

“Fabrication of CaO Nanoparticle’s; Characterization and its Efficient Fluoride Remediation”

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ABSTRACT

In the present study, micro-nanostructured calcium oxide (CaO) was successfully synthesised via a sol-gel-assisted precipitation-calcination technique for fluoride removal from groundwater. Fluoride contamination in drinking water is a severe public health and environmental hazard, and so there is a need for developing sustainable and effective treatment materials. Synthesised CaO was characterised systematically by XRD, FTIR, UV-Vis, SEM, HRTEM and EDS. XRD showed the production of a highly crystalline cubic phase of CaO nanoregime, while FTIR and UV-Vis spectroscopy revealed the surface functional groups and optical band gap features. SEM investigation revealed beehive-like surface morphology with well-defined microstructures from 1.00 to 10.00 μm , which are constituted of aggregated micro-nanoparticle domains. Micro/nano-structured features were validated by HRTEM, and the elemental purity of Ca and O was confirmed by EDS. Batch adsorption tests were carried out for evaluating fluoride removal effectiveness (78%). The performance improvement is ascribed to the synergistic impact of surface adsorption and CaF_2 precipitation, which is achieved by the micro/nano structure with a vast surface area and abundant occupied sites. The equilibrium data were correctly adapted to the Langmuir isotherm model, suggesting monolayer chemisorption via electrostatic attraction and ion-exchange mechanisms. The study provides a straightforward, cost-effective and environmentally friendly pathway for transforming a calcium precursor into a high-performance nanomaterial for the purification of ground water.

Keywords: HRTEM, FTIR, CaO micro/nano particles, Fluoride remediation, and SEM etc.

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INTRODUCTION

In the last few decades, there have been attempts to develop new energy transfer techniques for the synthesis of composite nanomaterials that are both economical and environmentally friendly, for example, the use of renewable energy sources or novel chemical processes that reduce waste and energy consumption [1]. These composite nanoparticles are crucial for the health and environmental conservation of human life [2]. Then the composite nanomaterials are becoming more essential in several areas, such as water purification, environmental health, biotechnology, and biological sciences [3]. They are also showing a big edge in several industries, including energy storage, medicine delivery, and agricultural advances. Composite nanomaterials are gaining importance in several domains such as these, as well as in various branches of biological sciences [4].

An adequate synthetic route is essential when designing composite nanostructured materials, driving the development of new methodologies over many decades [5]. Additionally, the current necessity for sustainable, efficient, and energy-saving methods in chemical reaction processing has motivated the search for alternative energy sources, moving away from micro-remediation technology methods [6]. Micro-remediation of smaller particles not only provides a much faster way of preparing standard materials, but it also assists in preparing

materials with smaller particles, which contributes to saving energy and meeting the needs for safer materials and processes for groundwater purification and environmental health [7]. For example, this method can save time and resources needed for standard remediation techniques; its use could make it a more sustainable option in environmental cleanup and groundwater purification [8].

The size, shape, stability, reduction agent, and applications of composite nanomaterials within the composite nanostructure are determined by the choice of stabilization and encapsulating agents used during synthesis [9]. Recently advances into the fields of composite materials and nanotechnologies are rapidly increasing the use of fabricated composite nanomaterials made from biological elements, which offer significantly improved structural, novel, and biological properties. Micromediated nanoparticle synthesis has recently gained significant popularity due to the widespread availability of fluorinated groundwater extracts and their various metabolic elements, which contribute to the remarkable durability and overall qualities of these materials in the underground water purifying sector [11-12].

Thereby, the majority of the aforementioned published reports have described CaO as one of the best NPs because it can clean groundwater on a large scale at very low concentrations and has excellent biocompatibility for its potential use in biological systems, pharmaceuticals, industrial processes, and many sectors of life [12-17]. Micro-remediation techniques using a wide range of materials have been reported to be effective over CaO nanoparticles, and these techniques are considered reliable, eco-friendly, and reproducible [18]. Further, these methods do not allow accumulation of CaO nanoparticles and do not interfere with groundwater fluoride scavenging and biomedical activities [19]. It has also been shown that calcium oxide nanoparticles are essential in decreasing and stabilizing fluoride, different phytochemicals, and soluble fluoride in ground water without the presence of harmful compounds [20-22]. Calcium oxide is an important commercial chemical with several uses. These phytochemical constituents are responsible for several biological activities, including antiproliferative, antioxidant, antibacterial, antifungal, anticancer, and antiviral effects [23-29].

The highlighted work focuses on the calcium oxide nanoparticles. The CaO-synthesized composite nanomaterial undergoes micro-remediation techniques and methods for forming the structure of nanoparticles. The conventional exterior features of ordinary calcium oxide microscopic particles and their development rate were evaluated and recorded using normal improvement procedures. The as-synthesized CaO micro-nanoparticle was studied for the purification of fluoride groundwater.



