Synthesis of ZnO and CuO nanoparticles via Sol gel method and its characterization by using various technique

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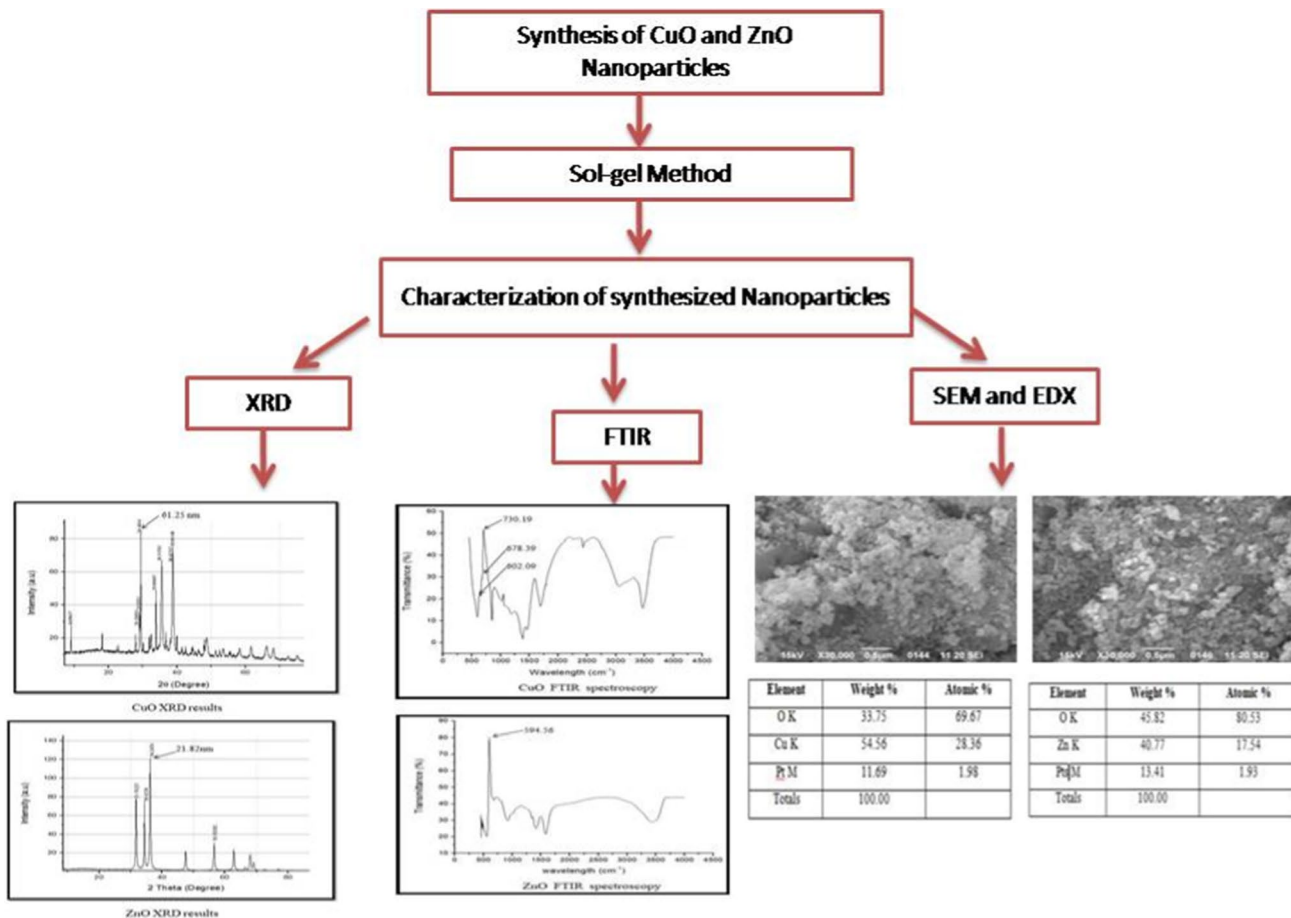
Abstract

Nanotechnology is a completely unique branch of technology that offers with substances in a very small size between (1–100 nm) with various crystal shapes. Metals have ability to produce large number of oxides. These metal oxides play a major role in many areas of chemistry, physics, material science and food science. In this research, Zinc Oxide (ZnO) and Copper (II) oxide nanoparticles were synthesized via sol–gel process using zinc nitrate and copper (II) nitrate as precursor respectively. The characterization of CuO and ZnO nanoparticles was done by using various techniques. X-ray Diffraction (XRD) indicates the crystallinity and crystal size of CuO and ZnO nanoparticle. Fourier transform infrared spectroscopy (FT-IR) was used to get the infrared spectrum of the sample indicating composition of the sample which contains various functional groups. XRD result shows the particle size of CuO at highest peak 29.4014^0 was 61.25 nm and the particle size of ZnO at highest peak $36.2476°$ was 21.82 nm. FT-IR spectra peak at 594.56 cm^{-1} indicated characteristic absorption bands of ZnO nanoparticles and the broad band peak at 3506.9 cm^{-1} can be attributed to the characteristic absorption of O–H group. The analysis of FT-IR spectrum of CuO shows peaks at 602.09, 678.39, and 730.19 cm^{-1} which refer to the formation of CuO. SEM images indicate the morphology of CuO and ZnO nanoparticles. Result of EDX characterization indicates that the both synthesized nanoparticles have good purity with very less amount of impurities. EDX data indicates that Cu content was 54.56%, oxygen content was 33.75% in CuO nanoparticles and Zn determined by EDX was 40.77 and O was 45.82 in ZnO.

