

Green synthesis of calcium oxide nanoparticles at different calcination temperatures

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nanoparticles

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Sofiya Bano and Sandhya Pillai

*Department of Nanotechnology, Christian College of Engineering and Technology,
Bhilai, India*

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Abstract

Purpose – The purpose of this paper is to prepare calcium oxide (CaO) nanoparticles by green synthesis technique and study its structural, morphological and optical properties. The prepared samples were calcined at, 200°C, 300°C and 500°C, and the variation in the properties at different temperatures were investigated.

Design/methodology/approach – Green approach has been used in the present work to synthesise the CaO nanoparticles using Murraya Koenigii leaf (curry leaves) extract as a capping agent. This technique involves the use of nontoxic reagents and natural products derived from various parts of plants.

Findings – Studies reveal that CaO nanoparticles with good optical properties can be synthesized successfully by green approach. The optical absorbance spectra show a broad absorption peak around 400–500 nm. FTIR studies confirm the presence of different functional groups that help in the stabilization of CaO nanoparticles. PL emission spectra show a high intensity emission peak at around 231 nm in addition to peaks at 375 nm and 400 nm. XRD studies show planes of CaO cubic phase. SEM images show a nonuniform distribution of spherical particles along with some clusters. EDX spectra confirm the presence of calcium and oxygen.

Practical implications – CaO nanoparticles have wide applications in optical devices as well as in phototherapy. It is thus interesting to devise new and eco-friendly techniques to synthesise these nanoparticles to suit these applications.

Originality/value – This work would provide a new insight into the preparation of different metal oxide nanoparticles by a very simple and cost-effective green technique without the use of toxic reagents.

Keywords CaO nanoparticles, Green approach, Capping agents, Murraya koenigii leaf extract

Paper type Research paper

Introduction

Technology in the twenty-first century requires miniaturization of devices into nanometer sizes. Nanotechnology is a specialised branch of science in which materials can be manipulated at nanometer size scale. As nanomaterials possess large surface area and consequently higher surface energy, they behave like an atom. These nano-sized materials have large surface-to-volume ratios in comparison with the bulk material. The bulk materials have constant physical properties, and these limit their applications in many fields. When bulk materials are manipulated in nano scale, they show enhanced and unique properties which are due to their size and morphology. Hence, they have wide applications in many fields like material science, photochemistry, electronics, space, energy and robotics (Kanude and Jain, 2017; Sorbiun *et al.*, 2018). Nanoparticles are more chemically reactive, make diffusion faster, are also feasible at lower temperatures and therefore exhibit enhanced performance in various fields like optical applications, catalysis etc (Gaffet, 2011; Siroki *et al.*, 2017; Kumar *et al.*, 2018). Metallic nanoparticles (MNPs) have attracted great



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attention in recent years because they possess unique characteristics such as optical properties, high surface area, surface plasmon resonance, mechanical strengths, low melting point, magnetic properties and good antibacterial properties and hence have vast applications in different areas such as catalysis, photography, in medical field as anticancer and antimicrobial agents, biotechnology and agriculture. Inorganic metal oxide nanoparticles like CaO have distinct features, are safe, stable and possess multifunctional properties (Kumar *et al.*, 2018; Ashok *et al.*, 2014; Balaganesh *et al.*, 2018). CaO is one of the most promising metal oxides, less expensive and easy to produce and hence finds application as catalysts in LEDs, remediation agent for toxic wastes, purification of hot gases, for CO₂ capture etc (Sorbiun *et al.*, 2018), additive in refractory (Mirghiasi *et al.*, 2014), doping material (Alavi and Morsali, 2010), pollutant emission control and flue gas desulfurization (Liu *et al.* (2010)). As it possesses unique structural and optical properties, CaO can be used as a potential drug delivery agent (Abraham and Sarathy, 2018), in photodynamic therapy (PDT), photo-thermal therapy (PTT), biomedical assessments like biodistribution, biolabeling (Butt *et al.*, 2015) and synaphic delivery of chemotherapeutic agents (Aseel *et al.*, 2018). Considering these applications of CaO nanoparticles, an eco-friendly, nontoxic and cost-effective method for synthesis of these nanoparticles would be of much interest.

