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Remediation of wastewater and innovative cheap cost filter for microbial and heavy metal removal using calcium oxide nanoparticles

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Problems with the environment have recently gotten worse because of crude hydrocarbons, microbial contamination, heavy metal pollution in water, and the price of pricey antioxidants. It motivated scientists to investigate the possibility of certain metal oxide nanoparticles for the treatment of water contaminants $Ca(OH)_2$ was used as a wet chemically generated precursor using the direct thermal breakdown technique to create CaONPs (calcium oxide nanoparticles). The samples were analyzed using TEM, IR, XRD, SEM, and Zetasizer. The particle size was in the 49.8 to 75.8 nm range. There is evidence that CaONPs have antimicrobial properties. The removal process of heavy metals by CaONPs was dependent on temperature and pH. CaONPs are a less expensive alternative to expensive sorbents for the elimination of metal ions. The effectiveness of the prepared calcium oxide nanoparticles was achieved as an effective and inexpensive adsorbent for the removal of various heavy metal ions (iron, lead, manganese, chromium, and copperII) from aqueous solutions, with a good opportunity to soon be integrated into technologies for treating polluted water and wastewater. CaONPs are a low-cost, versatile material utilized in a variety of environmental treatments.

Keywords: CaONPs; synthesis, characterization; Catalysis; filter

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INTRODUCTION

Nanotechnology has an important role in many fields. Nanoparticles can be synthesized by physical, chemical, and mechanical methods (Mughal & Hassan, 2022; Thakur & Thakur, 2022; Xu et al., 2022). CaONPs have found several applications in catalysis, adsorption, water purification, and as antibacterial agents (Hossini et al., 2022; Khan et al., 2023; de Jesus et al., 2024). CaONPs are particularly interesting since it is considered safe substance for human and animals (Hossini et al., 2022; Wang et al., 2022; Harris et al., 2023). Heavy metal pollution is of prevailing worry among the kinds of natural pollution because heavy metals have a high toxicity (Hafez and Fouad, 2020; Herath et al., 2022; Yang et al., 2022; Aziz et al., 2023). Chromium is widely used in a variety of industries, including material, metal finishing, and paint. Chromium is a hazardous, mutagenic, and cancer-causing chemical when present in excess (Andra et al., 2021; Singla, 2022; Ezzat et al., 2023). Lead poisoning usually has neurological or teratogenic repercussions. Neuronal necrosis is caused by organic lead (Bjørklund et al., 2024). Axonal degeneration and demyelination are caused by inorganic lead. Organic lead compounds are more readily absorbed and hence offer a larger risk (Wang et al., 2022). Manganese in drinking water sources produces neurotoxicity symptoms in individuals (Ávila et al., 2023). High levels of iron in the water generate an overload, which can lead to diabetes, hemochromatosis, stomach issues, and nausea. It also has the potential to harm the liver, pancreas, and heart (Yang et al., 2022). With recent advances in nanotechnology, various types of metal and metal oxide nanoparticles with antimicrobial activity have been synthesized. Metal nanoparticles containing magnesium oxide, copper, silver, iron, zinc oxide, nickel oxide, and calcium oxide exhibit antimicrobial properties (Herath et al., 2022; Ismail et al., 2022; Elnagar et al., 2023).

Large surface areas of CaONPs ensure a variety of interactions with the microbial surface. By studying the effect of calcium nanoparticles on four microbial strains, the activity of calcium nanoparticles against microbes was observed (Andra et al., 2021; Kumari et al., 2023). Applying wastewater in agriculture can be a source of biological contamination of soil, water, and plants including pathogenic microorganisms. Therefore, monitoring and evaluation of these risks are important to the search for environmentally friendly solutions (Pratap et al., 2023). It was clear from the study the effective properties of CaONPs, including the antimicrobial activity and the absorption of heavy elements from wastewater, so we recommend the use of CaONPs in the treatment of wastewater and the use of treated water in agriculture (Singla, 2022; Eddy et al., 2023). CaONPs have a huge capacity and are an excellent adsorbent for a variety of hazardous compounds (Abdullah et al., 2022; Ahmed et al., 2022; Jaiswal et al., 2022; Longos, 2022; Zhang et al., 2022; Kelle et al., 2023). The objective of this study is to provide a technique for eliminating heavy metals such as Cr^{+2}, pb^{+2}, Fe^{+2}, Cu^{+2}, and Mn^{+2} from aqueous solutions using CaONPs and to determine the ideal conditions for doing so. Also, the study determines how well calcium oxide nanoparticles may be used to cleanse wastewater that has been contaminated by microbes, as well as function of an antioxidant.

