Materials Science and Engineering, Volume 45, No. 1 (2020), pp. 182–190.
DOI: 10.32974.mse.2020.018

PREPARATION OF CALCIUM OXIDE BY A PRECIPITATION METHOD

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Abstract: CaO nanoparticles were prepared using precipitation method. Different concentration of $CaCl_2$ and NaOH were used as starting raw materials. The structure and microstructure of the prepared CaO powders were examined using several characterization techniques including X-ray diffraction (XRD), Scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS). Different crystallite sizes of nano and sub micro CaO were obtained based on the concentration of the reactants. Using precursors with low concentration leads to formation of product with small crystallite size.

Keywords: precipitation method, CaO particles

INTRODUCTION

Calcium oxide (CaO) is an important inorganic compound which is used in a wide range of applications, being of continuous interest in the field of materials research. It can be used as a catalyst, toxic-waste remediation agent, or as an additive in refractory and paint industries [1]. CaO nanoparticles can be used as an antimicrobial agent, a drug delivery agent as well as in various other biomedical applications [2]. Moreover, CaO is an attractive CO_2 absorber, due to its good kinetics and high capture capacity, even under low CO_2 partial pressure condition [3]. Calcium oxide has been regarded as one of the most promising candidates for carbon capture due to its high capturing efficiency, low running cost, and high abundance in the Earth's crust. Several different methods can be used for the preparation of CaO nanoparticles. Varying these methods, all physical and chemical properties of nano-CaO can be changed. The morphology, surface area and capturing efficiency can be carefully controlled under specific synthesis conditions [4].

Capturing CO_2 by using CaO based adsorbents has been attracted industrial sectors due to the high theoretical capacity of CO_2 capture, low cost, and potential use in large scale. The development of CaO-based adsorbents decreases over a number

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of cycles of CaO carbonation/calcination. To improve the sintering-resistant properties of CaO-based adsorbents, many factors should be considered such as decreasing the particle size and increasing the surface area, dispersing CaO on an inert support, as well as surface modification [5]. The aim of this research work is to produce CaO nanoparticles through precipitation method by manipulating the concentrations of the precursors, the stirring speed and the calcination temperature and time. Various characterization techniques will be carried out including XRD, SEM and EDX to investigate the structure, the morphological feature and the elemental composition.

1. MATERIALS AND METHODS

Calcium oxide (CaO) powder was produced according to the following reactions:

$$CaCl_{2\,(aq)} + 2\,NaOH_{(aq)} \longrightarrow Ca(OH)_{2(s)} + 2\,NaCl_{(aq)} \quad (1)$$

$$Ca(OH)_{2(s)} \xrightarrow{650^{\circ}C\ /1hr} CaO_{(s)} + H_2O_{(g)}. \quad (2)$$

$CaCl_2.xH_2O$ and NaOH with different concentrations (1, 0.5, 0.1 M) of $CaCl_2$ and (2, 1, 0.2 M) of NaOH were used as initial reagents for the synthesis of Ca $(OH)_2$ powder, using stoichiometric $Ca^{+2} : OH^{-} = 2 : 1$ mole ratio. The concentrations of both aqueous solutions are shown in *Table 1*. First, both aqueous solutions ($CaCl_2$ and NaOH) were heated up to 80 °C. At this fixed temperature, a given NaOH solution was added dropwise to a given $CaCl_2$ solution under stirring (1,300 rpm) by magnetic stirrer for 30 minutes. During this process, white precipitates formed. After that, each mixture was filtered and washed five times with distilled water then dried overnight at room temperature to obtain dry Ca $(OH)_2$ powder. The obtained powders were calcined at 650 °C under argon gas environment for one hour to produce a CaO powder. The experimental reagent is given in *Table 1*.

