

RESEARCH PAPER

## Efficient Synthesis of CeVO<sub>4</sub> Nanoparticles Using Combustion Route and Their Antibacterial Activity

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### ARTICLE INFO

#### Article History:

Received 29 February 2018

Accepted 18 March 2018

Published 01 April 2018

#### Keywords:

Antibacterial activity

CeVO<sub>4</sub>

Nanotechnology

### ABSTRACT

CeVO<sub>4</sub> (Cerium orthovanadate) nanoparticles were synthesized by urea-assisted simple and efficient combustion method. Phase formations of synthesized nanoparticles were characterized by thermogravimetric-differential thermal analyzer (TG-DTA). X-ray diffraction (XRD) pattern revealed the crystal planes and size of synthesized CeVO<sub>4</sub> nanoparticles. The average size, morphological shape and the crystalline nature of the nanoparticles were determined by scanning electron microscopy (SEM), transmission electron microscopic with selected area electron diffraction (TEM-SAED). Energy-dispersive X-ray spectroscopy (EDX) confirmed the presence of elemental composition and purity of the synthesized nanoparticles. Fourier transform Infrared spectroscopy (FT-IR) confirmed the possible stretching frequency on the surface of CeVO<sub>4</sub> nanoparticles. Surface area and porosity studies of synthesized nanoparticles were analyzed by Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) curve. Moreover, CeVO<sub>4</sub> nanoparticles evinced excellent antibacterial activity against Escherichia coli, Klebsiella pneumoniae, Pseudomonas aeruginosa, Proteus vulgaris, Salmonella typhi, Staphylococcus aureus, Streptococcus pyogenus, Bacillus subtilis, Streptococcus pneumoniae and Staphylococcus epidermidis. The studies describing the synthesis of CeVO<sub>4</sub> nanoparticles by efficient combustion method followed by the investigation of antibacterial activities may be useful for research opening a new arena in the field of nanobiotechnology.

#### How to cite this article

Kamble D. R, Bangale S. V, Ghotekar S. K, Bamane S. R. Efficient Synthesis of CeVO<sub>4</sub> Nanoparticles Using Combustion Route and Their Antibacterial Activity. J Nanostruct, 2018; 8(2):144-151. DOI: 10.22052/JNS.2018.02.004

### INTRODUCTION

Nowadays, the synthesis of nanosized rare earth orthovanadates (RVO<sub>4</sub>)/metal oxides has attracted much curiosity due to their miraculous and extensive applications in the field of catalysis,

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bioengineering and material science [1-5]. Amongst them, CeVO<sub>4</sub> nanoparticles has wide potential applications in various fields such as gas sensor [6-8], electrochromic material [9], photocatalyst [1,10], lubricating additives [11], luminescence

[12] and batteries fields [13]. Considering CeVO<sub>4</sub> nanoparticles have excellent physical and chemical properties, it was particularly important to seek a facile, efficient and energy saving preparation method. The scrutiny of the literature revealed some notable methods were used for synthesis of CeVO<sub>4</sub>. For example, sol-gel method [14], microwave assisted synthesis [8], ultrasound method [15], hydrothermal method [16], solid-state reaction method [17] and sonochemical method [18] have been reported. Therewith, metal oxide nanoparticles can be used as an anti-biotic, antioxidant, pesticide formulation, antimicrobial and antifungal agent when incorporated in textiles, coatings and plastics [19-22].

In this work, we rapidly synthesized CeVO<sub>4</sub> nanoparticles by simple and efficient combustion method using urea as a fuel. These synthesized nanoparticles were evaluated for antimicrobial activities by employing against some selected human pathogens. It was found that efficiently synthesized CeVO<sub>4</sub> nanoparticles evinced good biomedical application in nanobiotechnology.

