

Synthesis and Characterization of Porous Calcium Oxide Nanoparticles (CaO NPS)

A.S. Balaganesh, R. Sengodan, R. Ranjithkumar, B. Chandarshekar

Abstract: Calcium oxide nanoparticles (CaO NPs) gain great value in the areas of energy storage and drug delivery systems. Due to good porosity it finds its part in storage systems and its biocompatibility earns it a good value in drug delivery and gene transfection. In this present work, calcium oxide nanoparticles are prepared by means of simple precipitation method. Thus prepared particles are subjected to morphological, size and structural analyses. The X-ray diffraction studies revealed the polycrystalline nature of CaO nanoparticles. The SAED pattern confirms the polycrystalline nature. Transmission electron microscope shows that the size of the particles varies between 80 nm to 190 nm which is in good agreement with particle size analysis results.

Keywords: CaO NPs, Precipitation, XRD, TEM

I. INTRODUCTION

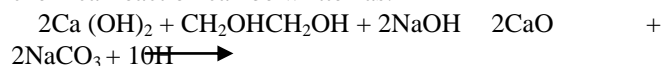
Application in various fields like catalysis, optoelectronics, sensors and environmental remediation [1–3] made nanoparticles a substantial one in production. Calcium oxide (CaO) is one of the promising metal oxide having many applications such as catalyst [4], dopant added to modify the electric and dielectric properties [5], remediation agent for toxic wastes [6, 7], for CO₂ capture [8–10], desulfurization of flue gas and emission control agent in pollution [11], purification of hot gases [12] etc. Calcium oxide is a high volume chemical, finds applications in numerous industries. In addition, calcium oxide is plenty in nature, inexpensive and easy to produce. Compounds and substances such as citric acid, glucose and certain dyes are purified using calcium oxide, before further refinement [13, 14]. Calcium oxide is used for balancing out acidic soil and is used in areas where rainfall washes the calcium from the soil. Calcium oxide finds its part in electronics as a desiccant in LEDs [13]. Drugs have grown beyond therapeutic agents to growth factors and have turned more quantized. Calcium oxide nanoparticles with its nano structures are very feasible to the applications in Drug Delivery Systems [15]. It is also used industrially as a dehydrating agent in the creation of steel, an absorbent, as a water softener, as a potential hydrogen regulator for waste water and in fertilizers [16]. The properties and applications of nanoparticles depends on their size and morphology [5]. The effects on the size, shape, uniformity and properties of the nanoparticles are by high temperature and nature of solvents [17–19]. Solution-phase methods provide a large degree of control over the nanoparticles prepared [5]. CaO

nanoparticles are prepared by various methods such as sol-gel [12], precipitation [18], hydrogen plasma-metal reaction [11], sonochemical synthesis [5], thermal-decomposition [6] etc.

II. EXPERIMENTAL TECHNIQUES

A. Preparation of CaO NPs

Initially, 3g of calcium hydroxide (Ca(OH)₂) was dissolved in 12.5 ml of ethyleneglycol and stirred vigorously and then 1 g of sodium hydroxide (NaOH) was added into the mixture. The solution was left to settle down for 5 hours after 10 minutes of sonication. The precipitate was filtered and obtained precipitate has been repeatedly washed with deionized water for 5 times and then calcined in 100°C, subsequently. Finally, calcium oxide nanoparticles of different size were obtained by calcining at 800°C. The chemical reaction can be written as:



B. Characterization

FTIR analysis was carried out by using [THERMOSCIENTIFIC Nicolet IR10] spectrometer. The XRD was recorded by using [RIGAKU Ultima IV] X-ray diffractometer. The surface morphology of CaO NPs was observed by using [JEOL JSM – 6390LV] scanning electron microscope, composition was analysed by using EDAX [JEOL JSM – 6390LV]. Size, shape and morphology were studied by using Transmission Electron Microscope [JEOL JEM 2100].

