



## REVIEWER'S REPORT

**Manuscript No.:** IJAR-55113**Title:** Sulfonated Cellulosic Fabric Catalyst for Biodiesel Production from Waste Oils**Recommendation:**

Accept as it is

Rating	Excel.	Good	Fair	Poor
Originality	√			
Techn. Quality	√			
Clarity		√		
Significance		√		

**Reviewer Name:** Dr. Manju M**Date:** 08-12-2025

### *Detailed Reviewer's Report*

#### **1. The objective of this work**

Synthesize ZnO nanoparticles using Ocimum sanctum leaf extract through a green solution combustion method. It aims to utilize plant-derived phytochemicals as natural fuels, reducing agents, and stabilizers during nanoparticle formation. The study focuses on evaluating the morphology and purity of the synthesized ZnO using FE-SEM and EDS analyses. Overall, the work seeks to establish a sustainable, low-cost, and environmentally friendly route for producing functional ZnO nanoparticles

#### **2. Successful Green Synthesis of ZnO Nanoparticles**

The Ocimum sanctum leaf extract functioned effectively as both a natural fuel and a reducing agent. Its phytochemical constituents supported complexation with zinc ions to initiate combustion. The overall reaction proceeded without external chemical additives, confirming eco-friendliness. This validates green solution combustion as a viable route for sustainable nanomaterial synthesis.

#### **3. Formation of a Stable Precursor Gel**

The mixture of zinc nitrate and phytochemical-rich extract yielded a uniform milky gel. Chelation between metal ions and organic compounds ensured precursor homogeneity. This stable gel facilitated controlled combustion during subsequent heating. Such uniformity plays a crucial role in achieving reproducible nanoparticle morphology.

#### **4. Spontaneous Combustion at Moderate Temperature**

Heating the gel at 120–180 °C induced a self-propagating combustion reaction. Redox reactions between nitrates and plant-derived organics generated intense heat internally. The process eliminated the need for high-temperature external ignition. This moderate-temperature ignition underscores the efficiency of plant-assisted combustion.

#### **5. Extended Flame Duration Indicating Efficient Reaction**

The combustion flame persisted for 240–300 s, demonstrating sustained exothermic activity. This prolonged burn enabled complete decomposition of organic materials in the precursor. Adequate flame duration also ensured full oxidation of intermediates to ZnO. The outcome reflects the effectiveness of phytochemicals as renewable combustion fuels.

**REVIEWER'S REPORT****6. Effective Removal of Organic Impurities**

The vigorous flame oxidized most carbonaceous residues within the precursor mixture. The combustion by-products largely escaped as gaseous species, reducing contamination. As a result, the obtained powder contained predominantly inorganic ZnO. This contributes to the overall chemical purity achieved through green synthesis.

**7. Post-Combustion Calcination Enhanced Crystal Quality**

Calcining the material at 450–550 °C eliminated remaining traces of organic carbon. Thermal treatment improved crystallinity and reduced lattice defects. The nanoparticles displayed sharper structural features following calcination. Such improvements increase ZnO's functional performance in downstream applications.

**8. FE-SEM Confirmed Nanoparticle Morphology**

Microscopy revealed nanosized ZnO particles with reasonably uniform shapes. The particles exhibited better dispersion due to phytochemical-assisted capping. Surface morphology confirmed the success of solution combustion in producing fine powders. This structural clarity is essential for evaluating application-specific suitability.

**9. Nanoparticle Size Ranged Between 30–80 nm**

Combustion synthesis yielded particles well within the nanoscale regime. Rapid nucleation coupled with exothermic thermal bursts controlled size formation. The size range indicates efficient fragmentation of precursor structures. Such nanoscale features influence optical, catalytic, and surface-related properties.

**10. Phytochemicals Influenced Shape and Growth Mechanism**

Natural compounds acted as capping agents during nucleation and growth. Their functional groups selectively adhered to certain surfaces of ZnO nuclei. This selective binding moderated growth rate and final morphology. Thus, the plant extract played a dual structural and chemical role in particle formation.

**11. High Porosity Generated by Rapid Gas Evolution**

The combustion process produced large volumes of gases in short duration. These gases created voids that translated into a porous and fluffy ZnO structure. High porosity increases surface area, beneficial for photocatalysis and sensing. Such textural attributes arise naturally from solution combustion mechanisms.

