A novel method of synthesis and microstructural investigation of calcium titanate powders

Xianjun Lei^{b,c,d}, Baoqiang Xu^{a,b,c*}, Bin Yang^{b,c}, Binbin Xu^{b,c,d}, Xutao Guo^{a,b,c}

^a State Key Laboratory of Complex Nonferrous Metal Resources Clear Utilization, Kunming University of Science and Technology;

^b National Engineering Laboratory for Vacuum Metallurgy;

^c Yunnan Provincial Key Laboratory for Nonferrous Vacuum Metallurgy, Kunming University of Science and Technology;

^d Faculty of Metallurgy and Energy Engineering, Kunming University of Science and Technology; Kunming 650093, P.R. China

Abstract

A novel method, using calcium chloride bihydrate ($\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ and TiO_2) as raw materials, to synthesize crystalline calcium titanate (CaTiO_3) has been investigated by thermodynamic calculation and X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), differential scanning calorimetric-thermogravimetric analysis (DSC-TG), high resolution transmission electron microscopy (HR-TEM) etc. Using XRD technique analyzed experimental results revealed success of thermal sintering process, in which the mass ratio of $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}/M_{\text{TiO}_2}=2:1$, holding temperature of 1073K, preservation time 2 hours, could be used to produce crystalline CaTiO_3 powders. Ratio of $\text{Ca/Ti/O}=1:1:3$ was verified by EDS pot-scan. According to SEM observation, the crystalline CaTiO_3 powders are mainly composed of many separated particles with average size approximately $10\mu\text{m}$ and have been demonstrated a strongly faceted morphology. The lattice plane, (112) is clearly observed in the HR-TEM micrograph and the lattice distance was measured as 0.27 nm.

Keywords: Calcium titanate; Perovskite structure; Thermal sintering method; Microstructure; Calcium chloride bihydrate

1. Introduction

Calcium titanate, CaTiO_3 , was discovered for the first time in mineral form in 1839 by Gustav Rose, a German mineralogist. As ceramic material, CaTiO_3 has been widely used in electronic devices and it is a key component of Synroc (type of synthesis rock used to store nuclear waste)[1]. Also, CaTiO_3 belongs to the important group of compounds with a perovskite structure; and is famous to be one of the important basic materials of ferroelectric ceramics. This compound is also used as a thermally sensitive resistor element owing to its negative temperature coefficient, and a refractory material with high corrosion receptivity against caustic solutions [2, 3]. Its most important features are high dielectric constant, large positive temperature of the resonance frequency, but also high dielectric loss that could be decreased by substitution of the A-site with trivalent ions [4]. Recently, CaTiO_3 was evaluated as a good biocompatible material [5], and CaTiO_3 with disordered structure has been reported to have photoluminescence properties [6, 7]. The above properties make it attract much more attention.

Various methods have been developed in the literatures to synthesize CaTiO_3 in past years. These methods can be divided into two groups, namely solid-state synthesis methods and wet chemical synthesis methods. As far as solid-state synthesis methods are concerned, CaTiO_3 was initially prepared by conventional solid-state reaction between TiO_2 and CaCO_3 or CaO at temperature of approximately 1623K [8]. Avvakumov [9] mechanochemically synthesized CaTiO_3