Fig. 1. Fluoride affected of Fingers (a) Fluoride affected of limbs (b)

Study Area

Agra district is the western most portion of Uttar Pradesh. Rajasthan bounds the district in the west, the state of Madhya Pradesh in the southern region and Etawah, Mathura, Firozabad, Etah and Mainpuri districts in north and east correspondingly. The district's roads and train routes provide excellent connectivity to it. by roads and train routes. Agra is a semi-arid region especially is bordered by Thar desert at its southwest, west and northwest sides. It is situated in north-central India near the end of the Aravalli series of hills (27° 10' N, 78°

05' E, 168 m above sea level). Summer temperatures range from 39–47°C in addition to relative humidity has been 18.5–48.5%, while winter temperatures are 2.5–10.5°C with humidity from 61–72%. It has an average annual rainfall of 761 mm, 90% of which falls between June and October. The soil of Agra is loose, sandy and calcareous. The region's hydrogeology features unconfined and semi-confined aquifers at depths of 2.4–22.4 meters below ground. The perennial Yamuna River borders the city to the north and northeast. Agra is characterized by alluvium—an assorted mixture of gravel, sand, silt, and clay deposited during the Quaternary period (Rai, 1996). The land is primarily used for agriculture.

Sample Collections

Fluoride water samples were collected from the fluoride-prone area of Baruali Ahir block, Patti Pachgai village, from August 2025 until December 2025 from groundwater sources such as hand pumps, open wells, and tube wells around Agra (see Figure 1). Samples were taken from 6 to 72 meters, respectively, in pre-washed 1-liter polyethylene-based bottles. Samples were stored at low temperature ($\sim 5^{\circ}\text{C}$) after collection and analysed within 45 h. Fluoride concentrations were evaluated by a fluoride ion selective electrode on an Orion EA 940 ion analyzer. pH was measured using a pH meter (CD Instruments, Bangalore, India) and electrical conductivity (EC) with a Systronics conductivity bridge. Total alkalinity (TA) total hardness (TH) and Chloride (Cl) were determined by argentometric, complexometric and acid-base titration techniques, respectively. Silica (SiO_2) and Phosphate (PO_4) were detected through spectroscopy by conducting the aforementioned ascorbic acid and molybdosilicate analysing procedures, respectively, following standard protocols (APHA, 1985). magnesium, Calcium, sodium and potassium were measured by atomic absorption spectrophotometer (Perkin Elmer 2380).



Fig. 2. Sample collection mixed fluoride ground water

Experimental Sections

Materials and Synthesis Method

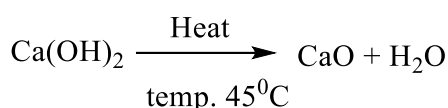
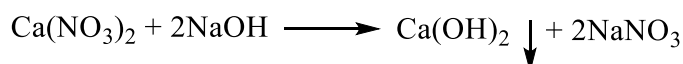
All chemicals used were analytical reagent grade. Calcium nitrate [$\text{Ca}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$] (Alfa Aesar) and sodium hydroxide (El Nasr Chemicals) were utilized. Glassware and solutions were prepared using deionized water. Calcium oxide was analyzed as KBr pellets ($4000\text{--}400\text{ cm}^{-1}$) on AMU, Aligarh spectrophotometers (Perkin Elmer 1600, Japan). UV-Vis spectra were recorded at ambient temperature in methanol with a PerkinElmer-Lambda 850 (Japan). Elemental analysis (O, Na, Ca, Cl) was performed via AMU Aligarh, SEM, and EDS. HRTEM particle size was measured on a Jeol 2100 plus 200 kV (Japan) in Chandigarh. SEM analysis used Model JSM 6510 LV (JEOL, Japan). Fluoride was routinely tested at Regional Water Analysis Labs in Aligarh and Agra.

Method for Synthesized CaO Nanoparticles

Precipitation Method

One of the most common methods for the synthesis of simple and economical CaO nanoparticles is the precipitation method.

Procedure: The precipitate of calcium hydroxide was generated by dropwise addition of sodium hydroxide (0.1 M) over a solution of calcium nitrate tetrahydrate made in distilled water with constant stirring of the mixture for 1 hour at 45°C using a hot plate magnetic stirrer. Then let the mixture stand in a quiet place for one whole day to allow the precipitate to form entirely. Next, centrifuge and filter the precipitate from Whatman filter paper No. 1; wash it several times with ethanol and distilled water to remove impurities; collect it and dry it for a few hours at about 100°C; and put the dried product in a muffle furnace heated at 500-800°C for two to four hours to obtain calcium oxide micro/nanoparticles. The generated CaO micro/nanoparticles were stored in a vacuum desiccator for further characterization.



