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EFFECT OF ULTRASONIC SOUND AND DOPING IN THE PARTICLE ARRANGEMENT AND MORPHOLOGY OF NANO CALCIUM HYDROXIDE

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ABSTRACT

The calcium hydroxide nanoparticle was prepared by sonicated method and characterized by XRD, SEM, and FTIR, from that analysis we studied that decreases in the particle size are observed with an increase in the pH of the solution. In high pH the concentration of OH- is high, in this study, we used NH_4OH as a reducing agent which has low alkalinity, In low alkalinity the defused double layer is thick and the repulsion between the particles is strong. This repulsion stops the agglomeration and decreases the size of the particle.

Keywords: Calcium Hydroxide, Nanoparticle, XRD, SEM, Diamond, Match-2.

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INTRODUCTION

Nanoparticles are a significant product due to their use in a variety of industries, including environmental remediation, optoelectronics, sensors, and catalysis.[1-3]One of the most promising metal oxides is calcium oxide (CaO), which finds use in a wide range of processes including catalysis[4], doping addition to altering electric and dielectric properties[5], toxic waste remediation[6,7], CO_2 capture[8–10], desulfurization of flue gas and emission control agent in pollution[11], purification of hot gases[12], and so on. Calcium oxide is used in many different industries. Furthermore, calcium oxide is abundant in nature, cheap, and simple to make. Calcium oxide is used to purify chemicals and compounds like glucose, citric acid, and certain colors prior to further refinement.[13,14] In places where rainfall washes the calcium out of the soil, calcium oxide is used to balance out acidic soil. In electronics, calcium oxide is used as a desiccant in LED's.[13] Drugs are becoming more quantized and function as growth factors rather than just medicinal agents. Because of their nanostructures, calcium oxide nanoparticles are highly practical for use in drug delivery systems.[15] In the industrial setting, it's also employed as a dehydrating agent in the steel-making process, an absorbent, a water softener, a possible wastewater hydrogen regulator, and in fertilizers.[16] The size and form of nanoparticles determine their characteristics and uses.[5] High temperatures and the type of solvents used have an impact on the size, shape, homogeneity, and characteristics of the nanoparticles.[17–19]There is a great deal of control over the manufactured nanoparticles using solution-phase procedures.[5] Numerous techniques, including solgel[12], precipitation[18], the hydrogen plasma-metal interaction[11], sonochemicalsynthesis[5], thermal breakdown[6], etc., are used to create nanoparticles. In the current work, CaOH nanoparticles were synthesized, which are hydrophobic nanoparticles of calcium hydroxide doped with metal groups, and were first synthesized CaOH by a sonochemical synthesis. In an effort to determine the most effective formulation, Sodium dodecyl sulfate (SDS), a biodegradable and biocompatible surfactant, was utilized as an emulsifier in each NP. The effect of ultrasonic sound and doping in the formation of morphology where studied.

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EXPERIMENITAL

Preparation of Calcium Hydroxide

Preparation of $Ca(OH)_2$nanoparticles by sonicated method by using $CaCO_3$ as a starting material and NH_4OH as a reducing agent. In this reaction, Sodium dodecyl sulfate (SDS) and ethanol are used as surfactants. The starting solution is kept under 0°C for 24 hrs. Then NH_4OH is added under ultrasonic sound using the instrument in an ultrasonic homogenizer (Bio logics. Inc model 3000) under a 60% pulsar rate. After being sonicated for about 15 min, the transparent solution was obtained. The solution was removed from all organic substances by reducing distillation. Then it was treated at 80°C for one hour under vacuum condition.

Synthesis of Cao-Sm

Each 0.01 mol prepared CaOH and 0–5% $Sm(NO_3)_3 \cdot 6H_2O$ by weight were weighed and dissolved in 50 ml C_2H_5OH solutions under a sonicator. Subsequently, 0.01 mol tartaric acid in 50 ml C_2H_5OH solution was added to the solution to form gel precursors. Then, 1 M 20 ml NaOH solution in C_2H_5OH solution was added to the precursor with continuous sonication for 30 min. The synthesized gel was filtered, washed with distilled water, dried, and further calcined in air at 600 °C for 2 h.