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Graphical Abstract

CuO and ZnO Nanoparticles characterization flowchart

1 Introduction

Nanotechnology is a completely unique branch of technology that offers with substances in a very small size between (1–100 nm) with various crystal shapes which include spherical nanoparticles, flower shaped, Nano rods, Nano ribbons, Nano platelets. The matchless physical and chemical attributes are end result from its excessive surface-to-volume ratio comparing with micro or nano-sized [1]. The uniqueness of nanoparticles in their physical properties and chemical properties make them subject of great attention. The properties of higher damping in nano particles, their mechanical stability, better thermal conductivity and higher strength are special characters [2]. Metal oxide nanomaterials having excessive surface area have attracted substantial interest for scientific studies because of their applications in the field of optical electronics, sensing devices and nano electronics [3]. Nanomaterials attracted many scientists because of their considerable chemical stability, magnetic behaviour, and biocompatibility. The nanomaterials act antibacterial, antifungal, antioxidant, and anticancer activities [4].

There are several methods that have been put forward for synthesis of these materials, namely chemical vapour condensation, arc discharge, hydrogen plasma-metal reaction, and laser pyrolysis with in the vapour phase, The other methods include Sol–gel synthesis, micro-emulsion, microbial processes which occur in liquid state, ball milling which is conducted in solid state [5, 6]. The synthesis process of nanoparticles defines largely their properties. The range of metal oxide is great and their range of properties and possible applications appear to be enormous [7].

Sol–gel process has several advantages. Solitary sol gel installation can yield materials at ultra-temperatures, synthesize nearly any substance, co-synthesize two or more substances with oneother, exactly observance the microstructure of the top outputs, and punctually dominance the physical ‹mechanical, and chemical characterizes of the ultimate outputs. [8]

In many literatures, it's far determined that Nano particles of ZnO and CuO gives higher overall performance as compared to that of bulk size [9]. Zinc is a necessary element to our health and ZnO nano particles additionally have right biocompatibility with human cells [10]. Recently ZnO and CuO is listed as safe material by FDA (food and drug administration, (USA) [11, 12].

Both copper oxide (CuO) and zinc oxide (ZnO) have shown varying antibacterial activity in opposition to microorganism species.They have been utilized in food packaging, wound healing and surface coatings because of their antimicrobial activities. However the anti-microorganism activities of these nano-substances are but to be in comparison against gram positive and gram negative bacteria species [13].

There are several nanomaterials that are applied for numerous food packages on commercial level. Titanium dioxide having a particle size of much less than a hundred nm is widely applied as food added substance and antimicrobial agent for food packaging and potential holders. Silver nanoparticles are applied as antimicrobial agent in food packaging, storage utensils, slicing board and refrigerator and moreover as food supplement. Zinc and Copper oxide are applied as nutritive added materials and moreover as antimicrobial operators in food packaging. [14]

Zinc oxide nanoparticles, which emerge as greater antibacterial as their particle size receives smaller, and chitin, that's a natural substance located with inside the shells of crustaceans like crabs and shrimps. [15]

The aim of this study was to synthesize zinc oxide and copper (II) Oxide of low dimension and analysed the size of particles by using XRD analysis at highest peak and study of various functional group present in synthesized nanoparticles at different peak by using FTIR. After reviewing various protocols [1, 3, 7–10] for synthesis of nanoparticles. This method has novel features, which are of great interest due to its low cost, easy preparation and feasibility. Synthesis of ZnO and CuO nanoparticles by improve sol gel technique is reported by Zinc nitrate as precursor, H_2O_2 (hydrogen peroxide) and calcified at 500 °C for 1 h & Copper (II) Nitrate as precursor, sodium hydroxide and calcified at 400 °C for 2 h. Further details are discussed in methodology. In this research, to study the optical and physical properties of ZnO and CuO nanoparticles synthesized by sol–gel method were characterised by various technique like XRD, FT-IR, SEM, EDX [16–18].

This Research opens a new pathway for understanding how formation of these nanoparticles influences their properties. Further understanding of process also promises advances in various nanomaterial design and synthesis methods, which can be further used for diverse industrial applications.