MATERIALS AND METHODS

Preparation of CaONPs

Drop-by-drop addition of aqueous sodium hydroxide (NaOH) in aqueous calcium chloride ($CaCl_2$) resulted in a supersaturated aqueous solution of CaONPs at 80°C for 60 minutes under rapid stirring (1300 rpm). The resultant white colloidal calcium hydroxide $Ca(OH)_2$ solution was washed in hydro-alcoholic suspension to remove sodium chloride, which improved its stability by reducing agglomeration. After passing the suspension through a 0.45-m filter syringe, it was dried for 24 hours at 80°C. These nanoparticles were calcined at 800°C for 30 min. Figure 1S (supplementary file) illustrates the preparation of CaONPs. The reactions that occurred in this work are shown in equations (1) and (2) (Kasirajan et al., 2022).

$$CaCl_2 + 2NaOH \longrightarrow Ca(OH)_2 + 2NaCl \quad (1)$$

$$Ca(OH)_2 \xrightarrow{\text{Calcination}} CaO + H_2O \quad (2)$$

Characterization of CaONPs

Fourier transforms Infrared spectrometer was carried out using Infra-Red Bruker Tensor 37, Spectral Range: 7500 to 370 cm-1. XRD (Bruker AXS GmbH, Germany) was recorded using an X-ray diffractometer. SEM (JEOL JSM-6390LV, USA) was used to detect the morphological characterization of CaONPs. CaONPs size was observed by Zetasizer (Malvern Panalytical Ltd, United Kingdom) (Amirsadat & Sharififard, 2022; Kasirajan et al., 2022).

Preparation of stock solution

Preparation of three different dilution of wastewater samples (50, 100, and 200 ppm) were prepared by mixing standard heavy metal solutions of ICP multi-element standard solution IV (Sigma-Aldrich). Stock solutions of these heavy metal solutions were prepared by mixing 5, 10, and 20 ml of both standards with 100 ml of distilled water. Standard solutions of HCl and NaOH were used for pH alterations (Thakur et al., 2022).

Batch adsorption set-up

The present work aimed to explore how an increase or decrease in pH and temperature can affect the adsorption phenomena of the divalent ions on the surface of CaONPs. First experiment: Stock solutions of these heavy metal solutions were prepared by mixing 5, 10, and 20 ml of both standards with 100 ml distilled water out of which sets of experiments were performed for elucidating the effect of different pH (5, 6, 7, 8) at same at 25° C using a pH meter) Jenway 3510 Standard Digital pH Meter, United States) equipped with electrodes. 1.0 M sodium hydroxide (NaOH) and 1.0 M hydrochloric acid (HCl) were used for the balance of pH levels to desirable values by adding them dropwise with the help of separate droppers. Second experiment, three stock solutions of heavy metals namely (Cu^{+2}, Mn^{+2}, Pb^{+2}, Cr^{+2}, and Fe^{+2}) were prepared (50, 100, and 200 ppm) at different pH values (5,6,7,8, and 10) and 25°C in a sealed conical flask (200 mL) and stirred continuously (150 rpm) in a thermostatic bath (WB-300 Series Stuart General-Purpose Digital Water Baths, Cole-Parmer, China) for 10 min at a pH 7 and different temperature (10, 25, 45, 65 °C), respectively. Thereafter, the sample was filtered by a novel filter (plastic tube open for two sides containing CaONPs layer, and cotton layer on the bottom). The initial concentration of heavy metals in the stock solution before and after the filtrate ration was estimated using the XRF instrument. Similarly, the optimized process parameters were achieved using the same procedure to investigate the adsorption conditions on the removal rate of $^{+2}$, Cr^{+2}, Fe^{+2}, Cu^{+2}, Mn^{+2}, and Pb^{+2} ions by CaONPs. Measurement of the initial concentration of heavy metals on an aqueous solution and filtrate solution by XRF instrument. In fact, a filter was created with only cotton layers, and after the filtration process and measuring the concentration of the elements, the values were subtracted from the final value of the filtration results with the filter that contains CaONPs layers. The Removal percent was calculated based on the equation: % Removal= $(A_0-A_t/A_0)*100$. Where A_O and A_t are the absorbance values from an initial concentration of heavy metal in the aqueous solution (200 ppm, 100ppm, 50ppm) and the final concentration of heavy metals on an aqueous solution after using a novel filter (CaONPs layer) (Talank et al., 2022).