III. RESULTS AND DISCUSSION

Figure 1 shows the FTIR spectrum of CaO NPs. The broad absorption band around 1480 cm⁻¹ and 3600 cm⁻¹ have attribution to the surface of the sample containing water molecules, due to way of handling while spectrum recording [21]. The carbonation of CaO nanoparticles are observed in 1400–1500cm⁻¹ broad band and the narrow band around 750cm⁻¹ is due to C–O bond [6, 11, 17]. The characteristic vibration of Ca–O is intensive around band 600 cm⁻¹ [4]. The presence of atmospheric CO₂ is noticed in small peaks around 2350 cm⁻¹ [22].

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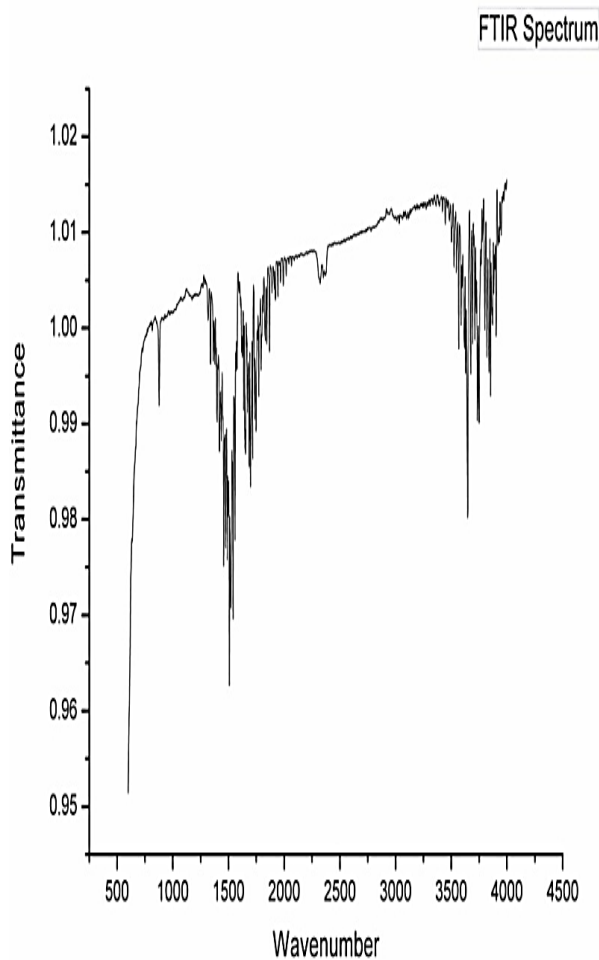


Figure 1 FTIR spectrum of CaO NPs

In order to prove that whether the prepared sample is CaO or not and to study the structure of the nanoparticles XRD analysis was performed and obtained XRD pattern of the sample is shown in Figure 2 which can be inclined to CaO (JCPDS card no.s 00-037-1497). The intense peaks observed at $2\theta = 28.5^\circ, 34.5^\circ, 47^\circ, 54^\circ, 63^\circ$ and 64° are respectively correspond to (111), (200), (220), (311), (222) and (400) orientation planes. The sharp peaks in the XRD pattern indicate the crystalline nature of CaO nanoparticles. No other characteristic peaks were observed. Thus the presence of pure CaO is observed in the as prepared particle. Crystallite size in the prepared sample was calculated by using the data from the XRD pattern by using the Scherrer formula,

$$D = \frac{k\lambda}{\beta \cos \theta}$$

Where,

D = crystallite size

λ = wavelength of the X-rays produced in the machine

β = width of a peak at half of its intensity

θ = angle of the corresponding peak

k = shape factor

The resulting crystallite size was evaluated to be about 51 nm.

