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Preparation and Characterization of Glass Ceramic Materials from Mixtures of Waste Oyster Shell and Recycled Soda-Lime-Silica Glass

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Abstract: Waste Oyster Shell (OS) and Soda-Lime-Silica Glass (SLS) waste has become one of the uncontrolled wastes which give negative impact to our environmental. To overcome that, the research on Preparation and Characterization of Glass Ceramic (GC) Materials from Mixtures of Waste Oyster Shell and Recycled Soda-Lime-Silica Glass has been reported. This research was one of the alternative ways in order to overcome environmental pollution issue and in other side it also gives the high profit to the industry to produce GC only by recycled the waste. The method uses in this research was Solid State Conventional Method and sintered at 900˚C for 2 hours before it has been characterized to study their properties. The data shows that the mass loss of the sample was increase due to the increasing of the OS content. FTIR analysis show the peak that show the deformation of SiO_2 non-bonding, Si-O-Si asymmetric stretching mode, bonding of C=O group. From XRD result, the sample indicating crystalline structure which can be seen the clear peak of calcite, quartz, devitrite, and cristobalite crystal phase on the 30OS70SLS sample which have related with the FTIR result. The maximum density of the sample was recorded at 2.7049 $gcm^{-3}$for powder density and 2.3410 gcm^{-3} for bulk density with acceptable percentage porosity value 13.47% for sample 30OS70SLS. Besides that, the SEM-EDX showed the existing of pores on the sample and also prove high element of Ca and Si on the 30OS70SLS sample.

Keywords: Porous Glass Ceramic, Oyster Shell, Soda-Lime-Silica, 30OS70SLS

1. Introduction

Recently there has been renewed interest in developed to the new idea in produce the product that give more profit to them and environmentally friendly. This is because people must reuse the solid waste to overcome the environmental problems due to illegal dumping and environmental pollution [1].

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So, what they do is recycling the waste that always increase day by day and produce new product. Another one scientific example of that idea is by mixing the waste of oyster shell and soda-lime silica glass to produce Porous Glass Ceramic (PGC). PGC is categorized as those ceramic that having high percentage porosity between 20% to 95% [2]. The use of this PGC is such as in lightweight, rigid, water and steam resistant, low transport, construction, filtration and bioimplants [3]. This PGC has high demand in around the world especially as construction material just because of their properties. This statement has been support by Sobey,2008 which has proved that PGC delivered the fire-resistance material, high temperature refractories and have good thermal and acoustic insulation properties which very suitable for concreate.

Due to the statement above, the aim of this research is to prepare porous glass ceramic from a mixture of waste oyster shell and recycle soda-lime-silica glass by using solid state conventional method which have been sintered at 900˚C for 2 hours at difference composition. The sample of porous glass ceramic then will be used to studies on the porosity values based on the relationship of bulk and powder density. Furthermore, from that sample we will observe the density, porosities value, crystal phase, the chemical bond, and also surface morphology of the PGC sample. By all of this characterization, we can find that which is the suitable composition that can be used by comparing all of the characterization data in this research.

2. Materials and Methods

2.1 Sample Preparation

Both of the raw material SLS glass and OS waste which has been collected at Muar, Johor were cleaned and dry to remove any foreign substance. Then the SLS glass were crush by using frit piston to make small pieces of glass while the OS was calcined in the oven for 6 hours at 1000˚C. The SLS glass and the OS then were grind repeatedly by using pestle and mortar to make it into finer powder before it had to be sieved through 50µm sieve mesh. Both of the powder sample were homogeneously mixed according to the ratio as shown in Table 1 below. The powder then was weighed for 2g prior to uniaxially pressed by using Hydraulic Pressure Machine. The sample was pressed at 3 tons consistently at 1 minutes. The pellet sample was put into the Electric Box Furnace (Protherm PLF) for the sintering process at 900˚C for 2 hours with rate 5˚C/min. The sample then ready to be used for study their characterization.

