NanoNEXT

Research Article

DOI: 10.54392/nnxt2545

Microstructural Analysis of Sol-Gel Synthesis of Alumina Nanoparticles

July Randhari [a], Prafulla Kumar Mallik [b], *, Sushant Kumar Senapati [a]

[a] Department of Mechanical Engineering, Indira Gandhi Institute of Technology, Sarang-759146, India
[b] Department of Metallurgical and Materials Engineering, Indira Gandhi Institute of Technology, Sarang, 759146, India
* Corresponding author Email: prafulla.mallik@gmail.com
DOI: https://doi.org/10.54392/nnxt2545
Received: 29-07-2025; Revised: 02-03-2025; Accepted: 15-12-2025; Published: 24-12-2025

Abstract: Alumina has been widely used in biomedical applications due to its superior chemical, mechanical, and wear properties, and exhibits good biocompatibility. However, its limited use in hip and knee replacement applications is due to its low fracture toughness and bio-inertness. This study focuses on the synthesis and characterization of alumina nanopowders using a cost-effective sol-gel method using aluminum isopropoxide, as a precursor. The powders obtained after drying the gel in an oven were calcined at 800°C for 1 hour in an electric furnace to produce the alumina nanopowder. During this process, parameters, such as temperature, pH, and stirring time, were carefully controlled to obtain the final product. X-ray diffraction, SEM, and TEM analysis were used to characterize the phases, composition, particle size, and morphology of alumina nanopowders. As a result, SEM and TEM analyses in conjunction with XRD data indicate that high-purity alumina nanopowders can be synthesized via the sol-gel process.

Keywords: Al_2O_3 nanoparticles, Sol-gel method, X-ray diffraction, Scanning Electron Microscope, TEM analysis

1. Introduction

Nanotechnology is a rapidly developing field with applications in science and technology for engineering new materials at the nanoscale [1]. The fundamental explanations for nanoparticles' unique qualities are quantum confinement, induced by electron mobility, and surface effects, generated by an increase in volume/area. Considering the needs of biomedical science for a new class of materials, alumina nanoparticles have been regarded as a suitable nanomaterial for various biomedical and biotechnology applications [2, 3]. Alumina is a biomaterial used in implants [4] due to its high inertness and higher compressive than tensile strength, which allows for more efficient compressive loading in biomedical applications [5, 6]. Furthermore, alumina provides the best tribological characteristics for articulating surfaces in orthopedic implants [7]. The reduction of alumina particle size to the nanometre scale further enhances its surface activity and mechanical response, which can improve biological interactions. The production of nanoscale alumina has been investigated using a number of methods that can be replicated in the lab. These methods differ in terms of precursors used, synthesis temperature, pressure, stirring speed, and pH, all of which are crucial for obtaining the required alumina phase [8]. Furthermore, each approach has different limits in terms of particle size, surface area, and porosity [9].

Alumina nanoparticles can be synthesized using several physical and chemical methods such as mechanical milling, spray pyrolysis, laser ablation, hydrothermal processing, combustion synthesis, and chemical vapour deposition. Among these techniques, the sol–gel method is widely preferred due to its low processing temperature, excellent compositional homogeneity, and precise control over particle size and morphology [10, 11]. It also improves the biocompatibility and bioactivity of scaffolds, mechanical strength, and prevents corrosion [12]. Several researchers have reported the synthesis of alumina nanoparticles using different sol–gel routes and precursors for biomedical applications [13-15]. Several synthetic techniques have been investigated to create nano-sized alumina with controlled phase, size, and shape. The acid leaching method successfully produced α-alumina powder with spherical morphology and particle size below 100 nm from calcined kaolin. Calcination at 1200 °C resulted in phase-pure α-alumina. Adding a surfactant during processing improved particle size refinement and uniformity [16].

This study emphasizes the role of processing additives in influencing nanoscale characteristics.