2 Processing methods

There are two approaches through which we are able to obtain the synthesis of nano materials. Bottom Up method produces small building blocks which one assembled into large structures. The foremost controlling parameters right here are the crystallinity, morphology, particle size, and chemical composition. Examples: chemical synthesis, laser trapping, self-assembly, colloidal aggregation, etc. The second method is, by "Top Down" method where the large physical compound is transformed into smaller nanostructures. Deposition of film and growth, nano imprint/lithography, etching technology, mechanical polishing etc. are number of the examples. Increase in surface-volume ration causes alternation in various thermal, mechanical and other properties. [19]

3 Materials and methods

3.1 Materials

All chemicals were of analytically grade and used without further purification. Copper (II) Nitrate [$Cu(NO_3)_2$, 98%], Sodium Hydroxide Pellets (NaOH, 99.8%), Zinc Nitrate [$Zn(NO_3)_2$ 99.9%], Hydrogen Peroxide (H_2O_2, 98.9%), Ethanol (C_2H_5OH, 99.9%), double distilled were purchased from SRL Pvt. Ltd. Mumbai (India). All reagents and chemicals were pure and used with no further treatment.

3.2 Methods

3.2.1 Synthesis of CuO nanoparticles

3.2.1.1 Chemicals Copper (II) Nitrate, Sodium Hydroxide pellets, ethanol.

3.2.1.2 Equipments Beaker, Magnetic Stirrer, Centrifuge, Dehydrator, muffle furnace, motal & pistal.

3.2.1.3 Synthesis The synthesis of CuO nano-particles via way of means of sol–gel technique was as fellow: 4.5 g of copper (II) Nitrate become dissolved in a 100 ml ethanol; 4.5 g of NaOH (Sodium Hydroxide) become dissolved in 200 ml ethanol. The solution of sodium hydroxide was added drop wise in the solution copper nitrate with non-stop stirring at room temperatures for 2 h. This ended with pH of 5.6 for the solution, which remained stable in the course of a stirring period of 2 h, as monitored by a pH-meter reactions arise and color of sample turns dark blue to black. The resulted pH of final solution was noted that 5.9. The pH was adjusted by using ethanol and not more than 8.0. Let the solution settles down and discard the upper layer. Transfer the gel into Centrifuge tubes and centrifuged at 10,000 rpm for 10 min. Discard the supernatant liquid and wash the sample with distilled water. Repeat the above step 2–3 times. The sample allowed to dry at 80 °C until the samples becomes dry then annealed the sample at 400 °C for 2 h in muffle furnace, crushed the annealed sample of CuO nanoparticles with Motal and Pistal. Fig. 1 shows the Flow Diagram of synthesis of CuO nanoparticles by sol- gel method.
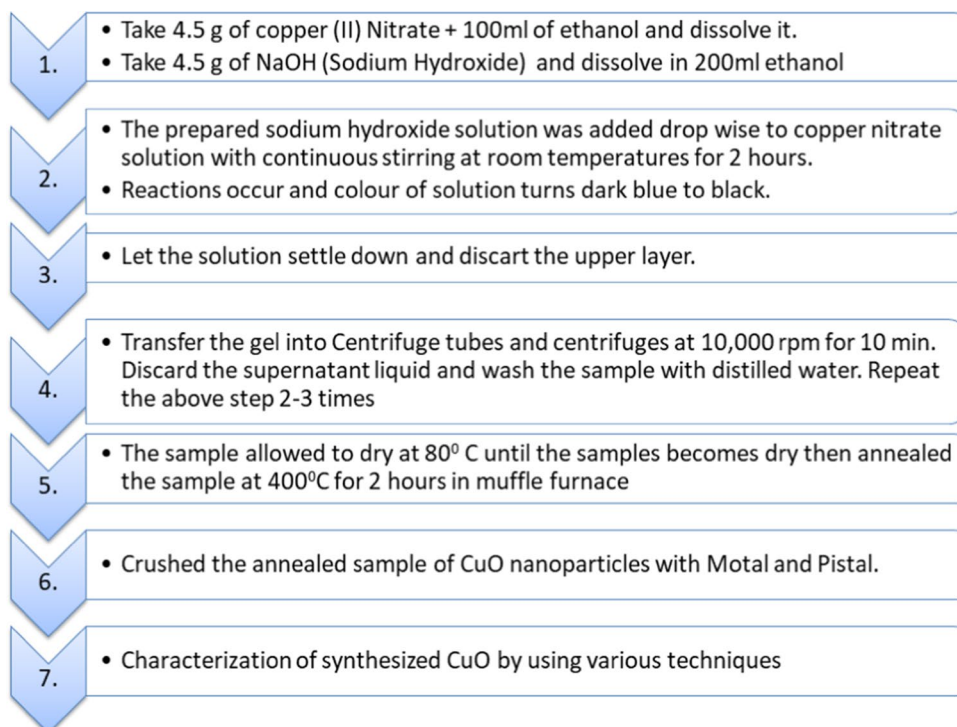
3.2.2 Synthesis Of ZnO Nanoparticles

