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Synthesis and study the properties of the wollastonite glass ceramics prepared from soda lime silica and cockle shell

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Abstract

In this paper, characterization of Wollastonite glass-ceramic consists of food wastes that are prepared by using SLS glass bottle and cockle shells, has been carried out. The cockle shells decompose the calcium oxide, CaO under thermal decomposition process, by eliminating carbon dioxide (CO_2) at the temperature 900°C. The XRF analysis of Wollastonite glass-ceramic has been done to determine its chemical compositions. The XRF result shows that, the major elements existed within the samples were CaO and SiO_2. But for the density, as the sintering temperature increase the density will be also increased.

Key words: Wollastonite, Cockle Shell, Soda lime silica, Glass, Ceramics

1 Introduction

Malaysia, a developing country produces around 29,000 tonns of municipal solid waste daily. In this country, more than 90 % of the total waste products were dumped off into landfills since it is thought to be the most prudent waste disposal option in Malaysia [1]. These landfills are filled up much faster than what has been planned. One of the factors that contributed to this problem is the lack of resource recovery such as recycling and other treatment options in the country's waste management system (Aja, 2014) [2]. Wollastonite ($CaSiO_3$) has a combination of interesting properties such as lack of volatile constituents, fluxing characteristics, low dielectric constant, low dielectric loss, thermal stability, low thermal expansion and low thermal conductivity. Wollastonite is a natural element that usually used in ceramic fabrications, medical materials for artificial bones and dental roots, high frequency insulators, filter material in resins and plastics, civil constructions, metallurgies, paints and frictional products. Since there is a wide usage of wollastonite especially in ceramic industries, producing wollastonite from waste materials is useful in order to minimize the waste products (Obeid, 2014) [3]. The economic and industrial growth causes a huge amount of waste produced. The waste product contains the valuable element, but need to be extracted. After that, the waste material can be used as the main element for the new products. For certain household wastes such as cockle shell and soda lime silica (SLS) glass bottles can be used as the source of calcium oxide (CaO) and SiO_2 respectively. The mixture of these two elements can produce other valuable product known as wollastonite, $CaSiO_3$ (Cornejo et al., n.d.) [4, 5]. Thus, the main purpose of this paper is to turn waste into wealth by producing the wollastonite glass ceramic from the waste sources such SLS glass bottles and waste cockle shell. In producing wollastonite, solid state method was used. This method is widely used for the preparation of polycrystalline solid from a mixture of solid starting materials because this method is simple and can save the time. This paper is to study the preparation and characterization of Wollastonite glass-ceramic production that created structure soda lime silica (SLS) glass and calcium oxide, CaO from cockle shells (CS).

2 Experimental

The raw materials utilized as a part of this study were cockle shell (CS) and soda lime silica (SLS) glass. Cockle shell (CS) wastes collected from the restaurant at Bangi (Malaysia) were washed with water, and kept at 200° C for dry in an oven for 2 hours. After that, the dried cockle shell (CS) was crushed so as to be pounded, a plunger and hammer were used and they were ground into powder by utilizing mortar and pestle. The cockle shell (CS) powder was undergone the heat treatments (calcination) process by the electric furnace at 900° C for 4 hours to remove the water as well as CO X and NO X in the cockle shell (CS). This process functions to decompose calcium carbonate ($CaCO_3$) to calcium oxide (CaO). Referring to work done by Mohamed et al., (2012) [6], cockle shell (CS) contains 95-99 % of $CaCO_3$ and its was converted into CaO after the calcination process. The heated cockle shell (CS) powder had been screened manually through a sieve to obtain an ideal size which is 63 μm.The reasonable soda lime silica (SLS) bottles were controlled basic of the raw material. At

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that point the cleaned soda lime silica (SLS) glass containers being crushed by utilizing a plunger and a sledge and were grounded by utilizing mortar and pestle and af-terward the soda lime silica (SLS) glass were sieved to get soda lime silica (SLS) powder size at 63 μm grain size. In this work, three different compositions of the samples were prepared. With a specific end goal to get the homogeneous powder mixture utilizing mortar and pestle to mixed the mixture that consist of 50 wt%, 40 wt% and 20 wt% of CaO acquired from cockle shell (CS) and 50 wt%, 60 wt % and 80 wt% of soda lime silica (SLS) glass powder. Table 1 shows the weight percentage (wt%) and the weight (g) of calcium oxide powder and (SLS) glass powder.