Table 1: Composition of OS and SLS sintered at 900℃

Batch Composition (wt.%)		Designation
Oyster Shell	Soda-Lime-Silica	
0	100	100SLS
10	90	10OS90SLS
20	80	20OS80SLS
30	70	30OS70SLS
40	60	40OS60SLS
50	50	50OS50SLS

2.2 Characterization Methods

Mass loss of the sample was calculated by using the mass before and after the sintering process which has been recorded while preparing the sample. This data used to plot graph in order to show the relation between the mass loss and oyster shell composition in the sample. The powder of the sample then will undergoes Fourier Transform Infrared (FTIR) by using Spectrum Two Machine to determine the organic component and the chemical bond in the sample. XRD is the one of the techniques that used for this research to know the crystallographic structure of the sample by using Bruker D2 PHASER 2nd generation X-ray Diffraction Machine. Next, we will observe the powder density and bulk density by using Newton EJ-303 Portable Balance and AccuPyc II 1340 gas displacement pycnometer Machine. The sample preparation for bulk density was on pellet form whereas for the powder density use the powder sample. By using this density data, we can calculated the porosity data of each sample. The formula to calculate the porosity value based on the powder density and bulk density was show in formula below:

$$Porosity = \left(1 - \frac{bulk\ density}{powder\ density}\right) \text{x } 100$$

For surface morphology, we use COXEM EM-30AX scanning electron microscopy (SEM). This machine will view the high resolution of clear images for the morphology structural including and also including the images of pores on the selected sample.

3. Results and Discussion

3.1 Mass Loss

The result on Figure 1 show that the percentage mass loss of the sample was increase due to the increasing of oyster shell content. The maximum mass loss is 12.86% at 50OS50SLS. Calcium carbonate ($CaCO_3$) was one of the main chemical compositions in OS. During the heat treatment process, decomposition of $CaCO_3$ to form calcium oxide (*CaO*) and Carbon Dioxide (CO_2) was occurred. The decomposition reaction of *CaCO3* during the heat treatment process was show in the formula below:

$$CaCO_3 \longrightarrow CaO + CO_2$$

This production of CO_2 during the sintering process will increase the mass loss of the sample. So, the trendline in Figure 1 show that if oyster shell content in the sample increase, the CO_2 gas release during the decomposition of $CaCO_3$ will be increase and caused the mass loss of the sample to increase.

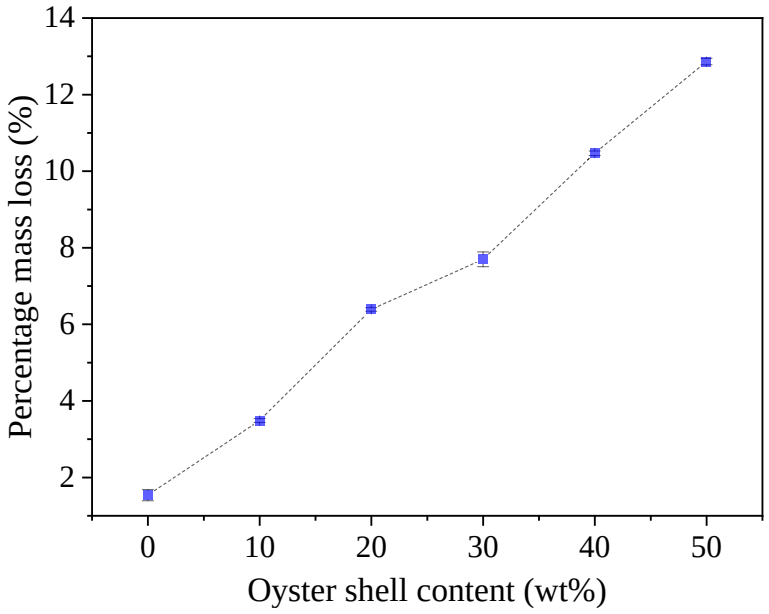

Figure 1: Graph of mass loss against OS content sintered temperature of 900℃.