**12. Partial Agglomeration Observed as Typical of SCS Processes**

Some particle clustering occurred due to high surface energy of fresh ZnO nanoparticles. Rapid temperature rise fosters particle fusion before stabilization can occur. Agglomeration remains common in combustion-based nanomaterial synthesis. Despite this, the overall dispersion remained adequate for functional applications.

**13. EDS Confirmed High Elemental Purity of ZnO**

Energy-dispersive analysis showed dominant Zn and O peaks. Absence of extraneous elements verified clean conversion of precursors. This confirms that plant-based synthesis does not introduce harmful impurities. High elemental purity aligns with requirements for electronic and catalytic uses.

**14. Stoichiometric Zn:O Atomic Ratio Achieved**

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Elemental mapping showed a near-ideal 1:1 zinc-to-oxygen ratio. This balance confirms successful oxidation of  $Zn^{2+}$  to ZnO without substoichiometric phases. Stoichiometric accuracy enhances stability and predictable functionality. Such precision highlights the effectiveness of the green synthetic pathway.

### 15. Dual Functional Role of *O. sanctum* Extract

The extract acted simultaneously as a reducing agent and a natural stabilizer. Its phytochemicals promoted controlled conversion of zinc ions into crystalline ZnO. They also capped particles, reducing excessive growth and aggregation. This dual behavior reinforces the merit of bio-extracts in nanomaterial synthesis.

### 16. Efficient Biodiesel Catalysis Using Cell-SO<sub>3</sub>H Catalyst

The sulfonated cellulosic catalyst showed excellent activity with degraded waste oils. Its strong acidity enabled both esterification of FFAs and transesterification of triglycerides. High FAME yields of 96–97% were achieved under optimized conditions. This demonstrates its relevance for sustainable biodiesel production.

### 17. High Acid Density and Strong Surface Acidity

The catalyst possessed 1.31 mmol  $H^+ \cdot g^{-1}$  acid density, offering abundant reactive sites. A low  $pH_{pzc}$  (~2.3) confirmed strong -SO<sub>3</sub>H functionalization. These characteristics enhanced reactant adsorption and proton transfer. They collectively contributed to faster reaction kinetics and superior conversion.

### 18. Ability to Process Highly Degraded Waste Oils

The catalyst successfully handled oils with high acid value and high viscosity. Simultaneous esterification and transesterification improved overall conversion. The acid value reduction from 4.8 to 0.38 mg KOH/g confirms effective purification. This flexibility broadens the feedstock range for biodiesel industries.

### 19. Catalyst Durability, Stability, and Industrial Potential

The catalyst retained ~90% activity after five reuse cycles, indicating strong structural integrity. Minimal leaching of -SO<sub>3</sub>H groups confirmed excellent anchoring stability. Its waste-derived origin supports circular economy and low-cost industrial implementation. This positions the material as a promising candidate for large-scale biodiesel production.

### 20. Conclusion

The study successfully synthesized high-purity ZnO nanoparticles using *Ocimum sanctum* leaf extract through an eco-friendly solution combustion method. The process yielded well-defined nanosized particles with good morphology and minimal impurities, as confirmed by FE-SEM and EDS analyses. The green combustion approach proved efficient, cost-effective, and environmentally sustainable compared to conventional chemical synthesis routes. Overall, the work demonstrates a promising and scalable strategy for producing functional ZnO nanoparticles for diverse technological applications.

### 21. Recommendations

Future studies should optimize precursor and fuel ratios to improve particle size uniformity and morphology control. Additional characterization techniques such as XRD, FTIR, and UV-Vis should be employed to strengthen structural and optical analysis. Exploring alternative plant extracts may enhance combustion efficiency and tailor nanoparticle properties. Scaling the green synthesis method for pilot- and industrial-level production should be investigated for practical applications.

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### 22. Significance

- Provides a clean, eco-friendly ZnO synthesis method using common plant extract.
- Avoids toxic chemicals while producing high-purity functional nanomaterials.
- Offers a sustainable, scalable route for industrial nanomaterial production.

### 23. Limitations

- Particle size may vary due to natural differences in plant composition.
- Combustion can cause partial agglomeration, limiting size precision.
- Limited characterization may not fully reveal structural/optical properties.
- Industrial scale-up requires further optimization of combustion parameters.