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The Effect of Lime Stone in the Probability of Formation Pores Structures in Glass Ceramic Based on Scoria Basalt Rocks

David Candra Birawidha[1], Slamet Sumardi[2*], Fany Oktaviando[3], Pulung Karo-Karo[4], Kusno Isnugroho[5], Yusup Hendronursito[6], Muhammad Amin[7]

[1,2,5,6,7] Research Centre for Mining Technology, National Research and Innovation Agency, Lampung, Indonesia
[3,4] Department of Physics, FMIPA, Universitas Lampung, Lampung, Indonesia

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ABSTRAK

Seiring berkembang pesatnya teknologi dibidang material, teknologi pembuatan material ringan juga semakin berkembang dimana tujuannya untuk mengurangi berat total tanpa mengurangi kekuatan mekanis dari suatu material. Beberapa parameter yang mempengaruhi dalam pembuatan material ringan adalah jumlah pori, berat material serta ketahanan fisik. Salah satu metode yang umum digunakan adalah dengan mencampurkan glass ceramic dengan foaming agent. Penelitian ini digunakan batuan basalt dari daerah Lampung Timur, Indonesia yang dicampurkan dengan batu kapur yang dilebur pada suhu 1100 °C dan 1300 °C. Kemudian sampel divariasikan antara kapur dan basalt dengan perbandingan sampel A (100% Basalt), sampel B (3:7), sampel C (5:5), dan sampel D (7:3) yang di bakar pada suhu 1100°C dan 1300°C. Berdasarkan hasil karakterisasi, sampel glass ceramic terbaik yang terbentuk adalah sampel B yang dibakar pada suhu 1300°C dengan jumlah pori sebesar 8% dan densitas sebesar 2,204 g/cm3. Berdasarkan hasil uji SEM, pori- pori dengan ukuran ≤0,5µm sebanyak 85% dan ukuran ≥0,5µm sebanyak 15%,dimana struktur glass ceramic terdeteksi fasa pyroxene dan lime dengan komposisi SiO2 sebanyak 21,61%.

ABSTRACT

This paper provides a Lightweight material is the result of technological problems in increasing the efficiency of finished products, saving manufacturing costs and environmentally friendly technology by reducing the amount of material used. There are many kinds of material manufacturing technology, ranging from the use of lightweight materials from the start, combining materials into composites and modifying the structure and characteristics of the material to make it lightweight. One commonly used method is to mix glass-ceramic with a foaming agent in purpose to modify the structure of material. The purpose of this study is to utillize basalt rock as source of glass ceramic and mixed with limestone to form cellular structure with optimal composition. The samples was crushed and sieve through 100 mesh afterward all material is mixed varied between basalt and lime with a ratio of sample A (100% Basalt), sample B (3:7), sample C (5:5), and sample D (7:3), which were burned at a temperature of 1100°C and 1300°C. After all sample reach designated temperature, all sample undergo annealed cooling in the furnace. Based on the characterization results, the best glass-ceramic sample formed with pores structure formation was sample B which is 70% addition of limestone in basalt mixture and burned at a temperature of 1100°C with a total pore size of 63% and a density of 0.92 g/cm3, where the glass-ceramic structure detected pyroxene and lime phases with a SiO2 composition of 14.61%. Basalt cellular ceramic is obtained in optimal condition with low density and higher percentage porosity.

1. INTRODUCTION

Material-making technology is growing with the pace of development of the times. Starting from rock-based materials, progressing to metals to polymers and composites as we know them today. The development of the material is also not limited to increasing its mechanical strength but has begun to expand to the aspect of dimensions to weight without having to reduce the advantages of its mechanical properties (Gao et al., 2018; Wang et al., 2019). One of the technologies currently developing, of which is a material that is light in weight but superior in its mechanical strength.

The use of lightweight materials has many positive sides, including reducing the cost of producing a product, reducing the consumption of basic materials so as to increase environmental friendliness and increase the savings in energy used. Based on the raw materials, the manufacture of lightweight materials that are commonly found in the market is those from aluminum, magnesium, or polymer alloys (Pu et al., 2018; Sorrentino et al., 2014). At first, light material technology focused more on the aerospace field and

*Corresponding author.
E-mail addresses: slametsumardi99@gmail.com (Slamet Sumardi)

then expanded to the field of machinery construction. And from the manufacturing technique, light materials are generally made into composites or modify the structure characteristic by made hollow with the bubble technique.