3.2.2.1 Chemicals Zinc Nitrate, H_2O_2 (Hydrogen peroxide), Double distilled water, ethanol.

3.2.2.2 Equipments Beaker, Magnetic Stirrer, Centrifuge, Dehydrator, muffle furnace, motal & pistal.

3.2.2.3 Synthesis The synthesis of ZnO nanoparticles via way of means of sol–gel technique was as fellowed: in this process 15.7 gm of zinc Nitrate was added to 500 ml of double distilled water with non-stop magnetic stirrer till Zinc Nitrate

Fig. 1 Flow Diagram of synthesis of CuO nanoparticles by sol- gel method

1.
- Take 4.5 g of copper (II) Nitrate + 100ml of ethanol and dissolve it.
- Take 4.5 g of NaOH (Sodium Hydroxide) and dissolve in 200ml ethanol

2.
- The prepared sodium hydroxide solution was added drop wise to copper nitrate solution with continuous stirring at room temperatures for 2 hours.
- Reactions occur and colour of solution turns dark blue to black.

3.
- Let the solution settle down and discart the upper layer.

4.
- Transfer the gel into Centrifuge tubes and centrifuges at 10,000 rpm for 10 min. Discard the supernatant liquid and wash the sample with distilled water. Repeat the above step 2-3 times

5.
- The sample allowed to dry at 80⁰ C until the samples becomes dry then annealed the sample at 400⁰C for 2 hours in muffle furnace

6.
- Crushed the annealed sample of CuO nanoparticles with Motal and Pistal.

7.
- Characterization of synthesized CuO by using various techniques

dissolved completely in distilled water. Then the solution was heated to 50 ℃ and 700 ml of alcohol (ethanol) was added slowly with non-stop stirring, ethanol used as solvent and reductant for manufacturing of virtuous nano-particle with narrow particle size distributions and to adjust the pH of sol. The pH of solution was near about 5.4. After this, add 6 ml of H_2O_2 in drop wise manner to the above solution and mixed it by using a magnetic stirrer until to get a clear solution. After adding H_2O_2 the pH of sol from 5.4 to 9 (Alkaline Condition) of the solution. Let settle down and discard the upper layer. Transfer the gel into Centrifuge tubes and centrifuges at 10,000 rpm for 5 min. Discard the supernatant liquid and wash the sample with distilled water. Repeat the above step 2–3 times. The sample allowed to dry at 80 °C until the samples becomes dry then annealed the sample at 500 °C for 1 h in muffle furnace, crushed the annealed sample of CuO nanoparticles with Motal and Pistal. Figure 2 shows the Flow Diagram of synthesis of ZnO nanoparticles by sol- gel method.

3.3 Characterization of ZnO and CuO nanoparticles

The characterization of synthesized nanoparticles was done by following analytical techniques, such as Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD).

FT-IR (Fourier Transform Infrared) spectroscopy is a technique which analyses Infra-red spectrum of a sample by absorption. The sample may be solid, liquid or in gaseous state. Magnesium oxide nano-particles show different peaks at different levels in FT-IR measurement, which correspond to different functional groups, and stretches [20].

XRD (X-ray Diffraction) is used for identification of phase of a sample possessing crystallinity. It also provides information on unit cell dimensions. The homogeneity and bulk composition is ascertained, based on Bragg's law which relates the wavelength of radiation with the diffraction angle and lattice spacing in crystalline sample [20].

SEM (A scanning electron microscope) is used to study the morphology of particle and is a type of electron microscope that creates images of nano-particles by scanning the surface of particle with a focused beam of electrons. The electrons interact with atoms in particles, producing various signals that contain information about the morphology and composition of the particles. [9]

EDX (Energy Dispersive X-Ray Analyse) is used to provide elemental identification and quantitative compositional information of synthesize nano-particles.

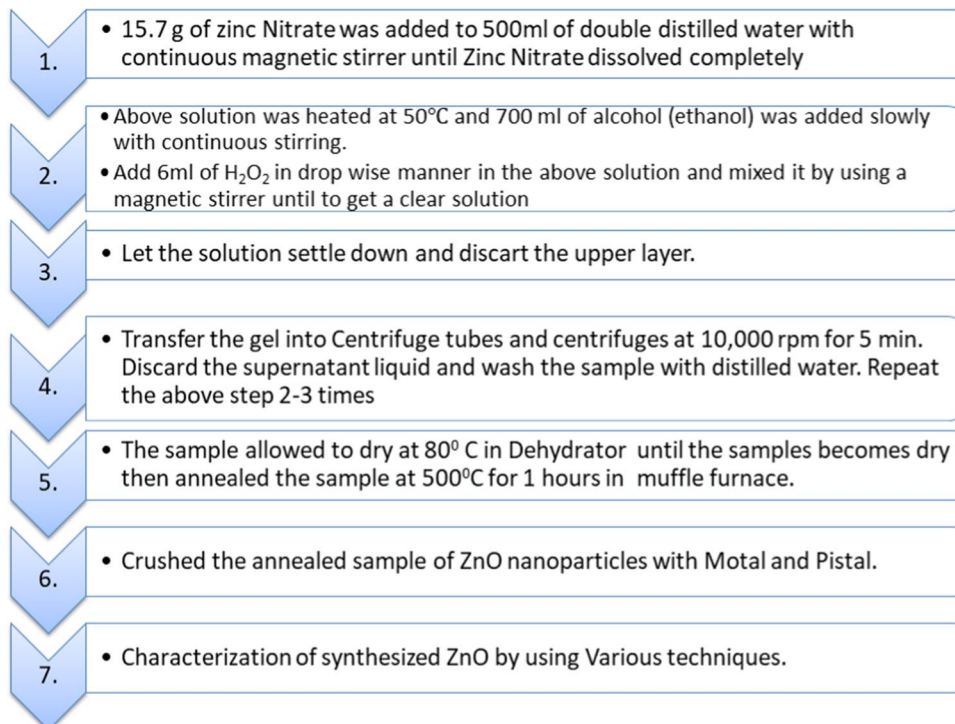**Fig. 2** Flow Diagram of synthesis of ZnO nanoparticles by sol- gel method

