EFFECT CALCINATION TEMPERATURE ON FORMED OF CALCIUM SILICATE FROM RICE HUSK ASH AND SNAIL SHELL

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ABSTRACT

Calcium silicate ceramics have been made using natural ingredients such as rice husk and snail shell with solid reaction techniques. The aim of this study to determine the characteristics of calcium silicate that have been sintered at a temperature of 900 ˚C, 1000 ˚C, and 1100 ˚C. Samples were characterized by X-Ray Diffraction (XRD), optical microscopy, and Fourier Transform Infra-Red (FTIR). The XRD results showed that $CaSiO_3$ (calcium silicate) along with the increase in calcination temperature and quantity was increased due to the increase of temperature of calcination. FTIR analysis showed that the Ca-Si-O bond was formed at wave number ranging from 848 cm^{-1} and 999 – 1001 cm^{-1}.

Keywords: Calcium silicate; rice husk ash; the shell of a snail.

Introduction

Calcium silicate is a combination of two elements, namely calcium and silica, known as the $CaSiO_3$ formula. Calcium silicate is the concern of researchers because the materials used are easily available and have used. Material from silica can be obtained from rice husk waste[1] and calcium as an alternative source can be obtained from industrial waste. Industrial wastes such as snail shells, shells, marble, calcium chloride waste, and portlandite-rich wastes.[2–4] The uses of calcium silicate include material for insulation in ceramic material thermal insulators,[5] for bone repair and regeneration,[6] as well as substitutes for chalk and kaolin.[7]

Calcium silicate can be synthesized using several methods. The methods that have been used are a sol-gel method,[8–10] hydrothermal method[6] and solid reaction method.[2,11,12] Solid reaction method has several advantages over other methods, environmentally friendly, non-toxic, and relatively cheaper.[2]

Research on calcium silicate using solid reaction methods has been carried out by several researchers examples research calcium silicate using rice husk ash and snail shell,[2] calcium oxide and silica commercial ingredients,[11] research on calcium silicate carried out by Istiyati & Asmi with eggshells and commercial silica as well as Budiman & Asmi[11,13] using calcium carbonate ($CaCO_3$) and silicon dioxide (SiO_2). The method used by the researchers was solid reaction by different variations in temperature and type of material.

Varies of Temperature has been done by several researchers. Phuttawong et al [2] conducted calcium silicate research on 800°C -1000°C calcination temperature variation and the results showed no effect of calcination temperature on calcium silicate formation. Research in Sari & Asmi[14] at 1000 °C temperature showed that the formation of calcium silicate ceramic phase was not optimal (<50%) As seen from the results of XRD. Ulfah & Asmi[15] conducted a study on the calcination temperature of 1100 °C, with the XRD test the study obtained insignificant calcium silicate which was characterized by

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only 1 peak of calcium silicate with a phase that was more perfect than the other phases.

This research uses silica material from rice husk ash and calcium from the snail shell as the basic material for making calcium silicate. The method used in this research is solid reaction method. Samples are also calcined at varying temperatures to see the effect of calcination temperature on calcium silicate. Characterization was done to determine the percentage of silica content in rice husk ash and calcium content in snail shells as the initial stage of making calcium silicate. Calcium silicate samples were then characterized to find out the functional groups formed in calcium silicate.

Methods

The materials used in this study were rice husk ash, snail shell, hydrochloric acid (HCl), DI water, ethanol, and NaOH. The tools used in this study were 200 mesh sieve, mortar and pestle, digital balance, beaker glass, ash-free filter paper, label paper, magnetic stirrer, hot plate, oven, stirring rod, hydraulic pressing, furnace, measuring cup, X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), Fourier Transform Infra-Red (FTIR).

Rice husk ash was then crushed and sieved with 200 mesh sieve to obtain a homogeneous size. The husk ash that has been sieved is taken partly for characterization with the XRF test. The snail shell used is *achatina fullica* snail shell. After obtaining a completely clean shell, then dry in the sun. The next step is to smooth the shell with mortar and pestle. After that, the sample is sieved with a 200 mesh sieve and characterized by the XRF test.

Rice husk ash (3.75 g) and snail shell (6.25 g) mixed using magnetic stirrer in 250 ml beaker with ethanol 98% solution for 5 hours. The homogeneous samples were then heated using an oven for 24 hours at a temperature of 100 ^{o}C. Then, the sample was crushed. The powder sample printed into pellets and sintered at 900 ^{o}C, 1000 ^{o}C, and 1100 ^{o}C.

