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Preparation and Characterization of Rare Earth Doped Nanoparticles for Biological Application

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ABSTRACT

We report the synthesis, antibacterial and antioxidant studies of hexagonal shaped lanthanum fluoride nanoparticles doped with Praseodymium (Pr^{3+}) and Holmium(Ho^{3+}) ions prepared with co-precipitation method using deionised water as solvent. The X-ray diffraction, SEM, TEM and selected area electron diffraction SAED pattern have been used for identification of crystal structure. Cell parameters are a = b = 7.0800AU and c = 7.2380AU and confirms with the JCPDS standard card (32-0483) of pure LaF_3 crystals. Antibacterial activity was assessed for both gram positive and gram negative bacterial strains. At 500µg/disc and 1mg/disc concentration the test substance showed very good activity against Pseudomonas aeroginosa and Salmonella typhi. Antioxidant activity was measured by DPPH free radical scavenging method. Synthesised nanoparticles showed very less antioxidant activity as compared to standard Ascorbic acid.

Keywords: Antioxidant activity, X-ray diffraction, SEM, TEM, etc.

INTRODUCTION

Rare earth elements are commonly used industrially in lasers, glasses, magnets and in many other applications. Lanthanum based fluorides are important materials for their optical properties. In the form of thin films or as nanoparticles, rare earth doped lanthanum fluorides show interesting up-conversion effect from near IR to visible light. Also X-ray luminescence has been observed on doped lanthanum fluorides. Luminescent properties of these materials can be used in many applications such as biological applications and for light emitting applications such as their use in diodes [1,2].

Lanthanum fluoride nanoparticles can be prepared by variety of ways. Hydrothermal synthesis has been widely employed for preparation of these materials [3-5]. Co-precipitation method [6, 7], micellar emulsion method [8] or using ionic liquid based synthesis [9] are been used. Among various host materials, lanthanum fluoride (LaF_3) possesses advantageous properties over the oxygen-based systems, as the former results from lower vibration energy and minimization for the quenching of the excited state of RE ions [10–15].

METHODOLOGY

LaF_3: Pr^{3+}, Ho^{3+} nanocrystals were synthesized by an aqueous route using microwave assisted technique operated at low power range. The method is simple and cost effective. Water soluble $LaCl_3$+ $PrCl_3$ + $HoCl_3$ (1 unit) and NH_4Cl (3units) are mixed to obtain a solution in 1:3 molar proportion [16]. A 10 ml homogeneous mixture is prepared in deionized water in a 100ml beaker using 0.192 mol of $LaCl_3$+ $PrCl_3$ + $HoCl_3$. To this 10ml solution of 0.576 mol NH_4F is added through separate syringe to avoid contamination. The complete setup was placed inside a conventional microwave oven set at low power range (in on-off mode set at 30sec) for around 30 minutes time. The low power range setting largely helped us avoiding spill off of the solution. A white ultrafine crystalline precipitate identified as doped LaF_3 (LFPH) nanocrystals appears almost instantly having settled down at the bottom of the beaker (Figure1). The precipitate was washed several times with de-ionized water and then dried in the microwave oven for about 15 minutes. The dried sample was then stored in sealed tubes for further characterization.

RESULTS AND DISCUSSION

1.**Structural Characterization:**
Well dispersed hexagonal geometry nanocrystals of LaF_3:Pr^{3+},Ho^{3+} have been synthesized in deionized water using precipitation method.XRD analysis shows hexagonal crystal structure with a = b = 7.080(A^0), c = 7.238(A^0), $\alpha = \beta = 90^0$ and $\gamma = 120^0$. The strongest peak is found to lie on (111) plane.

Traces of broad, hexagonal and spherical like nanocrystals have been observed. The average crystallite size is found to be 15nm by TEM measurements. TEM results are found to be in close agreement with the XRD studies with Debye Scherrer particle size of 11.17nm. The selected area electron diffraction (SAED) pattern shows four diffraction rings corresponding to the (110), (111), (300) and (221) reflections, which is in agreement with the hexagonal LaF_3 structure. The c/a ratio is found to approach unity in synthesized nanocrystals. SEM pattern shows dispersed particles with traces of aggregates. EDAX spectra confirmed the elemental components in the nanocrystals with certain trace elements. The FTIR spectrum has been used for identification of fundamental vibrational groups present in the material.

FIG 1. Synthesis of LFPH

2. Antimicrobial activity of the Lanthnum based nanoparticles LaF_3:Pr^{3+},Ho^{3+}

Preparation of sample: Lanthanum based nanoparticles were dissolved in water and used for antimicrobial activity. For antibacterial assay both

gram positive and gram negative bacterial strains were used. A total of five standard human pathogenic bacteria were procured from Government hospital. The organisms were maintained on nutrient agar slants and stored at 4 ° C with periodic sub-culturing.

