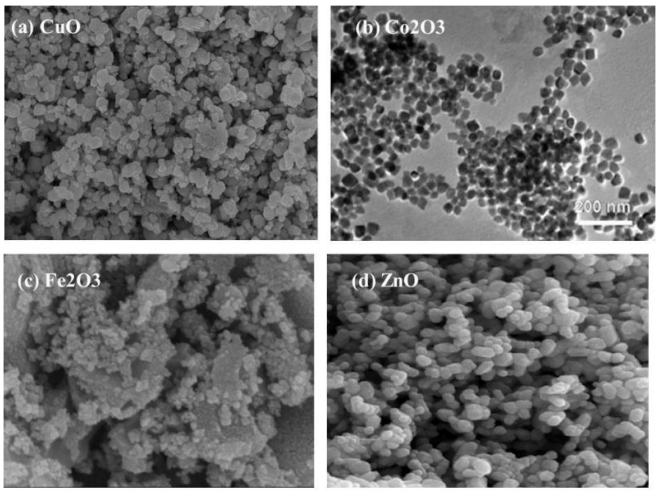

Figure 2 SEM micrograph of (a) Copper Oxide, (b) Cobalt Oxide, (c) Iron Oxide and (d) Zinc Oxide Nanoparticles

4. Conclusion

The chemical co-precipitation approach was used to successfully produce oxide nanoparticles of copper (CuO), cobalt (Co_2O_t), iron (Fe_2O_t), and zinc (ZnO) in this study. This method is effective for large-scale production. The crystalline nature of the produced nanoparticles was established through careful X-ray diffraction (XRD) analysis of their structural properties. The XRD patterns showed distinct peaks for each metal oxide, which means that the nanoparticles of pure oxide were successfully formed.

Scanning electron microscopy (SEM) was used for additional morphological analysis. A mostly spherical morphology was shown by the scanning electron microscopy pictures of the produced nanoparticles. Further evidence of a consistent and homogenous synthesis process was provided by the micrographs, which revealed that the nanoparticles were evenly spaced. The chemical co-precipitation approach is effective in creating metal oxide nanoparticles with acceptable features, as demonstrated by the observed morphology and structure of the nanoparticles.

The results show that chemical co-precipitation is a scalable and dependable way to make high-quality metal oxide nanoparticles, according to this study. This study's produced CuO, Co_2O_3, Fe_2O_n, and ZnO nanoparticles have attractive structural and morphological features that make them potential candidates for use in electronics, environmental remediation, catalysis, and other areas. The fabrication procedure for these nanoparticles could be fine-tuned in the future so that they have better, more application-specific characteristics.

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