Nano-sized alumina plates were also created utilizing the solution combustion approach, employing glycine and polyvinyl alcohol as organic fuels. The use of macromolecular fuels allowed for more control over particle morphology and facilitated the development of plate-like alumina nanostructures [17]. A new method for synthesizing γ-alumina nanocrystals with regulated size and shape involves employing oleylamine as a capping agent. The capping agent was crucial in controlling crystal development and preventing agglomeration [18]. Nanoscale α-alumina powder was made utilizing the urea-formaldehyde gel combustion process, with aluminium nitrate as the oxidant. Calcination at 1100 °C was necessary to generate the crystalline α-alumina phase, highlighting the importance of thermal treatment in phase change [19]. These studies clearly demonstrate that although various chemical routes can produce nano-alumina, high calcination temperatures and complex processing steps are often required. This creates the need for simpler and more cost-effective synthesis approaches with better control over particle size and phase purity. However, detailed microstructural studies linking synthesis conditions with phase formation remain essential. The present work focuses on the synthesis of alumina nanoparticles using aluminium isopropoxide via the sol–gel method and their detailed structural and morphological characterization using XRD and TEM techniques.

2. Experimental Procedures

2.1 Synthesis of Alumina Nano powder

Alumina nanoparticles were prepared by the sol-gel method using a stochiometric ratio of aluminum Isopropoxide, ethanol, and HCl as precursors. Initially, alumina isopropoxide (Loba Chemie Pvt Ltd.) was dissolved in ethanol (Sisco Research Laboratories) and stirred continuously for 20 minutes using a magnetic stirrer. To this solution, distilled water (Al: H_2O=1:4) was added and stirred for an additional 1 hour. After complete dissolution, the HCl solution (Merck Life Science Pvt Ltd.) was added dropwise and stirred for an additional 1 hour. During magnetic stirring, the magnetic capsule speed was 300 rpm, the solution temperature was 60 ℃, and the solution pH was 7.5. The product was kept for 24 hours to set the gel, and further, the gel was dried in an electric oven at 100 °C for 24 hours. The resulting powders were calcined in an electric muffle furnace for 2 hours at 800 °C (heating rate 10°C/ minute).

2.2 Characterization Techniques

The calcined alumina nanopowder synthesized by the sol-gel process was characterized using various characterization techniques to determine its composition, crystalline phase, and morphology. Figure 1(a-c) shows the sol-gel synthesis methods utilized in alumina nanopowders preparation process. X-ray diffraction (XRD) analysis was carried out to investigate the phases of the powder on a diffractometer (SHIMADZU, XRD 700) using Cu Kα radiation (λ=1.54 Å) at 40kV and 30 mA.

Figure 1 (a-c): Shows the sol-gel synthesis of alumina nanopowders after calcination

The XRD patterns were recorded over the 2θ range of 0°-100° with a step size of 0.02° and a counting time of 5s per step.

The microstructural and compositional characteristics of powder particles were investigated using High-resolution transmission electron microscopy (HRTEM; FEI Tecnai F30), which was conducted to analyze the presence of crystalline phases, composition, and morphology of the nanopowders. The well-defined dot and ring patterns confirmed the presence of crystalline phases, whereas the amorphous phase was observed as diffusing rings in the selective-area electron diffraction pattern (SAEDP).

3. Results and Discussion

3.1. XRD analysis

The XRD pattern of the synthesized alumina nanopowders confirms their crystalline nature. Figure 2 shows X-ray powder diffraction patterns of alumina nanopowders obtained by the sol-gel process. The dominant diffraction peaks correspond to the (222), (400), (440), and (511) planes of alumina. A weak peak corresponding to the (311) plane indicates the presence of a metastable γ-alumina phase. γ-alumina is thermodynamically more stable than θ-alumina but less stable than α-alumina due to its lower surface energy [20]. The observed peak broadening suggests the formation of nanocrystalline particles with small crystallite sizes. The average crystallite size calculated using the Scherrer equation ranges from 1.01 nm to 2.36 nm, indicating successful synthesis of ultra-fine alumina nanoparticles. Similar observations have been reported for sol–gel synthesized alumina nanopowders [21]. The presence of both crystalline and partially amorphous phases is typical for alumina prepared at moderate calcination temperatures.

The particle size of alumina nanopowders was calculated by Scherrer's formula, which is given by

$$t = \kappa\frac{\lambda}{\beta}cos\theta B \qquad (1)$$

Where, κ is a constant~0.9, λ is the wavelength of Cu K_α radiation = 1.54 Å, θ is the Bragg angle, and β is the full width of the diffraction peak at half maximum intensity.

The calculated average crystallite sizes ranged from 1.01 to 2.36 nm and were pure crystalline.