## MATERIALS AND METHODS

### *Synthesis of CeVO<sub>4</sub> nanoparticles*

In this study, CeVO<sub>4</sub> powder was successfully synthesized by solution combustion route using the starting reagents as Ce(NO<sub>3</sub>)<sub>3</sub>.6H<sub>2</sub>O, V(NO<sub>3</sub>)<sub>3</sub>.6H<sub>2</sub>O and Urea as a fuel. Urea possesses a high heat of combustion. It is an organic fuel providing a platform for redox reactions during the course of combustion. Initially cerium nitrate, vanadium nitrate and urea are taken in the 1:1:4 stoichiometric amount and homogenous paste was made. The paste formed was evaporated on hot plate at about 70 to 80 °C to get thick gel. This kept on a hot plate for auto combustion and heated at 170 to 180 °C. To obtain nanocrystalline CeVO<sub>4</sub> powder, this was sintered at 800 °C for 4 hrs. A fine dark brown colored material was obtained and this was carefully collected and packed for further characterization purposes.

### *Characterization techniques*

The synthesized CeVO<sub>4</sub> nanoparticles were characterized using thermogravimetric-Differential thermal analyzer (TG-DTA, PERKIN ELMER, USA, Diamond TG/DTA). The crystallinity and crystal phases were characterized by X-ray diffraction (XRD, Bruker, D8-Advanced Diffractometer) pattern measured with Cu-Kα

Radiation ( $\lambda = 1.5406 \text{ \AA}$ ) in the range of 20–90°. The morphology and composition of the synthesized CeVO<sub>4</sub> nanoparticles were examined by scanning electron microscopy (SEM, JEOL, JSM-6360), SEM coupled energy-dispersive X-rays spectroscopy (EDX, Bruker, XFlash 6130). Find the exact morphological structures and size of the CeVO<sub>4</sub> nanoparticles using transmission electron microscopic (TEM) with selected area electron diffraction (SAED) analysis is done by using a PHILIPS, CM200 with an accelerating voltage of 200 kV. The Fourier transform Infrared (FTIR) spectrum was recorded by JASCO 4100 in the range of 4000–400 cm<sup>-1</sup>. The specific surface area, pore size and volume were characterized by Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) analysis method at 77.40 deg. K (NOVA-100 Ver. 3.70).

### *Antibacterial activity of synthesized CeVO<sub>4</sub> nanoparticles*

The Minimum inhibition concentrations (MIC) of synthesized CeVO<sub>4</sub> nanoparticles were carried out by broth micro dilution protocol as described by Rattan [23]. DMSO (Dimethyl sulphoxide) was used as diluents to get desired concentration of drugs to test upon standard bacterial strains. Serial dilutions were prepared in primary and secondary screening. The control tube containing no antibiotic was immediately subculture by spreading evenly over a plate of medium suitable for the growth of the test bacterial pathogens and incubated overnight at 37 °C. The MIC of the control bacterial strain was measured to check the accuracy of the drug concentrations. The lowest concentration inhibiting growth of the bacterial pathogen was recorded as the MIC. The amount of growth from the control tube before incubation was compared. Subcultures might evince similar number of colonies indicating bacteriostatic, a reduced number of colonies indicating a partial or slow bactericidal activity and no growth if the whole inoculum has been killed. The test must include a second set of the same dilutions inoculated with a bacterial pathogen. A synthesized CeVO<sub>4</sub> nanoparticle was diluted obtaining 2000 µg/ml concentration, as a stock solution. In primary screening, 500, 250 and 125 µg/ml concentrations of the synthesized CeVO<sub>4</sub> nanoparticles were taken. The synthesized CeVO<sub>4</sub> nanoparticles found in this primary screening was further tested in a second set of dilution against all microorganisms. The CeVO<sub>4</sub> nanoparticles found

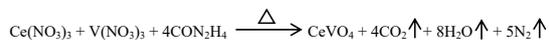
active in primary screening was similarly diluted to obtain 100, 50, 25, 12.5, 6.250, 3.125 and 1.5625 µg/ml concentrations. The highest dilution showing at least 99 % inhibition is taken as MIC.