Table 1: The composition of batch sample of wollastonite a glass ceramics

Weight percentage		**Weight (g)**	
Calcium oxide powder	SLS glass powder	Calcium oxide powder	SLS glass powder
50	50	10	10
40	60	8	12
20	80	4	16

To get the homogeneous mixture, the milling machine was used to mix the sample for 24 hours. Then again the sieve was used to sift all mixed powder to get the fine powdery form with the size of 63 microns. Then, the mixed powder was undergone the melt process using the elec-tric furnace at 1500° C for 4 hours. Finally the melt sample was quenched in the water to get the glass sample. In order to get the glass powder, the glass sample was undergone the crushing and grinding process. After that, the glass powder was shifted to get the fine powder. Then the sample was undergone the palletizing process. For every 20.0 g sample for each composition, 0.5 g was used to make a pallet by using pressing technique for 5 tons of pressure and holding it for a period of time usually in 10 to 15 minutes. After that, the pallet was undergone the heat treatment by using the electric furnace. The pallet was sintered at three different tempera-tures which are 700° C, 900° C and 1000° C.

3 Results and Discussion

3.1 Chemical Composition Analysis (XRF)

XRF system was used to determine the chemical composition of the sample. In the Table 2, the chemical composition of raw materials for XRF analysis is collected. This table shows the composition element of the sample in SLS glass powder, powder of raw CS and powder of CS af-ter undergoing pre-heating. From table 2, one can say that the major components of SLS glass are silica (SiO_2) and lime (CaO) with percentage of 59 % and 37 % and for the minor components of SLS glass that cover the other 4 % of the per-centage are ferric oxide (Fe_2O_3), sulphur trioxide (SO_3), potassium oxide (K_2O), scandium (III) ox-ide (Sc_2O_3), titanium dioxide (TiO_2), zirconium dioxide (ZrO_2), strontium oxide (SrO), copper (III) oxide (CuO) and zinc oxide (ZnO).

Based on the table, it can also seen obviously that CS has 99 % calcium carbonate ($CaCO_3$) but after undergone the calcination process, calcium carbonate ($CaCO_3$) turn into lime (CaO). The percentage lime after pre-heating is about 98 %. The result obtained is quite similar as reported by Mo-hamed et al. (2012) [6] where the lime (CaO) exist is 98.99 %.

Table 2: Chemical composition by XRF analysis of raw material

Element	**SLS glass (%)**	**Raw CS(%)**	**CS after calcination (%)**
CaO	37.183	-	98.919
$CaCO_3$	-	99.698	-
SiO_2	59.657	-	-
Fe_2O_3	0.874	-	0.067
SO_3	0.752	-	-
K_2O	0.400	-	0.622
Sc_2O_3	0.393	-	-
TiO_2	0.372	-	-
ZrO_2	0.188	-	-
SrO	0.093	0.286	0.337
CuO	0.053	-	0.022
ZnO	0.036	0.016	0.016
Co_2O_3	-	-	0.017

Table 3 represents the percentage of the main components that contained in each sample at room temperature. It shows that the major lime (CaO) is 72.805% at the composition 50% SLS, 50% CaO and the minor composition is 53.550% at composition 80% SLS 20% CaO. But for the silica (SiO_2) the highest percentage at the com-position 80% SLS 20% CaO is 26.887% and the lowest percentage is 13.442% at the composition 50% SLS 50% CaO. For the alumina (Al_2O_3), the major percentage is 17.727% at the composition 60% SLS 40% CaO and the minor percentage is 11.981% at the composition 50% SLS 50% CaO. From the table, the obvious element that can be detected is lime (CaO). The lime (CaO) increased from 80% SLS 20% CaO to 50% SLS 50% CaO. This is due to the increasing amount of lime (CaO). But for the SiO_2, it is decreased from 80% SLS, 20% CaO to 50% SLS and 50% CaO re-spectively. This is because of the soda lime silica glass has a silica, so the increase of silica due to an increase amount of SLS glass.