Table 1

Composition and concentration of the reactants in aqueous solutions

Sample No	$CaCl_2$ (mol/L)	NaOH (mol/L)
1	0.1	0.2
2	0.5	1
3	1	2

2. RESULT AND DISCUSSIONS

2.1. SEM and EDX characterizations of the samples

The samples were examined by scanning electron microscopy using a Carl Zeiss EVO MA10 equipment at 20 kV using a Bruker microprobe. The pictures are taken under BSD-SE mode. The measurement of the particle size was done by ImageJ. Characteristic results of the SEM and EDX investigation are shown in *Figures 1–3* and *Table 2*.

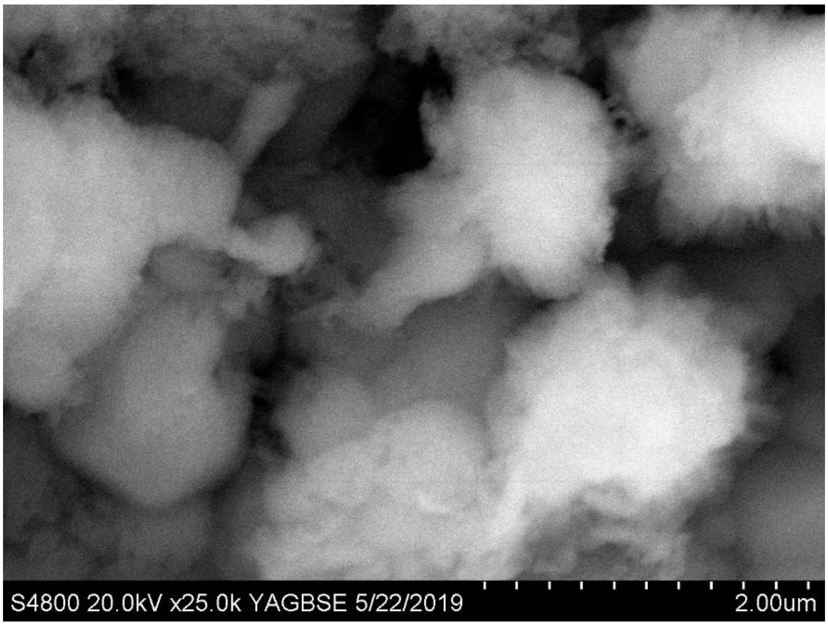

Figure 1

SEM image of CaO produced from Sample 1 (0.1M $CaCl_2$ + 0.2M NaOH)

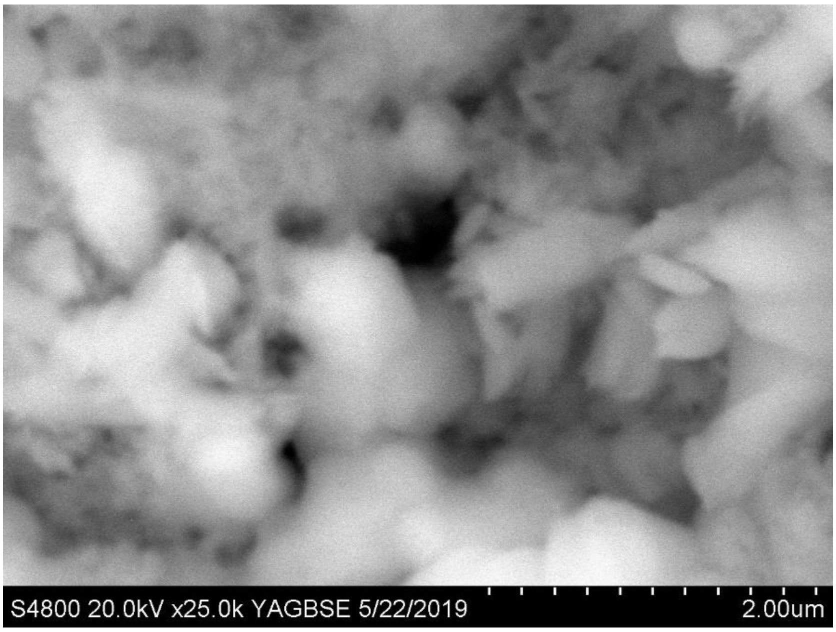

Figure 2

SEM image of CaO produced from Sample 2 (0.5M $CaCl_2$ + 1M NaOH)

