International Journal of
Social Science and
Education Research

ISSN Print: 2664-9845
ISSN Online: 2664-9853
Impact Factor: RJIF 8.00
IJSSER 2024; 6(1): 20-24
www.socialsciencejournals.net
Received: 13-11-2023
Accepted: 27-12-2023

Somveer
PhD Scholar, Dairy Engineering Division, ICAR-National Dairy Research Institute, Karnal, Haryana, India

FME Emerald
Principal Scientist, Dairy Engineering Division, ICAR-National Dairy Research Institute, Karnal, Haryana, India

Kumari M
Teaching Associate, Dairy Engineering Division, College of Dairy Science and Technology, Rajasthan University of Veterinary and Animal Sciences, Bikaner, Rajasthan, India

Rushikesh R Deshmukh
PhD Scholar, Dairy Microbiology Division, ICAR-National Dairy Research Institute, Karnal, Haryana, India

Prince
PhD Scholar, Dairy Chemistry Division, ICAR-National Dairy Research Institute, Karnal, Haryana, India

Ravikant V Vinchurkar
PhD Scholar, Dairy Microbiology Division, ICAR-National Dairy Research Institute, Karnal, Haryana, India

Lakshmaiah B
PhD Scholar, Dairy Microbiology Division, ICAR-National Dairy Research Institute, Karnal, Haryana, India

Corresponding Author:
Somveer
PhD Scholar, Dairy Engineering Division, ICAR-National Dairy Research Institute, Karnal, Haryana, India

A comprehensive review of characterization techniques empowering nanoparticle innovation for public benefits and sustainable future

Somveer, FME Emerald, Kumari M, Rushikesh R Deshmukh, Prince, Ravikant V Vinchurkar and Lakshmaiah B

DOI: https://doi.org/10.33545/26649845.2024.v6.i1a.73

Abstract

Nanoparticles (NPs) exhibit remarkable properties due to their diminutive size and high surface area, holding immense potential for revolutionizing various aspects of public welfare and sustainability. However, translating this potential into tangible applications hinges on thorough characterization, enabling precise knowledge of their physicochemical properties. The synthesis and application of NPs have witnessed significant growth, with particular emphasis on addressing societal challenges. From healthcare to environmental solutions, NPs play a crucial role. Key areas of focus include the analysis of physicochemical properties, surface functionalities, and structural attributes through advanced characterization tools. The integration of these techniques not only ensures the quality control of NP formulations but also guides the design of innovative solutions for diverse public welfare challenges. By scrutinizing aspects like biocompatibility, environmental impact, and potential toxicity, characterization techniques pave the way for safe and sustainable NPs formulations.

Keywords: Nanoparticles, characterization techniques, sustainability, biocompatibility, environmental impact

Introduction

In the modern world, everybody is concerned about the conservation of energy and researchers are viewing for new innovative methods for efficient usage of energy in minimum time. But without improvement in the heat transfer process, it is quite difficult to achieve. When a material especially metal, metal oxide gets reduced to nanoscale size, its exotic properties get developed at a significant level such as enlargement in surface area, constructive random molecular motion, surface tension, van der Waal's attractions, increase in active sites for reaction, etc. (Chauhan *et al.,* 2016) [4]. The proper dispersion of these NPs in base-fluids makes up the fluid with enhanced thermal conductivity and other thermo-physical properties. Characterisation of NPs is carried out after their synthesis so as to examine the desired properties obtained by NPs. The various techniques for characterisation are provided in Table 1.

Table 1: Characterisation techniques of nanoparticles (Dabhane *et al*., 2021a) [6]

S. No.	Characterisation techniques	Information
1.	Zeta Potential	Surface charge and stability
2.	Atomic Force Microscopy (AFM)	Surface topography and NPs size
3.	UV-Vis Spectroscopy	Stability and formation of NPs
4.	Field Emission Scanning Electron Microscopy (FESEM)	Shape and size
5.	Transmission Electron Microscopy (TEM)	Shape and size
6.	Fourier Transform Infrared Spectroscopy (FTIR)	Functional groups
7.	X-Ray Powder Diffraction (XRD)	Crystal structure and size
8.	Vibrating Sample Magnetometer (VSM)	Magnetic properties of NPs
9.	Thermo Gravimetric Analysis (TGA)	Thermal stability
10.	Dynamic Light Scattering (DLS)	Size distribution of NPs
11.	X-Ray Photoelectron Spectroscopy (XPS)	Chemical surface analysis
12.	Atomic Absorption Spectroscopy (AAS)	Elemental analysis
13.	Energy Dispersive X-Ray Spectroscopy (EDS)	Elemental analysis and purity
14.	Brunauer-Emmett-Teller (BET)	Surface analysis and stability

Characterisation of metal oxide nanoparticles

Suwanboon, *et al.* (2008) [14] observed that by increasing the Al content in Zn reaction mixture, the shape of ZnO NPs changed from rod-like to spherical and when ZnO was doped with Al, the crystallite and grain sizes were similarly reduced to the nanoscale range, with the lowest particle size of roughly 25-30 nm obtained at 10 mol% Al.