The Characterization and Discussion of Calcium Oxide Micro/Nanoparticles (CaONps)

The absorption range of the synthesized calcium oxide micro/nanoparticles was analyzed using a Fourier Transform Infrared (FTIR) spectrum recorded with a Perkin Elmer 1600 (manufactured in Japan) within the range of 4000–400 cm^{-1} . The surface morphology and particle distribution of the micro/nanoparticles were assessed using Scanning Electron Microscopy (SEM) with Model No. JSM 6510 LV (JEOL, Japan), operated at an accelerating voltage of 20 kV for the EDS detector. The particle size and shape of CaO micro/nanoparticles were characterized by High-Resolution Transmission Electron Microscopy (HRTEM) using a JEOL 2100 Plus emitter paired with a Gatan Tridiem GIF and a $2\text{k} \times 2\text{k}$ CCD camera, operating at 200 kV. The several digital micrograph programs were utilised to analyse images for particle size, shape, and lambda maxima, which were further determined using a PerkinElmer-Lambda 850 UV-Vis spectrophotometer. The powder phases present in the CaO micro/nanoparticles were identified by X-ray Diffraction (XRD) analysis.

Scanning Electron Microscopy (SEM) Analysis

The form, overall surface, and size texture of CaO micro/nanoparticles produced by calcium nitrate and sodium hydroxide were studied by scanning electron microscopy (SEM) examination. Fig. 1 (a) shows the SEM micrographs (1.00 μm) of CaO micro/nanoparticles, and Fig. 1 (b) shows the surface interactive image (10.00 μm) of particles based on their size and roundness. Figure 1 (a & b) shows the CaO micro/nanoparticles are quite uniform, well dispersed, and mostly big and honeybee-shaped, with an average particle size being 0.102 μm . The energy dispersive X-ray spectroscopy (EDS) Fig. 1(a & b) demonstrated a characteristic CaO existence absorption peak at roughly 3.5 keV without any sort of impurity [30-31].

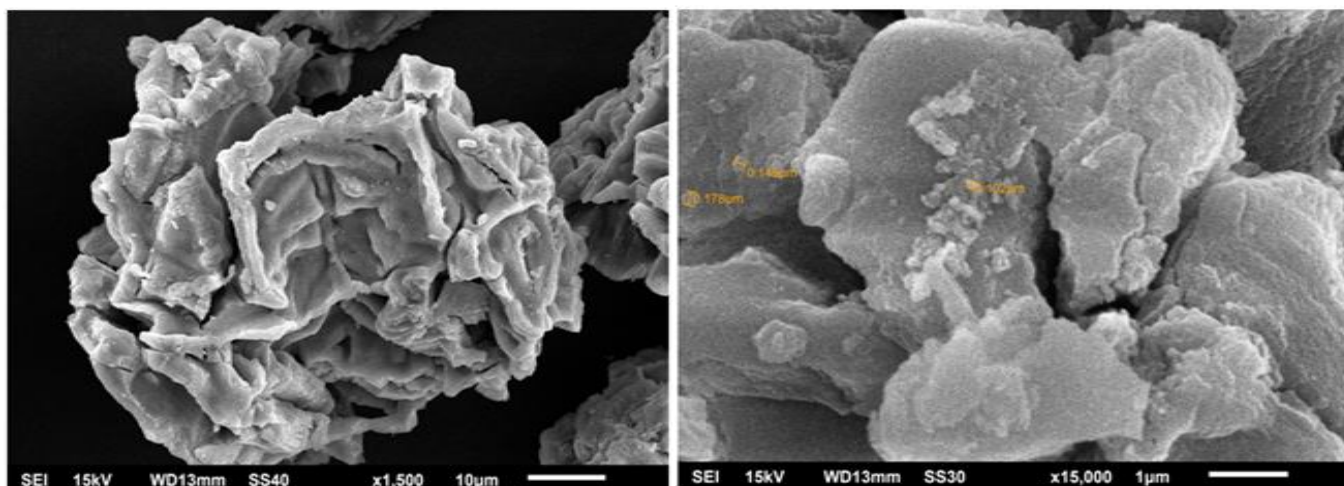


Fig. 3. Structure of Sem (a &b)

Table: EDX Analysis of CaO MPs

Element	Weight %	Atomic %
C K	10.30	16.14
O K	59.04	69.45
Ca	30.66	14.40
Totals	100.00	

