*Nanochem Res 3(2): 189-196, Summer and Autumn 2018*

RESEARCH PAPER

# Synthesis of $CeVO_4$ nanoparticles using sol-gel auto combustion method and their antifungal activity

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**ARTICLE INFO**

***Article History:***
*Received 9 February 2018*
*Accepted 16 May 2018*
*Published 1 August 2018*

***Keywords:***
*Antifungal Activity*
*$CeVO_4$ Nanoparticles*
*Nanotechnology*
*Photoluminescence*

**ABSTRACT**

Cerium orthovanadate nanoparticles ($CeVO_4$ NPs) were fabricated using urea-assisted facile sol-gel auto combustion method. X-ray diffraction (XRD) pattern revealed the crystal planes and size of the synthesized $CeVO_4$ NPs. The morphological shape and the crystalline nature of the NPs were examined by field emission scanning electron microscopy (FESEM). Energy-dispersive X-ray spectroscopy (EDX) affirmed the presence of elemental composition and purity of the fabricated NPs. Fourier transform infrared spectroscopy (FT-IR) confirmed the conceivable stretching frequency on the surface of $CeVO_4$ NPs. UV–visible diffuse reflectance spectroscopy (DRS) absorption spectrum indicated that the band gap is about 3.2 eV and the synthesized tetragonal $CeVO_4$ NPs exhibited a broad photoluminescence in the UV–visible region. Besides, these $CeVO_4$ NPs evinced antifungal activity against *Candida albicans*, *Aspergillus niger*, *Aspergillus clavatus*, *Trichophyton rubrum*, *Trichophyton mentographytes*, *Epidermophyton floccosum* and *Microsporum gypseum*. The studies describing the synthesis of $CeVO_4$ NPs by simple sol-gel auto combustion method followed by the investigation of antifungal activities may be useful for the research activities to open a new horizon in the field of nanotechnology.

**How to cite this article**
*Ghotekar S, Pansambal S, Pagar K, Pardeshim O, Oza R. Synthesis of $CeVO_4$ nanoparticles using sol-gel auto combustion method and their antifungal activity. Nanochem Res, 2018; 3(2):189-196. DOI: 10.22036/ncr.2018.02.008*

## INTRODUCTION

Nanomaterial of rare earth orthovanadates ($RVO_4$) have been broadly considered in recent years due to their imperative properties. Amongst them, $CeVO_4$ NPs have a tetragonal zircon-type structure belonging to the $I4_1/amd$ space group. Nowadays, $CeVO_4$ nanoparticles are utilized for potential applications in various fields such as photoelectric [1], gas sensor [2], luminescence [3] electrochromic material [4, 5], electrochemical sensor [6], photocatalyst [7], antibacterial agent [8], lubricating additives [9], and batteries fields [10]. Considering the magnificent chemical and physical properties of $CeVO_4$ nanoparticles, it was critical to seek a facile, rapid, efficient and energy saving preparation method. Heretofore, some notable methods were used for the synthesis of $CeVO_4$ NPs. Accordingly, a variety of techniques, such as electrospinning [1], sol-gel method [11], microwave assisted synthesis [12], precipitation method [13], hydrothermal method [2, 14], solid-state reaction method [15], sonochemical method [16], and ultrasound method

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[17] have been reported. Therewith, mixed metal oxide NPs can be used as an anti-biotic, antioxidant, pesticide formulation, antimicrobial and antifungal agent [18-24].

Sol–gel auto combustion is a swift and economically affordable synthetic route for the fabrication of nanomaterial and has been widely used for the fabrication of a variety of metal and metal oxide NPs, forming nano-sized, homogeneous, and highly reactive powders through mixing different elements at the atomic level.

In this work, we rapidly synthesized $CeVO_4$ NPs by simple and efficient sol-gel auto combustion method using urea as a fuel agent. These synthesized nanoparticles were assessed for antifungal activities by employing against some selected fungal strains. It was found that efficiently synthesized $CeVO_4$ NPs manifested good biomedical application in the field of nanomedicine.