ZnO NPs were synthesised using NaOH and Zn $(CH_3CO_2)_2.2H_2O$ at room temperature through sol-gel method and characterized for crystallography, morphology and functional groups using XRD, SEM and FTIR, respectively (Balushi *et al*., 2016) [3]. The uniform distribution of NPs with particle size of 100±4 nm was interpreted from SEM micrographs and the ZnO deformation and stretching were revealed from FTIR peaks obtained at 1516 and 523 cm^{-1}, respectively. A wet chemical route approach was used to synthesize ZnO NPs. The obtained peaks at 2θ values of 31.79, 34.44, 36.27, 47.63, 56.62, 62.91 and 67.96° were assigned to (100), (002), (101), (102), (110), (103) and (112) planes, which correlated to the hexagonal wurtzite structure of ZnO according to XRD with the crystalline size of 23.31 nm. The optical band gap energy for ZnO NPs was found to be in the range of 3.0 to 3.2 eV, which was slightly lower than the published band gaps values of 3.37 eV (Chauhan *et al.*, 2016) [14].

Vinay *et al.* (2019) [15] verified the presence of cow urine mediated Ag_2O NPs by XRD pattern analysis. TEM images revealed the size of NPs as 20 nm while the energy bandgap (E_g) of ~2.74 eV and a strong absorption peak at 430 nm wavelength supported the confirmation of Ag_2O NPs. The band at 520 cm^{-1} in the FTIR graph confirmed the presence of silver oxide bond as well.

Sumesh *et al.* (2019) [13] synthesised aluminium oxide (Al_2O_3) NPs from 'Muntingia Calabura' leaf using $Al_2(NO_3)_3.9H_2O$ as precursor. The presence of $\alpha\text{-}Al_2O_3$ NPs was confirmed from XRD data, whereas its purity and presence of functional groups were resulted from EDS and FTIR, respectively. SEM images also revealed the reduction of void gaps, fibre breakage and matrix breakage due to Al NPs addition. TGA results obtained after mixing NPs with sisal/coil/Al are shown in Table 2. By mixing of 0-3% mass concentration (wt.%) of Al NPs in sisal/coir composites, not only there was enhancement of initial T_{05} from 107.13°C to 127.76 °C, but also there was an improvement in residual percentage as well.

Table 2: TGA results of nano-aluminium/sisal/coir combinations

Mixing combination (nano-Al/sisal/coir)	Degradation temperature (°C) (T_{05})	Degradation temperature (°C) (T_{50})	Degradation temperature (°C) (T_{70})	Residual (%)
0%	121.66	386.16	493.19	27.15
1%	109.43	387.04	503.28	27.42
2%	107.13	388.34	518.29	27.85
3%	127.77	390.41	550.03	29.14

Ranjithkumar *et al.* (2021a) [10, 11] confirmed the hexagonal and leaf formations of ZnO NPs mediated from cow urine after analysis of FESEM images and the colloidal stability of -18.3 mV was obtained after zeta potential study. The XRD and FTIR graphs showed the formation of hexagonal wurtzite structure for ZnO NPs.

Characterisation of material doped nanoparticles

Chou *et al.* (2011) [5] examined the characteristics of AZO NPs with 0-2.5 wt. % Al content. It was observed that more irregular shapes and finer sizes of AZO NPs were obtained, as compared with ZnO NPs, when Al_2O_3 content was increased. Ahmad *et al.* (2012) [16]. Used the combustion approach to manufacture and characterise the AZO NPs along with pure ZnO and found that, as the Al doping level was increased, the crystallite size of NPs reduced from 25 to 11 nm. ZnO and AZO NPs had bond lengths of 1.981 and 1.976 Å, respectively which could be related to Al's smaller ionic radius. The release of enormous amounts of gaseous products explained the high porosity of AZO NPs. The particle size of undoped ZnO NPs was larger and spherical, whereas that of AZO NPs was smaller and ellipsoidal. FTIR analysis revealed a peak at 3440 cm^{-1}, which was attributed to -OH group stretching and bending vibrations at surface of AZO NPs. Amornpitoksuk *et al.* (2012) [2] revealed that without any dopant, the ZnO powders exhibited an urchin-like morphology, which changed to an agglomerated rod-like structure when the Ag doping was less than or equal to 0.5 mol %, whereas the urchin-like morphology re-developed as the Ag concentration was raised further.