3.2. TEM analysis

The compositional mapping of the synthesized alumina Nano powders provides clear evidence of elemental uniformity and phase purity. Figure 3 depicts compositional mapping of nanopowder. The elemental distribution maps show the presence of only aluminium and oxygen throughout the observed region, with no detectable impurity elements. This confirms that the sol–gel synthesis route using aluminium isopropoxide as the precursor produces chemically pure alumina nanoparticles. The uniform spatial distribution of aluminium and oxygen also indicates homogeneous hydrolysis and condensation during the sol–gel process. Such chemical homogeneity is essential for biomedical applications, where the presence of trace impurities can adversely affect biocompatibility and long-term performance.

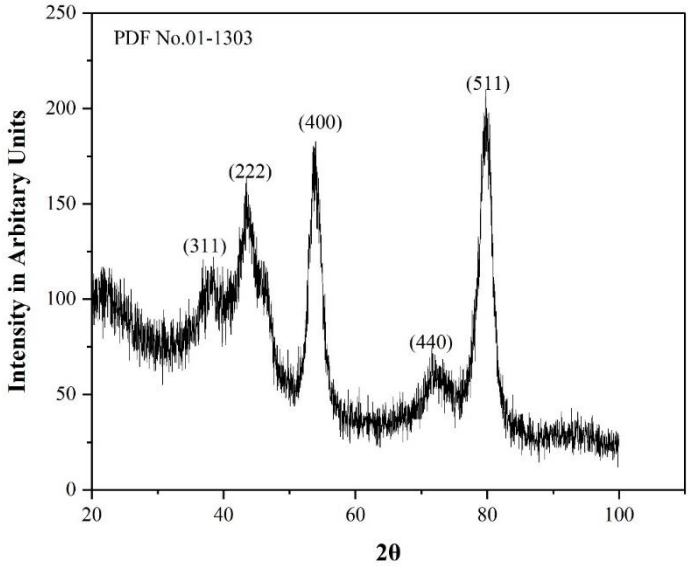

Figure 2. X-ray powder diffraction patterns of alumina nanopowders obtained by the sol-gel process.

Table 1. The particle size determination of α-alumina nanopowders

No. of peaks	2θ1	2θ2	2θB	B = $\theta_1\theta_2$(c)	Average particle size(nm)
1	54.8	37	45.90	8.9	1.01
2	70.73	62.33	66.53	4.20	2.36

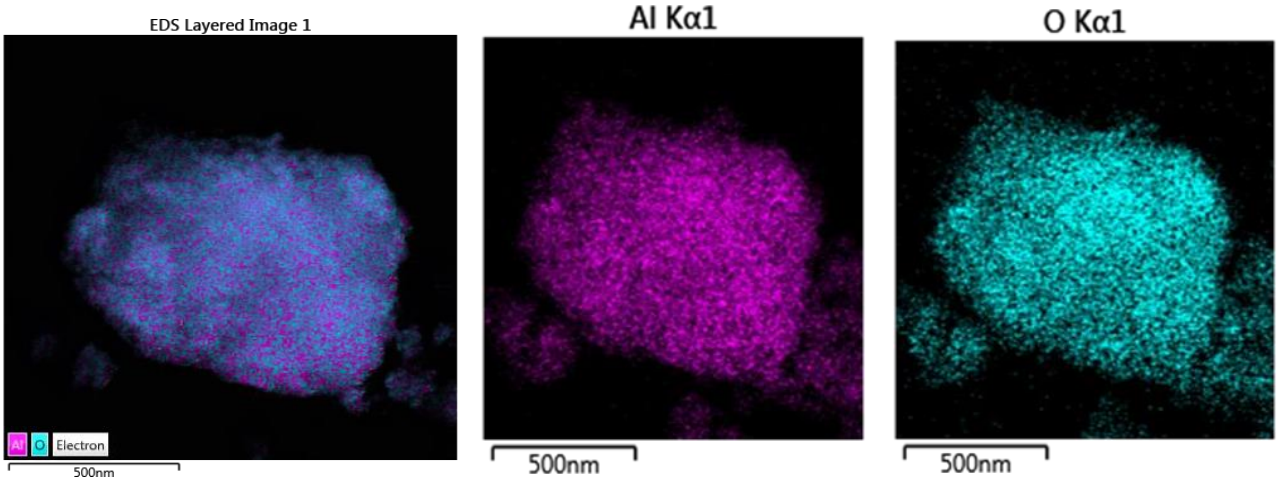

Figure 3. Compositional mapping of alumina nanopowders.