Characterization of samples was carried out to determine the number of elements contained, the sample phase, the surface morphology of the material and the functional group. Analysis of the number of elements using X-Ray Fluorescence (XRF) at Hasanuddin University. The phase of the sample was identified by X-Ray Diffraction (XRD) in Advanced Mineral and Mineral Laboratory of Malang University. Functional group testing using Fourier Transform Infra-Red (FTIR) in Lambung Mangkurat University.

Result and Discussion

Characterization Of XRF

The results of the XRF characterization of samples of rice husk ash and snail shells are shown in Table 1. Silicon (Si) is the most dominant element among other elements in rice husk ash. This element is suitable as source silica at the synthesis of calcium silicate. Content of the element K is quite large and this can affect the formation of calcium silicate. For that, purification is necessary. The other elements can still be ignored because the content is below 2%.

Table 1. Elements of rice husk ash and snail shell

Elements	**% (weight)**	
	Rice Husk Ash	**Snail Shell**
Si	86.35	-
K	7.81	-
Ca	1.93	99.34
Px	1.57	-
Fe	0.82	-
Mn	0.79	-
Cl	0.51	-
Zn	0.07	0.06
Ti	0.05	-
Rb	0.03	-
Nb	0.02	-
Mo	0.01	-
Sr	0.01	0.26
Ru	0.01	-
P	-	0.16
Cu	-	0.1

From Table 1, the highest content in the composition of the chemical elements of the snail shell is Ca, which is 99.34%. The second element, which has a higher content, is Sr, and then there are other elements such as P, Fe, Cu, and Zn. Ca elements form compounds that bind to the oxygen element to form CaO with the highest content value of 99.35%. All elements are bound to oxygen elements and have different content values. P_2O_5 compounds have a content of 0.3%, besides that Fe_2O_3, CuO, ZnO, and SrO have a value of 0.098%; 0.055%; 0.045%; and 0.19%.

Analysis of XRD Spectra

Spectra of Sample using XRD are shown in Figure 1. Figure 1 shows that the peak of calcium silicate begins to form at calcined at 900 ^{o}C. But, calcium silicate formed fewer than other temperature of calcination. Calcined at 1000 ^{o}C formed calcium silicate ($CaSiO_3$) and at 1100 ^{o}C there were most of all calcium silicate. This indicates that the higher the calcination temperature, the higher calcium silicate formed. This indicated there was a chemical mechanism in the sample for calcined. $CaCO_3$ from snail shell and SiO_2 from rice husk ash react to formed $CaSiO_3$. The analysis of sample characterization is presented in Table 2. Calcium silicate calcined at 1000 ^{o}C is beta calcium silicate β-$CaSiO_3$ (JCPDS84-0654). The temperature at 1100 ^{o}C was detected new peaks. This indicated that new phase. Compared beta calcium silicate, alpha calcium silicate occurs at 1100 ^{o}C (JCPDS 74-0874). So, the are more peaks at a temperature of 1100 ^{0}C.[16–19]