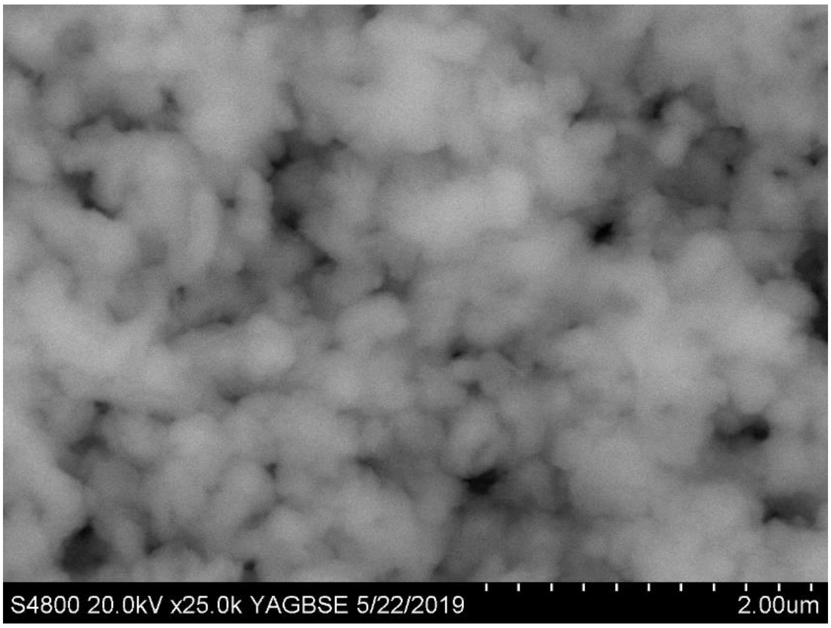

Figure 3
SEM image of CaO produced from Sample 3 (1M $CaCl_2$ + 2M NaOH)

According to EDX result shown in *Table 2*, using moderate concentration (0.5M) of $CaCl_2$ reactant leads to the formation of high amount of Ca and low amount of C and Cl, while using low concentration (0.1M) or high concentration (1M) of $CaCl_2$ resulted in production of high C content and low amount of Ca.

Table 2
The weight percentage and atomic percentage of the elements in the deposit of the product according to the EDX investigation

Elements	Weight percentage			Atomic percentage		
	Sample1	Sample2	Sample3	Sample1	Sample2	Sample3
C	8	5	7.3	15.6	10	14.2
O	39.5	35.5	35	55.4	54	52
Ca	52	59.3	57.5	28.7	35.9	33.7
Cl	0.5	0.2	0.2	0.3	0.1	0.1
Total	100	100	100	100	100	100

2.2. XRD investigation of the samples

The samples were measured by Bruker D8 Advance, Cu K-alpha radiation, 40 kV and 40 mA generator settings, parallel beam with Göbel mirror, Vantec-1 position sensitive detector with 1° opening, scanning rate 0.007° (2Th)/24 sec. In the investigation of three different preparation ratios as shown in *Figures 4–6*, the highest percentage of CaO powder was found to be 84.8% with a small amount of calcium carbonate and calcium hydroxide.

The results of XRD investigations are shown in *Figures 4–6*.