1. • 15.7 g of zinc Nitrate was added to 500ml of double distilled water with continuous magnetic stirrer until Zinc Nitrate dissolved completely

2. • Above solution was heated at 50°C and 700 ml of alcohol (ethanol) was added slowly with continuous stirring.
   • Add 6ml of H_2O_2 in drop wise manner in the above solution and mixed it by using a magnetic stirrer until to get a clear solution

3. • Let the solution settle down and discart the upper layer.

4. • Transfer the gel into Centrifuge tubes and centrifuges at 10,000 rpm for 5 min. Discard the supernatant liquid and wash the sample with distilled water. Repeat the above step 2-3 times

5. • The sample allowed to dry at 80° C in Dehydrator until the samples becomes dry then annealed the sample at 500°C for 1 hours in muffle furnace.

6. • Crushed the annealed sample of ZnO nanoparticles with Motal and Pistal.

7. • Characterization of synthesized ZnO by using Various techniques.

Fig. 3 XRD Pattern of synthe-size CuO Nanoparticles
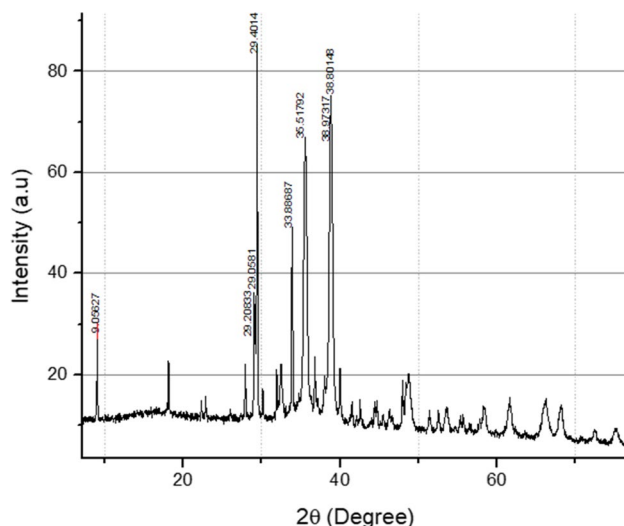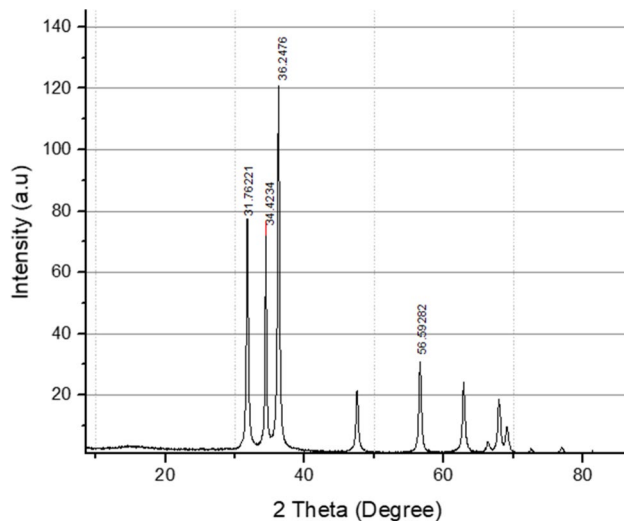

Fig. 4 XRD Pattern of synthe-size ZnO Nanoparticles.

4 Result and discussion

4.1 X-Ray diffraction analysis

The crystal structure and size of CuO and ZnO nano-particles were analysed by XRD. The sharp peak of Figs. 3 and 4 indicates that the nature of nano-particles was crystalline. X-Ray Diffraction Analysis or X-Ray Crystallography was used to identify the atomic or molecular structure of particles in which the crystalline atoms cause a beam of incident X-rays to diffract into many specific directions. The crystal structure of CuO and ZnO nano-particles prepared by sol–gel route has been showed in Figs. 3 and 4 at CuKα (λ = 1.54056 A) in the Bragg reflection 2θ range from 10° to 70° and 10° to 80° respectively. Sizes of the crystallite particles were calculated using Scherrer's equation.

$$\mathbf{D \; = \; K\lambda / \; \beta cos\theta}$$

where, D is the crystallite size of the particles, shape factor is 'K' (0.9 in the present work). 'λ' is the incident1 X-ray wave-length (1.54056 A, CuKα), θ is the diffraction angle and β is the full width half maximum.