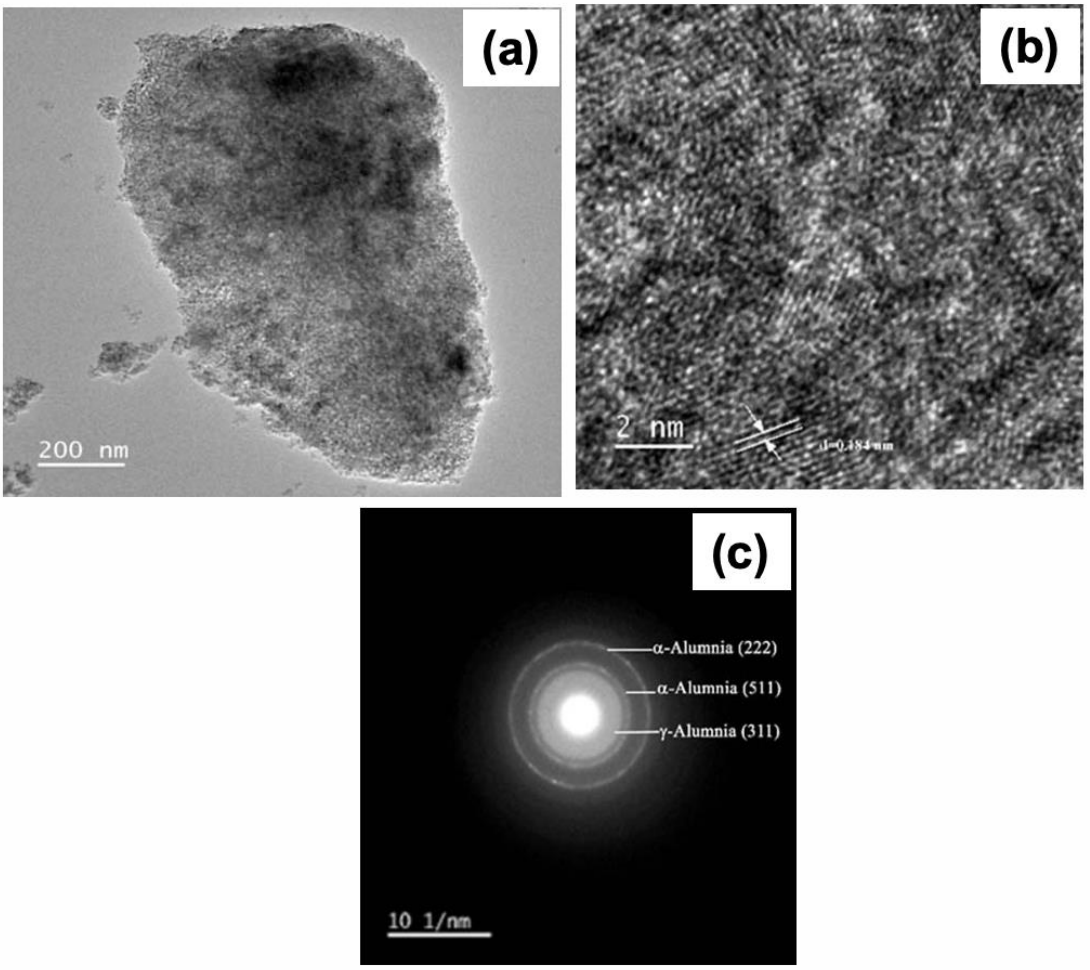

Figure 4 (a). Bright field TEM image of alumina nano powders, **(b)** HRTEM image of alumina nano powders, **(c)** SAED image of alumina nano powders.

The bright-field TEM image (Figure 4a) reveal that the alumina nanoparticles are nearly spherical in shape and uniformly distributed, with slight agglomeration. This agglomeration is commonly observed in oxide nanoparticles due to their high surface energy and strong interparticle interactions. The particle size observed in TEM images lies in the nanometre range, which is consistent with the crystallite size estimated from XRD analysis. The nanoscale particle size confirms the effectiveness of controlled pH, temperature, and calcination conditions used during synthesis. High-resolution transmission electron microscopy further confirms the crystalline nature of the synthesized alumina nanoparticles. Clear and well-defined lattice fringes are visible in the HRTEM image (Figure 4b), which indicates the good crystallinity. The measured interplanar spacing of 0.184 nm corresponds to the characteristic lattice planes of α-alumina, which agrees well with the XRD results. The presence of continuous lattice fringes across individual particles suggests that the nanoparticles are single crystalline in nature. Such crystallinity is important for achieving improved mechanical strength and wear resistance in biomedical ceramic materials.