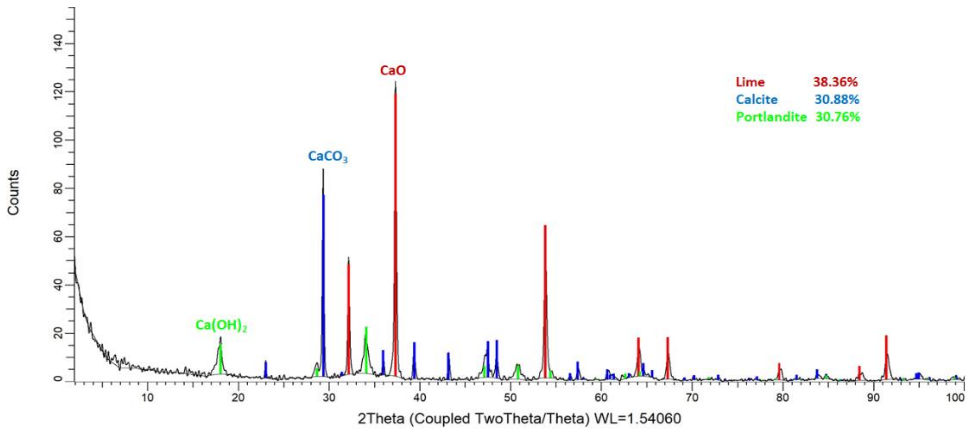

Figure 4

XRD diffractogram with the matched peaks of the calcined powder (Sample 1 with 0.1M $CaCl_2$ + 0.2M NaOH)

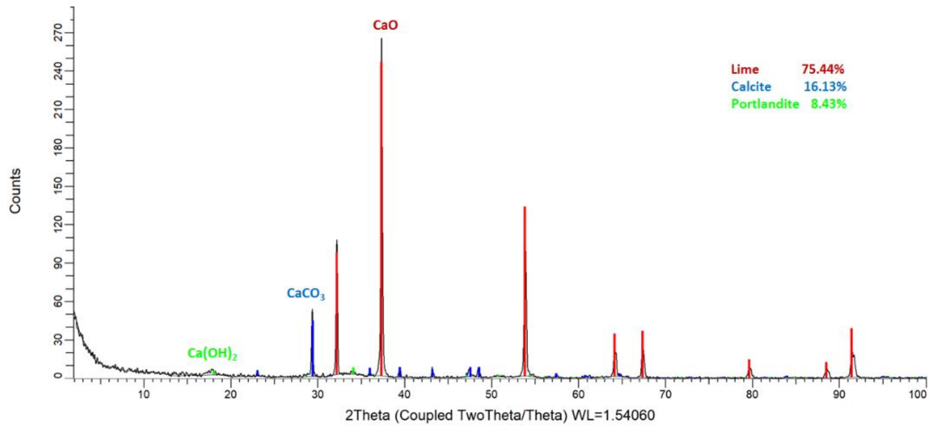

Figure 5

XRD diffractogram with the matched peaks of the calcined powder (Sample 2 with 0.5M $CaCl_2$ + 1M NaOH)

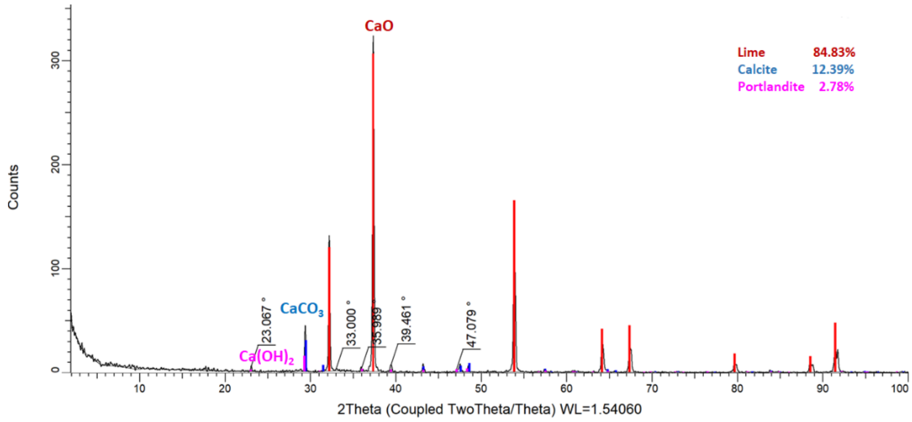

Figure 6
XRD diffractogram with the matched peaks for the calcined powder (Sample 3 with 1M $CaCl_2$ + 2M NaOH)

