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XRD and FTIR Characterization of Mixed Metal Oxide Nanoparticles Synthesized via Grape Leaf Powder-Assisted Calcination

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Abstract
Mixed metal oxide nanoparticles have gained significant attention due to their enhanced physicochemical properties compared to single metal oxides. In the present study, Co-Ni-Zn and Ni-Fe mixed metal oxide nanoparticles were successfully synthesized using a green synthesis approach with grape leaf powder as a natural reducing and stabilizing agent, followed by calcination at 500°C to achieve crystalline oxide formation. The structural properties of the synthesized materials were systematically investigated using X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR). XRD analysis confirmed the formation of nanocrystalline phases with crystallite sizes in the range of 50–70 nm. The Co-Ni-Zn system exhibited three distinct phases corresponding to Co_3O_4, NiO, and ZnO, while the Ni-Fe system showed the formation of NiO and Fe_2O_3 phases with possible $NiFe_2O_4$ spinel formation. FTIR spectra revealed characteristic metal–oxygen (M–O) stretching vibrations confirming the establishment of stable oxide lattices in both samples. The Ni-Fe sample exhibited additional M–O bands at 668 cm^{-1}, indicating spinel ferrite formation, along with fewer organic residues compared to the Co-Ni-Zn sample, suggesting better thermal decomposition and higher purity. The presence of phytochemical-related C–O stretching vibrations confirmed that grape leaf powder components act as effective capping agents, preventing nanoparticle agglomeration. The combined results demonstrate the successful synthesis of nanostructured mixed metal oxide systems with good crystallinity and stable surface chemistry using an environmentally benign approach.
Keywords: *Green synthesis; Grape leaf powder; Co-Ni-Zn mixed oxide; Ni-Fe mixed oxide; X-ray diffraction; FTIR spectroscopy; Nanocrystalline oxides.*

1. Introduction
Mixed metal oxide nanoparticles have attracted many interests because of their better physicochemical properties than single metal oxides. The incorporation of more than one metal ion to the same oxide lattice influences crystallinity, surface chemistry, electronic configuration, and charge distribution of these materials. Therefore, mixed metal oxides exhibit improved catalytic activity, magnetism, optics, and thermal stability. Mixed metal oxides can be used in applications including catalysis, sensors, environment protection, and functional coatings. Metal oxide nanoparticles are usually synthesized through the physical and chemical methods that are toxic, energy-consuming, and hazardous. Green synthesis using plant extracts is environmentally friendly and economically sustainable technique. Plant extracts contain flavonoids, phenolics, terpenoids, alkaloids, etc. that are natural reducing, stabilizing, and capping agents. They help to reduce metal ions to nanoparticles and avoid aggregation by capping nanoparticle surfaces. Grape leaves represent an agricultural waste that contains many biologically active substances such as polyphenols, flavonoids, tannins, organic acids, and sugars. Polyphenolic substances constitute the major component that explains high antioxidant and reducing abilities of grape leaves extract. The previous studies prove that the grape leaves extract is able to synthesize many metal nanoparticles such as titanium dioxide, iron, silver, and mixed metal oxides . Biogenic synthesis using plant extracts is considered to be the best alternative to traditional chemical synthesis of nanomaterials . The utilization of grape leaf powder in nanoparticle synthesis is not only environmentally responsible but also contributes to valorization of agricultural waste, following the goals

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of sustainable development. Among all synthesis techniques, solid-state calcination technique is preferable since it is simple, cost-efficient, scalable, and allows to control composition and crystallinity. It involves a mixture of metal salts with plant powder and subsequent heating when the organic matrix decomposes and metal species transform into crystalline oxides. The calcination temperature and time have great impact on crystallinity, phase purity, and particle size. The current work is devoted to the green synthesis of Co-Ni-Zn and Ni-Fe mixed metal oxide nanoparticles using grape leaf powder as a natural reducing and stabilizing agent, followed by calcination at 500°C. The obtained materials were characterized with the help of X-ray diffraction analysis (XRD) and Fourier transform infrared spectroscopy (FTIR) in order to determine crystalline phases, crystallite size, functional groups, and metal-oxygen bonds. Structural properties of two mixed oxide systems were compared to evaluate the effect of metal composition on crystallization.

