Producing High Purity of Metal Oxide Nano Structural Using Simple Chemical Method

M A Aseel¹, F H Itab¹ and F M Ahmed²

¹Physics Department, College of Science, Al-Mustansiryah University, Baghdad, Iraq.
²Physics Department, College of Education for Pure Science, University of Baghdad, Baghdad, Iraq.

e-mail: aseelalaziz@uomustansiriyah.edu.iq

Abstract. Calcium oxide nanoparticles and nanorods were prepared by using chemical method at two Molarities. The field emission scanning electron microscopy (FESEM) images suggested the formation of nanoparticles (50-60) nm and nanorods with diameter about 500 nm and length 3µm. The average surface roughness of CaO thin film was imaged by carried out atomic force microscope (AFM) and roughness found to be between (3.52-5.46) nm. XRD patterns show that the CaO was cubic crystalline nature and average grain size was found to be 70 nm. The direct optical band energy gap of all conditions to be ranged between (3.5-3.7) eV and thermal gravimetric analysis (TGA) was studied. Photoluminescence (PL) properties were investigated and found to have two emission bands.

1. Introduction
Calcium oxide (CaO) is an important inorganic material with a good physical properties and their power to resist hard process conditions. CaO is of special attention as they are not only constant under hard process conditions but also mostly note as safe material to human beings and animals [1]. CaO is a highly important wide-band gap insulator, least weight, perfect chemical resistance, rise secondary electron emission, rise thermal stability [2] and has the merit of non-toxicity and of being ready from easily available and economical portent and subsequently has large potential as solid bactericidal material [3]. Hence CaO is important of potential applications in different fields, such as catalysts, water treating, gas sensing and superconductors [4]. CaO nanomaterial with high surface area is important attention in adsorption materials. Recently all attempt have been studied CaO with increase surface area in different morphologies such as nanoparticles, nanorods and nanowire [5, 6]. Through different methods, chemicals methods [7], chemical bath depositions [8] and sol-gel [9]. The real dimensions such as diameters of (10-100) nm and lengths of a small micrometers supply a structural devices that can be very small, which have enhance for applications in biological devices, security monitoring and defence technology [10]. For the present work, we have studied CaO nanostructures by chemical methods under two molarities, result their simplicity, cost effective and low reaction temperature.

2. Experimental Part
Calcium oxide nanostructures were prepared by chemical technique at different molarities (0.4 and 0.6) mol using calcium chloride dihydrate (CaCl₂.2H₂O) was add to 100 ml of distilled water and use
sodium hydroxide (NaOH) and ureaCO(NH₂)₂ were added to 25 ml of distilled water with continuous stirring for 4 hr. and then the white precipitate was filtered and washed two times with distilled water and methanol and dried at 100°C. Finally the white colored material was calcinations at 700°C for 5 hr. in a vacuum oven to obtain CaO nanostructure. Morphology of CaO nanostructures were analyzed with field emission scanning electron microscope (FESEM) and Atomic Force microscope (AFM). Thermo gravimetric analysis (TGA) of CaO were investigation, crystal structure analysis was carried out by X-ray diffractometer (XRD-Philips) using (cuKα) radiation (λ= 1.5406 Å), a complete 2θ scan was made between 20° to 70°. From XRD data the average grain size of particles was calculated by Scherrer equation. The optical absorption of the colloidal CaO nanostructures were measured using spectrophotometer (CARY, 100 CONC plus, UV-Vis-NIR, Splitbeam Optics, Dual detectors) in the range of (200-900) nm.

3. Results and Discussion
Thermal Gravimetric Analysis spectrum was used to analyze the thermal behaviour of the CaO nanomaterial. Figure 1 shows the relation between the weight losses (%) with the thermal temperature. The another major weight loss was show in the temperature range of (300–450) °C concerning to decomposition of Ca(OH)₂ after calcinations at 700°C. From TGA the average grain size of particles was calculated by Scherrer equation. The optical absorption of the colloidal CaO nanostructures were measured using spectrophotometer (CARY, 100 CONC plus, UV-Vis-NIR, Splitbeam Optics, Dual detectors) in the range of (200-900) nm.