2.3. The correlation between the weight percentage, crystallite size and the concentrations of the reactants

Figure 7 shows the relationship between the concentrations of reactants and crystallite size of the produced CaO (see also *Table 1*). As follows from *Figure 7*, there is an approximately linear concentration dependence of the crystal size. It is in qualitative agreement with other experimental results obtained by precipitation techniques [6–16]. It is because the lower concentration of reagents leads to the smaller amount of matter attached to the nuclei in the growth phase. However, the line does not seem to pass through the origin (0; 0) – see *Figure 7*.

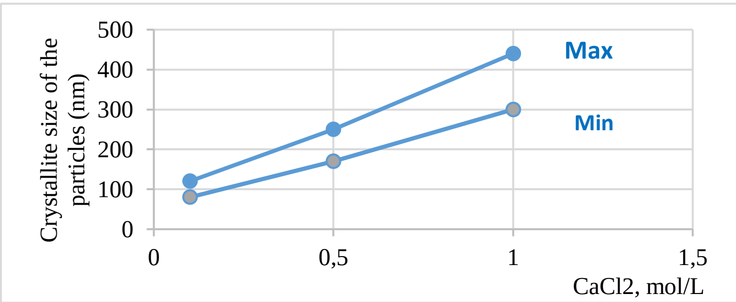

Figure 7
Crystallite sizes of the CaO as function of solution concentration

The connection between the concentrations of reactants and the composition of the final product is shown in *Figure 8*. One can see that the CaO content increases with the increase of the reactant concentrations. It is probably connected with *Figure 7*:

the increase in particle size decreases the specific surface area and does spontaneous capture of CO_2 or H_2O from the environment is decreased.

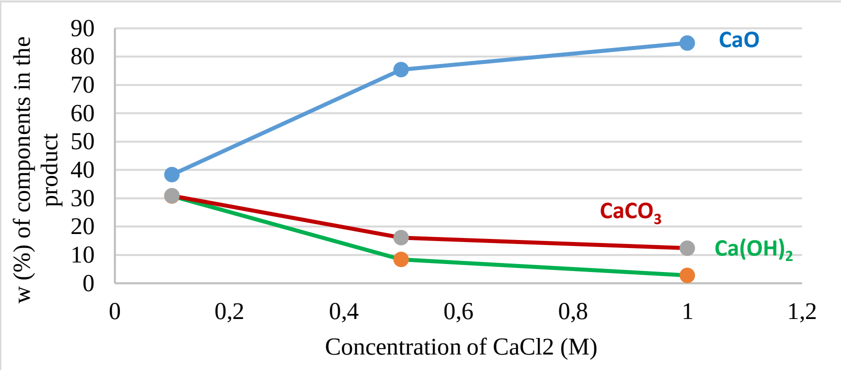

Figure 8

Composition of the final product vs the composition of the initial solution

CONCLUSION

Calcium oxide nano- and sub-micron particles were prepared by calcination of the product of chemical reaction between $CaCl_2$ and NaOH aqueous solutions. It is shown that about nano-range can be obtained only if a low concentration of the reactants is applied in the aqueous solution. The final product mostly contains CaO, although it is contaminated by $CaCO_3$ and Ca $(OH)_2$. This contamination decreases with decreasing the specific surface area of the particles, i.e. by increasing their size. Thus, the decrease in particle size and increasing at the same time the purity of the final CaO particles seems to be a challenge.

ACKNOWLEDGEMENTS

The authors are grateful to dr. Kristaly Ference of the University of Miskolc for his supporting in XRD analysis and discussions. We also thank to dr. Anna Sycheva and dr. Daniel Koncz-Horvath of the University of Miskolc for their helping in the investigation of SEM, EDX measurements and discussions in this research work.

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