Synthesis And Characterization of Nitrogen- Doped Cerium Oxide Nanoparticles Using Co- Precipitation Method

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Abstract-

- *Nanomaterials are being highly attracted by researchers to exploit their excellent properties for various applications.*
- *Cerium is the most excessive among rare earth elements, occupying at about 66 parts per million of the earth crust, and also presently a subject of great care due to its multiple applications, which include materials for catalysis, gas sensors, solid oxide fuel cells, ceramics, and oxygen storage.*
- *Numerous methods, including precipitation from solution , microwave assisted method , hydrothermal synthesis, sol-gel process , solvo thermal method have been used to prepare ceria nano particles with different morphologies.*
- *Among them, I had used co-precipitation method for the synthesis of cerium oxide nanoparticles because the method is simple, easy scale-up and cost effective.*
- *In this , I had synthesized cerium oxide nanoparticles using urea as capping and reducing agent, it was also known as carbamide and it is easily soluble in water. It is used as a nitrogen source for the synthesis of nanoparticles.*

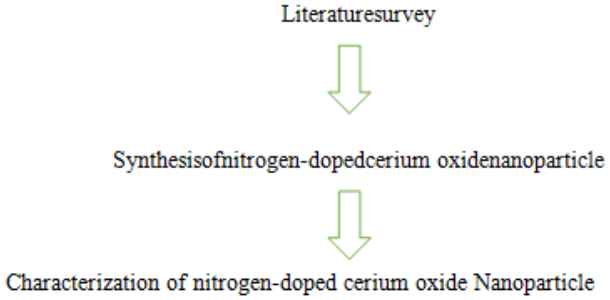
I. OBJECTIVES

- To synthesize nitrogen-doped cerium oxide nanoparticle using co precipitation method.
- To find the characterisation of the nanoparticle using UV-Visible spectroscopy, Scanning Electron Microscopy, X-ray diffraction analysis, Zeta potential, particle size analyzer and FTIR Spectrum analysis**.**

II. LITERATURE REVIEW

S.NO	Author name	Title of the work	Objective of the study	Inference
1.	Herbert ShyjiA(2019) - GCT	Synthesis and characterization of cerium oxide nanoparticle	To synthesis and characterize size, shape, chemical composition and crystal structure of the nanoparticle.	• Synthesis was carried out by Co-precipitation method. • Particle size was found to be 242nm. • In FTIR, CeO stretching band around 401cm-1 confirms the fingerprint region.
2.	M.Ramachandran, R.Subadevi, M.Sivakumar(2018)	Role of pH on synthesis and characterization of cerium oxide nanoparticles by modified co-precipitation method	Energy Materials Lab	Cerium oxide (CeO2) nano particles were synthesized using co-precipitation method.

III. METHODOLOGY



Preparation of cerium oxide nanoparticle synthesis via co-precipitation method:

- Cerium Oxide nanoparticle was synthesized using co-precipitation method.

- 1.6g(15ml) of Sodium hydroxide is adding dropwise to the precursor of 4.75g(100ml) cerium nitrate solution which was prepared using mixed solvent.
- Then it was stirred about 350rpm, where white precipitate will be formed.
- After the complete addition of NaOH, the solution was turned into pink colour.
- Again it was under stirring for about 350rpm for 3hrs.
- Finally, yellow precipitate formed, it was filtered and calcined for about 24hrs. Thus cerium oxide nanoparticle is obtained.

Preparation of nitrogen-doped cerium oxide nanoparticle synthesis via co-precipitation method

- Nitrogen-doped Cerium Oxide nanoparticle was synthesized using co- precipitation method.
- 1.6g(15ml) of Sodium hydroxide is adding dropwise to the precursor of 4.75g(100ml) cerium nitrate solution which was prepared using mixed solvent.
- Then it was stirred about 350rpm, where white precipitate will be formed.
- After the complete addition of NaOH, the solution was turned into pink colour.
- Then 0.5g of urea was added to the solution after 45minutes.
- Again it was under stirring for about 350rpm for 3hrs
- Finally, yellow precipitate formed, it was filtered and calcined for about 24hrs. Thus cerium oxide nanoparticle is obtained.