From the peak width and intensity the small particle size and better crystallinity of CuO and ZnO can be easily seen in Figs. 3 and 4. The higher intensity peaks in XRD pattern of the prepared CuO samplewas observed at 2θ- 29.4014, 35.51792, 38.80148, 38.97317 (Fig. 3) and in crystallography, XRD refers to monoclinic shape of CuO crystal as claimed

Table 1 Calculated Diffraction angle at highest peak and crystallite size of CuO and ZnO

Sample	Diffraction angle at highest peak	Average size
CuO	29.4014°	61.25 nm
ZnO	36.2476°	21.82 nm

Fig. 5 FTIR Spectroscopy of synthesized CuO Nanoparticles
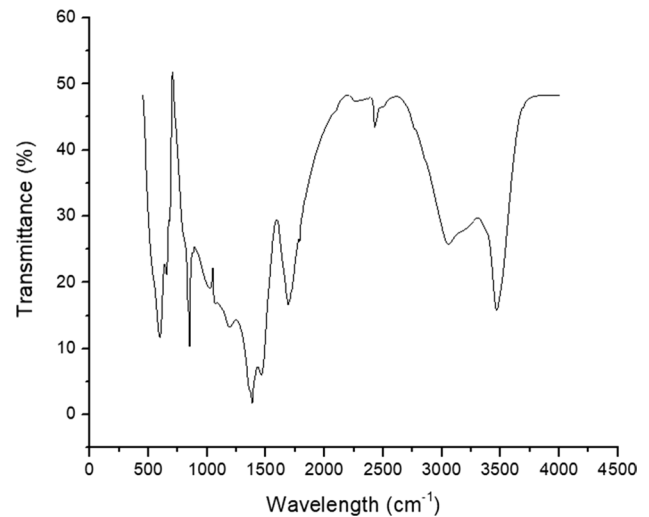
in JCPSD card no. 01-007-2551.) Average particle size of the nano-particle at highest peak (29.4014°) was determined by using Debye –Scherrer's equation and it was found to be 61.25 nm.

The higher intensity peaks in XRD pattern of the prepared ZnO sample was observed at 2θ- 31.7621, 34.4234, 36.2476 (Fig. 4). XRD pattern of ZnO shows sharp peaks, referring to the crystalline nature. The synthesised ZnO nanoparticles showed a single phase with clear diffraction peaks according to the information provided by reported data (JCPDS card no 36-1451). Average particle size of the ZnO nanoparticles was determined as 21.82 nm at highest peak (36.2476°) according to the Debye–Scherrer's equation. The crystallinity of ZnO refers to a wurtzite hexagonal structure which was result of penetration of two hexagonal lattices.

XRD analysis data also help to calculate the Elastic strain. The elastic strain results indicate that if the particle size was less than 20 nm than they have more strain and greater than 20 nm particles have less strain, meaning thereby that the smaller particles have higher strain and bigger particles have lesser strain. These values were in agreement with the literature values [21]. From the Table 1 : it was clear that both the CuO and ZnO have less strain. The crystalline size of the ZnO particle was 21.82 nm that was less than CuO particles i.e.61.25 nm because the concentration of chemical used in synthesis process varies and annealing temperature & time in muffle furnace also differ.

4.2 Fourier transform infrared spectroscopy (FT-IR)

FT-IR spectroscopy analysis has been done to observe the chemical and structural nature of the particles. The Infrared absorption band identifies the various functional group of the molecule. Both synthesized nano-particles CuO and ZnO has been scanned from 4000 to 400 cm^{-1}. The region from 4000 to 1500 cm^{-1} was functional group region and region from 1500 to 667 cm^{-1} was fingerprint region. The various phase and functional group in nanoparticles were studied by using FT-IR technique [22].

The analysis of FTIR spectrum of CuO shows peaks at 602.09, 678.39, and 730.19 cm^{-1} which refer to the formation of CuO as shown in Fig. 5. A broad absorption peak noticed at 3308.2 cm^{-1} attributed to O–H stretching of the moisture content. [23, 24] FTIR measurement of CuO nano-particles shows different peaks at different levels which corresponds to different functional groups and stretches present in it. The absorption peaks at 730, 891, 918 indicates the presence of C = C functional group which belongs to alkene class. (Source: the Libretext Libraries, University of California).