2. Materials And Methods

2.1.Materials

Analytical reagent (AR) grade metal salts were used as received. Cobalt(II) chloride hexahydrate ($CoCl_2 \cdot 6H_2O$, 237.93 g/mol, Merck) was used as the cobalt source. Nickel(II) chloride hexahydrate ($NiCl_2 \cdot 6H_2O$, 237.69 g/mol, Qualigens) was used as the nickel source. Zinc sulfate heptahydrate ($ZnSO_4 \cdot 7H_2O$, 287.55 g/mol, Merck) was used as the zinc source. Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$, 270.30 g/mol, Merck) was used as the iron source. Fresh grape leaves (Vitis vinifera) were collected, washed with deionized water, dried at 60-70°C for 24 hours, and grinded to a powder. Deionized water was used throughout the whole synthesis.

2.2.Preparation of Grape Leaf Powder

Fresh grape leaves were washed several times with deionized water to remove dust and other impurities. Then, they were dried in a hot air oven at 60-70°C for 24 hours till the constant weight was reached. The dried leaves were grinded to a powder with the help of mortar and pestle and stored in airtight containers at room temperature for further use. Grape leaves are known to be a good bio-template for nanoparticle synthesis; the phytochemicals of grape leaves provide reduction and stabilization of metal ions.

2.3.Green Synthesis of Co-Ni-Zn and Ni- Fe Mixed Metal Oxide Nanoparticles

Co-Ni-Zn and Ni-Fe mixed metal oxide nanoparticles were synthesized using grape leaf powder as a green reducing and stabilizing agent, followed by calcination. The synthesis was conducted according to the fixed stoichiometric ratio of biomass to metal precursor (0.01 mol metal precursor per 20 g grape leaf powder).

2.3.1. Synthesis of Co-Ni-Zn Mixed Oxide (Sample 1)

To obtain Co-Ni-Zn mixed oxide, 2.38 g of $CoCl_2 \cdot 6H_2O$ (0.01 mol), 2.38 g of $NiCl_2 \cdot 6H_2O$ (0.01 mol), and 2.87 g of $ZnSO_4 \cdot 7H_2O$ (0.01 mol) were added to 20 g of powdered grape leaf. The mixture was grinded for 20-30 minutes to ensure homogeneity. The powder mixture was transferred to porcelain crucible and dried in a hot air oven at 80°C for 6-8 hours to remove moisture. Afterward, it was calcined in a muffle furnace using the program heating (ramp to 200°C at 5°C/min with 1-hour holding, ramp to 500°C at 10°C/min with 3-hour holding) in order to obtain crystalline metal oxide nanoparticles. After the calcination process, the furnace was cooled down naturally to room temperature. The obtained calcined product was grinded again to powder and kept in airtight glass vials in a desiccator.

2.3.2. Synthesis of Ni-Fe Mixed Oxide (Sample 4)

To obtain Ni-Fe mixed oxide, 2.38 g of $NiCl_2 \cdot 6H_2O$ (0.01 mol) and 2.70 g of $FeCl_3 \cdot 6H_2O$ (0.01 mol) were added to 20 g of powdered grape leaf. The same procedure as described above was repeated (grinding, drying at 80°C for 6-8 hours, calcination at 500°C with the same heating program, natural cooling, and collecting).

2.4.Reaction Pathways and Formation Mechanism

The formation of mixed metal oxide nanoparticles occurs through biomass-assisted coordination and thermal decomposition. Functional groups in the grape leaf lignocellulosic matrix (hydroxyl, carboxyl, phenolic groups) coordinate with metal ions, forming

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stabilized biomass-metal complexes. Calcination leads to the decomposition of organic matrix and transformation of coordinated metal species to metal oxide phases.