In terms of economic factors and alternative basic materials, the technology for making lightweight materials is starting to look at the potential for underutilized and abundant raw materials. One that is being developed is basalt rock which is combined with the bubble technique in the softening phase to form a lighter weight but maintain its original properties (Isnugroho et al., 2018; Marangoni et al., 2014). Basalt rock itself have massive and hard nature, aphanitic texture and consisting of volcanic glass minerals, plagioclase, pyroxene, amphibole, and black minerals, in the softening phase which is close to its melting point, by applying the bubble technique of trapped gas during the cooling process, the density of processed basalt can be reduced by having a hollow texture (Marangoni et al., 2014; Sun et al., 2020). The existence of basalt rock itself on earth is very abundant. It can be said that 90% of the surface rocks on Earth were originally basalt, where along with the cooling of the lava and other volcanic processes, the rocks turned into other types of rock. Its presence in Indonesia itself is massive, mostly in the islands of Sumatra, Java, and Kalimantan, and its use, especially in Indonesia itself, is still very simple, limited to construction and building materials (Hendronursito et al., 2019; Isnugroho et al., 2018).

In the manufacture of lightweight materials based on basalt or other silica-based materials, the presence of gas in the process of forming a porous structure in the process of making lightweight materials plays an important role (König et al., 2016; Pereira da Costa et al., 2020; Rincón et al., 2017). There are many kinds of elements or materials that trigger the formation of gas, commonly called foaming agents, which are often the object of research by researchers. Some of the foaming agent elements used in the formation of the porous structure of silica-based materials include $CaCO_3$, fly ash, Mn_xO_y, Carbon, Nickel slag, soda-lime for the formation of a sol-gel reaction (Guo et al., 2016; Lee, 2013; Wu et al., 2006). Through foaming technology in the formation of cellular structures on silica-based materials, the density of the material has decreased from its original value. With a flat porous body structure that forms a certain structural pattern, the pressure force experienced by the material can still be evenly dispersed throughout the cellular structure (Benedetti et al., 2021; Nečemer et al., 2019).

In this study, the foaming process in gas formation that occurs during the mineral softening process, namely basalt scoria, is carried out by raw limestone, which is unlike the previous research conducted by Marangoni and Sun when utilizes basalt, the gas formed comes from burning glass or carbon (Marangoni et al., 2014; Sun et al., 2020). Limestone was chosen as a foaming agent because Sumatra, especially Lampung province, has abundant reserves to be utilized (Isnugroho et al., 2019, 2020). During the sintering process, lime ($CaCO_3$) will react with oxygen to release CO_2 gas to form quicklime (CaO) (Jiang et al., 2019; Megawati et al., 2019). The gases formed at the softening temperature of the basalt scoria, which is above 1000°C, will be trapped in the basalt scoria body structure to form a porous cellular structure after the cooling process of the material. By varying the percentage of lime used and the softening temperature of the basalt mineral, it is hoped that the optimal value for the formation of the best porous cellular structure will be obtained (Hesky et al., 2015; König et al., 2017; Petersen et al., 2017). With the creation of a lightweight material made from basalt, the potential for increasing the added value which is previously have very low and generally just for basic construction material can be increased. Therefore the aims of this study is to utillize basalt rock as source of glass ceramic and mixed with limestone to form cellular structure with optimal composition.

2. METHOD

In this study, the basic materials used were scoria-type basalt from East Lampung and limestone minerals from West Lampung. First step preparation sample was cleaning material then all materials were milled separately in a Ball Mill Type TR6-Z-A-D112.M4. When its finish, each material then sieved through a 100 mesh sieve to achieved homogen particle dimensions. For the initial characterization of basic materials like basalt raw and limestone, XRF analysis was carried out to determine the initial chemical composition of minerals using XRF PANalytical type: Minipal 4 which operates at a voltage of 30 kV. After that, all the basic ingredients were mixed with four variations of the composition which is for sample A consist of 100%wt basalt, sample B consist of 30%wt basalt and 70%wt limestone, sample C was 50%wt basalt and limestone each and sample D was 70%wt basalt and 30%wt limestone.