## EXPERIMENTAL SECTION

*Synthesis of $CeVO_4$ NPs*

All analytical purity grade reagents were used as received without any purification. In this investigation, $CeVO_4$ powder was effectively synthesized by sol-gel auto combustion method using the precursor as $Ce(NO_3)_3.6H_2O$, $V(NO_3)_3.6H_2O$ and urea as a fuel agent. Urea is an organic fuel providing a platform for redox reactions during the course of auto combustion because it possesses a high heat of combustion. Initially vanadium nitrate, cerium nitrate and urea are taken in the 1:1:4 stoichiometric ratio and homogenous paste was made. The paste formed was evaporated on hot plate at about 70 to 80 $^0$C to get thick gel. This kept on a hot plate for auto combustion and heated at 180 to 190 $^0$C. To obtain nanocrystalline $CeVO_4$ powder, this was sintered at 800 $^0$C for 4 h (Fig. 1). A fine dark brown colored powder was obtained and this was carefully collected for further characterization purposes.

*Characterization techniques*

The crystal phases and crystallinity of $CeVO_4$ NPs were characterized by X-ray diffraction (XRD, Brukar, D8-Advanced Diffractometer) pattern measured with Cu-Kα Radiation (λ= 1.5406 Å) in the range of 10–80$^o$. The morphology and composition of the synthesized $CeVO_4$ NPs were examined by field emission scanning electron microscopy (JEOL JSM-6701), and FESEM coupled energy-dispersive X-ray spectroscopy (EDX, Bruker, XFlash 6I30). UV-vis DRS absorption spectra of $CeVO_4$ NPs were performed using JASCO spectrophotometer V-770. Spectral analysis of photoluminescence was measured on FP-8200 spectrofluorometer. Functional group and structural properties was observed by Fourier transform Infrared (FT-IR) spectrum (FT-IR 4600).

*In-vitro antifungal activity of the synthesized $CeVO_4$ NPs*

Antifungal activity of the synthesized $CeVO_4$ NPs was examined against fungal strains (*Candida*