For Co-Ni-Zn System:

$CoCl_2 \cdot 6H_2O \rightarrow CoO + 2HCl + 5H_2O$ (upon heating)

$NiCl_2 \cdot 6H_2O \rightarrow NiO + 2HCl + 6H_2O$

$ZnSO_4 \cdot 7H_2O \rightarrow ZnO + SO_2 + \frac{1}{2}O_2 + 7H_2O$

Final: CoO + NiO + ZnO (or mixed oxide phases)

For Ni-Fe System:

$NiCl_2 \cdot 6H_2O \rightarrow NiO + 2HCl + 6H_2O$

$FeCl_3 \cdot 6H_2O \rightarrow Fe_2O_3 + 6HCl + 3H_2O$

Final: $NiO + Fe_2O_3$ (or $NiFe_2O_4$ spinel)

During calcination, the intimate mixture of metal salt precursors in the biomass matrix leads to the formation of closely coupled oxide phases.

2.5.Characterization Techniques

2.5.1. X-ray Diffraction (XRD)

X-ray diffraction analysis was carried out to determine the crystalline phases, phase purity, and crystallite size of the synthesized nanoparticles. XRD patterns were obtained on a powder X-ray diffractometer using Cu Kα radiation (λ = 1.5406 Å), operated at 40 kV and 40 mA. The scan range was 5-90° (2θ) with the step size of 0.02° in the continuous scanning mode. Phase identification was performed by matching diffraction peaks with standard JCPDS (Joint Committee on Powder Diffraction Standards) cards. The average crystallite size was estimated by the Debye-Scherrer equation:

$$D = K\lambda / \beta \cos\theta$$

where D is the crystallite size, K is the shape factor (0.94 for spherical particles), λ is the X-ray wavelength (1.5406 Å), β is the full width at half maximum (FWHM) of the diffraction peak in radians, and θ is the Bragg angle.

2.5.1. Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared spectroscopy was used to identify functional groups, metal-oxygen bonds, and organic residues in the synthesized nanoparticles. FTIR spectra were recorded in the wavenumber range of 400-4000 cm^{-1} with the help of spectrometer equipped with ATR (attenuated total reflectance) accessory. The spectra were analyzed to detect characteristic absorption bands of various functional groups and metal-oxygen bonds.

3. Results And Discussion

3.1.XRD Analysis

3.1.1. Co-Ni-Zn Mixed Oxide (Sample 1)

The XRD pattern of Co-Ni-Zn mixed oxide nanoparticles revealed diffraction peaks at 2θ values of 36.6°, 37.1°, 43.3°, 44.8°, 62.9°, and 75.3°. The diffraction peak at 43.3° corresponds to NiO (JCPDS Card No. 47-1049), which confirms the formation of the nickel oxide phase. The diffraction peaks at 36.6° and 44.8° correspond to Co_3O_4 (JCPDS Card No. 42-1467). Diffraction peaks at 37.1° and 44.8° may indicate the formation of ZnO (JCPDS Card No. 36-1451). Diffraction peaks at 62.9° and 75.3° confirm the formation of NiO once again. The presence of distinct diffraction peaks for Co_3O_4, NiO, and ZnO without any peak shift proves the formation of the physical mixture of three distinct oxide phases in the Co-Ni-Zn system. The diffraction peak broadening indicates the nanocrystalline nature of the material. According to the Debye-Scherrer equation, the average crystallite size was calculated to be about 50-60 nm.