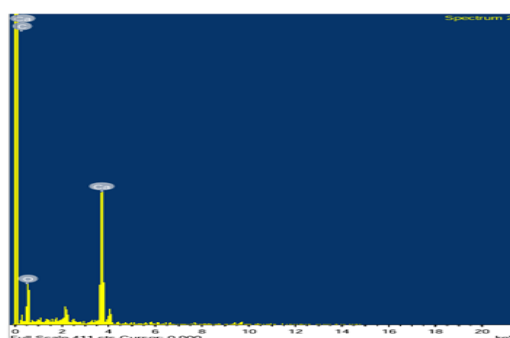


Fig. 4. EDX Spectra of CaO MPs

XRD Analysis

The XRD spectroscopy has been used for the evaluation of a normal crystalline morphology of the CaONPs. The standard X-ray diffraction pattern of CaO NPs has been characterised by XRD. The XRD pattern of CaONPs reveals significant diffraction peaks at $2\theta = 32.20, 36.00, 37.40$ and 38.00 , which indexed the planes (111), (111), (200) and (200) correspondingly. These planes are characteristic of a cubical crystal system with face-centred calcium of space group Fm-3m. The measured pattern showed a lattice constant of $a = 4.81 \text{ \AA}$ (calculated density of 3.350 g/cm^3), which matched the database Cross-References code ICSD: 064996. The crystalline character of the synthesised calcium oxide micro/nanoparticles was shown by the X-ray diffraction experiments [32-33].