RESULTS AND DISCUSSION

XRD

The XRD pattern of the sample shows in Fig.-1. The sharp peak of XRD shows the samples have a highly crystal nature. All peaks can be indexed according to the trigonal structure. The absence of additional peaks suggests no impurity contents within the empirical 3% detection limit of commercial XRD. The lattice constants were extracted and the unit cell volume was calculated. Using the formula,

$$\frac{1}{d^2} = \frac{(h^2 + hk + k^2) + \sin^2 \propto +2(hk + kl + hl)(\cos^2 \propto -\cos \propto)}{a^2(1 - 3\cos^2 \propto +2\cos^2 \propto)}$$

From Table-1, we confirm the cell parameter of $Ca(OH)_2$ is a=3. 5925 Å, c=4.908 Å The ultra-sonicated sound breaks the particles into atom levels so the arrangement of atoms gets controlled and the sample formed in the nano range also the size distribution is equal most of the particles are formed in the 60 nm range. The hkl values of all peaks are arranged in position and make a perfect crystal structure. Ultra-sonicated sound plays a main role in the arrangement of lattice. The space group of $Ca(OH)_2$ is $P^{-3}1m$.

Table-1: Cell Parameter for $Ca(OH)_2$

CellParameter	Å
A	3.5925
B	3.5925
C	4.9082

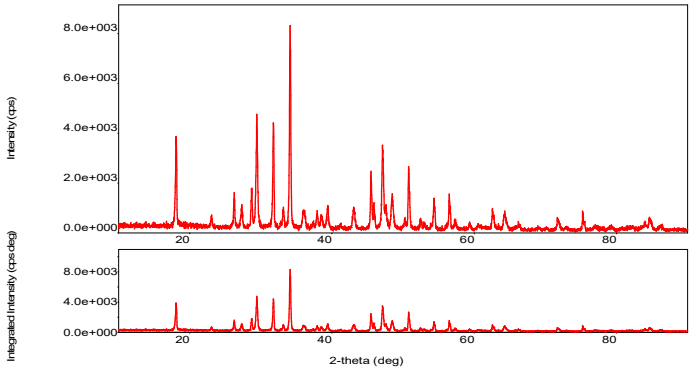

Fig.-1: XRD Pattern for $Ca(OH)_2$

Table-2: Atomic Site Positions for $Ca(OH)_2$

Ion species	X	y	Z
O	0.333	0.667	0.746
Ca	0.000	0.000	0.000
H	0.333	0.667	0.574

Effect of Ultra Sound in Particle Lattice Arrangement

When compared with NaOH, Ammonium hydroxide has low alkalinity, due to this the precipitation of particles is slow, and when ultrasonic sound is passed through the solution it breaks the particles. The slow particle precipitation and combined effect of ultrasound increase the zeta potential and decrease the particle size. Whenever the zeta potential increases the atomic flocculation or colloidal is decreased this leads to smaller size particles. Compared to previous studies size of the nanoparticle is small. This reducing agent and ultra-sonic sounds play an important role in the crystal lattice arrangement, with this combined effect the particles are arranged in a single array, this gives smaller size particles.

Table-3: hkl values of $Ca(OH)_2$

Angle [°2Th.]	hkl
18.097	001
28.694	100
34.098	011
36.03	022
45.869	012
50.778	110
54.318	111

Effect of Doping

In our study we used doping, with pure sample Sm were used as doping materials, the doped sample shows the cubic shape where the lattice parameter of the doped materials is changed. The lattice cell parameter of the doped sample is a = 8.066 Å and shows the Fd^{-3} space group the atomic distance of doped material is Ca-O = 1.7463Å, Sm-O = 0.0051Å. The doping of Sm is 34.5%. The 2 theta values of 56.23Å and 59.57Å are indicated for doping which is common to Sm, the doublet peaks show that doping exists in the sample because of lattice order displacement.