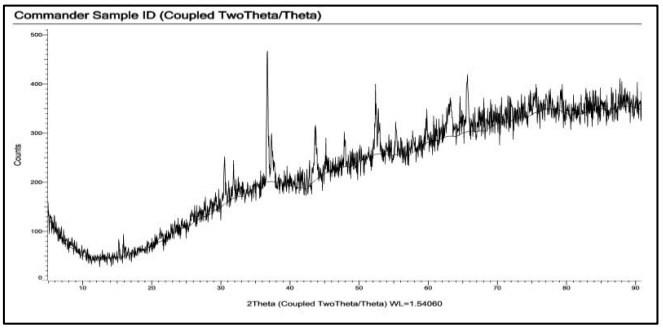

Figure 1 RD Spectra: Co-Ni-Zn Mixed Oxide (Sample 1)

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3.1.2. Ni-Fe Mixed Oxide (Sample 4)

The XRD pattern of Ni-Fe mixed oxide nanoparticles showed diffraction peaks at 2θ values of 31.2°, 35.6°, 37.1°, 43.3°, 53.7°, 57.3°, and 62.9°. The diffraction peak at 37.1° with high intensity (~1241 counts) corresponds to NiO (JCPDS Card No. 47-1049), which confirms the formation of the nickel oxide phase. The diffraction peaks at 35.6° and 53.7° correspond to Fe_2O_3 (JCPDS Card No. 39-1346), which confirms the formation of the iron oxide phase. The diffraction peaks at 31.2° and 57.3° indicate the formation of $NiFe_2O_4$ spinel (JCPDS Card No. 10-0325). The sharp and intensive diffraction peaks indicate the higher crystallinity compared to Sample 1. The average crystallite size was estimated to be 60-70 nm using the Debye-Scherrer equation. The higher crystallinity in the Ni-Fe sample (sharper diffraction peaks with higher intensity ~1241 counts) indicates better crystal growth and phase purity.

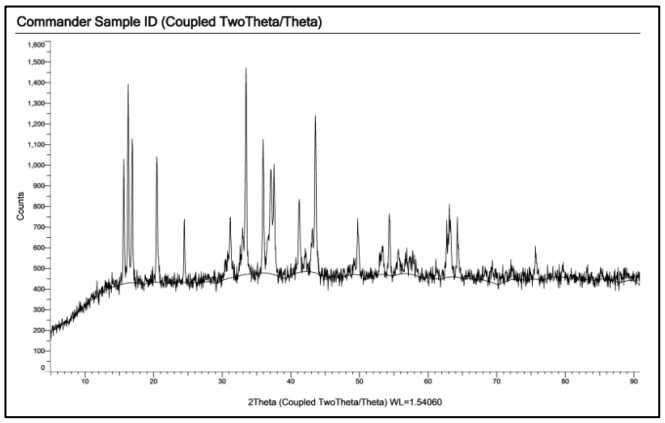

Figure 2 XRD Spectra: Ni-Fe Mixed Oxide (Sample 4)

3.1.3. Comparative XRD Analysis

Comparative XRD analysis showed distinct difference between two mixed oxide systems. The Co-Ni-Zn system revealed three distinct phases (Co_3O_4 + NiO + ZnO) without any peak shift, which proves the formation of physical mixture. In turn, the Ni-Fe system showed the possible formation of $NiFe_2O_4$ spinel together with the formation of NiO and Fe_2O_3 phases. The higher crystallinity observed in Ni-Fe sample indicates better crystal growth, while the Co-Ni-Zn sample showed smaller crystallite size (50-60 nm) compared to Ni-Fe (60-70 nm). Both samples exhibited nanocrystalline nature with crystallite sizes in the range of 50-70 nm; it confirms successful synthesis using grape leaf powder as a stabilizing agent.As shown in Table 1 Comparative XRD Analysis of Co-Ni-Zn and Ni-Fe Mixed Oxides

Table 1 Comparative XRD Analysis of Co-Ni-Zn and Ni-Fe Mixed Oxides

Parameter	Co-Ni-Zn Mixed Oxide (S1)	Ni-Fe Mixed Oxide (S4)
Phases identified	Co_3O_4 + NiO + ZnO	NiO + Fe_2O_3
Major peak (2θ)	43.3°	37.1°
Crystallite size	~50–60 nm	~60–70 nm
Spinel formation	Possible	Yes ($NiFe_2O_4$)
Crystallinity	Moderate	Higher
Peak broadening	Moderate	Moderate