![Figure 1. TGA spectra of CaO.](image1)

XRD pattern of the CaO nanomaterial at 0.4 M is shown in figure 2. It is seen that peaks appear at 32°, 37.02°, 54° and 64° corresponding reflecting planes are (111), (200), (220) and (311) respectively.

![Figure 2. XRD pattern on CaO.](image2)
The diffraction patterns detect a good polycrystalline quality without any foreign material and can be indexed to cubic crystalline structure according to JCPDS file number (37-1497). There is not any other clear phase in the XRD pattern which means high purity of the obtained CaO nanoparticles. The average particle size of CaO nanoparticles can be estimated using Scherrer equation was about 70 nm and same as reported by [12]:

\[ \text{Crystallite size} = \frac{0.94 \lambda}{\text{FWHM} \cos \theta} \]

Where \( \lambda = 1.54\text{Å} \) is the wavelength of X-ray, FWHM is the broadening of the diffracted peak at half maximum and \( \theta \) is the Bragg angle.

The FESEM of CaO nanomaterial studied by chemical method at different molarities (0.4 and 0.6) is shown in figure 3.

As previously mentioned, increased molarities of nanomaterial that effect on phase formation and morphology of acquired powder. It is clear in sample (a) at 0.4 M was consisted of uniform and smooth CaO nanoparticles with average grain size around (50-60) nm. In sample (b) at 0.6 M was consisted of CaO nanorods are exhibited hexagonal surface, the mean diameter to be about 500 nm and mean length is about 3 µm.

Figure 4 shows the formation of CaO nanostructure at different molarities (0.4 and 0.6). AFM micrograph proves that the grains are uniformly distributed within the scanning area (2000×2000) nm with individual columnar grains extending upwards.
This surface topography is important for many applications such as for applications in optoelectronic devices, solar cells, gas sensors and catalysts. The CaO nanostructures have spherical shaped with good dispensability, homogeneous grains aligned vertically. The roughness of CaO increasing with the molarities, this was perhaps due to scattering mechanism in which affect in the particle size. Symmetry of the particle size of CaO is important because the nanoparticle size influence to the electrical conductivity of CaO [2]. The estimated values of root mean square of surface roughness average and average grain size are listed in table 1.

| Molarities (mol) | Grain Size (nm) | Roughness Density (nm) | Root Mean Square (nm) |
|------------------|-----------------|------------------------|-----------------------|
| 0.4              | 85.93           | 3.52                   | 3.52                  |
| 0.6              | 100.2           | 5.46                   | 7.41                  |

The Uv-Vis spectrum of CaO Nanostructures at room temperature with different concentration deposited on quartz substrate is shown in figure 5. The regime absorption peak at 230 nm can be associated to band transition in the band gap region [8]. Absorption of CaO was found to be increased with an increase of molar concentration.

![Figure 5](image1.png)

**Figure 5.** Optical absorption for different molarity of CaO nanostructures vs. with wavelength.

The optical band gap of CaO was determined from $(\alpha h \nu)^2$ vs. $(h \nu)$ plot as shown in figure 6 and values have been obtained to be 3.7 eV and 3.5 eV for the molarities 0.4 and 0.6 mol respectively. The internal figure shows the optical band gap decreases with increase of molar concentration due to the increase of density of localize state in the conduction band [11].

![Figure 6](image2.png)

**Figure 6.** The energy gap $(E_g)$ CaO nanostructures with different molarities (0.4 and 0.6) M.
Photoluminescence spectrum of these CaO nanostructures is studied. As shown in figure 7, CaO nanoparticles and nanorods correspond to different PL properties, the properties was recorded at 325 nm as an excitation wavelength.