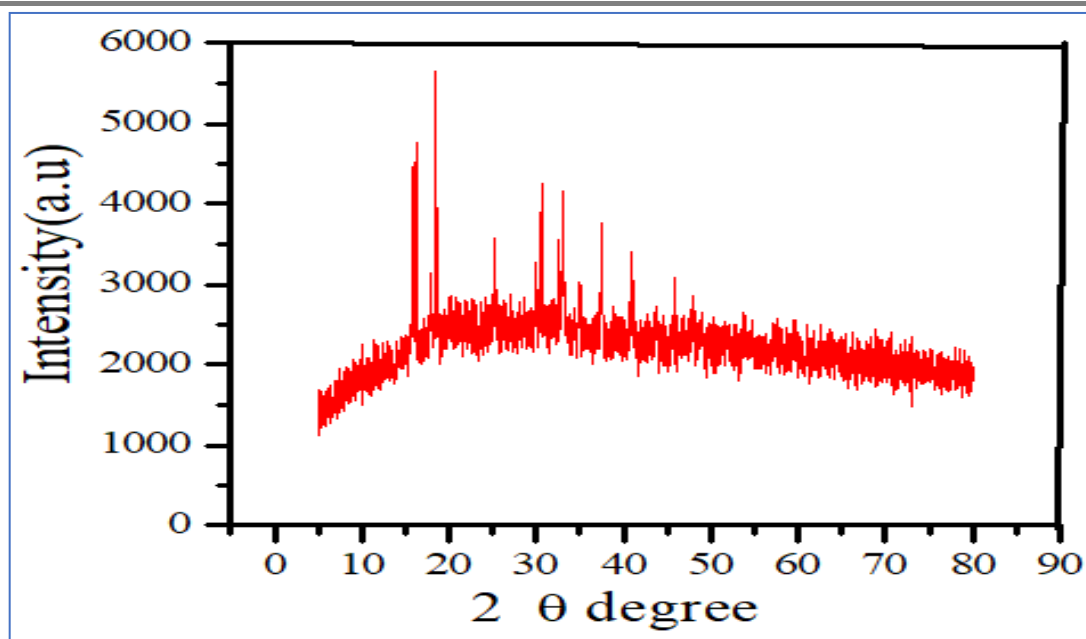


Fig. 5 Spectra of CaO NPs XRD

UV-Visible Analysis

To improve efficacy, an understanding of the development rate of the created calcium oxide nanoparticles when they integrate into a particular application is required. The reaction mixture takes on a colour change during the course of the reaction, which is the first indication of formation of calcium oxide micro/nanoparticles (CaONPs) from the ion preparation (CaNO_3). Formation and evolution of the CaO micro/Nps frequency bands at different time periods in the λ_{max} range of 380–480 nm in the methanol solution. This effect is attributed to unique vibrational alterations in electronic energy levels caused by the interaction between the incident electromagnetic spectrum and the electron clouds surrounding the surface of CaO micro/nano particles [34–36]. Moreover, research has shown that an absorption band can change to a higher or lower wavelength in an absorption spectra, depending onto the shape, size, aggregation state, and surrounding electrostatic environment of the standard particles [37–38]. Hence the change of bands would affect the creation as well as the sizes of CaO micro/nano particles [39]. Both band shifts were capture across the spectrum with increasing time relative to the mixing solution ratios. In the spectra the bands having the highest shift in absorption maxima were observed at 480 nm. Therefore, with increase in exposure time, the bandwidth and absorption maxima rise, which results in increase in CaONP size [40–41]. In addition to this, aggregation affects the reduction process leading to the synthesis of CaO micro/NPs, through the availability, contact and stability of the generated nanoparticles. This synthesis is highly important to ensure their efficiency in many applications such as the purification of groundwater fluoride, environmental cleanup and medicine delivery [42–43]. Ultimately, due to the reduction process of CaO micro-NPs, a noticeable improvement in the overall rate is noticed during the interaction betwixt the reaction mixture and the micro-remediation approach. The microremediation irradiation can accelerate the reaction medium, which is different from the conventional microremediation at room temperature, therefore reducing the energy consumption and providing the same growth condition for CaO micro/nanoparticles [44–45]. To summarise, the increase of CaO micro-NPs' volumes eventually leads to additional calcium ions being reduced on their surface, leading to the formation of stable CaO micro/micronano particles. Thus, the optimal reaction mixing ratio in respect to the reduction process is leading to an increase in the particle size of the CaO micro/nano particles.

Fourier-Transform Infrared Spectroscopy Analysis

The produced CaO micro/nano particles were analyzed, corresponding to a conventional FTIR spectroscopy range of 4000 to 400 cm^{-1} evaluation to demonstrate exactly how multiple components interacted with the resulting micro/nano particle. The comparative FTIR spectra of the CaO micro-NPs are displayed in Fig. The stretching vibrations of the stretch of different metabolites present in the spectra, and CaO stretches have been

accountable for the band in the spectrum that appears in the range of 1606 to 1316 cm^{-1} between C-O (carboxylic group) stretching vibrations were detected [45, 35], and CaO vibration bends were detected at 1000 cm^{-1} as well as clusters near 1795 cm^{-1} , 875 cm^{-1} , and 557 cm^{-1} . The bonding systems interacting with the chemicals, which lead to the creation of the micro/nano particles through reduction processes, determine the width and forms of these bands in spectra. The symmetric and asymmetric stretching vibrations of groups may be responsible for the strong peaks seen in the spectra [46-47]. This phenomenon could be explained by the existence of the functional group and a carboxylate ion. In addition to the bending vibrations of the groups, distinctive strong peaks were also seen in the spectra. These might be attributed to the asymmetric stretching mode. The existence, interaction, and movement of the above bands are significant. The findings suggest that in the produced CaO micro/nano particles, active chemicals are thought to function as decreasing, peaking, and stabilizing factors for Ca ions.