Each sample is burned in a furnace per 200g in a cup made of graphite. The combustion process is carried out in the Iwata furnace with a heating rate of 10°C/minute and held for 90 minutes in the designated temperature which is 1100°C or 1300°C. All sample cooling process is carried out slowly inside the furnace until its reach room temperature and all sample then characterized using XRF PANalytical type: Minipal 4 which operates at a voltage of 30 kV for chemical composition, XRD PANalytical type: E'xpertPro which operates at a voltage of 40 kV phase determitation, SEM type: Quatro Thermo scientific for morphology appearence, then for density and porosity calculation was based in archimedes method, using reference calculations SNI 03-6433-2000 and ASTM D-C 642-97.

3. RESULT AND DISCUSSION

Result

At the material preparation stage, XRF characterization of basalt and limestone was carried out to determine the initial content of the two materials. In previous research, basalt from East Lampung has a dominant composition of SiO_2 and Al_2O_3 because these compounds are very important components in the reaction for the formation of scoria basalt rocks (Amin & Suharto, 2017). XRF results for basalt and limestone is show in Table 1.

Table 1. XRF Results for Basalt and Limestone

Chemical composition	Basalt (%wt)	Limestone (%wt)
MgO	4.561	0.199
Al2O3	18.820	0.246
SiO2	48.418	0.293
CaO	9.761	98.871
Fe2O3	12.595	0.307
Na2O	3.356	-
K2O	0.636	-
TiO2	1.329	-
MnO	0.194	-

Based on Table 1, the dominant elements in basalt such as SiO_2 and Al_2O_3 have a chemical composition of 48.418% and 18.820%, respectively, and the element Fe_2O_3 is 12.595%. For limestone itself, the dominant element, namely CaO, has a chemical composition of 98.871%. The results of XRF characterization is show in Table 2.

Table 2. The Results of XRF Characterization of a Mixture of Basalt and Limestone Samples after the Combustion Process

Chemical Composition	A	B	C	D
	(%wt)	(%wt)	(%wt)	(%wt)
MgO	4.561	1.26	1.34	1.42
Al_2O_3	18.82	3.12	4.16	6.64
SiO_2	48.418	14.61	16.08	19.43
K_2O	-	0.35	0.31	0.31
CaO	9.761	73.21	71.16	63.03
TiO_2	1.329	1.04	1.09	1.18
MnO	0.194	0.42	0.44	0.49
Fe_2O_3	-	3.48	8.14	12.96

Seen in Table 2, the dominant elements of the mixed sample are silica and calcium. For sample A with 100% basalt, the chemical composition did not change much because there was no oxidation-reduction process, and for samples B to D, along with the addition of basalt rock increased, the silica composition increases and calcium decreases. Diffractogram of sample XRD test results at a temperature of 1100°C is show in Figure 1.

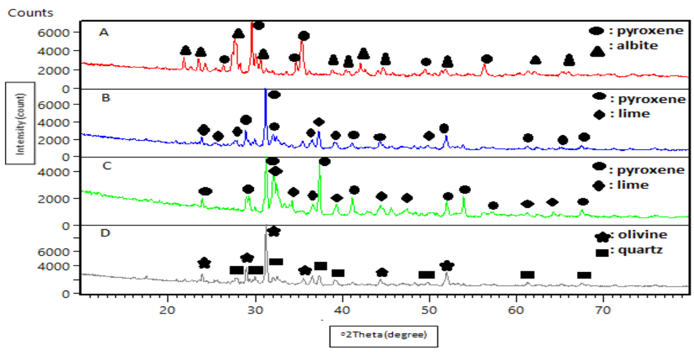

Figure 1. Diffractogram of Sample XRD Test Results at a Temperature of 1100°C

Based on Figure 1, with heating at a temperature of 1000°C, it can be seen that pyroxene (XY(Si,Al)$_2$O$_6$ growth was detected in samples A, B, and C accompanied by the growth of albite ($NaAlSi_3O_8$) and lime ($Ca(OH)_2$ depending on chemical composition dominant in the sample. Where at sample D, olivine (Fe_2SiO_4) and quartz (SiO_2) was detected different with the result from another sample. Diffractogram of sample XRD test results at a temperature of 1300°C is show in Figure 2.