The selected area electron diffraction patterns show distinct concentric rings as shown in figure 4c. It further confirms the polycrystalline nature of the alumina nanopowders. The diffraction rings correspond to the (222), (400), and (511) planes of α-alumina. In addition to these sharp rings, faint diffused rings are also observed, which indicate the presence of a small fraction of amorphous or metastable γ-alumina phase. The coexistence of crystalline α-alumina and amorphous γ-alumina is typical for alumina synthesized at moderate calcination temperatures around 800 °C. Similar observations have been reported in earlier studies on sol–gel derived alumina nanoparticles [22]. The presence of a minor amorphous phase may be beneficial for biomedical applications, as it can enhance surface reactivity and improve interfacial bonding when alumina is used as a coating or reinforcement material. Overall, the compositional mapping, HRTEM, and SAED analyses collectively confirm that the synthesized alumina nanopowders possess high purity, controlled nanostructure, and good crystallinity, making them suitable for advanced biomedical and structural applications.

4. Conclusion

In the present study, high-purity alumina nanoparticles were successfully synthesized using a simple and cost-effective sol–gel method with aluminium isopropoxide as the precursor. Careful control of synthesis parameters such as pH, temperature, stirring rate, and calcination conditions played a significant role in achieving phase-pure and nanosized alumina powders. The sol–gel route proved to be highly effective in producing chemically homogeneous alumina nanoparticles with controlled microstructure. X-ray diffraction analysis confirmed the formation of nanocrystalline alumina with dominant α-alumina phases. Transmission electron microscopy provided direct evidence of nanosized particle formation with near-spherical morphology and limited agglomeration. Elemental mapping confirmed the uniform distribution of aluminium and oxygen, indicating high chemical purity without detectable impurities. Overall, the results demonstrate that the sol–gel method is a reliable and scalable approach for producing alumina nanoparticles with properties suitable for advanced biomedical and engineering applications.

References

[1] D. Manyasree, P. Kiranmayi, R. Kumar, Synthesis, characterization and antibacterial activity of aluminium oxide nanoparticles, International Journal of Pharmacy and Pharmaceutical Sciences, 10(1), (2018) 32-35. https://doi.org/10.22159/ijpps.2018v10i1.20636

[2] P. Hassanpour, Y. Panahi, A. Ebrahimi-Kalan, A. Akbarzadeh, S. Davaran, A.N. Nasibova, R. Khalilov, T. Kavetskyy, (2018). Biomedical applications of aluminium oxide nanoparticles. Micro & Nano Letters, 13(9), 1227–1231. https://doi.org/10.1049/mnl.2018.5070

[3] Z. Al-Timimi, Z.J. Tammemi, Nanoparticles of Alumina (Al2O3): An Overview and Their Applications in Medical Surgery. Journal of Nanomedicine, 4(2), (2021) 1046. http://meddocsonline.org/

[4] P.Thomas, S.Barnstorf, B.Summer, G.Willmann, B.Przybilla, Immuno-Allergological Properties Of Aluminium Oxide (Al2O3) Ceramics And Nickel Sulfate In Humans. Biomaterials, 24(6), (2002). 959–966. https://doi.org/10.1016/s0142-9612(02)00432-5

[5] C. Piconi, A. Tampieri, Oxide ceramics for biomedical applications. Encyclopedia of Materials: Technical Ceramics and Glasses, 3,

(2021) 511-525. https://doi.org/10.1016/b978-0-12-803581-8.12101-0

[6] H.H. Al-Moameri, Z.M. Nahi, D.R. Rzaij, N.T. Al-Sharify, A review on the biomedical applications of alumina. Journal of Engineering and Sustainable Development, 24(5), (2020) 28-36. https://doi.org/10.31272/jeasd.24.5.5

[7] A. Udduttula, J.V. Zhang, P.G. Ren, (2019) Bioinert Ceramics for Biomedical Applications. In Biomedical Science and Technology Series (Wiley/Scrivener).