3.2.FTIR Analysis

3.2.1. Co-Ni-Zn Mixed Oxide (Sample 1)

The FTIR spectrum of Co-Ni-Zn mixed oxide nanoparticles showed several characteristic absorption bands. In the high wavenumber region, a weak and broad absorption band at 3418 cm^{-1} was attributed to O-H stretching vibrations of the surface hydroxyl groups related to the adsorbed water. The absorption band at 1630 cm^{-1} was attributed to C=O stretching or H-O-H bending vibrations and proved the presence of carbonyl groups and adsorbed water. The absorption band at 1385 cm^{-1} corresponded to C-H bending or carbonate vibrations and indicated the presence of residual organics from the grape leaf powder. In the low wavenumber region, strong absorption bands at 1045 cm^{-1} were attributed to C-O stretching vibrations of phytochemicals from grape

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leaf powder, which proved their role as capping agents. The most significant features were the absorption bands at 547 cm^{-1} and 469 cm^{-1}, which correspond to metal-oxygen (M-O) stretching vibrations. The absorption band at 547 cm^{-1} may be attributed to Co-O, Ni-O, and Zn-O stretching vibrations, while the absorption band at 469 cm^{-1} may be attributed to similar M-O bonds. The presence of the characteristic bands proves the successful synthesis of Co-Ni-Zn mixed metal oxide nanoparticles. Figure 3 FTIR Spectra: Co-Ni-Zn Mixed Oxide (Sample 1).

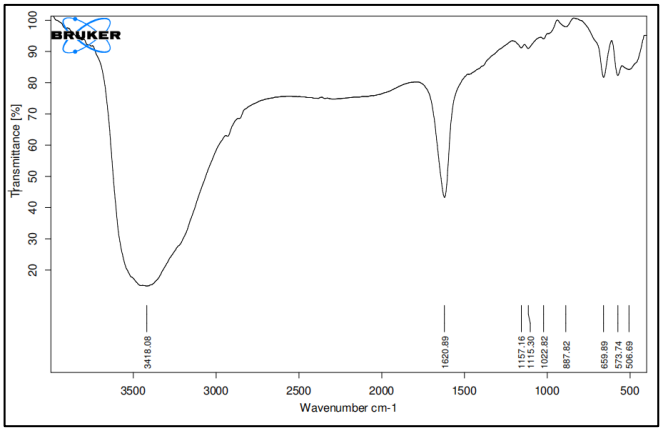

Figure 3 FTIR Spectra: Co-Ni-Zn Mixed Oxide (Sample 1)

3.2.2. Ni-Fe Mixed Oxide (Sample 4)

The FTIR spectrum of Ni-Fe mixed oxide nanoparticles revealed similar characteristic bands with some distinctions. The O-H stretching vibration was observed at 3394 cm^{-1}, shifted in comparison to the Co-Ni-Zn sample. The C=O/H-O-H bending vibration was observed at 1630 cm^{-1}, and C-O stretching vibrations were observed at 1045 cm^{-1}. The M-O stretching vibrations were clearly observed at 668 cm^{-1}, 547 cm^{-1}, and 469 cm^{-1} and corresponded to Ni-O and Fe-O bonds. The presence of the additional absorption band at 668 cm^{-1} is especially important, because it indicates the presence of Fe-O bonds, which relate to the $NiFe_2O_4$ spinel phase. It is notable that the Ni-Fe sample showed absence of C-H stretching and C-H bending peaks, indicating fewer organic residues and better thermal decomposition during calcination. As shown in Figure 4 FTIR Spectra: Ni-Fe Mixed Oxide (Sample 4).