## RESULTS AND DISCUSSION

### TG-DTA

TG-DTA curve recorded for thermal decomposition of CeVO<sub>4</sub> is shown in Fig. 1. The curve indicates that the slight weight loss in CeVO<sub>4</sub> powder due to little loss of moisture, nitrogen and carbon dioxide gas. The DTA curve of CeVO<sub>4</sub> recorded in static air and is shown in Fig. 1. The curve exhibited that CeVO<sub>4</sub> did not decompose,

but weight loss was due to dehydrogenation, decarboxylation and denitration and yield final product at 800°C. This weight change was in the synthesized powder was almost remain stable from the beginning as shown in reaction.



### Structural & crystallographic analysis

The synthesized CeVO<sub>4</sub> formation was confirmed by the characteristic peaks observed in the XRD patterns, as shown in Fig. 2. Such a powder XRD was carried out using monochromatic CuKα1 radiation (wavelength 1.5406Å), operating at a voltage of 40 KV and a current of 40 mA, in

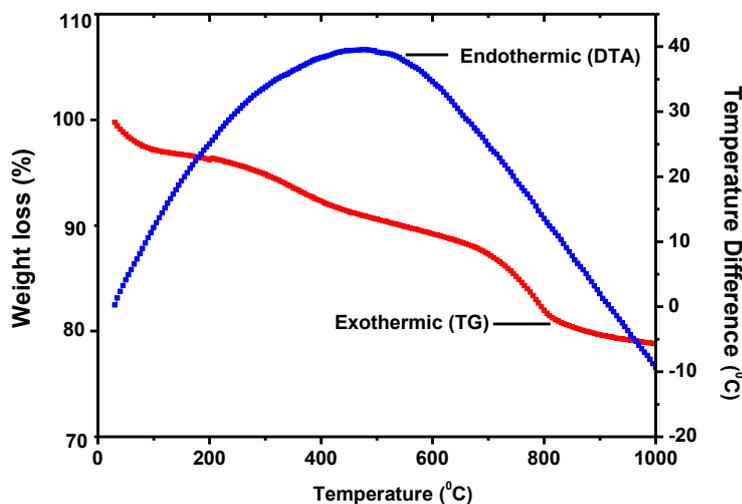


Fig. 1. TG-DTA curve of synthesized CeVO<sub>4</sub> nanoparticles.

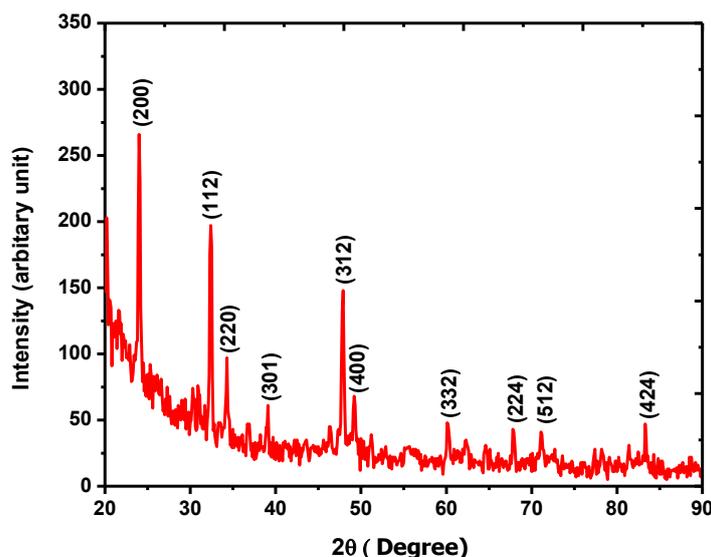


Fig. 2. XRD pattern of synthesized CeVO<sub>4</sub> nanoparticles sintered at 800 °C for 4 hrs.

the angular range 2θ of 20-90 deg. XRD analysis showed a series of diffraction peaks at 24.0°, 32.40°, 34.30°, 39.10°, 47.90°, 49.20°, 60.10°, 67.80°, 71.10° and 83.30°, corresponding to (200), (112), (220), (301), (312), (400), (332), (224), (512) and (424) crystal planes of tetragonal CeVO<sub>4</sub> nanostructures (JCPDS No. 12-0757). The sharp XRD peaks exposed that synthesized CeVO<sub>4</sub> nanoparticles are good crystalline in nature.