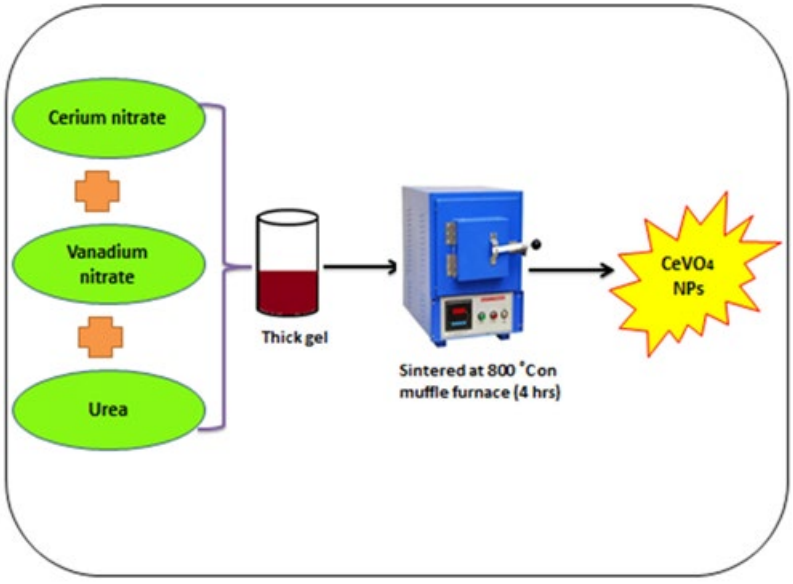


Fig. 1. Schematic diagram for the synthesis of $CeVO_4$ NPs

*albicans* MTCC 227, *Aspergillus niger* MTCC 282, *Aspergillus clavatus* MTCC 1323, *Trichophyton rubrum* MTCC 296, *Trichophyton mentographytes* MTCC 8476, *Epidermophyton floccosum* MTCC 7880 and *Microsporum gypseum* MTCC 2819) using the agar dilution protocol [25]. Determine the minimum inhibitory concentration (MIC), a stock solution of the synthesized $CeVO_4$ NPs was prepared in dimethyl sulfoxide and then incorporated in a specified quantity of molten sterile agar, i.e., dextrose agar for antifungal screening. The inoculums was prepared by taking a stock culture to about 100 mL of nutrient broth, in 250 mL clean and sterilized conical flasks. The flasks were incubated at 27 °C for 24 h before use. The plates were kept in aseptic condition at room temperature (at least 2 h) to allow diffusion of the solution properly into potato-dextrose-agar medium. Then, the plates were incubated at 25 °C for 48 h. The highest dilution showing at least 99 % inhibition zone is taken as MIC. Greseofulvin was used as a reference drug for antifungal activity. The experiments were performed in triplicate in order to minimize the errors of whole method.

## RESULTS AND DISCUSSION

*Structural and crystallographic analyses*

The synthesized $CeVO_4$ NPs formation was affirmed by the characteristic peaks observed in the XRD profile, as shown in Fig. 2. Powder XRD of fabricated $CeVO_4$ NPs was carried out using monochromatic CuKα1 radiation (wavelength 1.5406Å) in the angular range 2θ of 10-80 deg. XRD profile exhibited a series of diffraction peaks at 18.16°, 24.02°, 30.26°, 32.40°, 34.20°, 36.78°, 39.02°, 43.50°, 46.32°, 47.82°, 49.22°, 55.52°, 60.16°, 62.38°, 67.82° and 71.10°, corresponding to (101), (200), (211), (112), (220), (202), (301), (103), (321), (312), (400), (420), (332), (204), (224) and (512) crystal planes of tetragonal $CeVO_4$ nanostructures (JCPDS No. 12-0757). The sharp XRD peaks indicated that the synthesized $CeVO_4$ nanoparticles are good crystalline in nature. The size of the $CeVO_4$ NPs formed were calculated using Debye–Scherrer's equation which was around 45-95 nm.

*FESEM microphotographs*

Morphology of the synthesized $CeVO_4$ NPs was examined by the FESEM technique. It can be seen that the average crystal grain size of the $CeVO_4$ NPs was mainly 50-95 nm having quasi-hexagonal shape except slightly agglomeration (Figs. 3). This result exceeded to the literature result in which tetragonal structure of $CeVO_4$ NPs was prepared by precipitation method [13].

*EDX studies*

The elemental composition of the synthesized $CeVO_4$ NPs was analyzed by investigating the EDX, as shown in Fig. 4. This was carried out

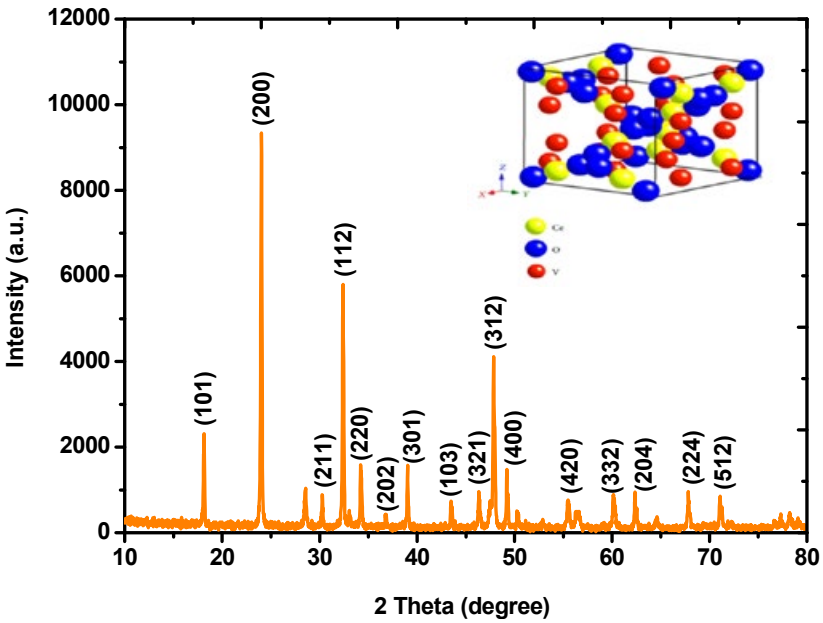


Fig. 2. X-Ray diffraction profile of the synthesized $CeVO_4$ NPs at room temperature