There are various physical and chemical techniques for synthesis of nanoparticles such as microplasma-assisted (Lin *et al.*, 2018), laser ablation (Becker *et al.*, 1998), sonochemical (Okitsu *et al.*, 2005), coprecipitation (Nande *et al.*, 2018), chemical reduction method (Guzmán *et al.*, 2008) etc. But these conventional methods lag because of their various limitations like using toxic chemicals such as polyvinyl pyrrolidone (PVP), hydrazine monohydrate etc. which acts as capping agents. This leads to toxicity in the environment and hinders their applications especially in biomedical field. Also their high cost, high pressure and temperature are other disadvantages in the synthesis of nanomaterials. Keeping these limitations in mind, a novel synthesis technique has been introduced, i.e. green synthesis of nanomaterials which is more beneficial than chemical synthesis methods (using chemical capping and reducing agents). Green nanotechnology is gaining importance in recent times as an alternative efficient, cost-effective and eco-friendly technique for synthesis of metal oxide nanoparticles. Green synthesis is nontoxic and gives high yield of products. For the synthesis of these environmental-friendly nanoparticles, many natural capping and reducing agents are being used such as plant, fungi, algae etc. Plant extract-mediated green synthesis of nanoparticles is cost effective, gives natural capping and reducing agents, provides stability and also enhances rate of reduction to the synthesized nanoparticles. These plant extracts contains many bioactive compounds like sterols, enzymes, lignins, amino acids which act as a reducing and capping agent that reduces metal ions (Vani *et al.*, 2017, Kalpana and Rajeswari, 2018, Some *et al.*, 2018, Khatoon *et al.*, 2017, Ramola *et al.*, 2019).

Murraya Koenigii, generally known as curry leaves, belongs to Rutaceae which represent more than 150 genera and 1,600 species. It is used as flavoring agents in curries and has many applications especially in the field of medicine. It possesses good antibacterial, antioxidant, antidiabetic properties which are of great importance in medicine (Kanude and Jain, 2017). Hence, in the present work, Murraya Koenigii leaf extract was chosen as a natural capping agent for the synthesis of CaO nanoparticles, which has not been attempted earlier.

The paper reports the synthesis of CaO nanoparticles by green approach using Murraya Koenigii leaf extract, at three different calcination temperatures – 200°C, 300°C and 500°C. The optical, structural and morphological properties of the CaO nanoparticles were investigated, and the effect of calcination temperatures on the properties of the nanoparticles was studied.

Experimental details

Preparation of Murraya Koenigii leaf extract

For the preparation of leaf extract, 50 gm of *Murraya Koenigii* leaf is washed with distilled water and then boiled for about half an hour in 100 ml distilled water to get its extract. The solution was filtered using Whatmann filter paper to get *Murraya Koenigii* leaf extract.

Synthesis of calcium oxide nanoparticles using leaf extract

10 ml of 1M calcium chloride solution and 10 ml *Murraya Koenigii* leaf extract were taken, and the solution was stirred using a magnetic stirrer for half an hour to which 10 ml of 1M NaOH solution was added dropwise while stirring. The precipitate was then filtered, and the filtrate was allowed to dry in an oven for one hour at 90°C. The dried precipitate was repeatedly washed with distilled water to remove the basicity of the solution. To get calcium oxide nanoparticles, calcinations were done in a muffle furnace at 200°C, 300°C and 500°C for three hours.

Characterization techniques

XRD studies were performed at NIT Raipur using PANalytical 3 KW X'pert powder-multifunctional X-ray diffractometer. SEM and EDX studies were also carried out at NIT Raipur using ZEISS EVO 18 scanning electron microscope and INCA 250 EDS with X-MAX 20 nm detector. Optical absorbance spectral studies were done using ELICO-SL210 UV-VIS spectrophotometer. PL studies were done at Pandit Ravishankar University Raipur using RF-5301PC SHIMADZU Spectrofluorophotometer. FTIR studies were also performed at Pandit Ravishankar University Raipur using Bruker Alpha FTIR spectrometer.

Results and discussion

XRD studies

The X-ray diffractograms of CaO nanoparticles calcined at different temperatures are presented in Figure 1(a), (b) and (c), and the corresponding data are enlisted in Tables 1–3 respectively. The observed diffraction peaks have been indexed using standard JCPDS NO.77-2376 corresponding to CaO cubic phase. The peaks with 2θ value 31.79°, 37.86°, 64.80°, 66.94° and 79.51° correspond to the crystalline planes (111), (200), (311), (222) and (400) respectively in the case of CaO nanoparticles at 200°C calcination temperature. Peak positions at almost the same 2θ values were observed for CaO nanoparticles calcined at 300°C and 500°C. No peaks corresponding to impurities were detected implying the purity of the synthesised CaO nanoparticles. The lattice constants were calculated and were found to be in good agreement with the reported values.