Khan *et al.* (2013) [9] synthesised AZO NPs using sol-gel technique and after characterization, the authors noticed that size of AZO NPs decreased while energy band gap increased when compared to undoped ZnO as shown in Table 3. SEM images showed increased agglomeration with increased Al concentration in AZO NPs. EDS data also confirmed the presence of C, O, Zn and Al. The interpretations from optical absorption spectra revealed that an increase in optical band gap was obtained with increase in Al doping concentration having a maximum value of 3.44 eV at 2.0 at.%.

Table 3: Variation in crystallite size and energy band gap with aluminium concentration

Aluminium concentration (wt %)	Crystallite size (nm)	Band gap (eV)
0.0	32.0	3.28
0.5	27.2	3.37
1.0	24.1	3.40
1.5	14.9	3.44

The doping ratio of Al in AZO NPs affects its phase composition, structure, size and after investigating these effects, Alkahlout *et al.* (2014) [1] found that incorporation of Al in ZnO using UV-vis spectroscopy and by observing a blue shift due to Burstein-Moss effect. The XRD analysis revealed the crystalline structure of AZO NPs with a single zincite phase. For an Al-to-Zn ratio of 0.5 mol %, NPs of 12 nm size were obtained and particle size as well as BET surface area were found to increase by further increasing the doping ratio. Presence of blue shift in UV-vis spectroscopy indicated the presence of Al in ZnO. Mallika *et al.* (2014) [17] synthesised AZO NPs using sol-gel technique and PVA as a chelating agent and the impact of Al doping on structural and optical characteristics was investigated. The hexagonal wurtzite structure of ZnO was verified by XRD patterns and the quantum size confinement effects were validated by the blue shift observed in the UV-vis absorption spectra as Al doping was increased. It was concluded that the strength of emission peaks fell monotonously with Al doping.

Chromium-doped Al_2O_3 was successfully synthesised through the combustion method by using $Al_2(NO_3)_3.9H_2O$, ammonium dichromate and sucrose as precursors (Farahmandjou and Golabiyan, 2015) [7]. The XRD data and SEM images confirmed the crystallite size of 35 nm and the presence of foamy as well as agglomerated NPs, respectively. The mass content and atomic content were found to be 1.89% and 0.81%, respectively based on EDS results. Rameshkumar *et al.* (2020) [18] prepared AZO NPs by co-precipitation method. The authors observed nano rod-like shape of NPs using SEM images, whereas the XRD analysis resulted in hexagonal wurtzite structure with mean crystallite size of 52 and 21 nm for 0.5 and 1.5 at. % Al, respectively. The optical absorbance was found between 380 and 392 nm for 0.5 and 1.5 at.% of Al doped NPs, respectively and the peaks obtained at 3400, 1500, 540 and between 425-440 cm^{-1} in FTIR analysis revealed the presence of hydroxyl groups, carbonyl groups, Al and ZnO, respectively. Ranjithkumar *et al.* (2021b) [10, 11] studied the surface morphology, structural and thermal properties of Ag-doped ZnO NPs synthesised using cow urine. The XRD pattern revealed the formation of hexagonal wurtzite structure and the images obtained from FESEM and TEM revealed the plate-like morphology of Ag-doped ZnO NPs. From the TGA curves, it was concluded that the thermal stability of cow urine mediated Ag-doped ZnO NPs was up to 643.23 °C melting point and could be used for various applications below this temperature.

Shreema *et al.* (2021) [12] used green synthesis strategy to prepare Ag-ZnO NPs from fresh leaf extract of *Morinda citrifolia.* FTIR results indicated that large absorption peaks associated with Ag-ZnO were detected at 3401,1598, 1385 and 538 cm^{-1} which showed carboxylic acid and hydroxyl stretch vibrations, carbon-carbon bond (C = C) and carbon-nitrogen bond (C-N) stretching of benzene/amide linkage and aromatic amines/alkyl halides and stretching of zinc-oxygen bond, respectively. The UV-vis absorption spectra showed the absorption peak at 396 nm and corresponding bandgap energy as 3.13 eV for Ag-Zno NPs. When compared to pure ZnO, SEM examination revealed that Ag-ZnO NPs had spherical shape with reduced aggregation and perfect separation, whereas XRD pattern revealed the particle size for Ag-ZnO NPs was smaller than pure ZnO.