#### SEM microphotographs

Morphologies and sizes of synthesized CeVO<sub>4</sub> nanoparticles were examined by SEM techniques. It can be seen that the average crystal grain size of the CeVO<sub>4</sub> nanoparticles was mainly 51-93 nm except slightly agglomeration (Fig. 3). This result exceed to the literature result which tetragonal

structure of CeVO<sub>4</sub> nanoparticles was prepared by ultrasound method [13].

#### TEM images

TEM images and SAED pattern of the synthesized CeVO<sub>4</sub> nanoparticles were characterized. Fig. 4 indicates the hexagonal shape of CeVO<sub>4</sub> nanoparticles with size 45-95 nm. The SAED pattern shows a number of bright spots, which confirmed that, the as-synthesized CeVO<sub>4</sub> nanoparticles are single phase nature of the material.

#### EDX studies

The composition of synthesized CeVO<sub>4</sub> nanoparticles has been analyzed by investigating the energy-dispersive X-ray spectroscopy (EDX), as shown in Fig.5. This was carried out to understand the composition of the cerium,

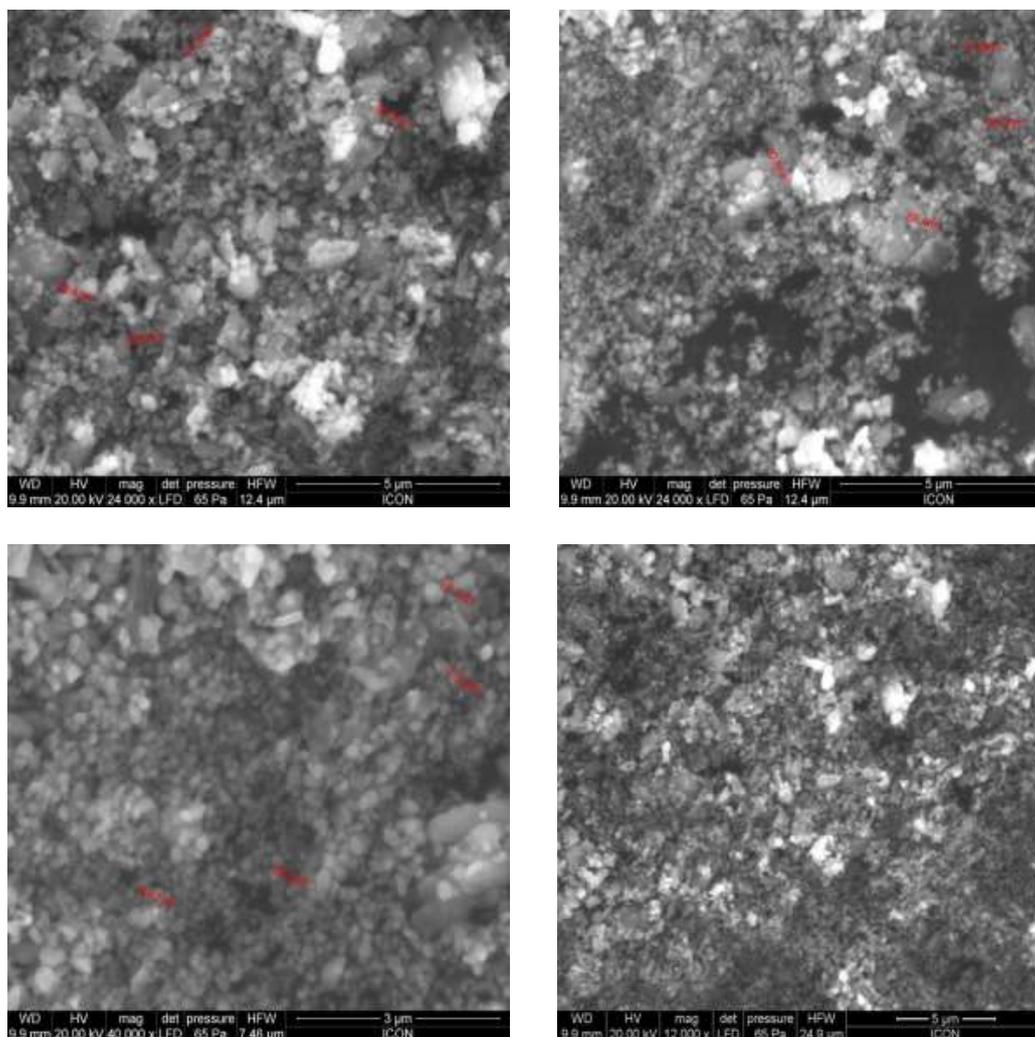


Fig. 3. SEM images of synthesized CeVO<sub>4</sub> nanoparticles sintered at 800 °C for 4 hrs.

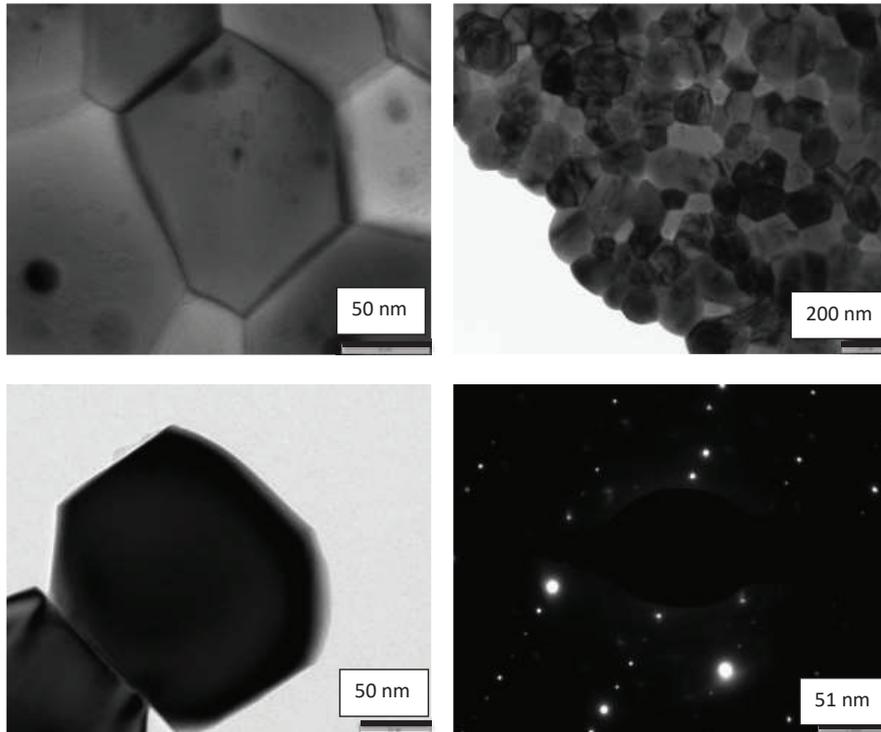


Fig. 4. TEM images with SAED pattern of synthesized CeVO<sub>4</sub> nanoparticles.