Microbial decontamination of wastewater

Microorganisms: The microbial strains isolates used throughout the present work namely: *Candida albicans* (ATCC10231) as yeast, *Staphylococcus aureus* (ATCC6538), *Listeria sp (ATTC13932) Pseudomonas aeruginosa* (ATCC 13867) as Gram-positive bacterium, and *[Escherichia coli* (ATCC8739) and *Burkholderia cepacia* (NCTC 10661), *Salmonella sp* (ATCC14028), as Gram-negative bacteria.

Minimum inhibitory concentration (MIC): The antimicrobial activity of CaONPs (50, 100, 150, and 200 µg/l) was investigated using the minimum inhibitory concentration (MIC) values (Kasirajan et al., 2022).

Antimicrobial Activity of CaONPs against Contamination of Water: The antimicrobial activity was investigated according to ASTME 2149-01 (Standard Test Method for Determining the Antimicrobial Activity of Immobilized Antimicrobial Agents Under Dynamic Contact Conditions). The antimicrobial activity of the CaONPs was expressed as the reduction percent of each test organism after 24 h incubation with the CaONPs under test according to the following formula: R(%)= [(B-A)/B]*100. Where R% is the reduction percentage of the microbial colony number, A is the number of microbial colonies in the flask containing the CaONPs after 24 h incubation (by culture 20 micro liter of susbintion on Muller Hilton agar plates), and B is the initial number of the microbial colonies in the flask before the CaONPs addition (by culture 20 micro liter of susbintion on Muller Hilton agar plates and the plate were incubated at 37 0C for 24-48 hours).

RESULTS AND DISCUSSION

Characterization of the CaONPs

Microscopic observation: The CaONPs morphology was examined by scanning electron microscope (SEM) and Transmission Electron microscope (TEM) (Miranda et al., 2022). The average diameter of the CaONPs was obtained using Image-J software (Bansal et al., 2022). The SEM images revealed that CaONPs had a fine diameter between 35 and 95 nm (Ameen et al., 2022). The TEM images revealed that CaONPs had a fine diameter between 49.07, 94nm (Figure 1).

Dynamic Light Scattering (DLS): The hydrodynamic diameters of nanoparticles were measured using dynamic light scattering (DLS). The distribution intensity in Figure 2S (supplementary file displays the result of the regularization algorithm applied to the data. Here, the contributions in terms of %intensity signal are shown versus particle diameter. For an ideal monodisperse sample, it would be expected that the peak in the intensity distribution was very close to the z-average size from the cumulant fit. the mean size peak by intensity is slightly different from the cumulant size (Buonomenna et al., 2022). The observations of the DLS value was 89.61 nm for the peak intensity.

The XRD pattern of CaONPs: All reflection peaks in Figure 3S (supplementary file can be readily indexed to a pure cubic phase of CaONPs with a space group of Fm-3 (ICSD 75785) (Shi et al., 2022). The crystallite size diameter (D) of the CaONPs has been calculated by the Debye–Scherrer equation (1) ($D = K\lambda/\beta \cos \theta$), where β FWHM (full-width at half-maximum or half-width) is in radian and θ is the position of the maximum of the diffraction peak (Ikram et al., 2022). K is the so-called shape factor, which usually takes a value of about 0.94, and λ is the X-ray wavelength (1.54060 for Cu Ka1). The average crystallite size of CaO is 55 nm.

FTIR spectra of CaONPs: The existence of functional groups was verified using a SHIMADZU IR Prestige 21 spectrometer set to screen at room temperature between 250 and 800 cm^{-1} (Jadhav et al., 2022). CaO's typical absorption bands are observed in the range of 300-800 cm^{-1} (Chauhan et al., 2022). The average value of lattice vibration is shown by the peaks at 306, 320, 325, and 344 cm^{-1} in the frequency distribution. According to, the IR band peaked at 424 cm^{-1}, which might be attributable to vibrations in the CaO lattice. C-H stretching corresponds to the band's appearance of 435 cm^{-1}. The absorbed band at 450 cm^{-1} is linked to the Ca-O bond as well (Figure 4S, Supplementary file).