from milled mixtures of $\text{CaO}[\text{Ca}(\text{OH})_2]\text{-TiO}_2$ (rutile). Mi et al. [10] obtained crystalline CaTiO_3 by grinding CaO-TiO_2 mixtures (both rutile and anatase) and heating them up to about 1223K. Evans et al. [4] monitored with variable X-ray powder diffraction the reaction in $\text{CaCO}_3\text{-TiO}_2$ milled mixtures and showed that pure CaTiO_3 is obtained at 1193K. CaTiO_3 powders obtained by these methods present several problems, such as high-processing temperatures, inhomogeneity and contamination by impurities with a nonuniform particle sizes distribution [4], which had negative impact on their electrical properties. In order to minimize these problems, wet chemical methods have been employed to synthesize CaTiO_3 powders with desired stoichiometry, such as: sol–gel [11], co-precipitation [12], combustion method [13], organic-inorganic solution technique [14] and hydrothermal process [15]. However, they are complicated and require organic solvents or precursors. Over the last couples of years, new methods have been developed in order to overcome the drawbacks and difficulties of those mentioned above. For example, a polymer precursor method was developed or synthesized materials were treated using adapted microwave oven [16, 17]. A very interesting approach to synthesis of CaTiO_3 with microtubular structure and rectangular cross-section was recently reported by Li et al. [18] Such morphology of CaTiO_3 is highly difficult to be obtained basing on bulk starting materials, therefore, Li et al. successfully used titanate nanofibers. In addition, Yang et al. [19] have recently published results of their work, reporting successful synthesis of hollow crystals of CaTiO_3 along with explanation of the mechanism of their formation.

Recently, CaTiO_3 has been formed in extraction processes of metallic titanium from titanium dioxides [20-22]. In their experiments, they both used a chemical reagent calcium chloride (CaCl_2), and finally the CaTiO_3 crystals were found. Chen et al. and Alexander et al. thought that the formation of CaTiO_3 because of the reaction TiO_2 with the electrolyte CaCl_2 has occurred at the perimeter of each TiO_2 particle while the particle interiors have been reduced to Ti_2O_3 and calcium ions are most readily available at the surface of the particles, hence the surfaces react to form the CaTiO_3 , necessitating oxygen removal from, and reduction of, the particle centers. Also, they suggested that the CaTiO_3 had disadvantages for the completed reduction of titanium oxides. In contrast, in Xu et al. experiments, when the additive CaCl_2 and TiO_2 mixed and heated at 1273 K, CaO stemmed from the decomposition of $\text{CaCl}_2\cdot 2\text{H}_2\text{O}$ which is owing to the strong moisture absorption of CaCl_2 would make TiO_2 transform to CaTiO_3 . This is coincide with Okabe's experiments, in which CaTiO_3 phase also appeared in sintered products when the mixture of TiO_2 and flux CaO was sintered at 1073 K in atmosphere [23]. What's more, they supported that the CaTiO_3 phase has a facilitation impact on reduction processes. Accordingly, a new method to synthesize CaTiO_3 was put forward in this paper based on a series of reactions between $\text{CaCl}_2\cdot 2\text{H}_2\text{O}$ and TiO_2 and thermodynamic calculation and experimental investigation preparing CaTiO_3 were carried out in this paper.

2. Experimental details

2.1 Materials

Anatase titanium dioxide was used as raw material, the chemical reagent calcium chloride dehydrate ($\text{CaCl}_2\cdot 2\text{H}_2\text{O}$) as the additive, the pure CaTiO_3 (Aladdin) as a contrast sample, shown in Table 1.

Table 1. Materials used in experiments

Material	Form	Purity or content/%
TiO_2	Powder(A.R)	> 99%

$\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$	Lump(A.R)	> 99%
CaTiO_3	Powder	> 99.5%
HCl	Aqueous	35.00 ^a

^a concentration of solution

2.2 Experimental procedures

Experimental procedures are presented in Fig. 1. After grinding $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ into powders in an agate mortar with a pestle rapidly, the mixture of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ powders and 10g TiO_2 powders with different mass ratio were preformed into the cylinder feeds about $\Phi 18 \text{ mm} \times 3 \sim 7 \text{ mm}$ at the pressure of 2 MPa respectively. Then these feeds were put into a porcelain boat and heated to the setting temperatures for different holding time at ramp rate 10 K/min in resistance furnace. The sintered products were washed by low content hydrochloric acid (5%) and deionized water repeatedly for 10 min. The product was dried in a vacuum oven at 353 K for 12 hours, then the CaTiO_3 powders sample can be obtained.

Fig.1. Schematic diagram of the experiment.