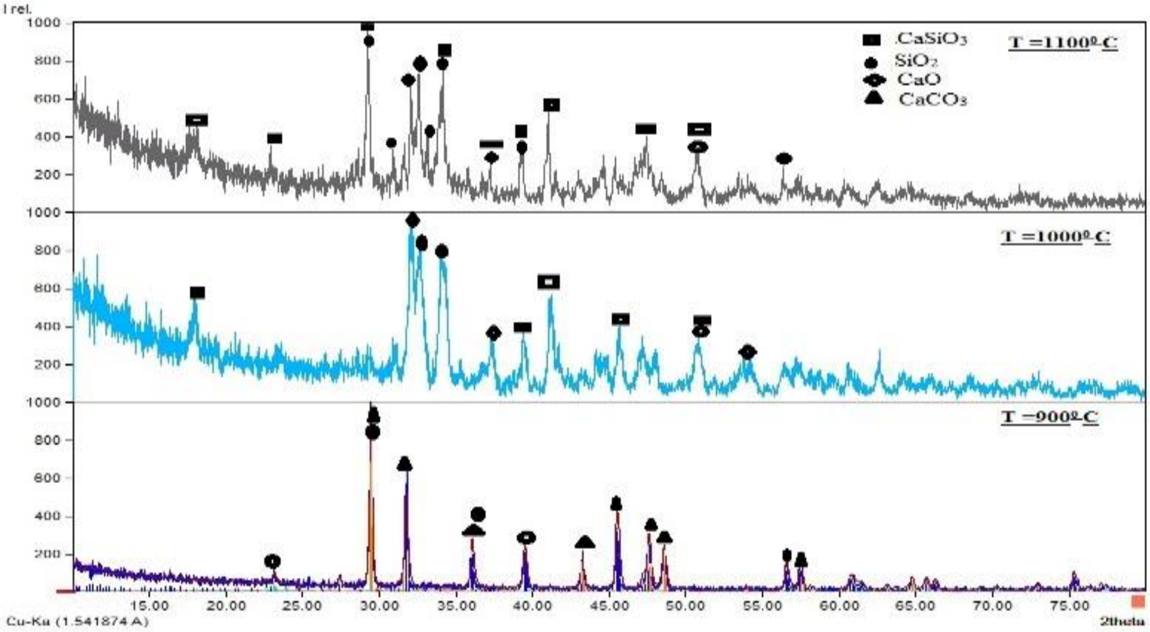

Figure 1. Spectra pattern of calcium silicate at 900 ^{o}C, 1000 ^{o}C, and 1100 ^{o}C

Table 2. The results of the sample characterization analysis using XRD

Temperature	Formation	Phase	Crystal System	(%)
900 ^{o}C	$CaCO_3$	Calcite	Trigonal	77.3
	$CaSiO_3$	Calcium silicate	Triclinic	11.7
	SiO_2	Cristobalite	Monoclinic	11.1
1000 ^{o}C	$CaSiO_3$	Calcium silicate	Triclinic	56.5
	SiO_2	Cristobalite	Monoclinic	31.5
	CaO	Lime	Cubic	12.0
1100 ^{o}C	$CaSiO_3$	Calcium silicate	Triclinic	72.3
	SiO_2	Cristobalite	Monoclinic	23.2
	CaO	Lime	Cubic	4.5

Based on Table 2, sample calcined at a temperature of 900 ^{o}C there is an element of $CaCO_3$ which is more dominating than other elements with a quantity of 77.3%, then the element $CaSiO_3$ with 11.7%, and SiO_2 11.1%. This indicated that at calcinating temperature

900 °C just little $CaCO_3$ transformed $CaSiO_3$. At 1000 °C, the element $CaSiO_3$ is higher with a quantity of 56.5% compared to the element SiO_2 which is valued at 31.5% and CaO 12.0%. Calcined at 1100 °C, the element $CaSiO_3$ has increased to 72.3%, and SiO_2 and CaO have decreased by 23.2% and 4.5%. the results indicated that calcined at temperature 1100 °C can increase calcium silicate. Quantity of phase from XRD peaks using software Match!. Figure 1 indicated that from 900 °C to 1000 °C transformed form higher crystallinity to lower crystallinity.[20,21]

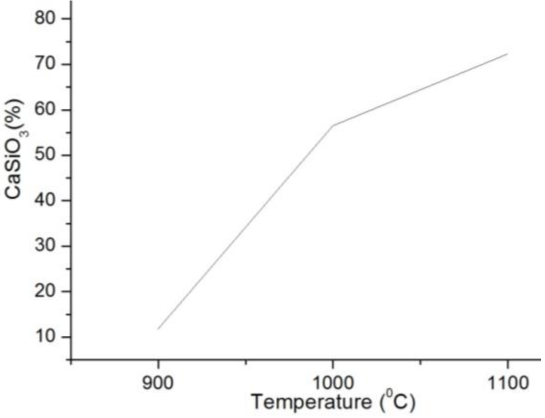

Figure 2. Percentage of $CaSiO_3$ with temperature 900 °C, 1000 °C, and 1100 °C

$CaSiO_3$ has increased in quantity from 900 °C to 1100 °C. This makes it possible for the basic ingredients of CaO and SiO_2 to be formed to have little impurities and contaminants and very small grain sizes so that at a temperature of 900 °C $CaSiO_3$ (Calcium silicate) has been formed. The element $CaCO_3$ is more dominating at 900 °C due to thermal reaction at this temperature is not optimal. The increase in sample quantity also indicates that the higher the calcinating temperature the quantity of calcium silicate will also increase as shown in Figure 2. This indicated calcination temperature can effect to formed calcium silicate. This is supported by previous reported.[2]

Characterization Of FTIR

The calcium silicate FTIR spectrum shows the absorption band at several wave numbers both at the calcination temperature of 900 °C, 1000 °C, and at the calcination temperature of 1100 °C. At 900 °C calcination temperature, absorption bands occur at wave numbers 507.7 cm^{-1}; 711.7 cm^{-1}; 874.7 cm^{-1}; 1090.8 cm^{-1}; 1411.0 cm^{-1}; and 1800.4 cm^{-1}. Absorption bands at a temperature of 1000 °C occur at wave numbers 517.9 cm^{-1}; 848,1 cm^{-1}; 872.7 cm^{-1}; 929.8 cm^{-1}; 1001.2 cm^{-1}; and 1417.1 cm^{-1}. Absorption band at a temperature of 1100 °C occurs at 526.1 cm^{-1}; 848.2 cm^{-1}; 899.2 cm^{-1}; 999.1 cm^{-1}; and 1419.1 cm^{-1}.