Removal of heavy metals from aqueous solutions

Effect of pH on heavy metal ions adsorption: Removing of heavy metals from contaminated water depends largely on the pH of the solution (Duan, et al., 2020). Consequently, the effect of pH of Cr^{+2}, Fe^{+2}, Cu^{+2}, Mn^{+2}, and Pb^{+2} ions (50 ppm, 100, 200) at various values of pH (2, 5, 7, 8, and 10) with "30 min" contact time (Figure 2). The removal % is low at (pH < 7) where the concentration of H^+ ion was high, and therefore the surface of the sorbent becomes highly positive charged, thus lessening the centripetal force among sorbent and Cr^{+2}, Fe^{+2}, Cu^{+2}, Mn^{+2}, and Pb^{+2} particles (Park et al., 2017). An increase in the removal percent

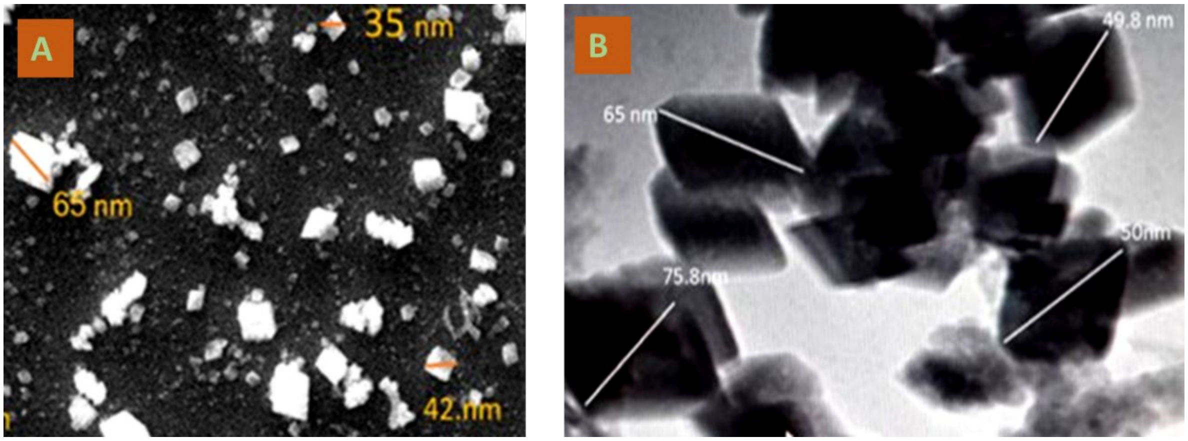

Fig. 1. Scanning electron microscope (A), TEM images of CaONPs (B)