Particles sizes were calculated from peak broadening analysis using Debye-Scherrer's formula

$$D = k\lambda / \beta \cos \theta$$

where k is the Scherrer constant (0.94 for spherical particles), λ is the wavelength of x-rays used (1.54Å) and β is the full width at half maximum (FWHM) of the peak under consideration.

Dislocation density (δ) = $1/D^2$ where D is the average particle size. The micro strain was determined by the relation, micro strain (ϵ) = $\beta \cos(\theta)/4$ where β is the FWHM (full width at half maximum). The values of average particle size, dislocation density and micro strain for the CaO nanoparticles synthesized at the three calcination temperatures are presented in Table 4.

It can be observed from the table that the average crystallite size of the prepared samples lies in the nano range. The crystallite sizes were found to increase at higher calcination

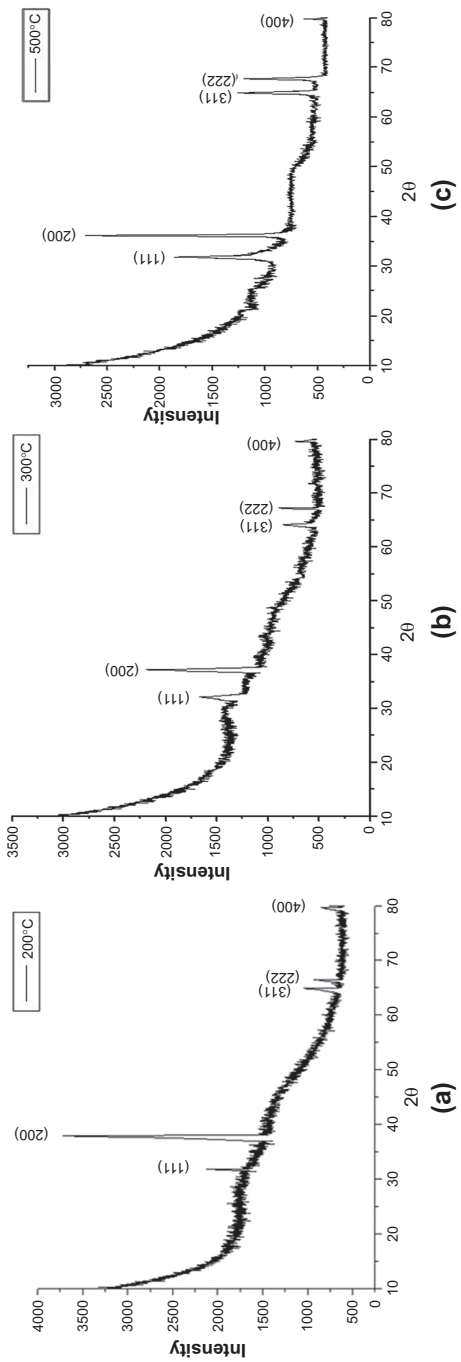


Figure 1.
X- ray diffractograms
of CaO nanoparticle at
(a) 200°C, (b) 300°C and
(c) 500°C calcinations
temperatures

temperatures which may be due to the coalescence of grains with heat treatment. This takes place due to the movement of atoms to favourable positions and thus merging into adjacent particles resulting in larger grain growth (Ungula *et al.*, 2017; Kim *et al.*, 2011). The dislocation density is higher for CaO calcined at 200°C as the particle size is lower in this case. The number of interfaces in a given volume is more in materials with lower particle size and correspondingly shows higher dislocation density (Ubale and Belkhedkar, 2015). The micro strain gives information about the defects present in the lattice, and its values are lower for CaO nanoparticles calcined at higher temperatures as the numbers of defects are reduced on heat treatment.

SEM studies

Figures 2(a), (b) and (c) show the SEM micrographs of CaO nanoparticles synthesised at 200°C, 300°C and 500°C calcinations temperature. Nonuniform distributions of spherical particles forming clusters are seen in the case of CaO synthesised at 200°C.