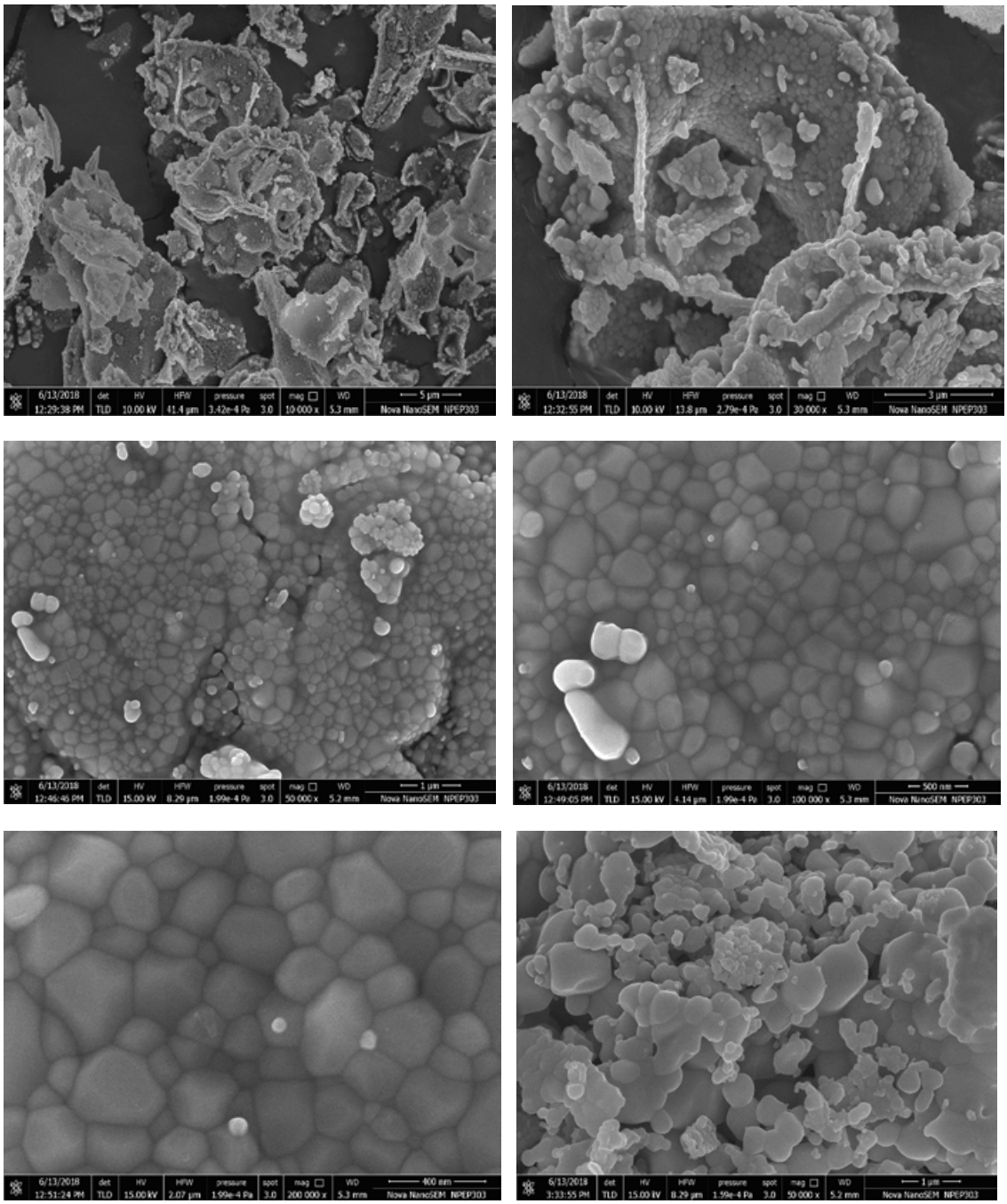

Fig. 3. FESEM images of the synthesized $CeVO_4$ NPs

to understand the elemental composition of the vanadium, cerium and oxygen in the fabricated nanomaterial. There was no unidentified peak are observed in EDX. This quantitative data affirms the NPs purity, composition and formation of $CeVO_4$ NPs.