Antibacterial activity was tested in triplicate using the standard paper disk diffusion method [17,18]. Stock solution of synthesized nanoparticles was prepared by dissolving in 50 mg/ml of methanol. The test concentrations of lanthanum nanoparticles 50µg, 100µg, 500µg and 1 mg per disk were applied to sterile paper disks (6 mm in diameter). The discs were dried before they were placed onto agar plates that had been seeded with reference bacterial strains. The diameters of the inhibition zones (diameter of inhibition zone minus diameter of disk) were measured in millimeters after incubation at 30^0C for 24 hours. Solvent (sterile distilled water) control disks without lanthanum nanoparticles prepared in the same manner were never observed to inhibit bacterial growth.

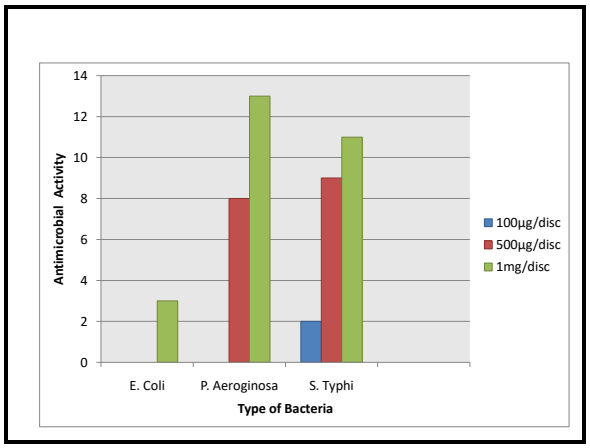

Fig.2:Antimicrobial activity of LaF_3:Pr^{3+},Ho^{3+}

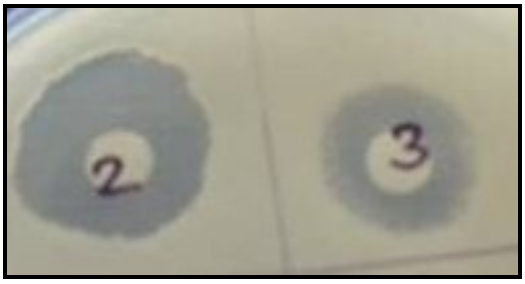

Fig.3:Antimicrobial activity of LaF_3:Pr^{3+},Ho^{3+} nanoparticles against *Pseudomonas aeroginosa* at 1mg/disc (2) and 500µg/disc(3) concentrations.

3. Antioxidant activity of the Lanthnum based LaF_3:Pr^{3+},Ho^{3+}nanoparticles

The antioxidant activity of lanthanum nanoparticles was checked on the basis of scavenging effect of the stable 2,2-diphenyl-1-picrylhydrazyl (DPPH) free radical activity[19].The standard antioxidant ascorbic acid was taken for comparison. Here standard ascorbic acid solution (1ml) and different concentrations (50, 100, 200, 400, 500 µg/ml in methanol) of 1ml of lanthanum nanoparticles were mixed with 3 ml of 0.4M DPPH solution. These mixtures were kept in dark for 30 minutes and after that the absorbance was measured at 517 nm using UV-Visible Spectrophotometer. In this assay ascorbic acid was used as a positive control. Lower absorbance of the reaction mixture indicated higher free radical-scavengingactivity.

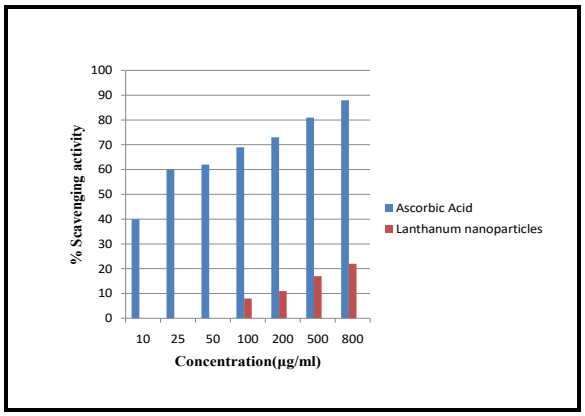

Fig.4:Antioxidant activity of LaF_3:Pr^{3+},Ho^{3+}

CONCLUSION

New rare earth doped lanthanum fluoride nanoparticles (LFPH) with enhanced properties have been grown. Attempt has been made to study biological application of the synthesized material.The results of antimicrobial activity of synthesized nanoparticles LaF_3: Pr^{3+}, Ho^{3+} were promising. At 500µg/disc and 1mg/disc concentration the test substance showed very good activity against *Pseudomonas aeroginosa* and *Salmonella typhi*. The slight inhibition of *E. coli* bacterial strain was observed at very high concentration but this activity was negligible. In Antioxidant activity test of LaF_3: Pr^{3+}, Ho^{3+} nanopartoicles both ascorbic acid and

$LaF_3:Pr^{3+},Ho^{3+}$ nanoparticles showed dose dependent activity. Lanthanum nanoparticles showed less significant amount of DPPH free radical scavenging effect compared to ascorbic acid. These biological analysis indicates that $LaF_3:Pr^{3+}$, Ho^{3+}nanoparticles are potential candidates for biomedical applications like biolabeling or biotagging.

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Conflicts of interest: The authors stated that no conflicts of interest.

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