2.3 Characterization

The phase of products was identified by X-ray diffraction (XRD) using a Rigaku (Japan) D/max-3B X-ray diffractometer equipped with graphite-monochromatized $\text{Cu K}\alpha$ radiation and quantitative analysis results (wt%) were analyzed by Reference Intensity Ratio (RIR) method of XRD. Scanning electron microscope (SEM) of S3400N and its Energy Dispersive Spectrometer (EDS) were used to detect element distribution of O, Ca and Ti by spot-scan method. NETZSCH STA 409PG/PC was used to carry out differential scanning calorimetric (DSC) and thermogravimetric analysis (TG) for the mixture feeds. Atomic scale observation on a grain boundary and a lattice fringe of microcrystals were performed by using a high resolution transmission electron microscopy (HR-TEM) (JEM-3010, 300kV, Japan). On HR-TEM observations, the powders were dispersed in acetone.

3. Results and discussion

3.1 Thermodynamics behavior of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ and TiO_2

$\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, which is similar with calcium chloride (CaCl_2), has a strong ability of moisture absorption to form some stable compounds of $\text{CaCl}_2 \cdot n\text{H}_2\text{O}$ ($n=4,6$). When it is heated, $\text{CaCl}_2 \cdot n\text{H}_2\text{O}$ ($n=4, 6$) begin to dehydrate to $\text{CaCl}_2 \cdot \text{H}_2\text{O}$ step by step. $\text{CaCl}_2 \cdot \text{H}_2\text{O}$ which is an unstable compound would further dehydrate with temperature increasing as followed reactions (1)-(3) at standard conditions.

These reactions are consistent with Xu et al. [20] reported under vacuum conditions. In standard conditions, Gibbs free energy changes with temperature of above reactions (1)-(4) were calculated and the reaction curves as shown Fig.2. These calculation results indicated the dehydration reaction of both $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ and $\text{CaCl}_2 \cdot \text{H}_2\text{O}$ are occurred when temperature higher than 500K, and $\text{CaCl}_2 \cdot \text{H}_2\text{O}$ could be decomposed to CaO to which the temperature up 950K. The calculation also illustrated when temperature increased reaction (4) would be happened easily, which could imply more and more CaTiO_3 will be formed as the reaction between CaO and TiO_2 . It is especially true when $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ and TiO_2 were mixed and heated at the temperature up to 950 K.

Fig.2. The ΔrG_T -T curves of reactions (1)-(4) in standard conditions.

It was reported that, On the other hand, CaCl_2 began to melt when temperature rose to 1047 K, and CaO could be dissolved in large amount of molten CaCl_2 [24], which could improve the reaction processes. Liu et al. [25] studied CaTiO_3 at high temperature suggested that linear thermal expansion was observed for CaTiO_3 to 1373 K, which might imply the structure of CaTiO_3 will keep stable low that temperature. B.J. Kennedy et al. [26] suggested that CaTiO_3 exhibit an orthorhombic structure with space group $pbnm$ below 1380 K. These were the guarantee of the stable and well crystalline CaTiO_3 were obtained. Consequently, we chose the experiment temperature vary from 1073 K to 1273 K.

3.2 Experimental investigation of preparing CaTiO_3

Fig.3.XRD patterns of sintered products from feeds with different $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}/M_{\text{TiO}_2}$ at 1273 K for 3 h: (a) $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}/M_{\text{TiO}_2}=1:1$; (b) $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}/M_{\text{TiO}_2}=2:1$; (c) $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}/M_{\text{TiO}_2}=3:1$; (d) $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}/M_{\text{TiO}_2}=4:1$.