3.2 FTIR

The FTIR vibration range mode wavelength in Figure 2 was obtained from 400 cm^{-1} to 1500 cm^{-1}. According to the FTIR spectra show on the graph there are three chemical bonding that will be highlight for this research is SiO_2 non-bonding at 514 cm^{-1} [4]. The existing of this SiO_2 bonding is because it is one of the dominant compounds contain in SLS. Second is *Si-O-Si* asymmetric stretching mode at 1021 cm^{-1}. It is due to the deformation of *Si-OH* during the sintering process. From the existing of *Si-O-Si* bonding, it shows that the Si^{4+} and O^{2-} ions were realigned in order to form the α-quartz [5]. And the last one is *C=O* bonding at 875 cm^{-1}. The bonding of *C=O* group show the existing of the carbonate ion which implied of the involvement of $CaCO_3$ on the sample.

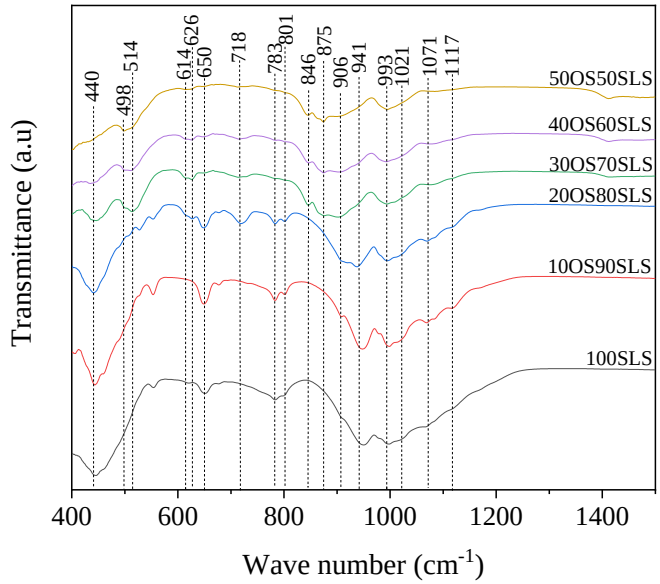

Figure 2: FTIR spectra of OS mixed with SLS with different composition sintered at 900℃.

3.3. XRD

For the XRD, we have plot the intensity vs 2Θ graph of each sample composition as shown in Figure 3. From that graph above it show that the broad hump characteristic of all of the sample indicate crystalline structure pattern. Some of the crystalline peak that can be seen in the graph is calcite, quartz, devitrite and cristobalite. Calcite is a species of carbonation products precipitated by carbonic reaction. The highest peak that shows the calcite crystalline phase is at the 30OS70SLS sample. This data can be related with the FTIR data which show the C=O bonding which is the functional group of forming calcite crystalline [6].

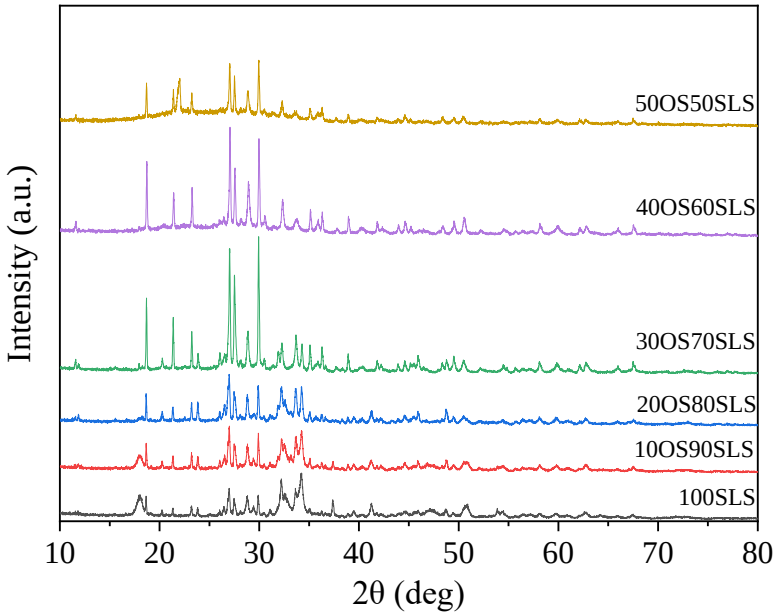

Figure 3: XRD patterns for different composition of OS and SLS.