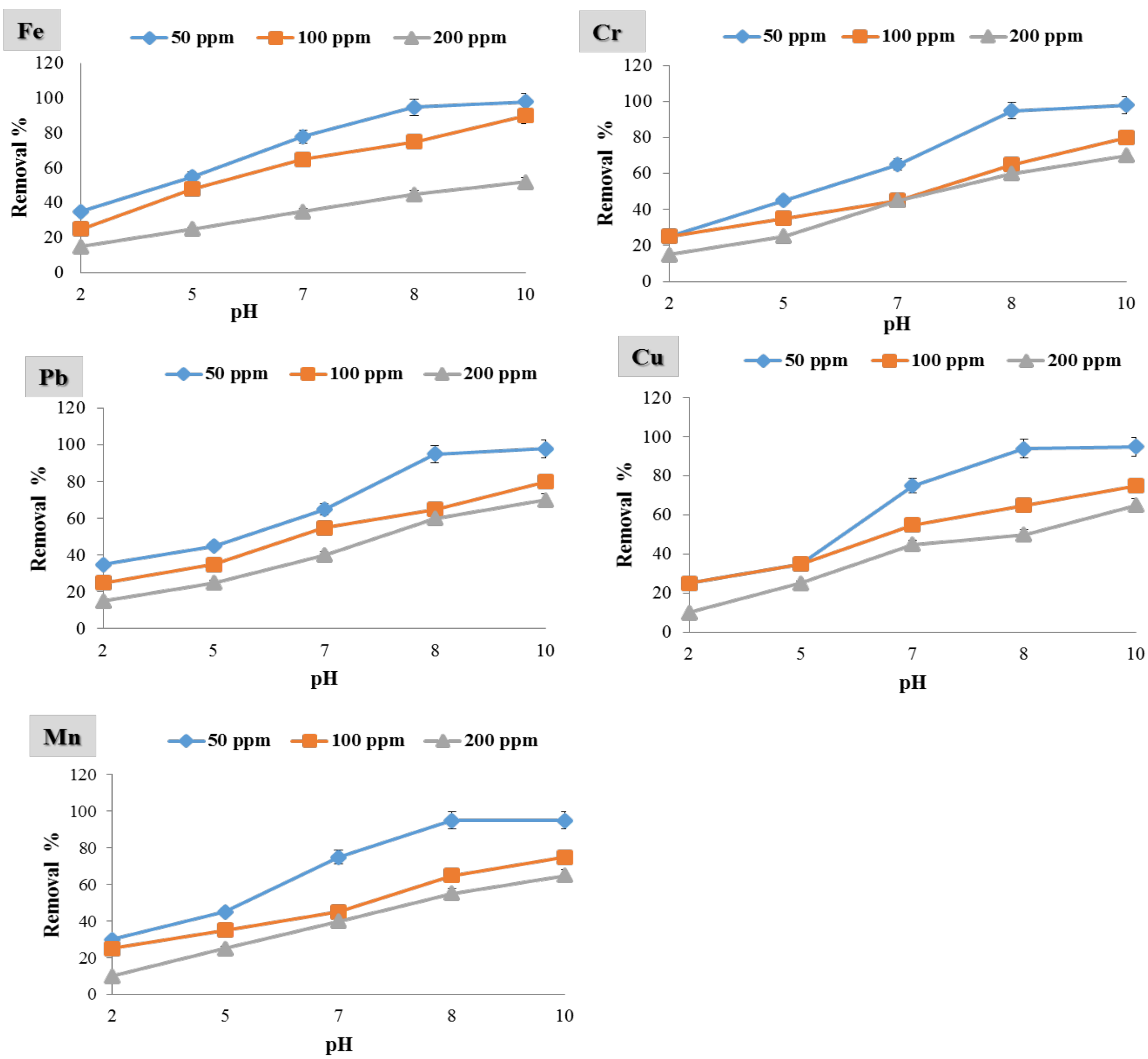

Figure 2. Effect of pH on heavy metals removal percentage using different concentrations (50, 100, and 200 ppm).

of heavy metals ions at pH (7-10) (Abdulraheem et al., 2020). "pH>5", the concentration of "H_3O^+ hydronium particle" is extremely low, and the sorbent's surface is highly negatively charged, the destinations for Cr^{+2}, Fe^{+2}, Cu^{+2}, Mn^{+2}, and Pb^{+2} particles are effectively accessible (Borggaard et al.,2019). So the competition between the proton and the ion is zero or very low, and the removal % Cr^{+2}, Fe^{+2}, Cu^{+2}, Mn^{+2}, and Pb^{+2} ions are expanded at that point (Ghorbani et al., 2020), at pH bend is moved to the soluble area (pH>8) the removal% of Cr^{+2}, Fe^{+2}, Cu^{+2}, Mn^{+2}, and Pb^{+2} inset most remains constant.

Effect of temperature on heavy metal ions adsorption: High temperatures will make it is necessary to increase the adsorbate's dispersion rate while shifting temperatures will affect the adsorbent's capacity to maintain harmony with a certain adsorbate (Nozari et al., 2022). The adsorption of different concentrations of Cr^{+2}, Fe^{+2}, Cu^{+2}, Mn^{+2}, and Pb^{+2} ions by CaONPs was presented in (Figure 3). Adsorption of metals increases as temperature increases (Li et al., 2020). The process of adsorption has advantages being low cost, easy, and less time-consuming, and is likely to be incorporated soon as clean water and wastewater treatment techniques (El-Sayed, 2020).