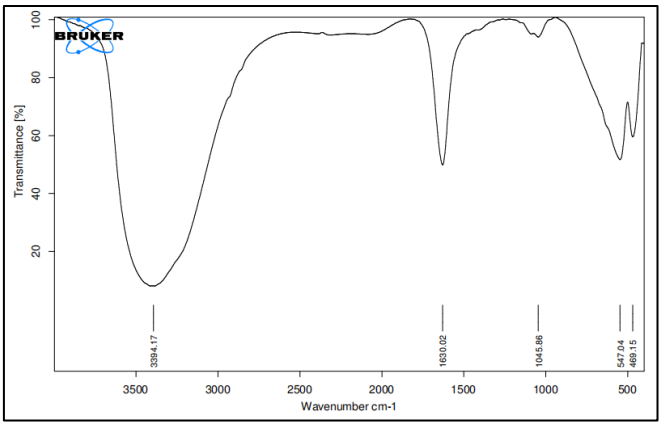

Figure 4 FTIR Spectra: Ni-Fe Mixed Oxide (Sample 4)

Table 2 Comparative FTIR Analysis of Co-Ni-Zn and Ni-Fe Mixed Oxides

Wavenumber (cm^{-1})	Functional Group	Co-Ni-Zn (S1)	Ni-Fe (S4)
3394–3418	O–H stretching (water)	Present	Present
1630	C=O / H–O–H bending	Present	Present
1385	C–H bending / carbonate	Present	Absent
1045	C–O stretching (phytochemicals)	Present	Present
668	M–O stretching (Fe–O)	Absent	Present
547	M–O stretching	Present	Present
469	M–O stretching	Present	Present

3.2.3. Comparative FTIR Analysis

The comparative FTIR analysis revealed some differences between the two samples. Both samples

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revealed characteristic M-O stretching vibrations, which confirmed the successful formation of metal oxides. The additional M-O band at 668 cm^{-1} in the Ni-Fe sample confirmed the formation of Fe-O bonds in ferrite spinel structure. It correlates with the XRD results and proves the possible formation of $NiFe_2O_4$ spinel. The Ni-Fe sample showed fewer organic residues (absence of 1385 cm^{-1} bands), indicating better thermal decomposition and higher purity. The Co-Ni-Zn sample contained more organic residues, which probably relates to the presence of multiple metal species, which influenced the decomposition of biomass components. The presence of C-O stretching vibrations at 1045 cm^{-1} in both samples indicates that phytochemicals from grape leaf powder are effective capping agents, which avoid nanoparticle aggregation. The presence of the surface hydroxyl groups (O-H at 3394-3418 cm^{-1}) in both samples increases surface reactivity and can be useful for catalytic and photocatalytic applications.