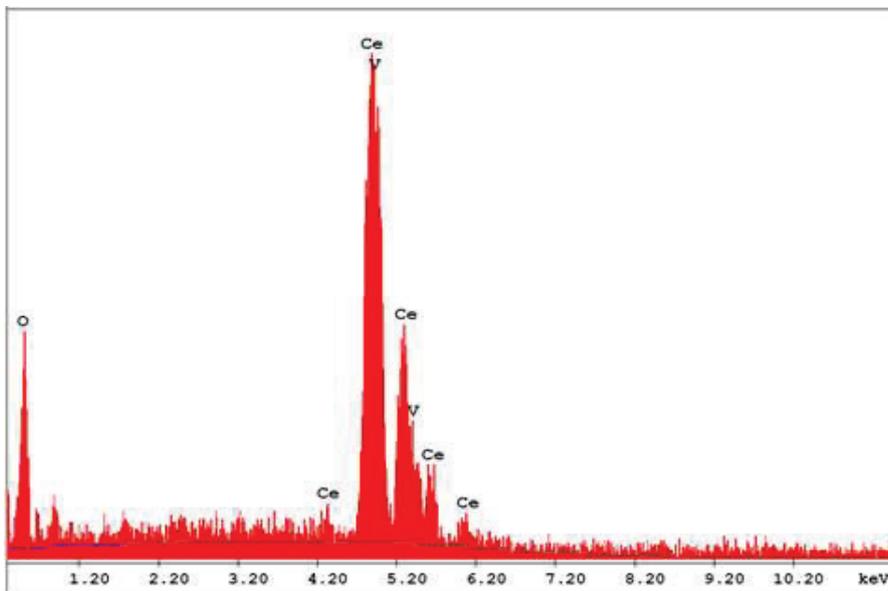


Fig. 5. EDX pattern of synthesized CeVO<sub>4</sub> nanoparticles sintered at 800 °C for 4 hrs.

vanadium and oxygen in the synthesized material. There was no unidentified observed in EDX. This quantitative data confirms the purity, composition and formation of CeVO<sub>4</sub> nanoparticles.

#### *Vibrational properties*

Fig. 6 represents the FTIR spectrum of CeVO<sub>4</sub> nanoparticles synthesized from combustion method. The IR band at 810 cm<sup>-1</sup> is attributed to the Re-O-V vibrations of ReVO<sub>4</sub>. Residual water

and -OH group are detected around 3449 cm<sup>-1</sup>, corresponding to the O-H stretching frequency due to the bending vibration of associated water. It suggested that the CeVO<sub>4</sub> nanoparticle was successfully fabricated by facile and efficient combustion method.

*Surface area and porosity studies*

The specific surface area of the CeVO<sub>4</sub> nanoparticles calculated using multipoint BET

equation is 16.52 m<sup>2</sup>/g and according that the CeVO<sub>4</sub> nanoparticles have solid and smooth surface. Furthermore, the corresponding BJH analyses indicate that the cumulative pore volume was 0.005 cm<sup>3</sup>/g and the pore size of which estimated from the peak position was 3.32 nm possesses a relatively narrow pore size distribution. Most of the pores ranged from 1.36 nm to 34.79 nm, as shown in the inset of Fig. 7. Therefore, these particles are polycrystalline in

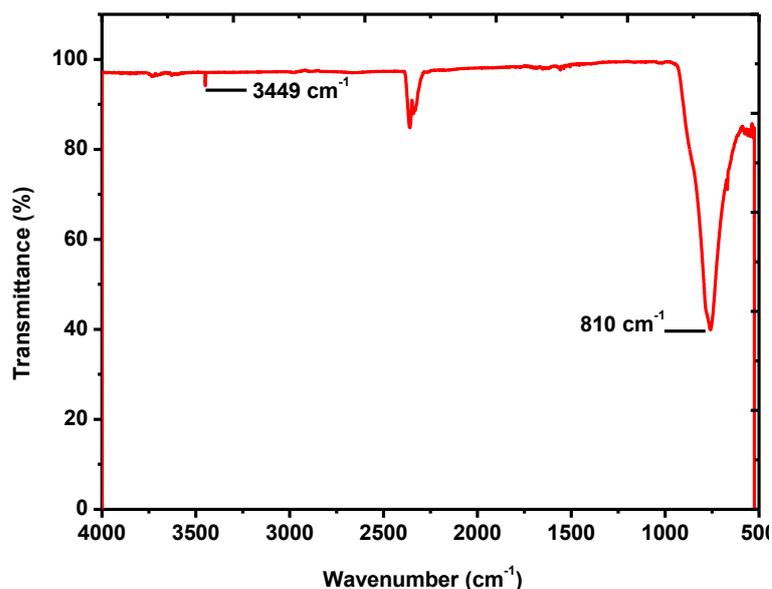


Fig. 6. FTIR spectra of synthesized CeVO<sub>4</sub> nanoparticles.