IV. MATERIALS AND INSTRUMENTS

The chemicals which were used for the synthesis of nitrogen-doped cerium oxide nanoparticles are

1. Cerium nitrate
2. Sodium hydroxide
3. Urea as nitrogen source
4. Mixed solvent (ethanol)

The instruments used for nanomaterial synthesis are

1. Magnetic stirrer
2. pH meter
3. Hot-air oven
4. Centrifuge

SYNTHESIS OF CERIUM OXIDE NANOPARTICLES

Fig1:4.75g(100ml)Cerium nitrate And 1.6g(15ml) NaOH solution

Fig2:Cerium nitrate under stirring at 350rpm

Fig 3:Addition of NAOH at the time Mixing(after 30 mins)

SYNTHESIS OF CERIUM OXIDE NANOPARTICLES

Fig4:Formation of pink Precipitate

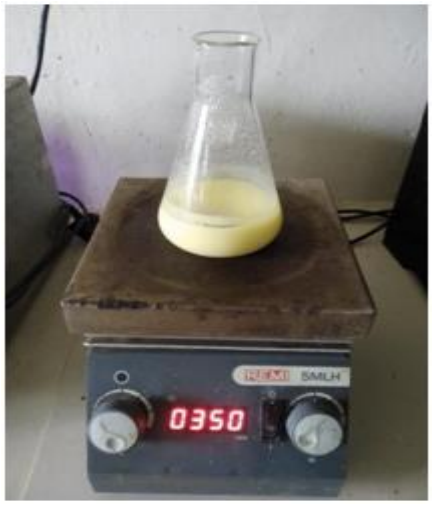


Fig5:Formation of yellow precipitate

Fig6:After centrifugation

CENTRIFUGE AND ITS WORKING PROCEDURE:

A Centrifuge works by using the principle of sedimentation.
PROCEDURE:

1. The test tube samples were inserted evenly into one of the portals.
2. If needed based on the number of samples we are testing, insert test tubes filled with water for balance.
3. Tubes opposite from one another were placed inside the machine to keep the gravity in the centre.
4. The lid was selected and select the desired settings.
5. The centrifuge was filled and wait for it to complete the cycle.
6. When the centrifuge has stopped spinning, take out the balances and samples.

Speed : 5000rpm
Time : 5minutes

Fig7:Centrifuge

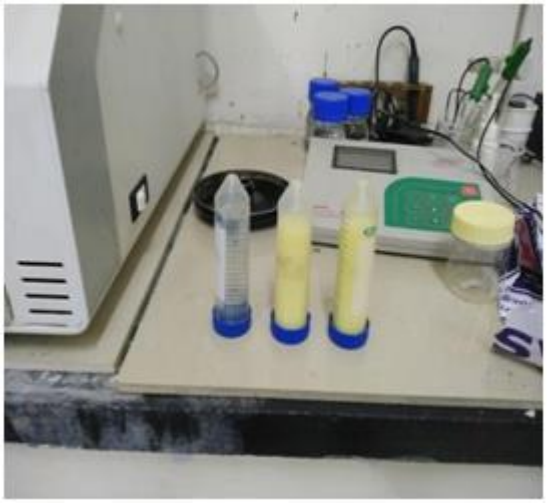


Fig8:Before centrifugation

Fig9:After centrifugation

SYNTHESIS OF CERIUM OXIDE NANOPARTICLES

Fig10:Settled nanoparticles

Fig11:Synthesized nanoparticles

Fig12:Synthesized Nanoparticles

CHARCTERIZATION OF NITROGEN-DOPED CERIUM OXIDE NANOPARTICLES

1. Particle size analyzer
2. Zeta potential
3. FTIR spectrum Analysis
4. X-Ray diffraction
5. Scanning electron microscopy
6. UV-VIS Spectroscopy Analysis

SONICATOR

Sonication uses sound waves to agitate particles in a solution,such disruptions can be used to mix solutions, speed the dissolution of a solid into a liquid, and remove dissolved gas from liquids.

V. RESULTS

The average crystalline size of the prepared nanoparticles was estimated using the Debye-Scherer equation as

$$D = 0.9\lambda/(\beta\cos\theta)$$

Where

D = crystalline size
λ = wavelength of the incident X-rays
θ = diffraction angle of the peak of the cubic phase β = full width half maximum of the peak

The average crystalline size of the cerium oxide nanoparticle will be 31nm.

VI. CONCLUSION:

- The present study is mainly focussed on the synthesis and characterization of nitrogen-doped cerium oxide nanoparticle. It was synthesized by using co- precipitation method.
- Nitrogen-doped cerium oxide nanoparticle were in the range of 286.3nm.
- Zeta potential analysis shows that the average value is in the range of -23.0mV.
- FTIR analysis shows that the spectral peaks clearly indicates the nitrogen-doped cerium oxide stretching.
- The XRD peaks indicates the formation of pure-phase CeO2 in a cubic fluorite structure.

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