4. Discussion

The comprehensive structural characterization confirms the successful synthesis of Co-Ni-Zn and Ni-Fe mixed metal oxide nanoparticles using grape leaf powder as a green reducing and stabilizing agent. The combination of XRD and FTIR analyses provides additional information about the material properties and proves the reliability of the green synthesis technique. X-ray diffraction analysis proved the crystalline nature of both synthesized materials with the presence of diffraction peaks corresponding to the oxide phases. Absence of any impurity peaks indicates high phase purity and proves effective transformation of precursor salts into oxide lattices during calcination. The crystallite sizes estimated with the help of the diffraction peak broadening fell in the nanometer range (50-70 nm), which confirms the nanocrystalline domain formation. The variation in crystallite sizes in case of Co-Ni-Zn (50–60 nm) and Ni-Fe (60–70 nm) may be attributed to different ionic radii and coordination preferences of metal ions. Further, the structural information provided by XRD results indicates some important differences in the two systems under study. In the Co-Ni-Zn system, three different phases are identified without any peak shift, thus proving a physical mixture and absence of any solid solution formation. This means that the metal oxides Co_3O_4, NiO, and ZnO do not inter-diffuse in such conditions. On the other hand, Ni-Fe system indicates the formation of spinel $NiFe_2O_4$, which means that Fe^{3+} ions get incorporated into the NiO structure. The formation of spinel is due to the similarity in ionic radii and coordination preferences of Ni^{2+} and Fe^{3+} ions. Further, the FTIR spectroscopic data clearly indicates the formation of oxides as indicated by the presence of metal-oxygen vibration bands in the lower wavenumber region. Bands found at 668, 547, and 469 cm^{-1} are due to vibrations of metal-oxygen bonds. The presence of extra M-O vibrations at 668 cm^{-1} in case of Ni-Fe is particularly indicative of the formation of $NiFe_2O_4$ spinel, as it corresponds to Fe-O vibration frequencies. Further, the presence of C-O vibration frequencies at 1045 cm^{-1} in both the cases proves that the phytochemicals from grape leaf powder act as capping agents and prevent nanoparticles from agglomeration. Further, it was observed that Ni-Fe sample contained lesser organic residue than the Co-Ni-Zn sample. This may be due to different thermal stability of the metal salts used, or due to the catalytic effect of iron species on degradation of organic matter. Higher crystallinity of Ni-Fe sample indicates that iron promotes crystal growth, whereas the presence of multiple metal species in the Co-Ni-Zn sample inhibits crystallite growth, thereby giving rise to smaller particle size. These observations show that the synthesized nanoparticles have suitable structural features that allow them for functional investigations. Their high crystallinity and phase purity, combined with the presence of phytochemical capping agents, indicate the possibility of their use in catalysis, photocatalysis, and antimicrobial activities. Further, the formation of $NiFe_2O_4$ spinel in case of Ni-Fe sample will impart additional magnetic property, while the physical mixture of Co_3O_4, NiO, and ZnO in case of Co-Ni-Zn system may provide a synergic effect for catalytic applications.

Conclusion

In the current study, Co-Ni-Zn and Ni-Fe mixed metal oxide nanoparticles were successfully synthesized using grape leaf powder as a green

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reducing, stabilizing and capping agent using solid-state calcination method at 500°C. The synthesized materials were characterized using XRD and FTIR spectroscopy to determine their structural features.

Successful Synthesis: Grape leaf powder served as an excellent green reducing, stabilizing and capping agent for synthesis of both Co-Ni-Zn and Ni-Fe mixed metal oxide nanoparticles. Presence of phytochemicals (flavonoids, phenolics, terpenoids) in grape leaf powder facilitated the reduction of metal ions and prevention of nanoparticle agglomeration.

XRD Confirmation:XRD patterns confirmed the formation of nanocrystalline phases in both systems:
Co-Ni-Zn: Co_3O_4 + NiO + ZnO (physical mixture of three phases)
Ni-Fe: NiO + Fe_2O_3 (possible $NiFe_2O_4$ spinel formation)

Crystallite Size: The crystallite sizes varied between 50 and 70 nm, thus proving their nanocrystalline nature. The Co-Ni-Zn system had smaller crystallite size (50–60 nm), while the Ni-Fe system had larger crystallite size (60–70 nm).

FTIR Confirmation: FTIR spectra showed characteristic M–O stretching vibrations:
Co-Ni-Zn: 547, 469 cm^{-1}
Ni-Fe: 668, 547, 469 cm^{-1}

$NiFe_2O_4$ Spinel: The Ni-Fe sample showed an additional M–O band at 668 cm^{-1}, characteristic of Fe–O stretching vibrations in spinel ferrite structures, confirming $NiFe_2O_4$ spinel formation.

Comparative Assessment: The Ni-Fe sample showed better purity with fewer organic residues (absence of 1385 cm^{-1} peaks), indicating more effective thermal decomposition and higher phase purity compared to Co-Ni-Zn.

Green Synthesis Advantage: Use of grape leaf powder as the synthesis medium offers sustainable and cost-effective way of synthesis of nanomaterials, which is in line with the principles of green chemistry.

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