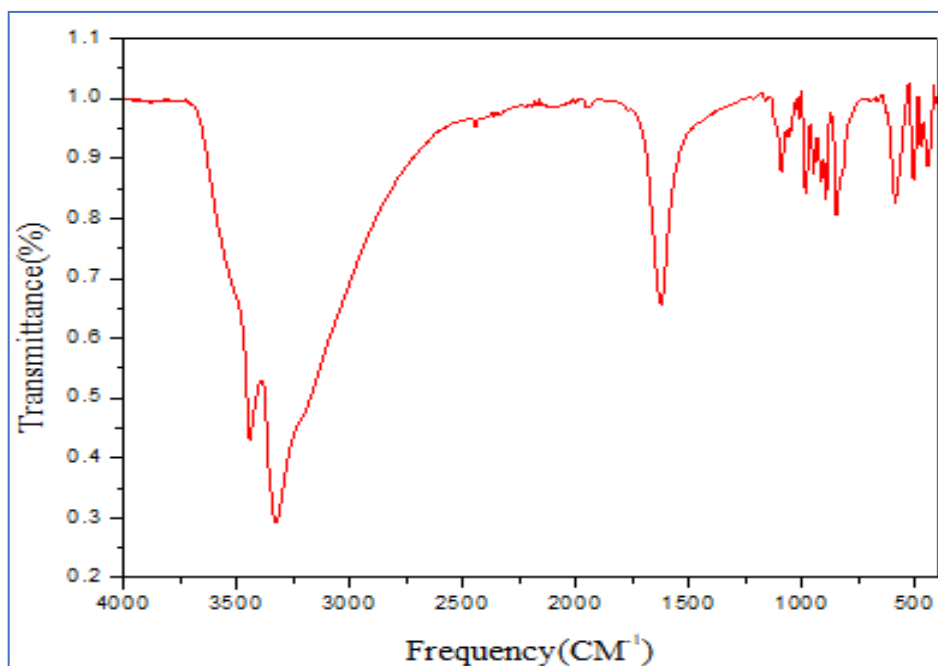


Fig. 6. FTIR spectra of CaO NPs

High-Resolution Transmission Electron Microscopy

HRTEM spectra (Fig. 7) confirming significant presence of calcium and oxygen corroborate the results obtained by SEM-EDS. The separate signals of Ca and O corroborate the presence of CaO on the surface. The elemental mapping of Ca and O in the given figure shows the CaO mixed oxide particles are uniformly dispersed in the outer coating layer. The HRTEM study of CaO is described in detail in the images, elemental mapping, and EDS spectra: Fig. 4 shows the CaO micro particles are closely packed, and in the higher resolution images the structures layered on top of each other further illustrate the extent of deposition and the anchoring of the metal oxides within the cavities. The micro/nanoparticle size within the nanoregime validated the microcrystalline structure of the NCs, consistent with the analytical findings. The HRTEM spectrum likewise shows large levels of calcium and oxygen in the NCs, in agreement with the SEM-EDS data. The measured calcium content confirmed the reported elemental makeup of the particles of the CaO remediation. In Fig. 7 is presented the region chosen for elemental mapping, which results indicate notably greater quantities of calcium and oxygen. The mixed ratio confirmed the homogeneous mixing of CaO micro/nano particles and the modified CaO mixed oxide surface in the cavity.

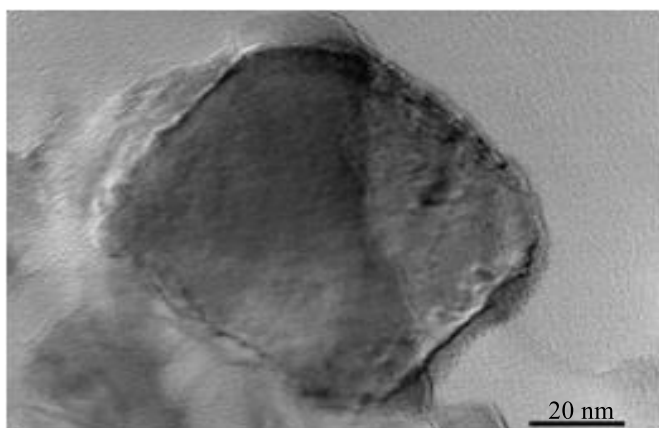


Fig. 7. HRTEM Image of CaO nanoparticle

5. Batch Adsorption Studies

Utilizing 1 g of calcium nitrate, 100 ml of groundwater and 250 ml conical flasks were used for batch adsorption testing. At room temperature, the flask was shaken, and by changing the pH of the solution from 1 to 7 while maintaining a sorbent dosage of 1 g/100 ml for a 1.30 hrs contact time, the impact of the starting pH was investigated, and 0.1 N NaOH was used to change the groundwater pH [48]. Then that the consequence of adsorbent dose and contact time on the sorbent's sorption capacity was investigated within the range of 1 to 6 g and 1 to 6 h [49-50]. Then the following formula was used to calculate the adsorption removal % of different physicochemical parameters and metals in samples from Pattipachgai village groundwater: % Removal = (Initial concentration – final concentration) / initial concentration x 100

$$\% \text{ Removal} = \frac{\text{Initial concentration} - \text{Final concentration}}{\text{Initial concentration}} \times 100 \quad (1)$$

Effect of Contact Time

The effect of contact duration on our material's and the final composite's adsorption capacity is shown in Fig. 8. It was observed that activated carbon had an adsorption rate of less than 4%. The literature predicts that activated carbon will have a lower adsorption capacity than our used material for the elimination of pollutants. The consequence of adsorbent dose and contact time on the sorbent's sorption capacity was investigated within the range of 1 to 6 g and 1 to 6 h. Following the time, the solution was filtered through Whatman filter paper no. 42, and the resulting filtrate was determined for elements like oxygen and calcium as well as physico-chemical characteristics [51-52]. Our used material, in contrast, exhibits a significant ability to remove Ca from aqueous solutions. This phenomenon occurs because the surface of calcium oxide and the oxygen atoms in its micro/nanoparticles provide additional adsorbing sites, which explain the increased adsorption capacity. The calcium can also interact with these sites electrostatically [53].