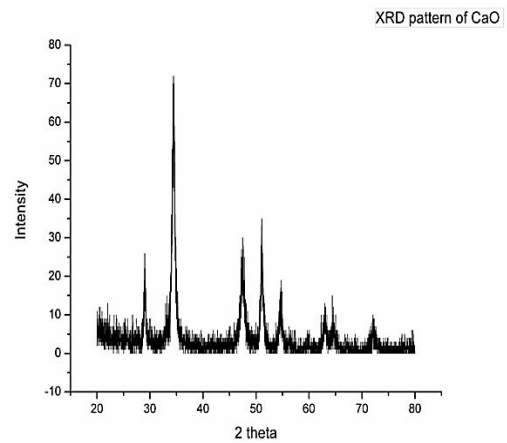


Figure 2 XRD pattern of CaO NPs

Figure 3 shows the SEM image of CaO nanoparticles. The bright areas of the picture reveal high emission of secondary electrons when exposed to electron beam of SEM. This is due to high surface to volume ratio in those areas [24]. The SEM morphology revealed the agglomeration of CaO nanoparticles. It can be seen from the micrograph that the synthesized sample composed of grains with no regular shape. The average grain size of CaO nanoparticles varies from few nm to 0.5 microns. The interaction between nanoparticles with large surface area and high surface energy results in agglomeration [12]. Individual particles seem to be nano sized plate like crystals and they partially fused to form hard agglomerates.

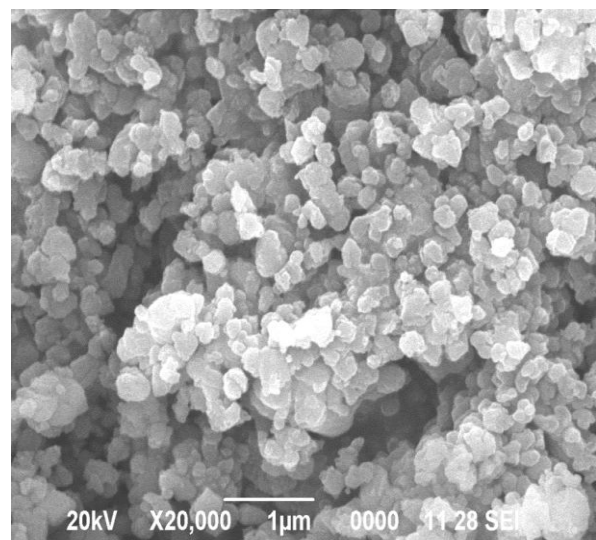


Figure 3 SEM images of CaO NPs

The observed micropores can be ascribed to the release of the CO_2 from the CaO microspheres. The presence of pores in the CaO microsphere would present efficient catalytic activity in biodiesel production by transesterification reaction from vegetable oils. Easy separations from the biodiesel products are due to advantages of CaO nanoparticles such as high surface area, uniformity, porosity and large size.

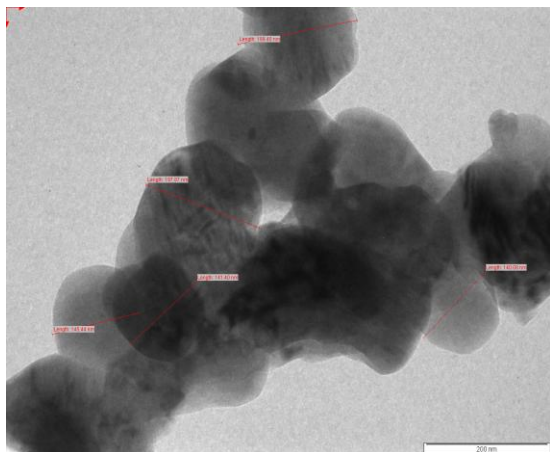


Figure 4 TEM image of CaO nanoparticles

The pores present in the CaO microspheres could effectively be used as a carrier in Drug Delivery Systems (DDS). Figure 4 below shows the TEM image of the prepared CaO nanoparticles. The size of nanoparticles range between 80 nm and 190 nm. It also revealed the agglomeration of nanoparticles. The X-ray diffraction and TEM studies revealed the polycrystalline nature of CaO nanoparticles [23].

IV. CONCLUSION

Porous calcium oxide nanoparticles were obtained by simple and cost effective precipitation method. The X-ray diffraction studies revealed the polycrystalline nature of CaO nanoparticles. The scanning electron microscope images revealed that the synthesized particles are agglomerated. Transmission electron microscope shows that the size of the particles varies between 80 nm to 190 nm. The obtained results indicate that porous CaO microsphere can be used in transesterification reaction of biodiesel production from vegetable oils and it can be used as effective drug carrier in DDS due to its bioactive and biocompatible nature.

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