[8] D.F. Niero, O.R.K. Montedo, A.M. Bernardin, Synthesis and characterization of nano α-alumina by an inorganic sol–gel method. Materials Science and Engineering B, 280, (2022) 115690. https://doi.org/10.1016/j.mseb.2022.115690

[9] J. Konstanty, D. Tyrala, Particle Sizing and Surface Area Measurements: A comparative assessment of commercial air permeability and laser light diffraction instruments. Applied Sciences, 14(11), (2024) 4802. https://doi.org/10.3390/app14114802

[10] K. Agrawal, G. Singh, D. Puri, S. Prakash, Synthesis and characterization of hydroxyapatite powder by sol-gel method for biomedical application, Journal of Minerals & Materials Characterization & Engineering, 10(8), (2011) 727-734.

[11] A. Ulatowska-Jarza, D. Andrzejewski, K. Maruszewski, H. Podbielska, W. Strek, Advantages of sol-gel technologies for biomedical applications, In Optical and Imaging Techniques for Biomonitoring IV 3567.SIPE Digital Library, 3567, (1999) 50-58. https://doi.org/10.1117/12.339188

[12] R. Gupta, A. Kumar, Bioactive materials for biomedical applications using sol–gel technology. Biomedical Materials, 3(3), (2008) 034005. https://doi.org/10.1088/1748-6041/3/3/034005

[13] F. Ashrafi, S.A. Babanejad, A. Ghasemi, Synthesis of alumina nano powder using sol-gel method and chelate precursor, Asian Academic Research Journal of Multidisciplinary, 2, (2015) 2319-2801.

[14] M. Farahmandjou, N. Golabiyan, Synthesis and characterization of Alumina (Al2O3) nanoparticle prepared by simple by sol- gel method, International Journal of Bio-Inorganic Hybrid Nanomaterials, 5(1), (2016) 73-77.

[15] R. Rogojan, E. Andronescu, C. Ghitulica, B. S. Vasile, Synthesis and characterization of alumina nano-powder obtained by sol-gel method. UPB Buletin Scientific, Series B: Chemistry and Materials Science, 73(2), (2011) 67-76.

[16] S. Bhattacharyya, P.S. Behera, Synthesis and characterization of nano-sized α-alumina powder from kaolin by acid leaching process. Applied Clay Science, 146, (2017) 286–290. https://doi.org/10.1016/j.clay.2017.06.017

[17] W. Liu, X. Liu, P. Zhang, Z. Wang, X. Li, M. Hu, Nano-sized plate-like alumina synthesis via solution combustion. Ceramics International, 45(8), (2019) 9919–9925. https://doi.org/10.1016/j.ceramint.2019.02.034

[18] D. Mrabet, M. Vu, S. Kaliaguine, T. Do, A new route to the shape-controlled synthesis of nano-sized γ-alumina and Ag/γ-alumina for selective catalytic reduction of NO in the presence of propene. Journal of Colloid and Interface Science, 485, (2016) 144–151. https://doi.org/10.1016/j.jcis.2016.09.021

[19] R. Halder, S. Bandyopadhyay, Synthesis of fast sinterable oxygen deficient nano α−alumina & its optical properties. Journal of Alloys and Compounds, 735, (2017) 2092–2101. https://doi.org/10.1016/j.jallcom.2017.11.297

[20] K.M. Parida, A.C. Pradhan, J. Das, N. Sahu, Synthesis and characterization of nano-sized porous gamma-alumina by control precipitation method. Materials Chemistry and physics 113(1), (2009) 244-248.

[21] S. Said, S. Mikhail, M. Riad, Recent processes for the production of alumina nano-particles. Materials Science for Energy Technologies, 3, (2020) 344-363. https://doi.org/10.1016/j.mset.2020.02.001

[22] H. Yang, M. Liu, J. Ouyang., Novel synthesis and characterization of nanosized γ-Al2O3 from kaolin, Applied Clay Science, 47(3-4), (2010) 438-443. https://doi.org/10.1016/j.clay.2009.12.021

Author Contribution Statemen

July Randhari: Conceptualization, methodology, investigation, Writing original draft. Prafulla Kumar Mallik: Conceptualization, supervision, methodology, investigation, Writing original draft. Sushant Kumar Senapati: Writing Review and Editing. All the authors read and approved the final version of the manuscript.

Acknowledgement

This research was supported by the Science and Engineering Research Board (SERB) project No.EEQ/2016/000546 and MODROB project grant No.9-40/RIFD/MOD/Policy-1/2018-19, AICTE, Govt.of India.

Does this article screened for similarity?

Yes

Conflict of interest

The Author's declares that there is no conflict of interest anywhere.