Wavenumber ranges of 507 - 526 cm^{-1} are vibrations of O-Si-O bonds.[8,22] This bond is a bond that forms silica bonds (SiO_2). O-Si-O bonds undergo dip shifts due to different calcination temperatures. At 900 °C calcination temperature, the bond occurs at wavenumbers 507.7 cm^{-1}, at a temperature of 1000 °C occurs at wavenumbers 517.9 cm^{-1}, while at a temperature of 1100 °C occurs at wavenumbers 526.1 cm^{-1}. Wave numbers 1411.0 cm^{-1}; 1417.1 cm^{-1}; and 1419.1 cm^{-1} indicates the vibration of C-O bonds that form CO_3.[23]

$CaCO_3$ were seen at 900 °C at wavenumbers 874.7 cm^{-1}. However, this bond changed to Ca-O at 1000 °C and temperature of 1100 °C with a wavenumber of 872.7 cm^{-1} and 899.2 cm^{-1} which corresponds to the XRD results in the previous discussion. C = O groups of carbon dioxide in the FTIR spectrum appear at a calcination temperature of 900 °C, this could be due to the interaction between calcium oxide and calcium silicate during the sample preparation process (13). $CaSiO_3$ is indicated at wavenumbers 848 cm^{-1} (at the temperature of 1000 °C and temperature of 1100 °C) and at wavenumbers 999-1001 cm^{-}(8,9,20). $CaSiO_3$ at 900 °C was not clearly seen in the FTIR spectra and were clearly seen when the calcination temperature was increased. This means that $CaSiO_3$ has not been optimally formed at 900 °C. it can be caused by $CaSiO_3$ very little at 900 °C so that bond vibration $CaSiO_3$ detected at very low absorbance. Previous research found that the formation of $CaSiO_3$ will be optimal above 900 °C. [2] This supports the results obtained in the XRD.

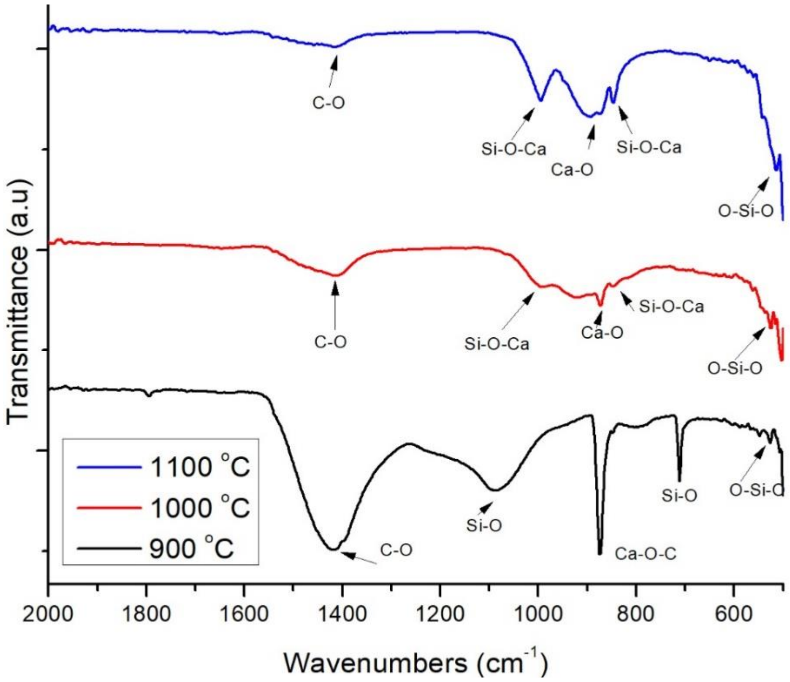

Figure 3. Spectra FTIR of calcium silicate at 900 ^{o}C, 1000 ^{o}C, 1100 ^{o}C using wavenumbers of 500 - 4000 cm^{-1}

Conclusion

Calcium silicate successfully formed from rice husk ash and snail shell by solid-state reaction. Silica content found in rice husk ash was 86.35% and the calcium content of the snail shell was 99.34%. phase of calcium silicate formed at 900 ^{o}C is $CaCO_3$, $CasiO_3$ (Calcium Silicate) increases with higher calcination temperature. The functional group was characterized using FTIR which results at 900 0C formed functional groups O-Si-O, Si-O, Si-O-Si, and Ca-O-C. The heating temperature in range 900-1100 °C could be effect the quantity of calcium silicate formed.

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