D Value (Å)		Relative intensity		hkl	Lattice constant (Å)	
Obs	Rep	Obs	Rep		Obs	Rep
2.8119	2.7759	59	42	(111) _C	$a = 4.870$	$a = 4.808$
2.3742	2.4040	100	100	(200) _C	$a = 4.748$	$a = 4.808$
1.4374	1.4497	28	12	(311) _C	$a = 4.767$	$a = 4.808$
1.4060	1.3879	25	13	(222) _C	$a = 4.870$	$a = 4.808$
1.2044	1.2020	23	5	(400) _C	$a = 4.818$	$a = 4.808$

Table 1.
XRD data of CaO
nanoparticle at 200°C
calcination
temperatures

D value (Å)		Relative intensity		hkl	Lattice constant (Å)	
Obs	Rep	Obs	Rep		Obs	Rep
2.7920	2.7759	76	42	(111) _C	$a = 4.835$	$a = 4.808$
2.4206	2.4040	100	100	(200) _C	$a = 4.841$	$a = 4.808$
1.4532	1.4497	38	12	(311) _C	$a = 4.819$	$a = 4.808$
1.3928	1.3879	40	13	(222) _C	$a = 4.824$	$a = 4.808$
1.2040	1.2020	32	5	(400) _C	$a = 4.816$	$a = 4.808$

Table 2.
XRD data of CaO at
300°C calcinations
temperature

D value (Å)		Relative intensity		hkl	Lattice constant (Å)	
Obs	Rep	Obs	Rep		Obs	Rep
2.8091	2.7759	68	42	(111) _C	$a = 4.866$	$a = 4.808$
2.4741	2.4040	100	100	(200) _C	$a = 4.948$	$a = 4.808$
1.4354	1.4497	46	12	(311) _C	$a = 4.761$	$a = 4.808$
1.3778	1.3879	20	13	(222) _C	$a = 4.773$	$a = 4.808$
1.2009	1.2020	20	5	(400) _C	$a = 4.804$	$a = 4.808$

Table 3.
XRD data of CaO at
500°C calcinations
temperature

Sample	Average particle size (nm)	Dislocation density δ (10^{14} cm^{-1})	Micro strain ϵ (10^{-4})
CaO at 200°C	68	2.16	5.03
CaO at 300°C	69	2.10	4.96
CaO at 500°C	75	1.77	4.55

Table 4.
The values of
crystallite size,
dislocation density and
micro strain of CaO at
different calcination
temperatures

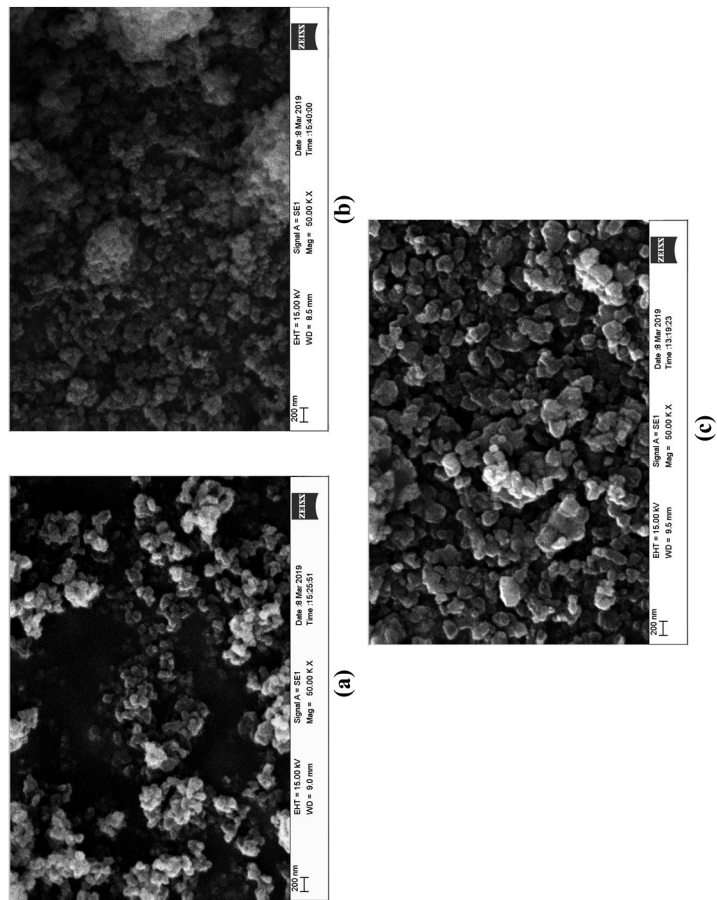


Figure 2.
SEM micrograph of
CaO nanoparticles
synthesised at (a)
200°C, (b) 300°C and (c)
500°C calcinations
temperature

Agglomeration and formation of clusters are more prominent in the case of CaO nanoparticles calcined at 300°C and 500°C. Denser distribution of spherical particles is also seen. The particles sizes appear to be larger than those calcined at 200°C. This is in agreement with the absorbance data obtained for CaO nanoparticles at the three temperatures. A decrease in band gap at higher calcinations temperature suggests an increase in particle size.