3.4 Density

Density of the porous glass ceramic sample is divided into two which is powder density and bulk density. From the Figure 4 it shows that both powder density and bulk density is increase until content of oyster on the sample archive to 30 wt% and slowly decrease back when the oyster shell content up to 40 wt%. The maximum value of powder and bulk density is 2.7049 $gcm^{-3}$and 2.3410 gcm^{-3}. This data is due to the formula of density and mass loss. When the mass loss of the sample increase, the relative density of the sample will also increase. The reason why at 40 wt% content of oyster shell the density of the sample starts to decrease just because the energy to heat the sample during the sintering process which is 900˚C is not enough to convert all $CaCO_3$ content in the sample into CaO and CO_2 gas.

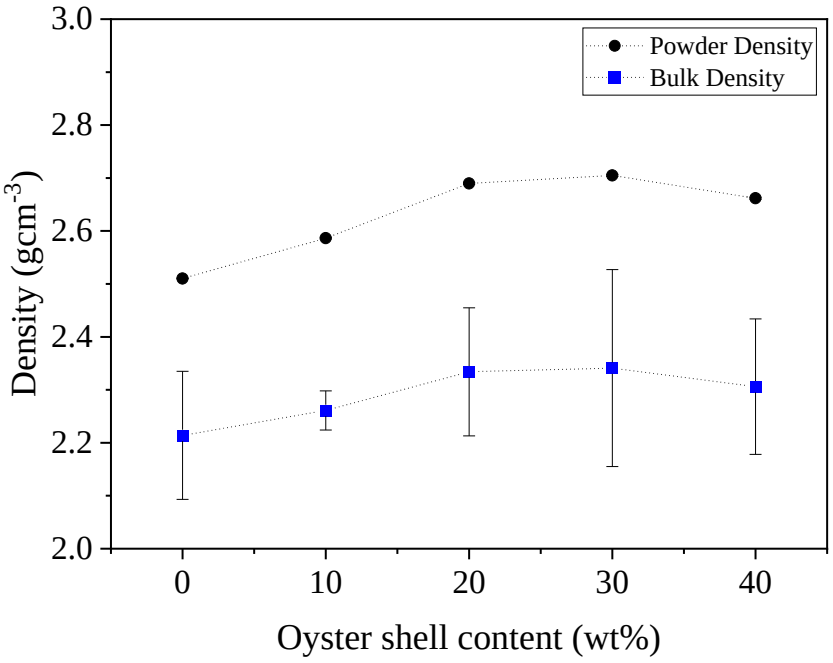

Figure 4: Bulk density and Powder density versus OS content.

3.5 Porosity Value

The porosity value of the sample was calculated by using the relation between bulk and powder density. As show in Figure 5, the percentage porosity was increase dramatically with the increasing of oyster shell content. The highest point of porosity value is at 30 wt% of oyster shell content which is 13.47%. This pore is produced from the heat reaction of $CaCO_3$. During the sintering process at 900˚C, $CaCO_3$ will decomposes into CaO and CO_2 gas. This CO2 gas which releases during the decomposition of CaCO3, will cause the bubble of gas which then will become a pore in the porous glass ceramic sample. The porosity value starts to decrease when the oyster shell increases up to 30 wt% because the heat energy during the sintering process was not enough [7]. This heat energy during the heat treatment process is used to decomposes all of the $CaCO_3$ contain in that sample. This show that for sintering process at the 900˚C the best composition for with high porosity value is at 30 wt% of oyster shell and 70 wt% of SLS glass.