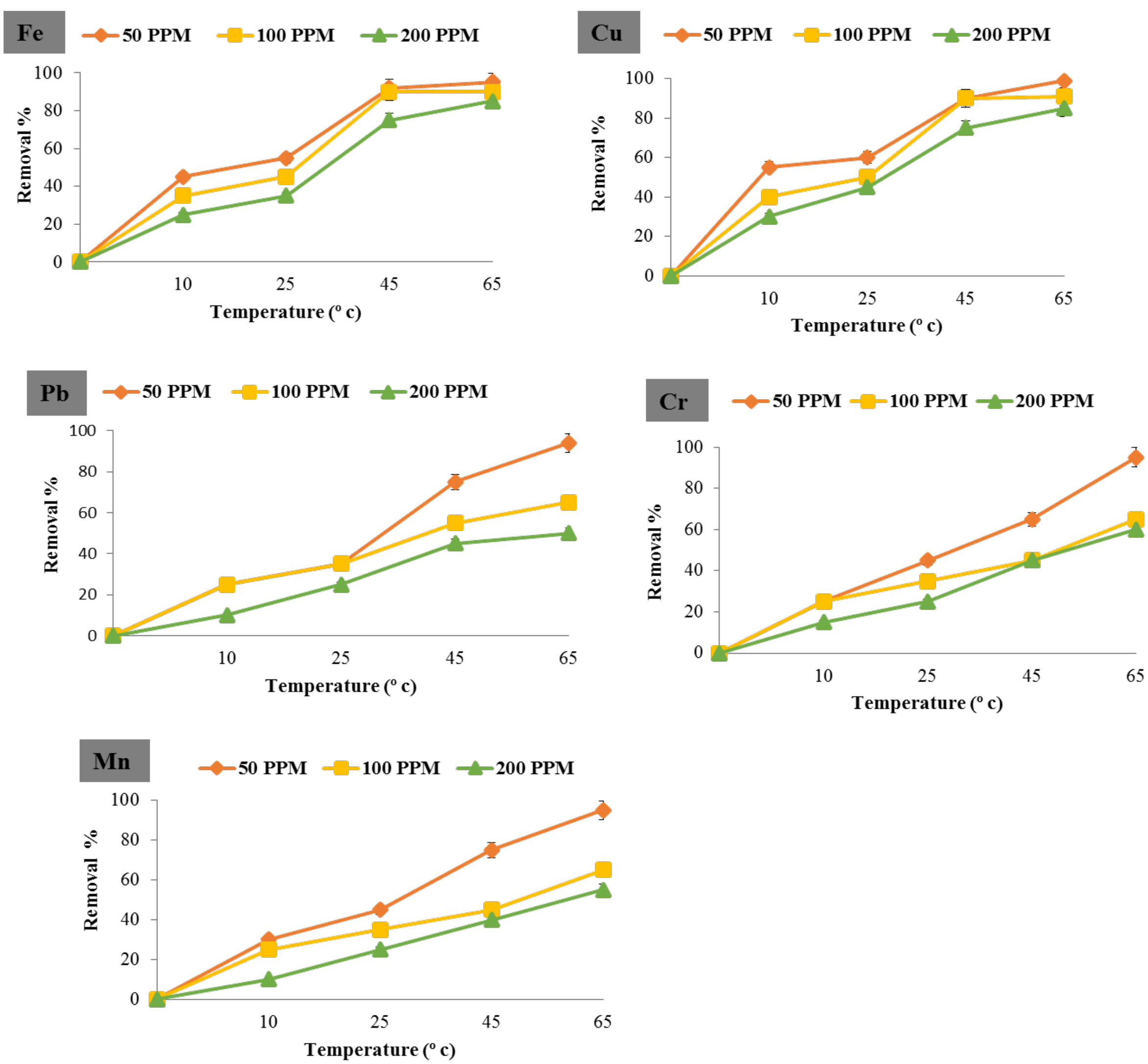

Figure 3. Effect of temperature on heavy metals removal percentage using different concentrations (50, 100, and 200 ppm).

CaONPs as Decontaminants of Wastewater: Alkalinity and active oxygen species are associated with CaONPs, which show a high antibacterial activity (Ghaffari et al., 2023). It has been verified that the antibacterial mechanism of CaONPs is brought about by the generation of superoxide on the surface of these particles, and an increase in pH value by the hydration of CaONPs with water. CaONPs damage the cell membrane and then cause the leakage of intracellular contents which in turn leads to the death of the bacterial cells (Khan et al., 2023). CaONPs initiated the sensitivity changes in (*E. coli, Staphylococcus epidermidis, Burkholderia Cepacia,* and *Candida albicans*) induced by active oxygen. However, the strong antibacterial activity of CaONPs could be observed in the absence of any ROS production (Chauhan et al., 2022). The antimicrobial effect of different concentrations with CaONPs against *Burkholderia cepacia*, *Staphylococcus aureus, E. coli,* and *Candia albicans* was verified by growth reduction using the OD values (Figure 4) CaONPs showed a high microbial activity (ROS) activity due to the active oxygen species (Kumari et al., 2023).