EDX studies

Figures 3(a), (b) and (c) show the EDX spectra of CaO nanoparticles at 200°C, 300°C and 500°C calcinations temperature. The presence of Ca and O are confirmed in all the cases. The atomic percentages are presented in the adjacent tables. Excess oxygen is observed at all the calcination temperatures.

Absorbance spectral studies

Figure 4 presents the absorbance spectra in the range 400–700 nm of CaO nanoparticles at different calcinations temperatures. A broad absorption peak in the range of 400–500 nm is observed in all the cases. It can also be observed that the absorption edge is shifted to higher

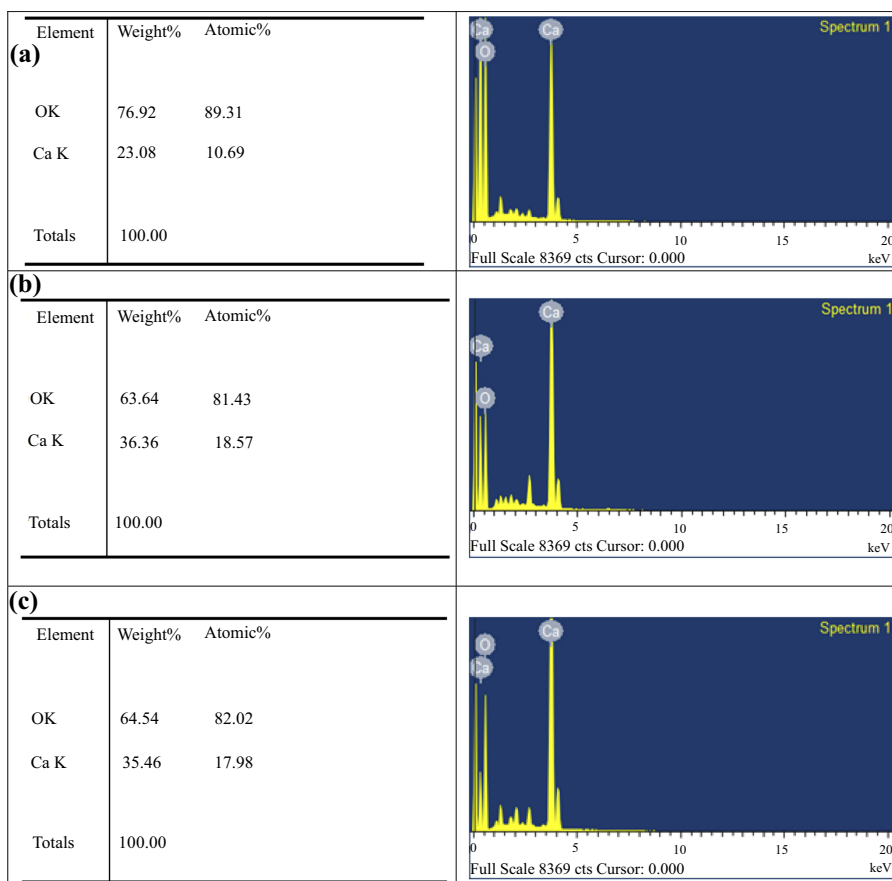
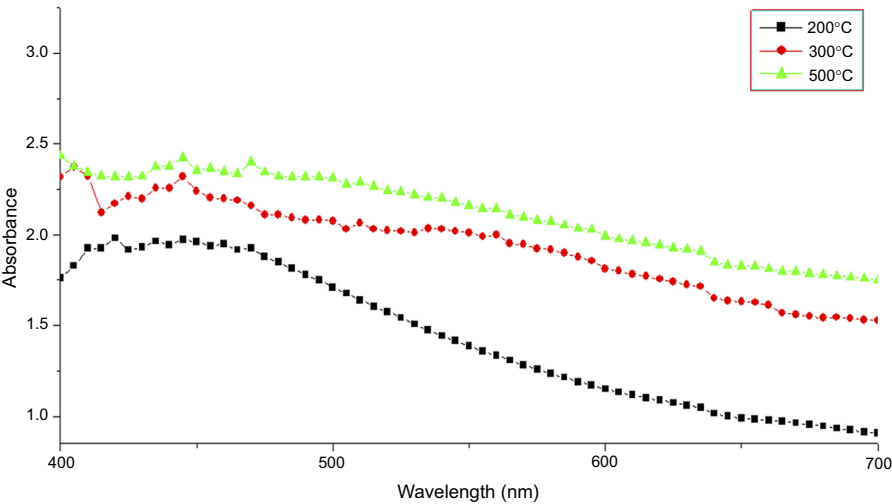