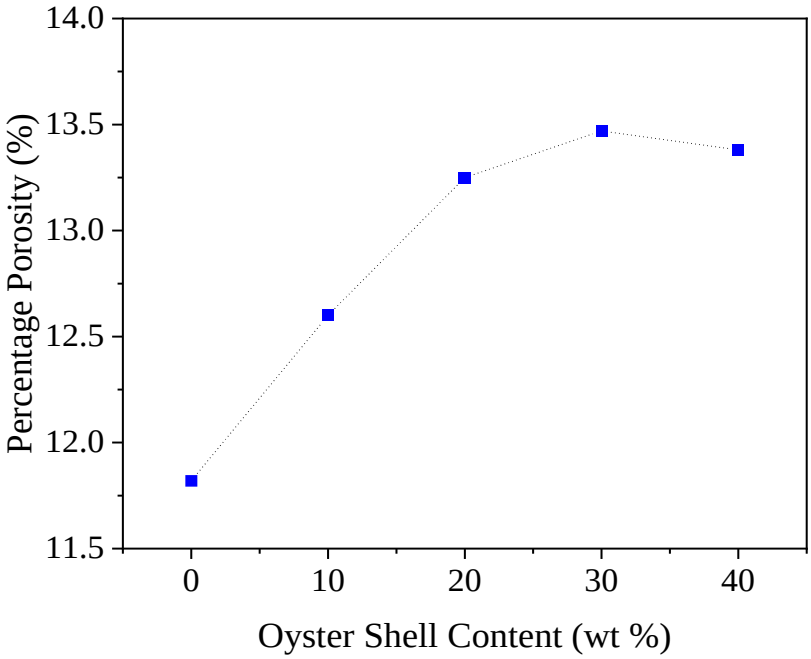

Figure 5: Porosity versus Oyster content graph.

3.6 SEM-EDX

From the previous characterization we know that the best composition for this research was the 30OS70SLS. So that, 30OS70SLS sample is use to observe the surface morphology of that sample and find out the characterization of that sample. Figure 6(a) show the SE images for 30OS70SLS with 3 selected area A, B and C. The element found in that selected area is show in the Table 2. Overall data in table 2 show that the high element show in the sample is Carbon, Oxygen, Sodium and Silicon and Calcium. Figure 6(b) show that there are many pores in that sample. These pores proved that during the sintering proses there was existing of CO_2 gases which come from the decomposition of $CaCO_3$ on the sample. Because of this decomposition also, there we can observe the existing of CaO composition on the surface of the sample. Table 2 show the data of the spectrum on the selected area which have high element of Ca and Si on the surface of the sample. This has been proven by the element data of spectrum 1, spectrum 2 and spectrum 3 in Table 2.