*UV–Vis diffuse reflectance spectrum and photoluminescence of CeVO4 NPs*

Fig. 5 demonstrates UV-Vis DRS of $CeVO_4$ NPs. It can be seen that the nanomaterial has good absorption capacity in the visible region (400–700 nm). Additionally, the band gap energy is the

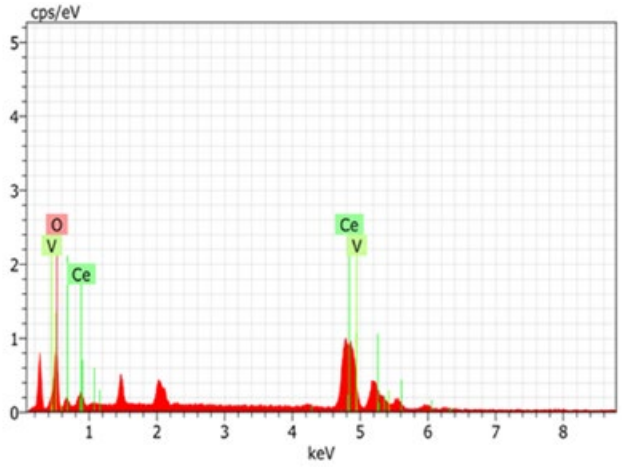


Fig. 4. EDX spectrum of the synthesized $CeVO_4$ NPs

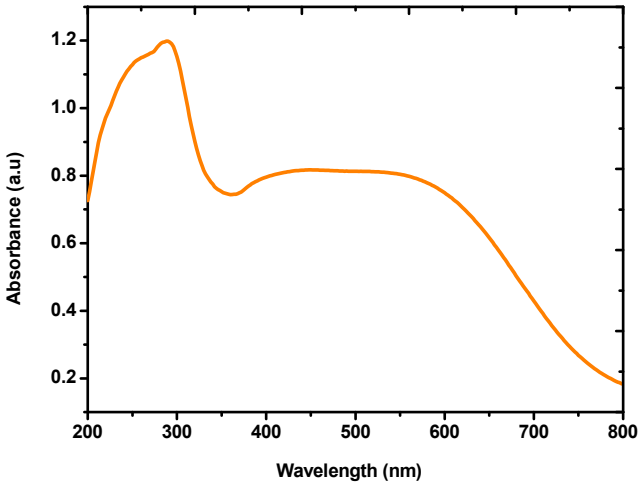


Fig. 5. UV-Vis DRS spectrum of the synthesized $CeVO_4$ NPs

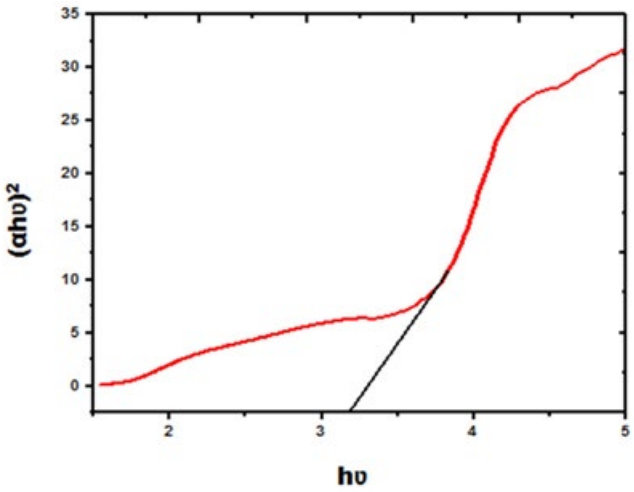


Fig. 6. A plot of $(\alpha h\upsilon)^2$ versus photon energy $(h\upsilon)$

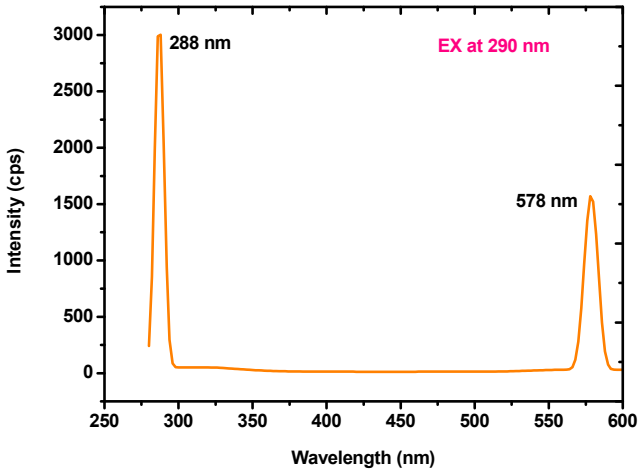


Fig. 7. Fluorescence spectra of the synthesized $CeVO_4$ NPs

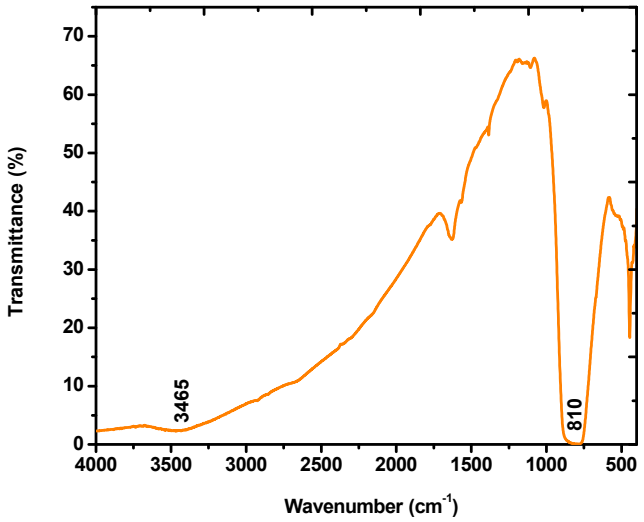


Fig. 8. FT-IR spectrum of the synthesized $CeVO_4$ NPs

Table 1. Minimum inhibition concentration of $CeVO_4$ NPs against fungal pathogens

| Test pathogens | MIC (µg/ ml) of $CeVO_4$ | MIC (µg/ ml) of Reference drug |
|---|---|---|
| *C. albicans* (MTCC 227) | 250 | 500 |
| *A. niger* (MTCC 282) | 250 | 100 |
| *A. clavatus* (MTCC 1323) | 250 | 100 |
| *T. rubrum* (MTCC 296) | 100 | 100 |
| *T. mentographytes* (MTCC 8476) | 100 | 100 |
| *E. floccosum* (MTCC 7880) | 250 | 100 |
| *M. gypseum* (MTCC 2819) | 500 | 100 |

criteria of material selectivity for the photocatalyst. The plot of $(\alpha h\upsilon)^2$ versus photon energy ($h\upsilon$) was obtained to determine band gap of $CeVO_4$ NPs (Fig. 6). The band gap was found to be 3.2 eV suggesting that the synthesized material using sol-gel auto combustion method is useful for photocatalytic applications. Fig. 7 indicates fluorescence spectrum with an excitation wavelength of 290 nm. The spectrum exhibited broad band peak of emission at 288 nm and 578 nm (yellow color).

*Vibrational properties*

To further affirm the formation of the CeVO4 crystal structure using FT-IR spectroscopy as shown in Fig. 8. The broad IR band at 810 $cm^{-1}$ is attributed to the V-O-V vibrations of $ReVO_4$. Residual -OH group and water are detected around 3465 $cm^{-1}$, corresponding to the O-H stretching frequency due to the bending vibrational frequency of associated water. It suggested that the $CeVO_4$ NPs was successfully fabricated by simply sol-gel auto combustion method.