Figure 3.
EDX spectra of CaO
nanoparticles
synthesised at (a)
200°C, (b) 300°C and (c)
500°C calcination
temperatures

Figure 4.
Absorbance spectra of
CaO nanoparticles at
three different
calcination
temperatures

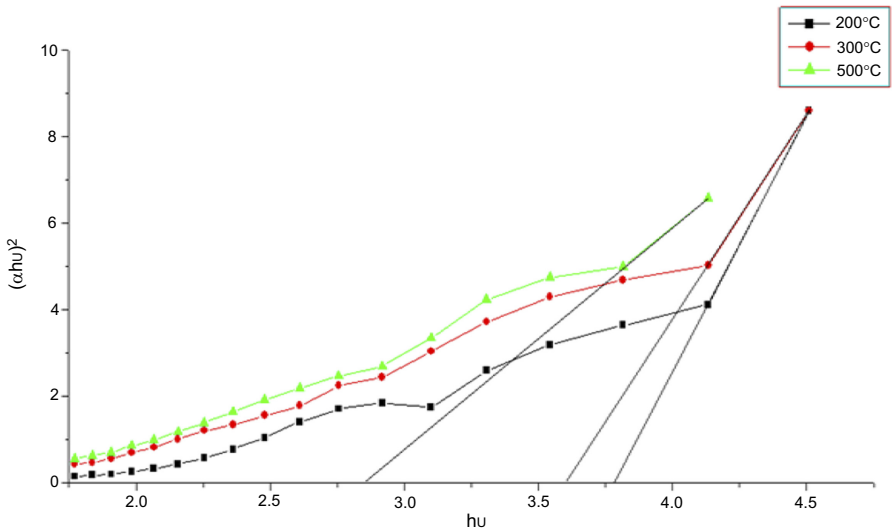


wavelength at higher calcination temperatures. There are several reports in literature that increasing calcination temperature results in bigger grain size and lesser strain. Subsequently, the absorption edge shifts to higher wavelength, and the optical bandgap decreases ([Abd-Elnaiem *et al.*, 2017](#); [Abdel-Rahim *et al.*, 2015](#), [El-Sebaï *et al.*, 2012](#)).

The relation between the absorption coefficient α and the incident photon energy $h\nu$ for direct band gap materials is given by the relation, $\alpha = c(h\nu - E_g)^{1/2}/h\nu$ where E_g is the optical band gap and c is a constant. Thus, a plot between $(\alpha h\nu)^2$ vs $h\nu$ (Tauc's plot) gives the band gap value E_g of the material.

[Figure 5](#) shows the Tauc's plot of CaO nanoparticles at different calcinations temperatures. The corresponding band gap values are shown in [Table 5](#). The band gap

Figure 5.
Tauc's plot of CaO
nanoparticles at
different calcination
temperatures



values have been found to decrease with increasing calcination temperatures. This can also be confirmed with a shift in the band edge toward longer wavelengths at higher calcinations temperatures. The decrease in bandgap with increase in calcination temperatures can be attributed to the presence of surface dangling bonds surrounding the crystallites. Studies have shown that under heat treatment, process of crystallization takes place and dangling bonds are produced. At higher calcination temperature, the crystallites break down increasing the number of surface dangling bonds (Hasegawa and Kitagawa, 1978). As a result, concentration of localized states in the band structure increases gradually. The width of the localized states increases thereby reducing the optical energy gap (Abd-Elnaiem *et al.*, 2017).

Photoluminescence studies

Figure 6 presents the PL emission spectra of CaO nanoparticles at different calcination temperatures. Three prominent emission peaks are observed at all calcination temperatures. At 468 nm excitation, a sharp emission peak at around 231 nm is observed which may be due to excitonic recombination. Excitons (bound electrons holes pairs attracted to each other by Coulomb force) are generated in the conduction and valence bands of the material, a result of absorption of photons of energy equal or greater than its band gap energy. As these charge carriers have a very short life time ($\sim 10^{-8}$ s), they recombine with each other. Two peaks one around 375 nm and the other around 400 nm may be due to band edge emission. Defect-related weak emission between 480 and 500 nm is observed at 365 nm excitation. These defect-related green emission may be due to oxygen vacancies. It can also be observed from the figure that the PL emission intensity is higher at higher calcination temperature. This is in

S.No	Sample	Band gap (eV)
1	CaO at 200°C	3.78
2	CaO at 300°C	3.60
3	CaO at 500°C	2.86