Figure 6 displays the FTIR spectra of the synthesized ZnO in the range of 4000–400 cm^{-1}. FTIR spectrum of pure ZnO nanoparticles, the peak at 594.56 cm^{-1} was the characteristic absorption of Zn–O bond and the broad band peak at 3506.9 cm^{-1} can be attributed to the characteristic absorption of O–H group. These data were similar to the results

Fig. 6 FTIR Spectroscopy ofsynthesize ZnO Nanoparticles
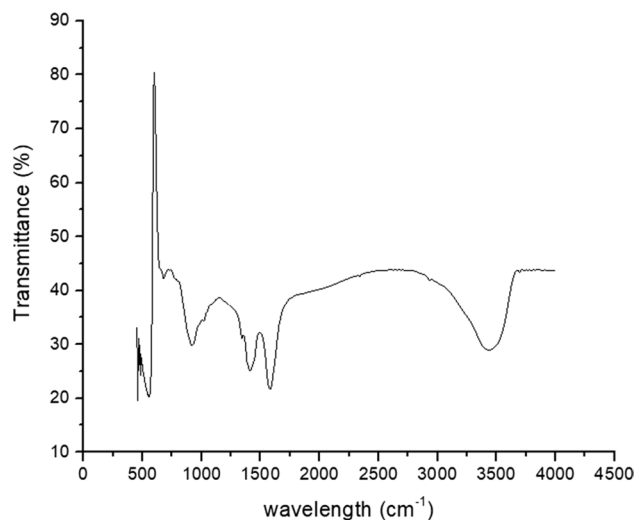
observed by others [9]. The peak at 594.56 cm^{-1} was allotted to Zn–O [25, 26]. Absorption peak between 507 and 597 indicates the presence of C-I stretching which belongs to halo compounds.Bending Peak at 903 and 1001 shows the presence of C＝C group belong alkene class. (Source: The Libretext Libraries, University of California)

4.3 SEM (scanning electronic microscope)

Morphology of CuO and ZnO Particles synthesized by sol–gel method and the size of the formed CuO and ZnO nanoparticles has been studied by SEM technique at different magnification shown in Figs. 7 and 8 respectively. Precursor concentration used during synthesis process plays an important role on morphology of nanoparticle. Figure 7 diplays

Fig. 7 SEM morpholgy of CuO Nanoparticles
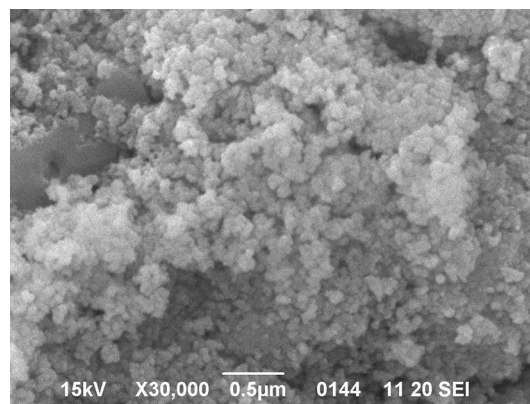
Fig. 8 SEM morphology of ZnO Nanoparticles.
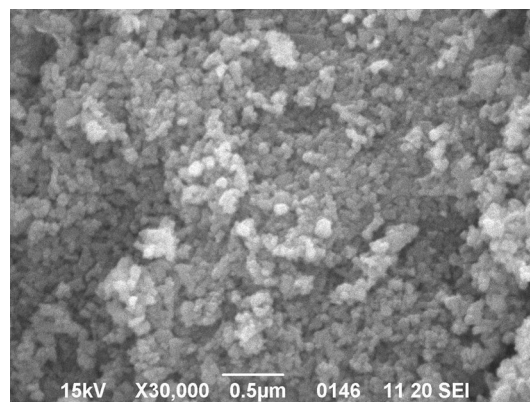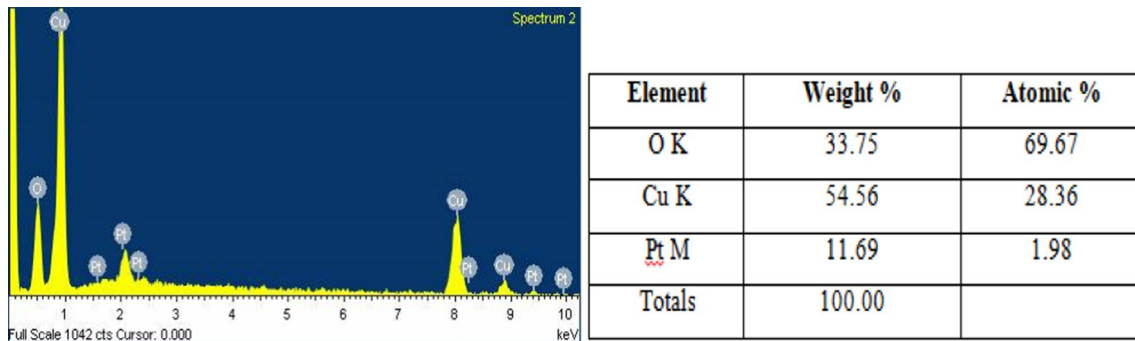
Fig. 9 EDX graph and Composition of CuO Nanoparticles