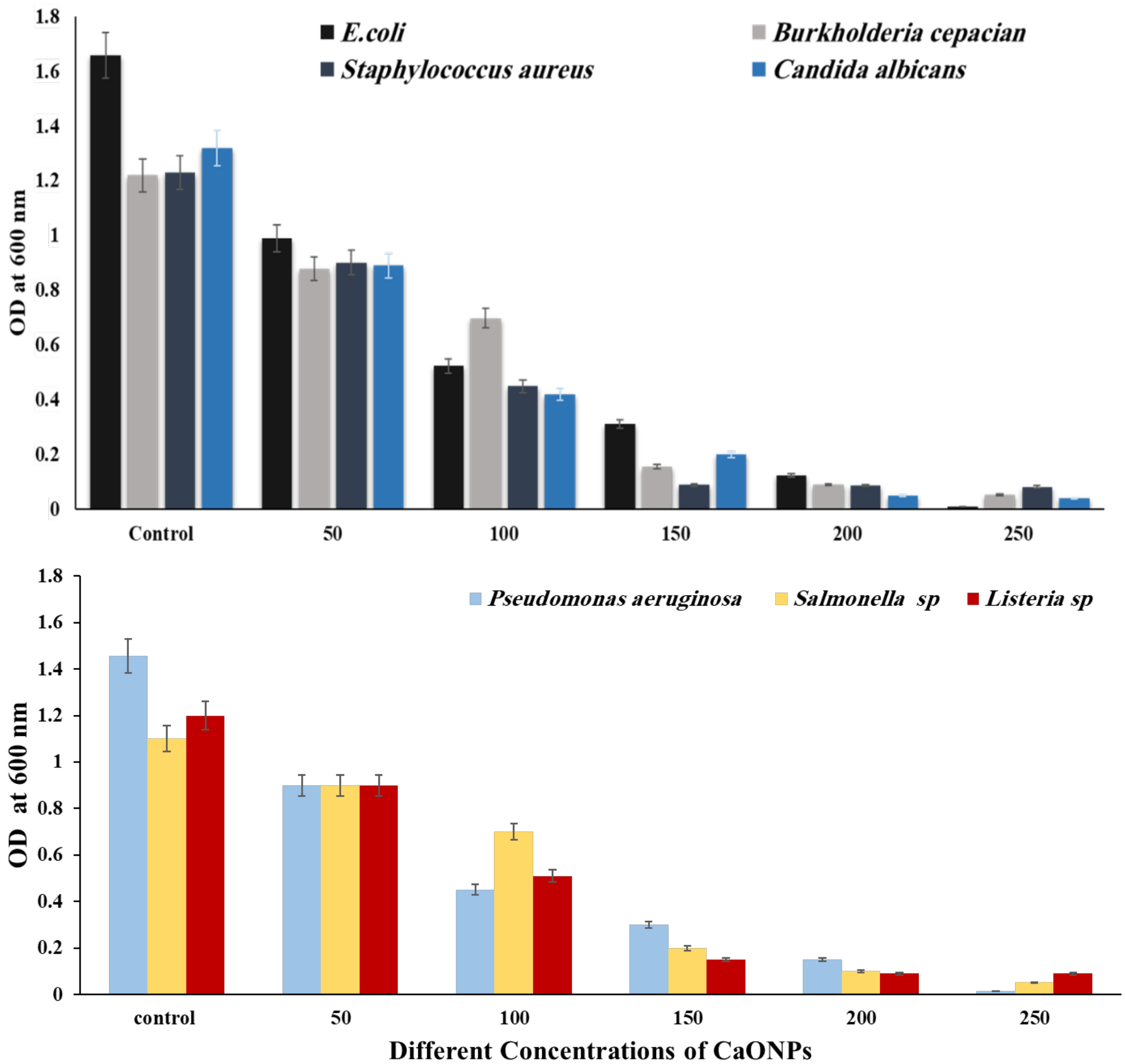

Figure 4 . Remediation of microbial contaminated synthetic water as affected by different CaONPs concentrations

Damage to the cell membrane causes leakage of contents into the cells and potentially leads to cell death. The concentration of CaONPs (250 ppm) was able to kill all tested microorganisms (Chaudhary et al., 2019). On the other hand, the CaONPs were used to treat wastewater containing many microorganisms (50000 total counts) containing different types of bacteria, molds, and yeast. CaONPs at 200ppm lead to a reduction of the total microorganisms (99.9%) (Ganesh et al., 2024). An overview on metal oxide incorporated bionanocomposites and their potential applications. Nanostructures & Nano-Objects, 38, 101126. This results in calculation by log 10 reduction after after 48 hours of incubation at 37^{0}C (Figure 5). These nanoparticles also are low-cost and available materials. These properties make them promise antibacterial agents.