Fig.3 shows XRD patterns of different sintered products corresponding to feeds with different mass ratio $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}:M_{\text{TiO}_2}$. Fig. 3(a) indicates the sintered products contained CaTiO_3 and TiO_2 phases, but the strongest peak corresponding to TiO_2 phase, illustrating part of TiO_2 was worked out and transformed into CaTiO_3 . With increase of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, peaks corresponding to TiO_2 phase disappeared completely and all intense peaks corresponding to CaTiO_3 can be observed when the mass ratio $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}/M_{\text{TiO}_2}=2:1$, as Fig.3(b). However, CaCO_3 and $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ peaks appeared when the mass ratio $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}:M_{\text{TiO}_2} = 3:1$ from fig.4(c). The reason of the formation of CaCO_3 should be the reaction between CO_2 in air and Ca(OH)_2 which resulted from moisture absorption of CaO , the hydrolysis product of $\text{CaCl}_2 \cdot \text{H}_2\text{O}$, and the existence of $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ stemmed from moisture adsorption of the excessive $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ in products. The production of CaCO_3 has few influences on the purity of CaTiO_3 because the sintered products would be washed by low content hydrochloric acid (5%), then CaCO_3 will be removed. Finally, as shown in Fig.3(d) a large number of peaks of $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ are observed when the mass ratio $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}:M_{\text{TiO}_2} = 4:1$. It is noteworthy that the peak intensity of CaTiO_3 do not increase when the mass ratio of $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}:M_{\text{TiO}_2}$ more than 2:1, these experimental results suggest pure CaTiO_3 is synthesized at 1273 K for 3 h. Thus, it is concluded that the crystalline CaTiO_3 can be obtained when mass ratio $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}:M_{\text{TiO}_2}$ is more than 2:1.

Fig.4.XRD patterns of sintered products from feeds at different temperature with mass ratio $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}/M_{\text{TiO}_2}=2:1$ for 3h: (a) 1073 K; (b) 1173 K; (c) 1273 K.

Fig.4 shows XRD patterns of sintered products from feeds with mass ratio $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}/M_{\text{TiO}_2}=2:1$ for 3h at different holding temperatures, in which the CaTiO_3 phase has already formed perfectly. When the holding temperature at 1073 K, the XRD pattern just shows the peaks corresponding to CaTiO_3 due to the reaction between TiO_2 and CaO which is from the decomposition of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ according to Fig.2, as shown in Fig.4(a). It is also manifested that the intensity of CaTiO_3 phases increase with temperature going up, as far as Fig.4(b) and Fig.4(c) are concerned, these might be in agreement with the linear thermal expansion of CaTiO_3 at high temperature suggested by Liu et al. [25]. Accordingly, higher sintering temperature make peaks of CaTiO_3 phase stronger implies the CaTiO_3 particles with the more grown and perfect crystalline.

Fig.5.XRD patterns of sintered products of mass ratio $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}} / M_{\text{TiO}_2} = 2:1$ under different holding time at 1073 K: (a) 1h; (b) 2h ; (c) 3h.

Similarly, longer temperature holding time led to the more grown and perfect crystalline of CaTiO_3 from Fig. 5 at 1073 K. Moreover, enough holding time is necessary to obtain much more pure phase of CaTiO_3 . It is clear that there was still TiO_2 phase existing, **quantitative analysis result of RIR method indicated TiO_2 phase accounted for 2.09% in weight ratio as shown in Fig.6(a)**, even though phases of CaTiO_3 (98%) are dominant in the sample when the holding time is 1 hour as shown in Fig.5(a). When the holding time was prolonged more than 2h, the peak of TiO_2 disappeared and the intensity of CaTiO_3 (100%) phases increased as shown Figs.5(b-c).

Fig.6. Quantitative analysis results (RIR method) bar graphs of calcium titanate product and reagent sample.

Fig.7.XRD patterns of sintered products: (a) the sample after being washed by 5%hydrochloric acid; (b) the calcium titanate reagent sample.

In order to investigate the purity of synthesized CaTiO₃ (which is obtained with the mass ratio of $M_{CaCl_2 \cdot 2H_2O} : M_{TiO_2} = 2:1$ at 1273 K for 2 hours) by contrasting with commercial CaTiO₃ reagent sample, the XRD patterns as shown in Fig.7. Noticeably, TiO₂ phase exists even if CaTiO₃ and Ca(TiO₃) phases are dominant in CaTiO₃ reagent sample. And quantitative analysis results represent 3.05%TiO₂ phase, 67.1%CaTiO₃ phase and 29.1%Ca(TiO₃) phase, respectively, in reagent sample as shown in Fig.6, compared with synthesized CaTiO₃(100%) according to Fig.6(b) and Fig.7(a), which indicates the synthesized CaTiO₃ has a higher purity.