Benefits and Applications of Metal-doped Nanoparticles

The metal-doped NPs possess following benefits over metal NPS

- **Enhanced Catalytic Activity:** Metal-doped NPs often exhibit superior catalytic properties, making them effective catalysts in various chemical reactions (Shreema *et al*., 2021) [12].
- **Tunable Properties:** The introduction of metal dopants allows for the fine-tuning of NPs properties, including optical, electronic, and magnetic characteristics.
- **Improved Stability:** Metal dopants can enhance the stability of NPs, making them more resilient to environmental factors and ensuring prolonged functionality.
- **Altered Reactivity:** The presence of metal dopants can modify the reactivity of NPs, expanding their applicability in different chemical and biological processes (Ahmad *et al*., 2012) [16].
- **Unique Optical and Electronic Features:** Metal-doped NPs can possess unique optical and electronic features, enabling applications in fields such as sensing, imaging, and electronics.
- **Antibacterial and Antimicrobial Properties:** Certain metal dopants exhibit antibacterial and antimicrobial properties, making them valuable for medical applications, including drug delivery and antimicrobial coatings (Khan *et al*., 2013) [9].
- **Magnetic Resonance Imaging (MRI) Contrast Agents:** Metal-doped NPs, particularly those containing paramagnetic metals, can serve as contrast agents in MRI, enhancing imaging capabilities.
- **Enhanced Photocatalytic Activity:** Metal-doped NPs can be used as efficient photocatalysts for applications

such as water purification and pollutant degradation under light exposure.

- **Biomedical Applications:** Metal-doped NPs show promise in biomedical applications, including targeted drug delivery, imaging, and therapeutic treatments.
- **Environmental Remediation:** Metal-doped Nps can be employed in environmental remediation processes, such as the removal of pollutants and contaminants from air, water, and soil (Chou *et al*., 2011) [5].

Conclusion

The advancement of NPs holds immense promise for transforming various facets of public welfare and sustainability due to their unique properties derived from their small size and high surface area. However, realizing the potential benefits of NPs requires a comprehensive understanding of their physicochemical properties, emphasizing the critical role of thorough characterization techniques. The synthesis and application of NPs have experienced substantial growth, addressing societal challenges across diverse fields, from healthcare to environmental solutions.

A broad range of characterization methods has been explored enabling researchers to scrutinize surface charge, stability, size, shape, crystal structure, magnetic properties, thermal stability, and other critical parameters. These techniques ensure the quality control of NPs formulations and guide the design of innovative solutions for addressing varied public welfare challenges. Of particular significance is the careful examination of biocompatibility, environmental impact, and potential toxicity, facilitated by these characterization techniques, thereby paving the way for the development of safe and sustainable NPs formulations. The subsequent sections delve into the characterization of metal oxide NPs, shedding light on specific studies and methodologies employed.

In conclusion, the synthesis and characterization of NPs, especially metal-doped variants, offer a myriad of benefits and applications. The enhanced catalytic activity, tunable properties, improved stability, altered reactivity, and unique optical and electronic features of metal-doped NPs make them invaluable across diverse domains. From antibacterial and antimicrobial applications to serving as MRI contrast agents, these NPs demonstrate versatility and potential for addressing current challenges in public welfare and sustainability. The thorough exploration of characterization techniques and their integration into the synthesis process is fundamental to unlocking the full potential of NPs for the betterment of society and the environment. This comprehensive review has underscored the pivotal importance of characterizing key aspects, such as physicochemical properties, surface functionalities, and structural attributes, using advanced techniques.