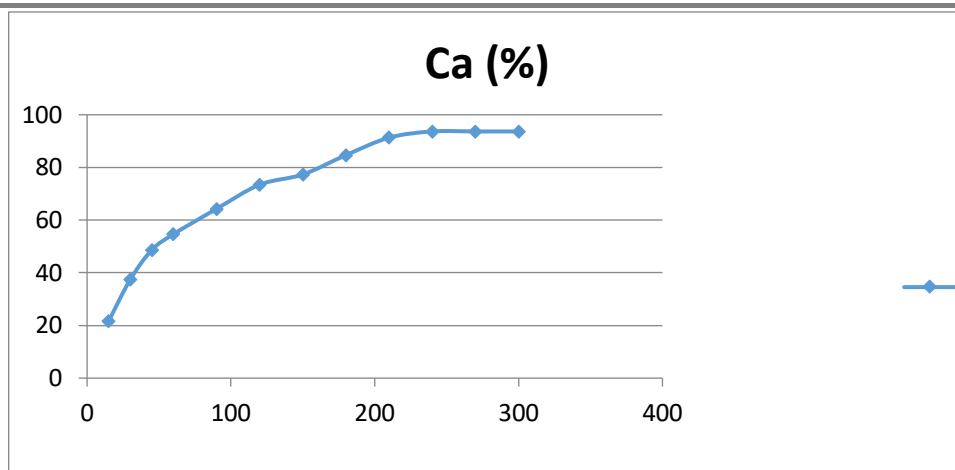


Fig. 8. The effect of contact time on the amount of Ca adsorbed on different.

Effect of pH

The effect of pH on the absorption of Ca onto the generated CaO was determined by assessing the absorption at pH values of 3, 4, 5, 6, and 7, as shown within Fig. 9. Then the composite was found to be able to successfully absorb Ca below pH 7 and above pH 3, as Ca did not adsorb to the CaO in firmly acidic environments. Figure 5 shows that pH 5 & 6 has the highest adsorption capacity. Adsorption isotherms are essential for comprehending the adsorption mechanism [54-55].

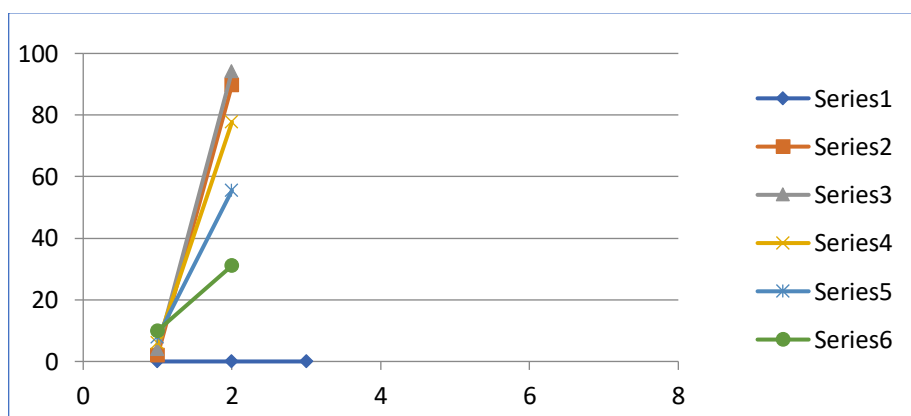


Fig. 9. The effect of Ph on the amount of Ca adsorbed on the CaO microstructure.