Table 3: Chemical composition by XRF analysis at the temperature 30° C

Element	**Percentage (%)**		
	80:20	**60:40**	**50:50**
CaO	53.550	65.572	72.805
SiO_2	26.887	14.805	13.442
Al_2O_3	15.019	17.727	11.981

Table 4 shows the chemical composition of 80% SLS and 20% CaO at three different temperatures. The major element that has been shown in the table and graph are lime (CaO), silica (SiO_2) and alumina (Al_2O_3). The other minor element are scandium (III) oxide (Sc_2O_3), ferric oxide (Fe_2O_3), potassium oxide (K_2O), strontium oxide (SrO), zirconium dioxide (ZrO_2), chromium (Cr_2O_3), zinc oxide (ZnO), zinc oxide (ZnO), sul-phur trioxide (SO_3) and titanium dioxide (TiO_2) for the temperature 700°C, 900°C and 1000°C. Table 4 shows the amount of SiO_2 and Al_2O_3 de-creases as temperature increases.

Table 4: Chemical composition by XRF analysis for 80 % SLS and 20% CaO for different temperatures

Element	Temperature (°C)		
	700	**900**	**1000**
CaO	66.011	55.150	53.193
SiO_2	14.851	27.367	27.062
Al_2O_3	17.137	14.652	16.392
Sc_2O_3	0.429	-	0.419
Fe_2O_3	0.641	0.852	0.928
K_2O	0.228	0.375	0.365
SrO	0.348	0.237	0.213
ZrO2	0.262	0.219	0.189
Cr_2O_3	0.093	0.142	0.142
ZnO	-	0.115	0.114
CuO	-	-	0.050
SO_3	-	0.655	0.564
TiO_2	-	0.238	0.369

Table 5 shows the chemical composition of 60% SLS and 40% CaO that have been sintered at different temperatures. Figure 4 clearly show the CaO decreases as the temperature increases. Other minor elements that have been detected are silica (SiO_2), alumina (Al_2O_3), scandium (III) oxide (Sc_2O_3), ferric oxide (Fe_2O_3), potassium oxide (K_2O), strontium oxide (SrO), zirconium dioxide (ZrO_2), chromium (Cr_2O_3), zinc oxide (ZnO), copper (III) oxide (CuO), sulphur trioxide (SO_3) and magnesium oxide (MnO).

Table 5: Chemical composition by XRF analysis for 60 % SLS and 40% CaO for different temperatures

Element	Temperature (°C)		
	700	**900**	**1000**
CaO	53.171	65.459	64.175
SiO_2	27.844	15.619	14.531
Al_2O_3	15.110	16.334	19.195
Sc_2O_3	1.613	-	-
Fe_2O_3	0.788	0.952	0.979
K_2O	0.310	0.230	0.206
SrO	0.185	0.449	0.335
ZrO_2	0.163	0.251	0.260
Cr_2O_3	0.109	-	0.195
ZnO	0.113	0.054	0.088
CuO	0.042	-	0.037
SO_3	0.460	0.569	-
Ag_2O	0.056	-	-
MnO	0.037	-	-
NiO	-	0.083	-

The chemical composition of 50% SLS and 50% CaO at different temperatures is shown in the Table 6. The obvious components that have been shown in the table and the graph are lime (CaO) and silica (SiO_2). The other minor components that have been detected are alumina (Al_2O_3), scandium (III) oxide (Sc_2O_3), ferric oxide (Fe_2O_3), potassium oxide (K_2O), strontium oxide (SrO), zirconium dioxide (ZrO_2), chromium (Cr_2O_3), zinc oxide (ZnO) and copper (III) oxide (CuO). Table shows that the amount of CaO and SiO_2 decreasing as temperature increases.

Table 6: Chemical composition by XRF analysis for 50 % SLS and 50% CaO at different temperatures

Element	Temperature (°C)		
	700	**900**	**1000**
CaO	84.671	75.450	73.467
SiO_2	13.258	12.135	12.709
Al_2O_3	-	10.080	11.500
Sc_2O_3	0.164	0.197	0.228
Fe_2O_3	0.869	0.905	0.694
K_2O	0.250	0.581	0.546
SrO	0.352	0.293	0.363
ZrO_2	0.195	0.158	0.218
Cr_2O_3	0.162	0.161	-
ZnO	0.045	0.041	0.241
CuO	0.034	-	0.034