Fig.8.DSC-TG curves of mixture with mass ratio $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}/M_{\text{TiO}_2}=2:1$.

In order to gain further insight into the thermal behaviors between $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ and TiO_2 , a test was carried out by NETZSCH STA 409PG/PC as shown in Fig.8. It can be seen that there are two main weight loss stages in the heating process of the mixture to 1373 K at the heating rate of 10 K/min and holding 1373K for 10 min. In the first stage, two continuous endothermic peaks should be quite related to volatilization of free-water at 332 K and dehydration reaction of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ to form $\text{CaCl}_2 \cdot \text{H}_2\text{O}$, where the strongest endothermic peak appeared according to the DSC curve at 455 K. These results are in accord with theoretical expectation. $\text{CaCl}_2 \cdot \text{H}_2\text{O}$, which is an unstable compound, would further dehydrate to CaCl_2 , CaO and HCl [20].

In the second stage when temperature goes up to 1056K, CaCl_2 begins to melt as the second strongest endothermic peak is shown on DSC curve, which could be in agreement with the results of Sajal Ghosh et al. [27] and M. Yamada et al. [28] From this time, the sample starts to lose weight rapidly and about 57.17% weigh is kept finally, which should be ascribed to volatilization of CaCl_2 and decomposition of $\text{CaCl}_2 \cdot \text{H}_2\text{O}$. This is again consistent with the results described in other reports [20, 24]. The endothermic peak at about 1106 K suggests liner thermal expansion of CaTiO_3 and formation of CaTiO_3 . This is perfectly correlated to the results of thermodynamic calculations. Moreover, the reaction of formation of CaTiO_3 from CaO and TiO_2 is exothermic, but no exothermic peak is observed on DSC curve that can confirm this reaction occurred. The exothermic peak on DSC curve is probably hidden under the endothermic peak [29,30]. The endothermic peak at about 1177 K in the curve may be attributed to the volatilization of CaCl_2 . When the temperature remaining 1373K for 10 min, only mass loss of 1.44% implies dehydration reaction of $\text{CaCl}_2 \cdot \text{H}_2\text{O}$ and volatilization of CaCl_2 almost complete.

3.3 Microstructure of synthesized CaTiO_3

The feeds with the mass ratio of $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}}:M_{\text{TiO}_2}=2:1$ at 1273 K for 2 hours were sintered, and the well purity crystalline CaTiO_3 powders were synthesized after being washed by low content hydrochloric acid (5%) and deionized water repeatedly, dried in a vacuum oven at 80°C for 12 hours. Scanning electron microscope (SEM) was employed to observe the surface morphology and particles size of the sample CaTiO_3 , and Energy Dispersive Spectrometer (EDS),

being used to detect element distribution of O, Ca and Ti by spot-scan method, were shown in Fig.9 and Table 2.

Fig.9. SEM images and EDS results of samples: (a) Low-magnification and (b) high-magnification SE SEM images of pure CaTiO_3 particles ; (c) Low-magnification and (d) high-magnification SE SEM images of samples with the mass ratio of $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}} : M_{\text{TiO}_2} = 2:1$ at 1273 K for 2 hours; (e) EDS spectra of the sample by spot-scan method.

Table 2. Composition of EDS points in Fig.9, wt%

Phase	O	Ca	Ti
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A	CaTiO ₃	33.13	28.85	38.03
B	CaTiO ₃	40.75	25.47	33.78