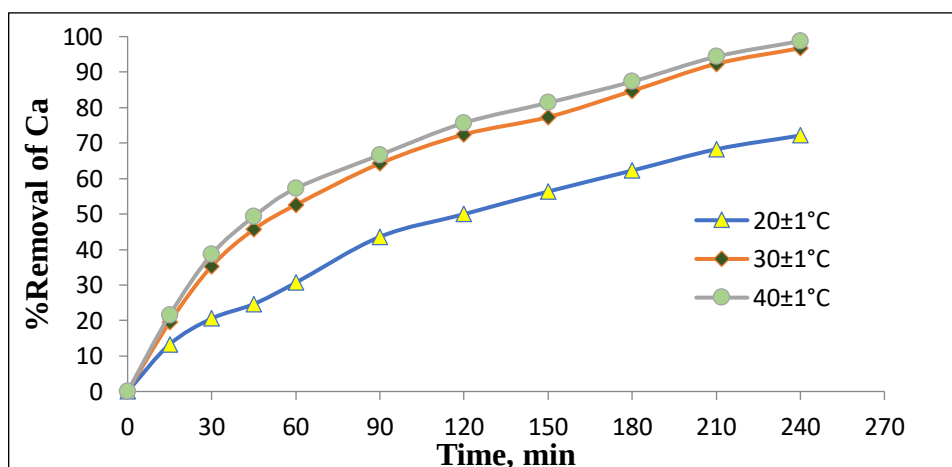


Fig. 10. Effect of temperature

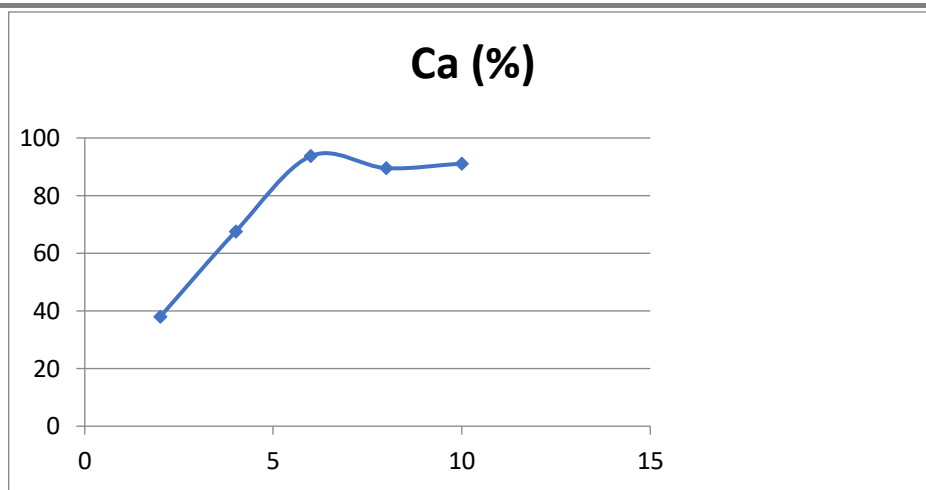


Fig. 11. The effect of dosage on the amount of Ca adsorbed on the miro/nano CaO

It becomes protonated and has a positive net charge on its surface in solutions with a pH lower than its PZC $\frac{1}{4}$ 4, which makes it unable to effectively adsorb Ca. Then, on the other hand, it may adsorb the positively charged calcium species when it is placed in solutions above its PZC $\frac{1}{4}$ 4 because deprotonation causes the net surface charge to become negative. Calcium exhibits as the neutral $\text{Ca}(\text{OH})_2$ species at pH 7, which has no desire to engage electrostatically with the positively charged CaO. Therefore, at pH 7, chromium adsorption is not facilitated by columbic or electrostatic interactions. At pH 5 and pH 6, the highest fluoride removal of CaO micro/nanoparticles was 85% and 87%, respectively [56-58].

Comparison table for Bulk and Nano material:

Parameters	Bulk-CaO	Nano-CaO
Adsorption(mg/g)	35.5	45.5
Removal Efficiency	66.3	78.0
Contact time(min.)	190	130
Dosage Required(g/L)	2.5	1.65
Surface Area(m^2/g)	14.8	86.1

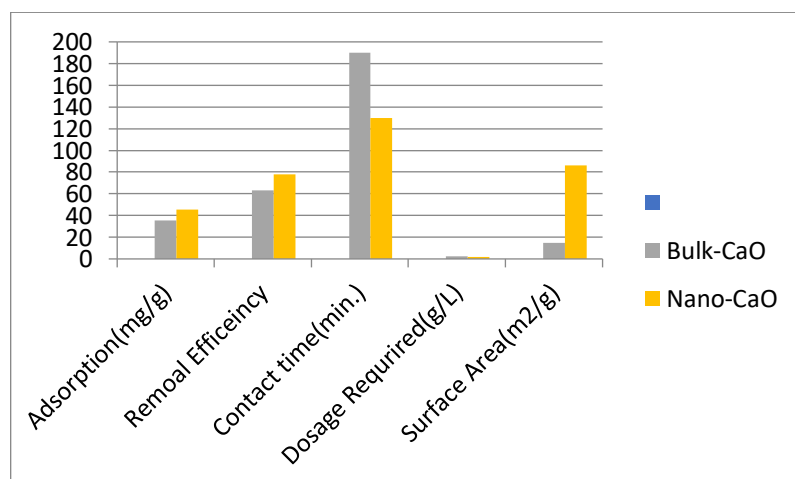


Fig. 12. Comparison graph of Bulk and micro-nano material

From this it is clear that our fabricated nano material are effective for remediation of fluoride in water sample.

CONCLUSIONS

In the present study, we report the fabrication of CaO micro/nano particles using precursor calcium nitrate oxide. After fabrication the powder sample was characterized by different techniques (FTIR, UV-Vis, SEM, EDS, and HRTEM). The results show the grain size of CaO micro/nano particles. The development of micro/nano particles seems to be an answer to the issue of fluoride contamination in groundwater. Consequently, major research efforts have been devoted to the development of sophisticated, low-cost, and sustainable functionalized sorbent materials and technologies with multifunctional capabilities for effective fluoride removal. In this study, a functionalized micro/nano remediation by calcium oxide micro/nano particles was specifically prepared for the simultaneous removal of fluoride from groundwater. The aim was to optimize the selectivity and effectiveness of fluoride removal from in the groundwater.

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Conflicts of Interest: The authors claim that there are no conflicts of interest.

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