3.2 Density

Table 7 contains the density for the three compositions of prepared samples that are 80 % SLS 20 % CaO, 60 % SLS 40 % CaO, and 50 % SLS 50 % CaO with the different temperatures at 30° C, 700° C, 900° C and 1000° C. From the Table 7, one may say that the highest density around 2.810 g/cm^3 at temperature 700°C for the composition 80% SLS 20 % CaO [7] and the lowest is 2.305 g/cm^3 at a temperature 30° C for composition 80% SLS 20% CaO. The density of the composition 80% SLS 20% CaO is increased when we increase the temperature from 30° C to 700° C and drop, from temperature 700° C to 900° C, then increased again at temperature 900° C to 1000° C. For the composition 60 % SLS 40 % CaO, it shows that the density is increased from the 30° C to 900° C and decreased from 900° C to 1000° C. It can be concluded that the density is increase to the temperature. For the all composition, we can see the density is increased from 900° C to 1000° C. Lastly, the density of the composition 50 % SLS 50 % CaO is linearly increased as temperature increases. When samples are at the lower temperature, possesses the amorphous phase but when the temperature increased, the samples are slowly intended towards the crystalline phase. This is why due to the density at the lower temperature (30°C) is lower than the density at higher temperature (1000°C). Since the density at the higher temperature is high, the formation of porosity is less in higher temperature leading to more compact sample.

Table 7: Value for density for all samples

Raw Material		Density (g/cm^3)			
SLS	CaO	30° C	700° C	900° C	1000° C
80	20	2.305	2.810	2.440	2.623
60	40	2.522	2.612	2.619	2.593
50	50	2.672	2.684	2.800	2.797

3.3 Linear Shrinkage

The glass powder was pressed into the pallet and sintered at various temperatures. The shape of samples becomes more rounded when it reached the main melting point of the sample. Table 8 shows the percentage shrinkage obtained for each sample. The highest value for percentage is 9.96%, which is of the sample 80% SLS, 20% CaO at temperature 700°C, while the lowest percentage value is 0.15% which is that of the sample 80% SLS, 20% CaO at temperature 700°C. Generally, the higher sintering temperature results the higher of the percentage of shrinkage [8]. There is a lot of porosity before sintering process is done and after sintering process porosity can be removed. With higher sintering temperature particle tend to grow excessively and therefore more porosity will be removed.

Table 8: Shrinkage percentage value of the sample

Sample	Temp	Diameter (mm)		Shrinkage %
		D_i	D_f	
80% SLS 20% CaO	700 °C	13.04	13.02	0.15
	900 °C	12.99	12.02	7.46
	1000 °C	12.95	11.66	9.96
60% SLS 40% CaO	700 °C	13.01	12.80	1.61
	900 °C	13.01	12.50	3.92
	1000 °C	13.06	12.08	7.50
50% SLS 50% CaO	700 °C	13.03	12.73	2.30
	900 °C	13.02	12.15	6.68
	1000 °C	13.03	12.05	7.52

3.4 X-ray Diffraction

XRD measurement of the samples was used in order to identify the crystalline phase of the sample. Figure 1 and 2 illustrate the XRD pattern of the crystalline structure of CS. Arogonite ($CaCO_3$) was found almost at all peak of the pattern for pure CS. This result was synchronized with Mohamed et al. (2012) [6], where XRD showed that the raw CS made up of aragonite ($CaCO_3$). But for the CS after undergoing pre-heating, the port landite ($Ca(OH)_2$) was found at the highest peak of the XRD pattern.