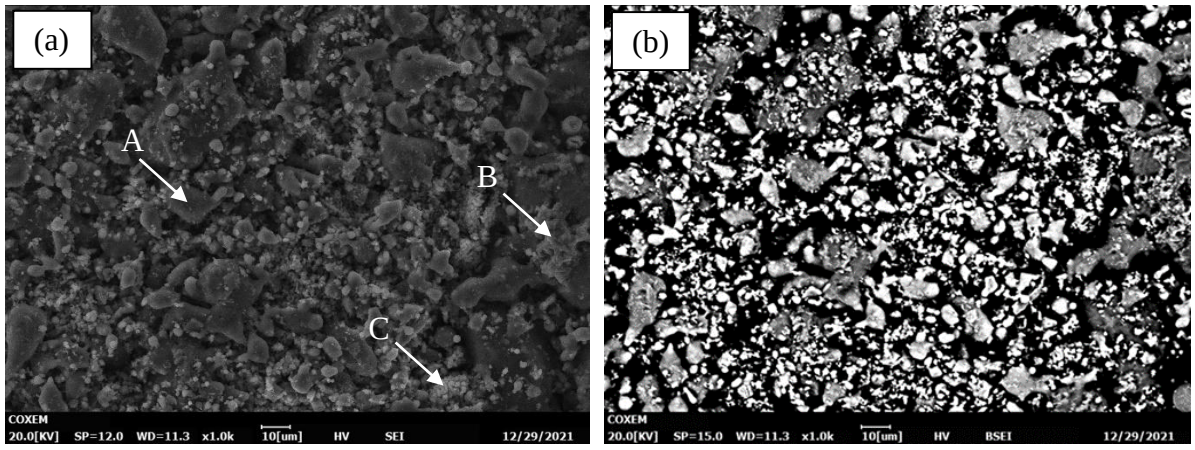

Figure 6: (a) SE image (b) BSE image of 30OS70SLS

Table 2: Elements in selected area

Element	Area A	Area B	Area C
	Atomic Percent (at%)		
C	13.97	12.85	13.82
O	59.18	59.91	60.28
Na	4.99	8.96	5.94
Si	9.80	11.10	9.91
Ca	10.42	6.33	8.88
Au	0.52	0.53	0.53
Mg	0.47	0	0
Fe	0.03	0.05	0.02
Al	0.30	0	0.32
Br	0	0.27	0
Rb	0.31	0	0.30
Total	99.99	100.00	100.00

4. Conclusion

At the end of this research, we notice that the sintering process give an effect on the mass loss of the sample. The data show that the mass loss of sample increase when the oyster shell content decrease due to the decomposition of $CaCO_3$ during the heat treatment process. FTIR spectra show the deformation of Si-OH, formation of Si-O-Si bond, bonding of C=O group and O-H stretching carboxyl group. This data can be related with the XRD result, it shows there are 4 crystalline phase that can be observe clearly at sample 30OS70SLS which is calcite, quartz, devitrite and cristobalite. The density graph also shows that the highest powder and bulk density value for 30OS70SLS which is 2.7049 gcm^{-3} and gcm^{-3}. This result to the highest porosity value at 13.47% for 30OS70SLS sample compared to other composition. The result was being support by the surface morphology of the sample by using SEM which show there are many pores and calcium element on the surface of the sample of 30OS70SLS. The formation of pores was due to the formation of bubble CO_2 gas from the decomposition of $CaCO_3$ to form CaO. From all of the result above, we can conclude that the porous glass ceramic sample with 30 wt% of oyster shell was the best composition for sintering at 900˚C for 2 hours compare to other composition.

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References

[1] Benzerga, R., Laur, V., Lebullenger, R., Gendre, L. L., Genty, S., Sharaiha, A., & Queffelec, P. (2015). Waste-glass recycling: A step toward microwave applications. *Materials Research Bulletin*, *67*, 261–265. https://doi.org/10.1016/j.materresbull.2014.07.037

[2] Guo, H., Gong, Y., & Gao, S. (2013). Corrigendum to 'Preparation of high strength foam glass-ceramics from waste cathode ray tube' [Mater. Lett. 64 (2010) 997–999]. *Materials Letters*, *107*, 9. https://doi.org/10.1016/j.matlet.2013.05.040

[3] Bernardo, E., Castellan, R., & Hreglich, S. (2007). Sintered glass-ceramics from mixtures of wastes. *Ceramics International*, *33*(1), 27–33. https://doi.org/10.1016/j.ceramint.2005.07.012

[4] Nandiyanto, A. B. D., Oktiani, R., & Ragadhita, R. (2019). How to Read and Interpret FTIR Spectroscope of Organic Material. Indonesian Journal of Science and Technology, 4(1), 97. https://doi.org/10.17509/ijost.v4i1.15806

[5] Zaid, M., Matori, K., Ab, A., Wahab, Z., & Rashid, S. (2017). Effect of sintering on crystallization and structural properties of soda lime silica glass. Science of Sintering, 49(4), 409–417. https://doi.org/10.2298/sos1704409z

[6] Seo, J. H., Park, S. M., Yang, B. J., & Jang, J. G. (2019). Calcined Oyster Shell Powder as an Expansive Additive in Cement Mortar. Materials, 12(8), 1322. https://doi.org/10.3390/ma12081322

[7] Chinnam, R. K., Bernardo, E., Will, J., & Boccaccini, A. R. (2015). Processing of porous glass ceramics from highly crystallisable industrial wastes. Advances in Applied Ceramics, 114(sup1), S11–S16. https://doi.org/10.1179/1743676115y.0000000053