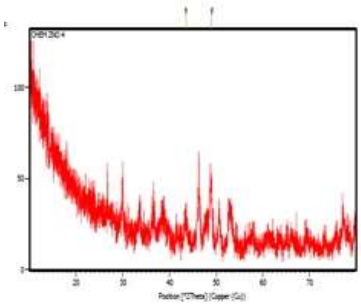

Fig.-2: Observed XRD Data for Doped Sample

SEM

The scanning electron microscope is used to find the shape and size of the nanoparticle. The most important morphology of the sample is studied using SEM. The SEM image of $Ca(OH)_2$ indicates the accurate morphology of Nanoparticles. The particles were formed in a spherical shape. The particlesare not equally distributed the size varies from 20-60 nanometers due to environmental factors like room temp atmospheric air moisture, etc., The morphology of the parent material was changed. When Sm was used as a doping material and CaOH was used as the parent material. The atomic radius of Ca is higher than Sm, so the doping takes place in a good manner. The lower atomic radius gives the best result in doping that leads to lattice disorder this lattice disorder gives the change in morphology.

FTIR

The sample shows adsorption in 498.44 cm^{-1}, 718.61 cm^{-1}, 1060.75 cm^{-1}, 1384.94 cm^{-1}, 1625.08 cm^{-1}, 2003.08 cm^{-1}, 2378.14 cm^{-1}, 2925.50 cm^{-1}, 3364.18 cm^{-1}, 3687.03 $cm^{-1}$and 3763.72 cm^{-1}. 1625

cm^{-1}appeared in the samples and was assigned to the stretching and bending vibrations of hydrogen-bonded surface OH groups of physically adsorbed water surface. The samples showed an absorption band at 498 cm^{-1} associated with C-H stretching. The peak arranged in 2952 cm^{-1} is associated with Ca-O-Ca bonding.