*Antifungal activity of $CeVO_4$ NPs*

The results of antifungal activity of the synthesized $CeVO_4$ NPs are presented in Table 1. The antifungal activity of the synthesized $CeVO_4$ NPs was determined *in-vitro* using an Agar plate method against selected strains viz. *C. albicans*, *A. niger*, *A. clavatus*, *T. rubrum*, *T. mentographyte*, *E. floccosum* and *M. gypseum* at different concentrations ranging between 100 µg/ml to 1250 µg/ml. Fabricated $CeVO_4$ NPs exhibited a moderate activity against *T. rubrum*, *T. mentographyte* and evinced excellent activity against *C. albicans* at concentration of 250 µg/ml reference standard Griseofulvin at concentration 500 µg/mL.

## CONCLUSION

In summary, we have successfully fabricated tetragonal $CeVO_4$ NPs via sol-gel auto combustion method to obtain biologically active nanomaterial. The synthesized $CeVO_4$ NPs were quasi-hexagonal in shape as observed in FESEM analysis. The DRS spectrum confirmed that the synthesized $CeVO_4$ NPs have a high absorption with 3.2 eV band gap. The synthesized $CeVO_4$ NPs has shown excellent antifungal activity against *Candida albicans* fungal strain and hence it may be useful for the treatment of Candidiasis. This result provides useful information of $CeVO_4$ NPs having no any side effects and play significant role in nanomedicine.

## ACKNOWLEDGEMENT

Author SG are thankful to Jaysingpur College, Jaysingpur, Savitribai Phule Pune University and Microcare Laboratory Gujrat for providing the technical, instrumental and biological activities supports.

## CONFLICT OF INTEREST

The author declare that there is no conflict of interests regarding the publication of this manuscript.

## REFERENCES

1. Yao L, Guo E, Sun K, Lu Q, Wang Q. Formation of one-dimensional CeVO4 nanobelts as an enhanced photoelectrocatalyst and density functional study. Materials Letters. 2018;231:11-5.
2. Chen L. Hydrothermal synthesis and ethanol sensing properties of CeVO4 and CeVO4–CeO2 powders. Materials Letters. 2006;60(15):1859-62.
3. Zhu L, Li Q, Li J, Liu X, Meng J, Cao X. Selective Synthesis of Mesoporous and Nanorod CeVO4 without Template. Journal of Nanoparticle Research. 2007;9(2):261-8.
4. Picardi G, Varsano F, Decker F, Opara-Krasovec U, Surca A, Orel B. Electrochemical characterization of optically passive CeVO4 counterelectrodes. Electrochimica Acta. 1999;44(18):3157-64.
5. Opara Krasovec U. Structural and spectroelectrochemical investigations of tetragonal CeVO4 and Ce/V-oxide sol-gel derived ion-storage films. Solid State Ionics. 1999;118(3-4):195-214.
6. Kumar JV, Karthik R, Chen S-M, Marikkani S, Elangovan A, Muthuraj V. Green synthesis of a novel flower-like cerium vanadate microstructure for electrochemical detection of tryptophan in food and biological samples. Journal of Colloid and Interface Science. 2017;496:78-86.
7. Phuruangrat A, Kuntalue B, Thongtem S, Thongtem T. Effect of PEG on phase, morphology and photocatalytic activity of CeVO4 nanostructures. Materials Letters. 2016;174:138-41.
8. Kamble DR, Bangale SV, Ghotekar SK, Bamane SR. Efficient Synthesis of CeVO4 Nanoparticles Using Combustion Route and Their Antibacterial Activity. Journal of Nanostructures. 2018;8(2):144-51.
9. Liu F, Shao X, Yin Y, Zhao L, Shao Z, Liu X, et al. Shape controlled synthesis and tribological properties of CeVO4 nanoparticles aslubricating additive. Journal of Rare Earths. 2011;29(7):688-91.
10. Zhang H-J, Shu J, Wei X, Wang K-X, Chen J-S. Cerium vanadate nanoparticles as a new anode material for lithium ion batteries. RSC Advances. 2013;3(20):7403-7.
11. Opara Krašovec U, Orel B, Šurca A, Bukovec N, Reisfeld R. Structural and spectroelectrochemical investigations of tetragonal CeVO4 and Ce/V-oxide sol-gel derived ion-storage films. Solid State Ionics. 1999;118(3):195-214.
12. Wang H, Meng Y, Yan H. Rapid synthesis of nanocrystalline CeVO4 by microwave irradiation. Inorganic Chemistry Communications. 2004;7(4):553-5.
13. Rahimi-Nasrabadi M, Ahmadi F, Fosooni A. Influence of capping agents additives on morphology of CeVO4 nanoparticles and study of their photocatalytic properties. Journal of Materials Science: Materials in Electronics. 2017;28(1):537-42.
14. Xie B, Lu G, Dai Q, Wang Y, Guo Y, Guo Y. Synthesis of CeVO4 Crystals with Different Sizes and Shapes. Journal of Cluster Science. 2011;22(4):555-61.
15. Watanabe A. Highly Conductive Oxides, CeVO4, Ce1−xMxVO4−0.5x(M=Ca, Sr, Pb) and Ce1−yBiyVO4, with Zircon-Type Structure Prepared by Solid-State Reaction in Air. Journal of Solid State Chemistry. 2000;153(1):174-9.
16. Mosleh M, Mahinpour A. Sonochemical synthesis and characterization of cerium vanadate nanoparticles and investigation of its photocatalyst application. Journal of Materials Science: Materials in Electronics. 2016;27(9):8930-4.