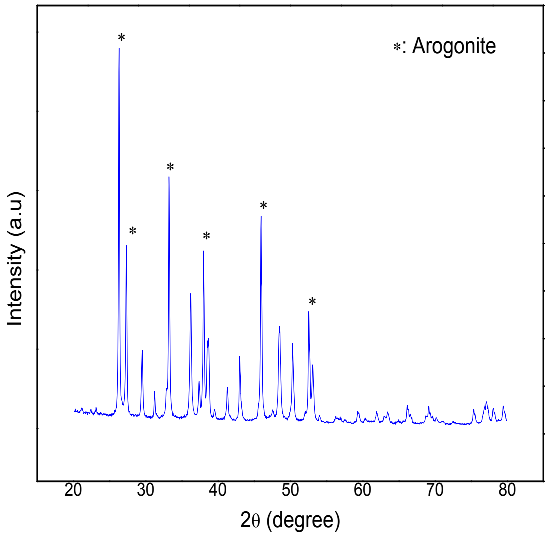

Figure 1: XRD pattern of raw CS

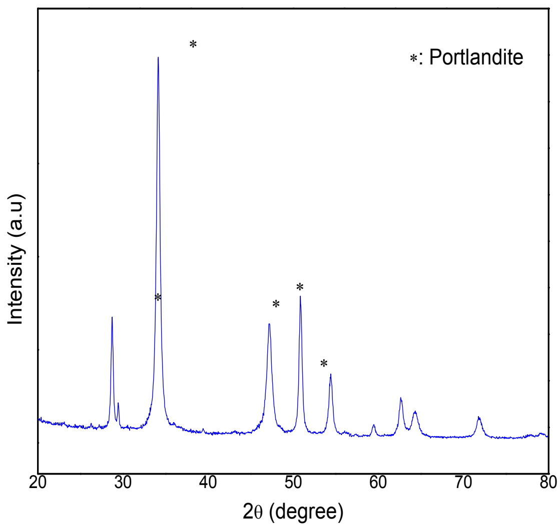

Figure 2: XRD pattern for CS after pre-heating

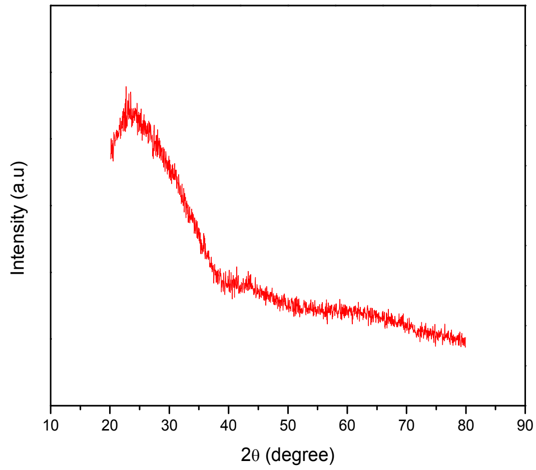

Figure 3: XRD pattern of SLS glass bottle

Figure 3 shows the XRD pattern of the SLS glass bottle. The pure SLS glass was found at a low angle in the range of 20°-30°, which indicates that the amorphous nature of glass sample. This finding was consistent with the finding obtained by (Brunner et al., 2006) [9]. At this range, the SLS glass possessed a short range and random arrangement of atoms. Figure 4 represents the XRD pattern for different composition at room temperature. There are no Wollastonite peaks at this temperature, which clearly shows that the compound is more amorphous at room temperature.

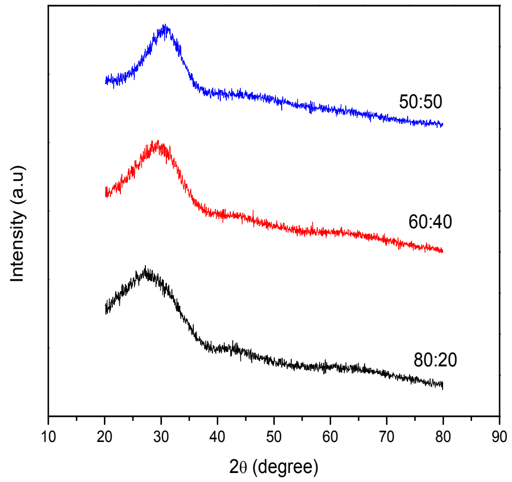

Figure 4: XRD pattern of the mixture SLS glass and CS at room temperature (30° C)

The XRD pattern of composition 80% SLS 20% CaO after sintered at three temperatures are shown in the Figure 5. From the graph in the Figure 5, it showed that the samples exist crystalline phases at 900°C and 1000°C. The highest peaks occurred at θ = 30°C, this peak is wollastonite. But for 700°C, there are no wollastonite peaks which indicate that the compound is more amorphous at this temperature.