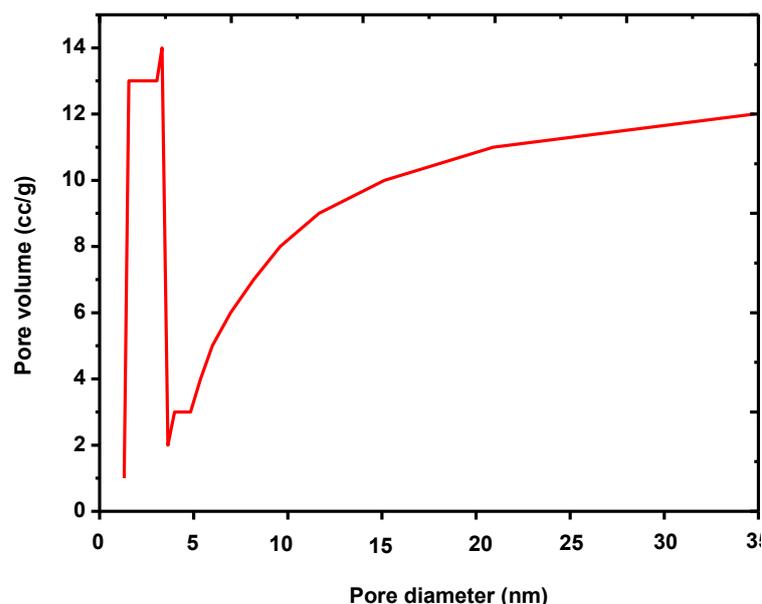


Fig. 7. BJH pore size distribution curves of synthesized CeVO<sub>4</sub> nanoparticles.

Table 1. Minimum inhibition concentration of CeVO<sub>4</sub> against selected bacterial pathogens.

Test pathogens	MIC (µg/ ml) of CeVO <sub>4</sub>	MIC (µg/ ml) of Reference drug
<i>E. coli</i> (MTCC-443)	50	100
<i>K. pneumoniae</i> (MTCC-109)	125	100
<i>P. aeruginosa</i> (MTCC-1688)	62.5	100
<i>P. vulgaris</i> (MTCC-8427)	100	100
<i>S. typhi</i> (MTCC-98)	100	100
<i>S. aureus</i> (MTCC-96)	125	250
<i>S. pyogenes</i> (MTCC-442)	200	100
<i>B. subtilis</i> (MTCC-441)	62.5	250
<i>S. pneumoniae</i> (MTCC-6305)	100	250
<i>S. epidermidis</i> (MTCC-12228)	100	100

nature.

#### Antimicrobial activity of CeVO<sub>4</sub> nanoparticles

The results of antibacterial activity of the synthesized CeVO<sub>4</sub> nanoparticles are presented in Table 1. Moderate to good antibacterial activity is observed against some bacteria. Synthesized CeVO<sub>4</sub> nanoparticles exhibited potent and good antibacterial activity against *E. coli*, *P. aeruginosa*, *S. aureus*, *B. subtilis*, *S. pneumoniae* and moderate activity against other bacteria with ampicillin was used as the reference drug.

#### CONCLUSION

The convenient utilization of urea as a fuel for the efficient synthesis CeVO<sub>4</sub> nanoparticles through a combustion method to obtain significantly biologically active material. The synthesized CeVO<sub>4</sub> nanoparticles were hexagonal in shape as observed in TEM analysis. The synthesized CeVO<sub>4</sub> nanoparticles have shown potential upshot against selected pathogenic bacteria. This encouraging result provides useful information for designing a much better medicinal compound using a synthesis of CeVO<sub>4</sub> nanoparticles with minimal side effects and play significant role in biomedicine.

#### ACKNOWLEDGEMENT

Authors are thankful to SAIF IIT Powai, Shivaji University Kolhapur and Microcare Laboratory Gujrat for providing the technical, instrumental and biological activities supports.

#### CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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