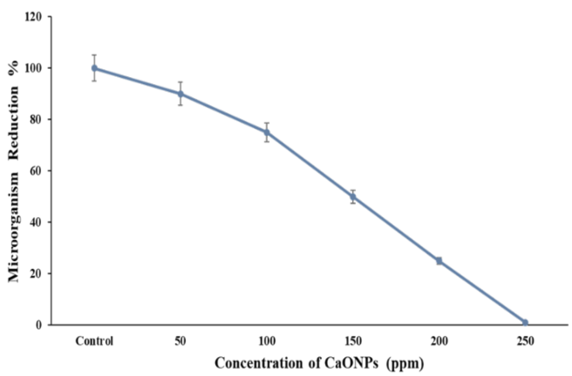

Figure 5. Wastewater treatment using different concentrations of CaONPs for 48 hours at 37 ^{0}C

CONCLUSION

This study showed that CaONPs can be produced by a direct thermal decomposition of $Ca(OH)_2$ as the precursor, at 800 °C for 30 min. CaONPs have antimicrobial effects. CaONPs can liquid heavy metals ion removal. CaONPs are a low-cost, versatile resource utilized in a variety of environmental treatments, including the generation of biodiesel and a new filter for eliminating heavy metals and microorganisms.

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SUPPLEMENTARY FILES

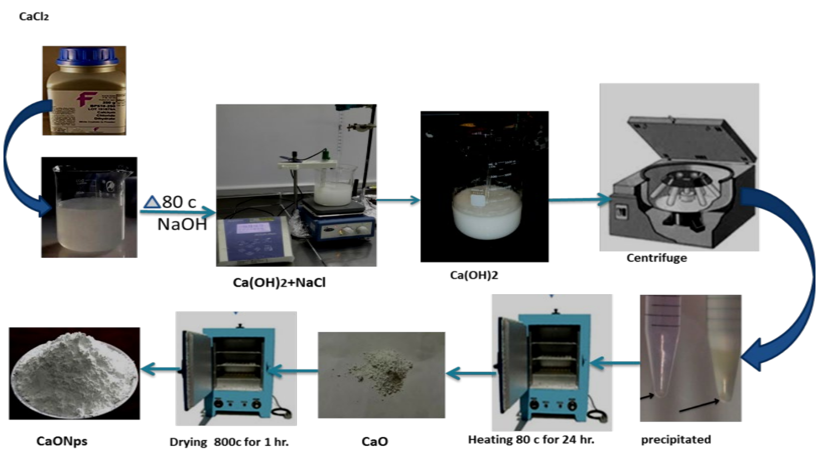

Figure 1S. Preparation of CaO NPs by direct Thermal decomposition method

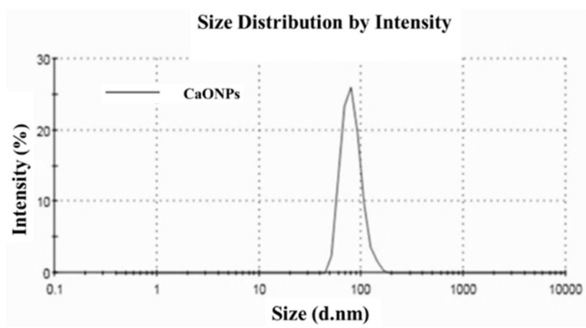

Figure 2S. Dynamic light scattering analysis of calcium oxide by Malvern Zetasizer

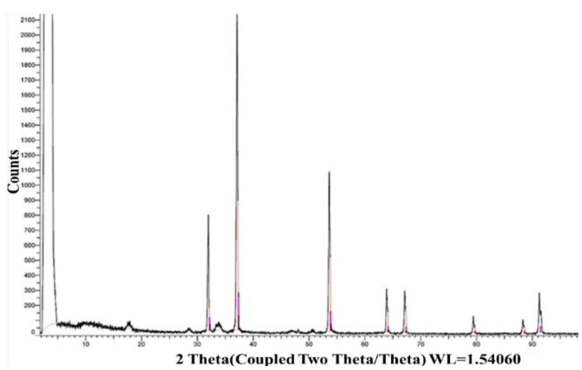

Figure 3S. The XRD pattern of CaONPs

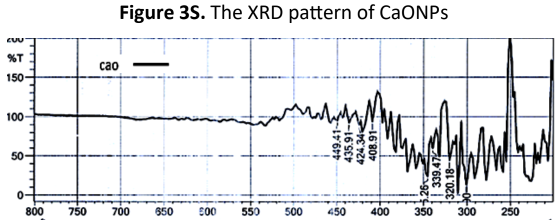

Figure 4S. FTIR spectra of CaONPs