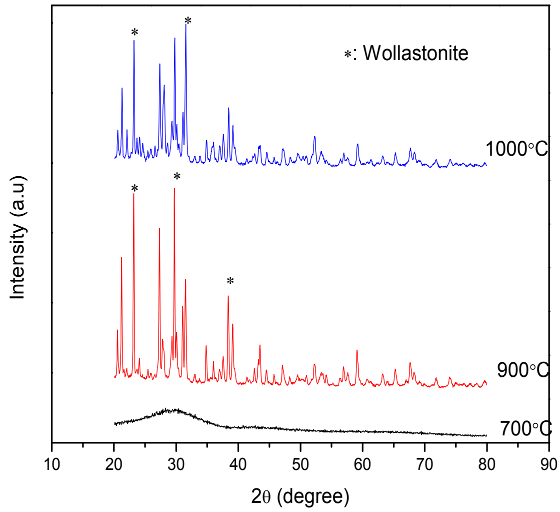

Figure 5: XRD pattern for composition 80 % SLS 20 % CaO at different temperature

Figure 6 and 7 show the XRD pattern for 60 % SLS, 40 % CaO and 50 % SLS, 50 % CaO. From the Figures 6 and 7, we have seen that at 700° C there is no peak presents. At this temperature, the compound is more amorphous but from 900°C to 1000°C it had a crystalline phase. Gehlenite was found at the highest peak and the highest peak obtained at θ = 30°. The result obtained is similar to the study of Zhang & Liu, 2013 [10].

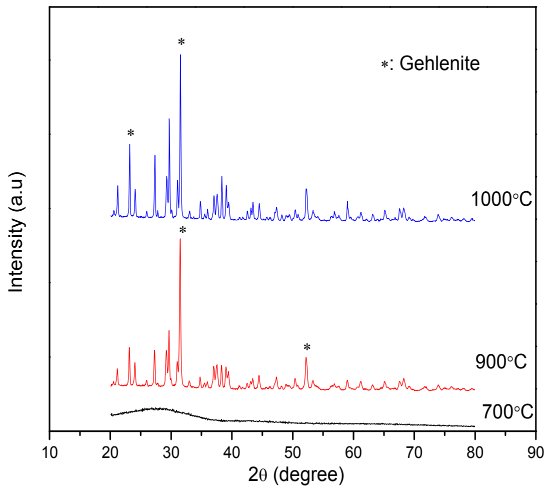

Figure 6: XRD pattern for composition 60 % SLS 40 % CaO at different temperature

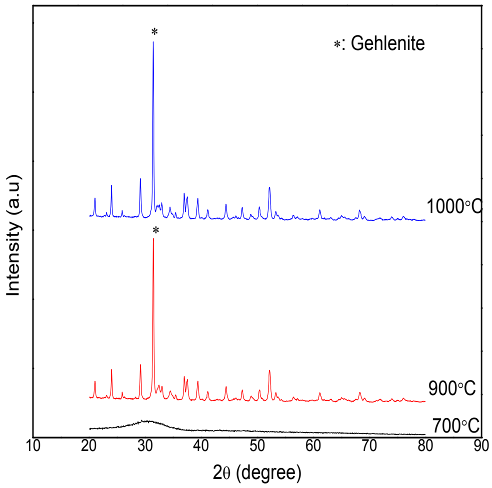

Figure 7: XRD pattern for composition 50 % SLS 50 % CaO at different temperature

4 Conclusions

Wollastonite glass-ceramic has been synthesized successfully from CS and SLS glass bottle. The three samples had been prepared for the composition of 80 % SLS 20 %, CaO, 60 % SLS, 40 % CaO and 50 % SLS, 50 % CaO. From the XRF results, one may conclude that the raw CS contains the CaO and the raw SLS glass contains the SiO_2. The density measurements, clearly shows that as the sintering temperature increased, the density also increased for the composition 50 % SLS, 50 % CaO. The shrinkage also follows the trend of density, so density is related to the effect of shrinkage occurring in the samples as the sintering temperature increase. From the XRD results it shows that at the lower temperature at 700° C, the sample is still in the amorphous phase. But when the temperature starts to increase from 900° C, the sample slowly formed crystalline. The gehlenite was found at the highest peak for the composition 50 % SLS, 50 % CaO. Based on the experiment and analysis, it indicated that the temperature at 900° C and sample for composition 50 % SLS, 50 % CaO is the best temperature and composition for Wollastonite to exist compare to the others temperature and composition.

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