The CaO nanostructures have two strong emission bands, the first peak observed at 440 nm due to exciton emission by the radiative annihilation. The second peak in the green region of the visible spectrum at 580 nm resulted by radiative recombination of an electron in the conduction band [8]. It is found that the intensity of the green visible emission is less than intensity of exciton emission from CaO nanostructures because the green visible have more oxygen vacancies. However, figure show that the intensity of CaO nanorods less than CaO nanoparticles because CaO nanorod have higher crystallization, less oxygen vacancy concentration and the diameter of particles increase than the CaO nanoparticles [13].

4. Conclusions
In this work pure CaO were prepared by chemical method with different molarities (0.4, 0.6) M. FSEM result show the formation two kinds of CaO nanoparticles with size in the range of (50-60) nm and CaO nanorod with a diameter of 500 nm and length about 3µm. The PL property show that the two types of bands, the first is an exciton emission in UV region and the second in the green region. The XRD result confirmed the high purity crystallinity of the CaO.

5. References
[1] Jagadesh D, Prashantha K and Shabadi R, " Star shaped sucrose capped CaO nanoparticles from Azadirachta indica: A novel green synthesis", synthesis and Reactivity in Inorganic and nano-metal chemistry, ISSN: 1553-3174, (2016).
[2] Albuquerque E L and Vasconcelos M S, "Structural Electronics and optical properties of CaO", Journal of Physics: Conference series 100, (2008).
[3] Kazemi H, Zandi K and Momenian H, " Sonochemical synthesis of Ca(OH)2 nanoparticles and its application in preparation of MWCNT- paraloid nanocomposite", Journal of nanostructures, vol. 5, (2015), 25-32.
[4] Mirghiasi Z, Bakhtiar F and Darezereshki E, " preparation and characterization of CaO nanoparticles from Ca(OH)2 by direct thermal decomposition method", Journal of industrial and Engineering chemistry, vol. 20, (2014), 113-117.
[5] Tahmasian A, Morsali A and Woojoo S,"Sonochemical synthesis of a one- dimensional Mg metal-organic framework: Anew precursor for preparation of MgO one-Dimensional nanostructure",Journal of Nanomaterials,3134567,(2013),7.
[6] Bult A R, Ejaz S, Barom J C, "CaO nanoparticles as a potential drug delivery for biomedical
application”, Digest journal of nanomaterials and Biostructures”, vol. 10, No. 3, (2015), 799-809.

[7] Amin M A and Morsali A,” Ultrasonic-assisted synthesis of Ca(OH)$_2$ and CaO nanostructures”, Journal of Experimental nan science, vol. 5, No.2, (2010), 93-105.

[8] Nirmala P N, Suresh, et.,” Influence of annealing temperature on optical properties of CaO thin film”, International Journal of Recent scientific Research, vol. 4, Issue 4, (2013), 425-427.

[9] Darcanova O, Beganskiene A and Kareiva A, ”Sol- gel synthesis of calcium nanomaterial for paperconsrvvation”,Chemija, vol. 26, No. 1, (2015), 25-31.

[10] Xing Tang Z, Claveau D, Corcuff R, ”Preparatuion of nano- CaO using thermal- decomposition method”, Elsevier, vol. 62, (2008), 2096-2098.

[11] Xing Tang Z, ”Sonication – Assisted preparation of CaO nanoparticles for Antibacterial agents”, Quim Nova, vol. 7, (2013), 933-936.

[12] Sundrarajan M, Suresh J, ”A comparative study on Antibacterial properties of MgO nanoparticles prepared under different calcination temperature”, Digest Journal of Nanomaterial and Bio structures, 7,3,P.983-989,(2012).

[13] Zhang H, Cao T and Cheng Y, ” synthesis of nanostructured MgO powder with photoluminescence by plasma- intensified pyrophdrolysis process of bischofite from brine”, DE Gruyter, Green Process synth, vol. 3, (2014), 215-222.