The SEM images of the reagent CaTiO₃ powders [Fig.9(a, b)] present that the crystalline CaTiO₃ particles have smaller particle size and are made up of a considerable amount of aggregated particles compared with CaTiO₃ sample. The particles with 2 μ m average size in Fig.9(a, b) look like round shape which is consistent with CaTiO₃ samples produced by mechanochemical synthesis [9,10]. In contrast the synthesized CaTiO₃, as shown in Fig.9(c, d), is mainly composed of mostly separated particles with approximate 10 μ m average size. What's more, it is easy to differentiate the size and shape of the particles, most of the particles are very regular in shape, this is especially true when we considered that the bigger particles in the centre of photographs exhibited a strongly faceted terraces morphology, which would be different from particles stemmed from other methods completely [8,9,30]. This observation can be understood as follows. Firstly, considering the size nonuniformity of CaCl₂·2H₂O powders in the feeds because they were rapidly grounded mechanically owing to their strong ability of moisture absorption so as to impartity of CaCl₂·2H₂O powders' size. Secondly, crystalline CaTiO₃, under this experimental condition, could be observed linear thermal expansion [24]. So, it can be easy for CaTiO₃ to grow. On the other hand, there was a combination of the two types of crystalline CaTiO₃, that is very smooth in the surface of these particles, this is a character of crystal. As far as EDS spectra can be concerned, which is shown in Fig.9(e), the crystal is composed of Ca, Ti, O and the ratio of Ca/Ti/O=1:1:3 is calculated from the data shown in Table 2.

Fig.10. (a) Low-magnification and (b, c) high-magnification SE SEM images of surface synthesized CaTiO₃ particle.

Interestingly, the synthesized CaTiO₃ particles are the nature of their surface faceting. The SEM images of surfaces demonstrate that the particles had major surface facets parallel to the growth axes, as would be expected. Analysis of a surface from the same sample use SEM developed this understanding further, Fig.10(b, c) showed sample images. The crystalline CaTiO₃ demonstrates a strongly faceted morphology, the CaTiO₃ formed terraces on a sub-micrometer scale as it grown. There is one possible explanation for this terracing is that they result from CaTiO₃ attempting to form a uniform layer maintaining faceted growth and one crystallographic orientation. It is understandable that the synthesized CaTiO₃ crystals have high degree of crystallinity. It can be concluded the well crystallized CaTiO₃ powders were formed and the purity of them were perfect. This was consistent with Fig. 6(b) and Fig.7.

Fig.11. (a) Low-magnification and (b) high-magnification Bright-field TEM photograph and (c) HR-TEM photograph of the synthesized CaTiO_3 particles.

Fig.11(a, b) show the TEM photographs of bright-field images for the synthesized CaTiO_3 particles. The synthesized CaTiO_3 particles are faceted and amorphous grains, and they keep the state of disperse. Therefore, it is very easy to differentiate the size and the shape of the particles. These were consistent with Fig.9. Fig.11(c) shows the HR-TEM photograph of the synthesized CaTiO_3 , in which, the lattice distance was measured as 0.27 nm, corresponding to the (112) plane. Such microstructural analysis gives us the crystals to be CaTiO_3 , and the crystals seem to be formed in (112) orientation. Such well-oriented single crystalline according to the orthorhombic unit cell, can explain why the intensities of (112) peaks in its XRD pattern were the highest. It can be concluded that the well crystallized CaTiO_3 phase is synthesized, so that its chemical composition may be approached to homogenous state.

4. Conclusion

Thermodynamic calculation indicated we can have synthesized CaTiO_3 via the sintered reaction between $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ and TiO_2 when the temperature was higher than 1073K, and the reason was that $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ would decompose to CaO which reacted to TiO_2 and transformed to CaTiO_3 . XRD, SEM and HR-TEM analyses confirmed that the pure CaTiO_3 crystal powders prepared by the sintering method in standard conditions. XRD analyses indicated the synthesis conditions of CaTiO_3 were the holding temperature was higher than 1273K, the mass ratio of $M_{\text{CaCl}_2 \cdot 2\text{H}_2\text{O}} : M_{\text{TiO}_2} = 2:1$ and heat preservation time was more than 2 hours. SEM images revealed that the crystallized CaTiO_3 powders were mainly composed of mostly separated particles which were seem to consist of many fine grains with average size approximately 10 μm and the nature of their surface faceting. HR-TEM analyses showed that the synthesized CaTiO_3 with their lattice distance was measured as 0.27 nm, corresponding to the (112) plane, and the crystals were inclined to grow

along (112) orientation.

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