Table 5.
Band gap of CaO
nanoparticles at
different calcination
temperatures

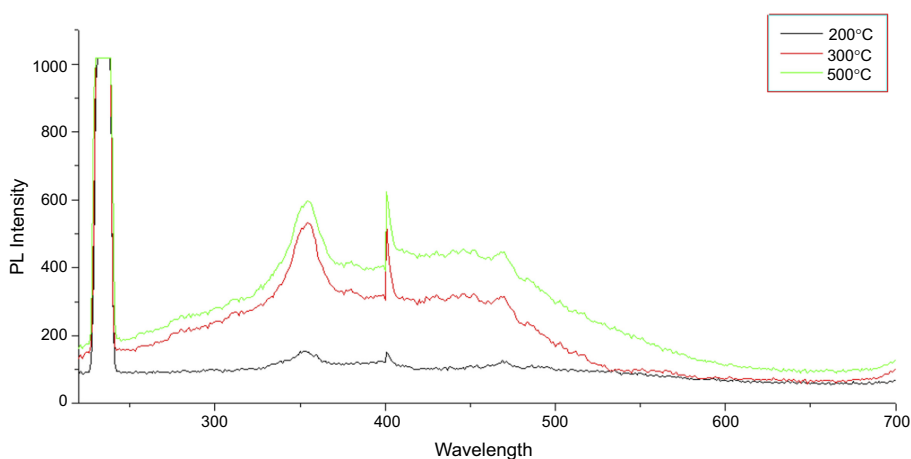


Figure 6.
PL spectra of CaO
nanoparticles at three
different calcination
temperatures