References

1. Alkahlout A, Al Dahoudi N, Grobelsek I, Jilavi M. Synthesis and characterisation of aluminium doped zinc oxide nanostructures via hydrothermal route, Journal of Materials. 2014;214:1-8.
2. Amornpitoksuk P, Suwanboon S, Sangkanu S, Sukhoom A, Muensit N, Baltrusaitis J. Synthesis, characterization, photocatalytic and antibacterial activities of Ag-doped ZnO powders modified with a diblock copolymer. Powder Technology. 2012;219:158-164.
3. Balushi NAA, Devi MG, Al Abri M. Preparation and characterisation of ZnO nanoparticles. Journal of Nanoscience and Technology. 2016;2(4):192-194.
4. Chauhan Z, Pradhan CB, Kumari M, Chetri DR, Kafley TN, Zangpo S, *et al*. Growth and characterization of ZnO nanoparticles. International Journal of Innovative Research in Computer and Communication Engineering. 2016;4(4):2320-9801.
5. Chou YH, Chau JLH, Wang WL, Chen CS, Wang SH, Yang CC. Preparation and characterisation of solid-state sintered aluminium-doped zinc oxide with different alumina contents. Bulletin of Materials Science. 2011;34(3):477-482.
6. Dabhane H, Ghotekar SK, Tambade PJ, Pansambal S, Ananda Murthy HC, Oza R, *et al*. Cow urine mediated green synthesis of nanomaterial and their applications: A state-of-the-art review. Journal of Water and Environmental Nanotechnology. 2021;6(1):81-91.
7. Farahmandjou M, Golabiyan N. Solution combustion preparation of nano-Al_2O_3: synthesis and characterization. Challenges in Nano and Micro Scale Science and Technology. 2015;3(2):100-105.
8. Khan A, Ali HM, Nazir R, Ali R, Munir A, Ahmad B, *et al*. Experimental investigation of enhanced heat transfer of a car radiator using ZnO nanoparticles in H_2O-ethylene glycol mixture. Journal of Thermal Analysis and Calorimetry. 2019;138(5):3007-3021.
9. Khan W, Khan ZA, Saad AA, Shervani S, Saleem A, Naqvi AH. Synthesis and characterization of Al doped ZnO nanoparticles. International Journal of Modern Physics: Conference Series. 2013;22(1):630-636.
10. Ranjithkumar B, Kumar ER, Srinivas M, Ramalingam HB, Srinivas C, Magesh G, *et al*. Evaluation of structural, surface morphological and thermal properties of Ag-doped ZnO nanoparticles for antimicrobial activities. Physica E: Low-dimensional Systems and Nanostructures. 2021;133:114801.
11. Ranjithkumar B, Ramalingam HB, Kumar ER, Srinivas C, Magesh G, Rahale CS, *et al*. Natural fuels (Honey and Cow urine) assisted combustion synthesis of zinc oxide nanoparticles for antimicrobial activities. Ceramics International. 2021;47(10):14475-14481.
12. Shreema K, Mathammal R, Kalaiselvi V, Vijayakumar S, Selvakumar K, Senthil K. Green synthesis of silver doped zinc oxide nanoparticles using fresh leaf extract Morinda citrifoliaand its antioxidant potential. Materials Today: Proceedings. 2021;47(9):2126-2131.
13. Sumesh KR, Kanthavel K. Green synthesis of aluminium oxide nanoparticles and its applications in mechanical and thermal stability of hybrid natural composites. Journal of Polymers and the Environment. 2019;27(10):2189-2200.
14. Suwanboon S, Amornpitoksuk P, Haidoux A, Tedenac JC. Structural and optical properties of undoped and aluminium doped zinc oxide nanoparticles via precipitation method at low temperature. Journal of Alloys and Compounds. 2008;462(1-2):335-339.
15. Vinay SP, Nagaraju G, Chandrappa CP, Chandrasekhar N. Novel Gomutra (cow urine) mediated synthesis of silver oxide nanoparticles and their enhanced photocatalytic, photoluminescence and antibacterial

studies. Journal of Science: Advanced Materials and Devices. 2019:4(3):392-399.

16. Ahmad K, Bhatti IA, Muneer M, Iqbal M, Iqbal Z. Removal of heavy metals (Zn, Cr, Pb, Cd, Cu and Fe) in aqueous media by calcium carbonate as an adsorbent. International Journal of Chemical and Biochemical Sciences. 2012;2:48-53.
17. Surapaneni KM, Priya VV, Mallika J. Pioglitazone, quercetin and hydroxy citric acid effect on cytochrome P450 2E1 (CYP2E1) enzyme levels in experimentally induced nonalcoholic steatohepatitis (NASH). European Review for Medical & Pharmacological Sciences. 2014 Sep 15, 18(18).
18. RameshKumar S, Shaiju P, O'Connor KE. Bio-based and biodegradable polymers-State-of-the-art, challenges and emerging trends. Current Opinion in Green and Sustainable Chemistry. 2020 Feb 1;21:75-81.