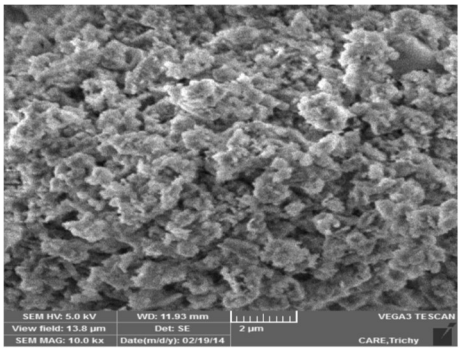

Fig.-3: SEM Images

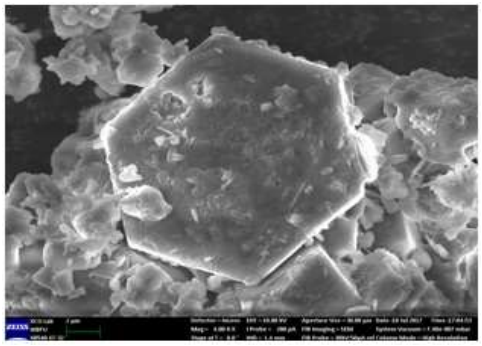

Fig.-4: SEM Images

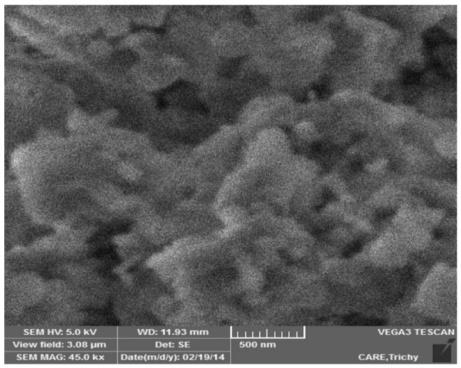

Fig.-5: SEM Images

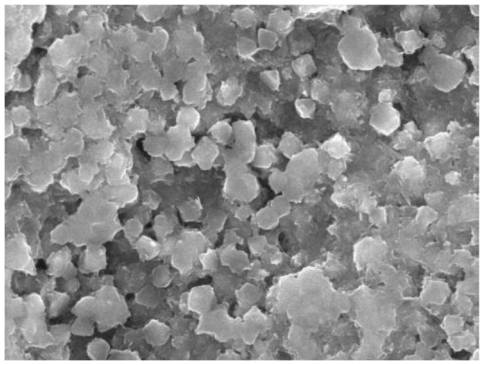

Fig.-6 Doped Sample

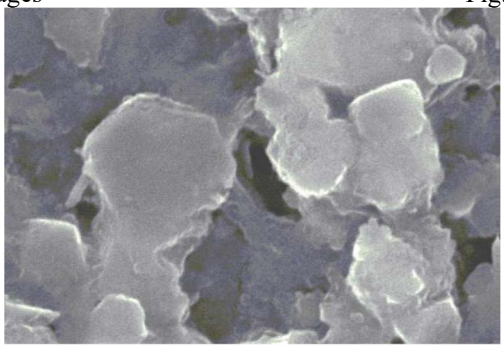

Fig.-7: Doped Sample

Fig.-3, 4 and 5: Represented for SEM Images of the Sample $Ca(OH)_2$
Fig.-6 and 7: Represented for SEM images of the Sample $Ca(OH)_2$ Doped Material

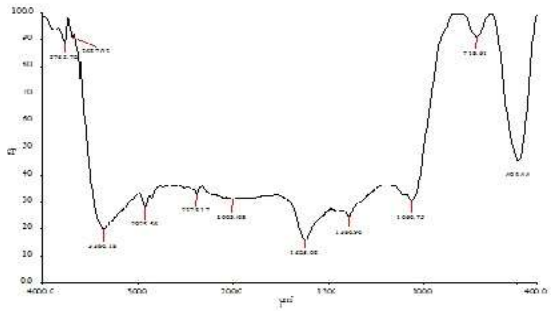

Fig.-8: FTIR Spectrum of the $Ca(OH)_2$

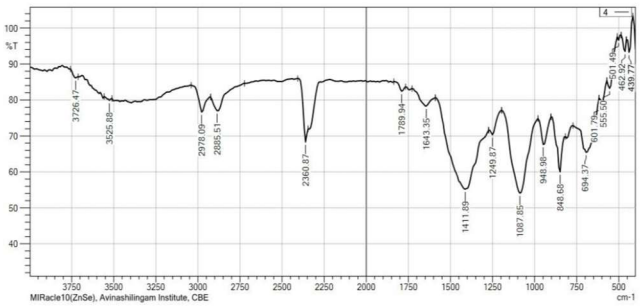

Fig.-9: FTIR Spectrum of Doped Sample

Table-4: FTIR Assignment for $Ca(OH)_2$

Wave Length	Function Group
3364 cm^{-1}	O-H bonding at hydroxyl groups
2925 cm^{-1}	Ca-O-Ca bonding
1625 cm^{-1}	OH bending
718 cm^{-1}	Ca-O bonding
498 cm^{-1}	associated with C-H stretching

CONCLUSION

The calcium hydroxide nanoparticle was successfully prepared by sonicated method and characterized by XRD, SEM and FTIR, from the analysis we concluded that a decrease in the particle size was observed with an increase in the pH of the solution[20] but we used NH_4OH as a reducing agent it has low alkalinity. In low alkalinity the defused double layer is thick and the repulsion between the particles is strong. This repulsion stops the agglomeration and decreases the size of the particle. When the ultrasound is introduced into these method vast amounts of micro air bubbles are formed due to ultrasonic cavitation.[23]

These micro air bubbles can disturb the ordered crystal sequence this is shown in the Ca-O distance as 2.4182 Å and O-H distances as 0.8442 Å and bond angles are 95.872and 67.863 respectively. From SEM images we concluded that particles are formed in spherical form and equally distributed in between the size of 20-60nm. This is due to the ethanol mixed with water as a surfactant. Because, ethanol-water mixture have less volume than the sum of their individual component's. The addition of even a few percent of ethanol to water sharply reduces the surface tension of water.

The reduced surface tension helps smaller-sized particles. From FT-IR we concluded that the functional groups present in the nanoparticle, the adsorption in 3364 cm^{-1} confirms the presence of the OH group in the particle; the OH group plays an important role in the formation nano nano-sized particles. The adsorption in 2925 cm^{-1} represents Ca-O-Ca bond presence and 718 cm^{-1} represents Ca-O bonding. Overall, the sample is confirmed it's in nano size, spherical shape, and mostly equally distributed.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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