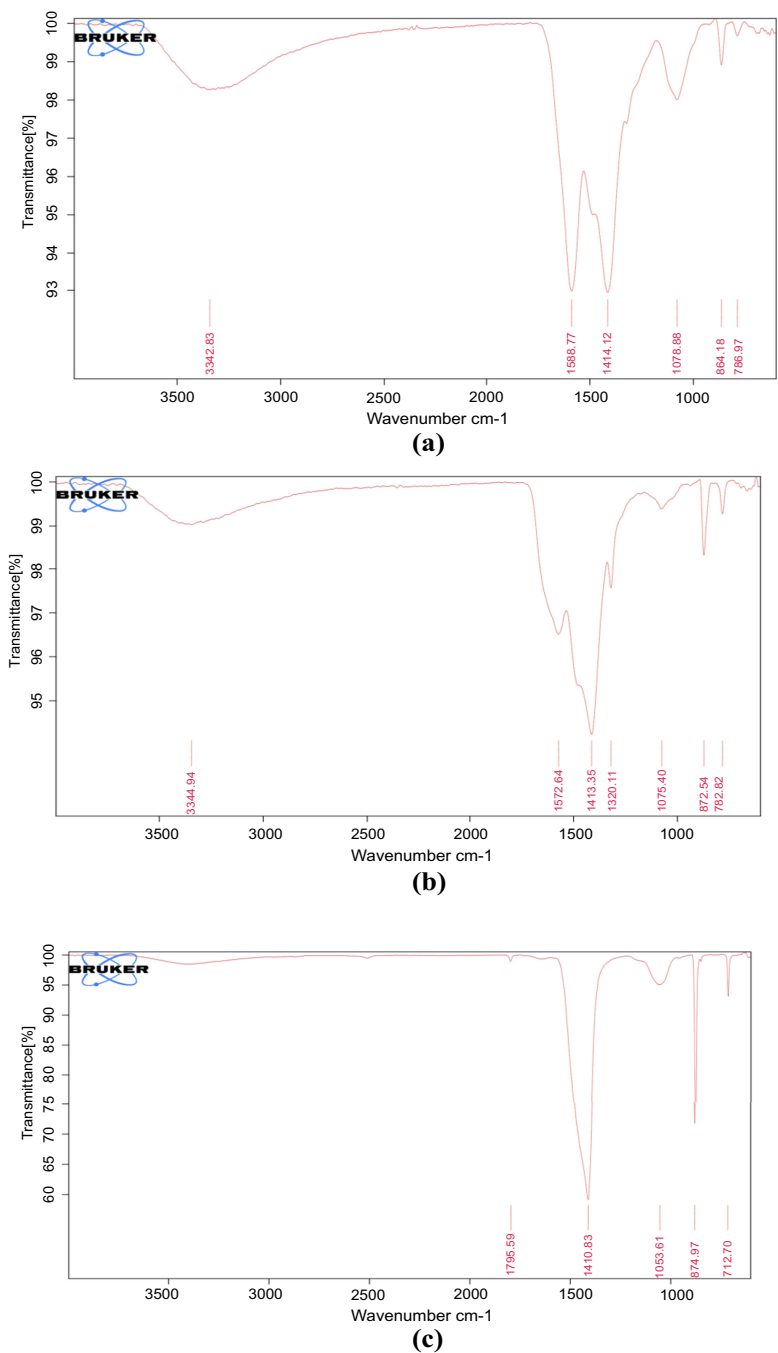


Figure 7.
FT-IR spectra of CaO
nanoparticles at
(a) 200°C, (b) 300°C and
(c) 500°C calcination
temperatures

agreement with the higher absorbance at higher calcination temperature from the optical absorption spectral studies (Aseel *et al.*, 2018; Nirmala *et al.*, 2013; Joya *et al.*, 2016).

Infrared spectroscopy

Infrared spectroscopy is an important technique in providing information about transitions in vibrational and rotational levels in molecules. Fourier transform infrared spectroscopy involves the interference of electromagnetic radiation and indicates the different functional groups present on the surface of the nanoparticles. The synthesised calcium oxide nanoparticles were characterized using Bruker Alpha FTIR spectrometer in the range of $4,000\text{ cm}^{-1}$ to 500 cm^{-1} . The FTIR spectra of CaO nanoparticles synthesised at 200°C , 300°C and 500°C calcination temperatures are shown in Figure 7(a), (b) and (c). It can be observed from Figure 7(a) that broad peaks are found at 3242 cm^{-1} , 1588 cm^{-1} , 1414 cm^{-1} , 1078 cm^{-1} , 786 cm^{-1} and a sharp peak at 864 cm^{-1} . These peaks indicate the presence of $-\text{N}-\text{H}$, $-\text{O}-\text{H}$ (carboxylic), $-\text{CN}$, $\text{C}=\text{O}$, $\text{C}=\text{N}$, $\text{C}=\text{C}$, $\text{C}-\text{O}$, $\text{C}-\text{C}$, $\text{Ca}-\text{O}$ (linkage) etc in the nano powder of CaO. The broadband at $1,414\text{ cm}^{-1}$ suggests the presence of $\text{C}-\text{O}$ bond which can be associated with the carbonation of CaO nanoparticles (Ramola *et al.*, 2019; Abraham and Sarathy, 2018).

In case of CaO nanoparticles synthesised at 300°C calcination temperature, peaks are almost same as those prepared at 200°C with a slight shift in their positions ($3,344\text{-}1$, $1572\text{-}1$, $1413\text{-}1$, $1075\text{-}1$, $872\text{-}1\text{-}1$). A sharp peak at $1,320\text{ cm}^{-1}$ is also observed. All these peak positions show the presence of carboxylic group, hydroxyl, amines and amides which confirm that the phytoconstituents help in the stabilization of CaO nanoparticles (Anantharaman *et al.*, 2016). In CaO nanoparticles prepared at 500°C calcination temperatures, in addition to the peaks at $1,410\text{-}1$, $1053\text{-}1\text{-}1$, a small peak at 1795 cm^{-1} corresponds to $\text{C}=\text{O}$ bonds from carbonate (Galván-Ruiz *et al.*, 2009) and a sharp peak at 712 cm^{-1} is observed which can be attributed to the presence of $\text{Ca}-\text{O}$ bonding which identifies the presence of calcium oxide (Anantharaman *et al.*, 2016). The broad bands around $3,340\text{ cm}^{-1}$ and $1,570\text{ cm}^{-1}$ are not visible for the CaO sample calcined at 500°C ; however a small hump at around $3,407\text{ cm}^{-1}$ is observed.

Conclusion

In this work, CaO nanoparticles were synthesized successfully by green synthesis technique using *Murraya Koenigii* leaf extract at 200°C , 300°C and 500°C calcination temperatures. XRD studies confirm the cubic phase of CaO. SEM studies show the presence of spherical particles along with some clusters for all calcinations temperatures. Presence of calcium and oxygen in elemental form is observed from EDX spectra. The optical absorbance spectral studies show that the band gap values decreases with increasing calcination temperature in all the cases. The PL investigation suggests emission between defect level, band edge emission as well as excitonic emission. The FTIR spectra show broad and sharp peaks confirming the presence of carboxylic group, hydroxyl, amines and amides in the synthesised nano powder of CaO.

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Corresponding author

Sofiya Bano can be contacted at: sofikgn786@gmail.com