Element	Weight %	Atomic %
O K	33.75	69.67
Cu K	54.56	28.36
Pt M	11.69	1.98
Totals	100.00	
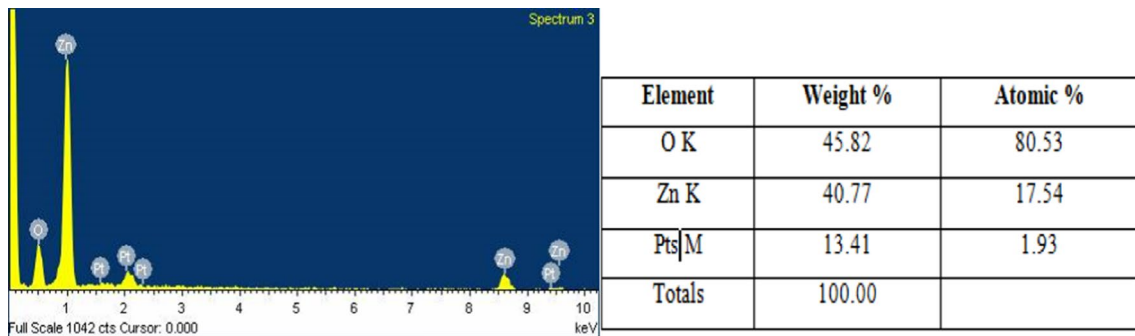
Fig. 10 EDX graph and compositionof ZnO Nanoparticles

Element	Weight %	Atomic %
O K	45.82	80.53
Zn K	40.77	17.54
Pts M	13.41	1.93
Totals	100.00	

the SEM image of CuO nanoparticle synthesized by sol–gel process. It indicates the significant and greater tendency of agglomerations. We collect images at different magnifications, most of the images show particle size between 61.25 nm to 66.12 nm.The smallest size reported at X30,000 magnifications was 61.25 nm shown in Fig. 7.

Figure 8 indicate the SEM morphology of ZnO Particle and image taken at × 30,000 magnifications were spherical in shape and having smooth surface. In the Present study Zinc Nitrate [$Zn(NO_3)_2$] has been used in synthesis process of ZnO with spherical shape via Sol–Gel Method. ZnO particle have different structure depend upon the precursor used during the synthesis process [7].The smallest size reported at × 30,000 magnification is 21.82 nm shown in Fig. 8.

4.4 Energy dispersive X-ray spectroscopy (EDX)

Result shown in Fig. 9 attained from the EDX characterization indicate that the CuO nano-particle has good purity i.e. Cu content is 54.56%, oxygen content was 33.75% with very less impurities. [27] In some studies it was found that if Sodium Hydroxide (NaOH) used as solvent, the EDX graph shows closer to stoichiometry as compared to ammonia hydroxide used as solvent [6]. The concentration of Cu was higher which claimed that synthesize CuO nanoparticle were nearly about stoichiometry.

Figure 10 indicate the Energy Dispersive X-Ray analysis of ZnO nanoparticle synthesize by Sol Gel Process and indicates that the nanoparticles were nearly about stoichiometry. The weight amount of Zn determined by EDX was 40.77 and O was 45.82 [27]. There were few traces of impurities present in the EDX spectra. The EDX data confirm the formation of ZnO nanoparticles.

5 Conclusion

The Sol gel synthesis was a bottom-up process for synthesis of nanoparticles. In this synthesis process, the final synthe-sized particles were formed by performing a many irreversible chemical reactions. In this paper, Copper oxide and zinc oxide may be synthesized via way of means of the use of sol- gel techniques that have been very natural in generating small sized nanostructures (<< 100 nm). ZnO and CuO nanoparticles had been effectively synthesized via way of means

of easy Sol–Gel approach. The organized ZnO nanoparticles have been characterised by the use of XRD, FT-IR, SEM, EDX techniques. The average particle size of CuO and ZnO is calculated as 61.25 nm and 21.82 nm respectively via way of means of the use of Scherrer's equation of substance dried at 80 °C. Elastic Strain of the synthesized CuO and ZnO nano-particles calculations concluded that the small particles have high strain and large particles have low strain; each the synthesized nano-particles size more than 20 nm so each particles have low strain. The evaluation of FTIR spectrum of CuO indicates peaks at 602.09, 678.39, and 730.19 cm^{-1} which consult with the formation of CuO. FTIR spectrum of pure ZnO nano-particles, the height of peak at594.56 cm-1changed into the function absorption of Zn–O bond. A SEM image indicates the good agglomeration of CuO nanoparticles. Zinc Nitrate [$Zn(NO_3)_2$] has been used in synthesis process of ZnO with spherical shape via Sol–Gel Method EDX analysis confirm the present of CuO and ZnO nanoparticles with less impurities and nearly about stoichiometry These CuO and ZnO nanoparticles provide super capability in future packages of food packaging and growing the shelf existence of the product. The approach has an excessive yield and may be used for big scale synthesis of ZnO and CuO nano particles.

Declarations

Competing interests The authors declare that there is no conflict of interests regarding the publication of this paper.

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