17. Zheng Y, Yang Q, Jiang J, Tang P, editors. Synthesis and Characterization of Nanoparticulate CeVO4 by Ultrasound Method and its Photocatalytic Activity. 2015 Asia-Pacific Energy Equipment Engineering Research Conference; 2015: Atlantis Press.
18. Ghotekar S. A review on plant extract mediated biogenic synthesis of CdO nanoparticles and their recent applications. Asian Journal of Green Chemistry. 2019;3(2):187-200.
19. Pansambal S, Deshmukh K, Savale A, Ghotekar S, Pardeshi O, Jain G, et al. Phytosynthesis and biological activities of fluorescent CuO nanoparticles using Acanthospermum hispidum L. extract. Journal of Nanostructures. 2017;7(3):165-74.
20. Savale A, Ghotekar S, Pansambal S, O. P. Green Synthesis of Fluorescent CdO Nanoparticles using Leucaena leucocephala L. Extract and their Biological Activities. Journal of Bacteriology & Mycology: Open Access. 2017;5(5).
21. Ghotekar S, Savale A, Pansambal S. Phytofabrication of fluorescent silver nanoparticles from Leucaena leucocephala L. leaves and their biological activities. Journal of Water and Environmental Nanotechnology. 2018;3(2):95-105.
22. Ghotekar SK, Vaidya PS, Pande SN, Pawar SP. Synthesis of silver nanoparticles by using 3-methyl pyrazol 5-one (chemical reduction method) and its characterizations. Int J Multidis Res and Deve. 2015;2(5):419-22.
23. Ghotekar SK, Pande SN, Pansambal SS, Sanap DS, Mahale KM, Sonawane B. Biosynthesis of Silver Nanoparticles Using Unripe Fruit Extract of Annona reticulata L. and its Characterization. World J Pharm and Pharm Sci. 2015;4(11):1304-12.
24. Aher YB, Jain GH, Patil GE, Savale AR, Ghotekar SK, Pore DM, et al. Biosynthesis of copper oxide nanoparticles using leaves extract of Leucaena leucocephala L. and their promising upshot against diverse pathogens. International Journal of Molecular and Clinical Microbiology. 2017;7(1):776-86.
25. Wiegand I, Hilpert K, Hancock REW. Agar and broth dilution methods to determine the minimal inhibitory concentration (MIC) of antimicrobial substances. Nature Protocols. 2008;3:163.