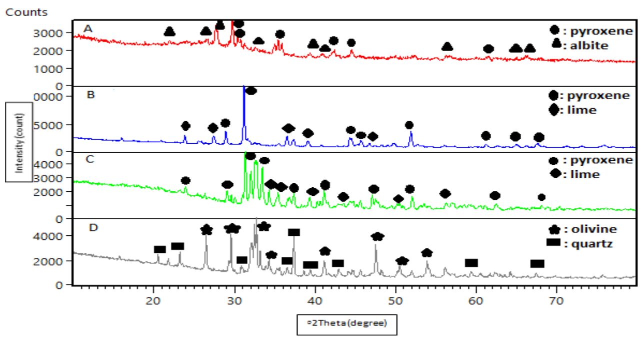

Figure 2. Diffractogram of Sample XRD Test Results at a Temperature of 1300°C

As seen in Figure 2, when burning at a higher temperature of 1300°C, the phase formed is not different from when it is burned at a temperature of 1100°C. It's just that when burned at a higher temperature, the amount of crystallization grows more. This is because the homogenization process takes longer at 1300°C which causes more dissolved chemical elements during the melting process it affects crystal growth to increase during the cooling process. SEM morphology appearance with Secondary Electron is show in Figure 3.

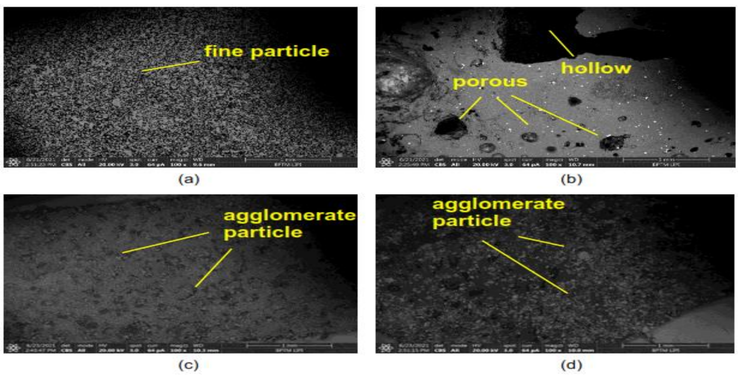

Figure 3. SEM Morphology Appearance with Secondary Electron Mode on Samples Burned at 1100°C, (a). Sample A, (b). Sample B, (c). Sample C, (d). Sample D

Based on the results of the SEM analysis in Figure 3 with a magnification of 100x, the sample burned at a temperature of 1100°C tends to undergo a sintering process, so that the glass-ceramic structure is not formed due to the melting process. The average powder particles in each sample variation only experienced agglomeration because the sintering conditions that occurred at a temperature of 1100°C and the gas which was expected to be trapped to form a cellular structure could not occur. Based on Figure 3(b), it is different for sample B, the melting process occurs in the powder particles formation. SEM morphology appearance with Secondary Electron mode on samples burned at 1300°C is show in Figure 4.

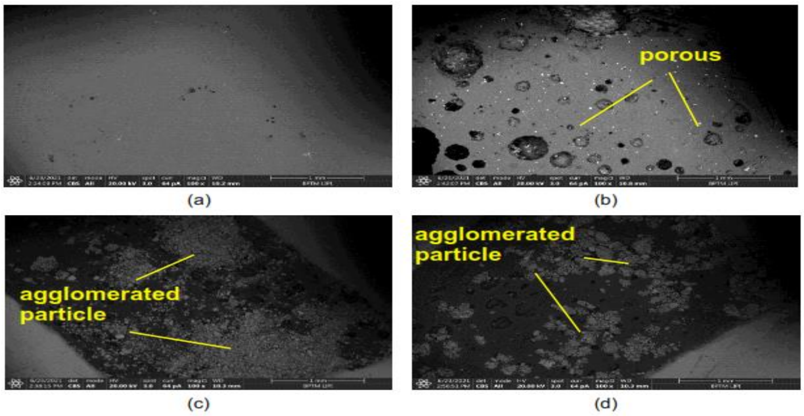

Figure 4. SEM Morphology Appearance with Secondary Electron Mode on Samples Burned at 1300°C, (a). Sample A, (b). Sample B, (c). Sample C, (d). Sample D.

Based on Figure 4, compared the results of combustion at a temperature of 1100°C for sample A with 100% basalt able to fuse to form a ceramic glass body structure. However, due to the lack of a stimulating element for a gas generation when the silica melting process occurs, the final result after cooling the ceramic glass body does not form a porous cellular structure (Figure 4(a)). As for sample B, with a higher temperature condition of 1300°C, the resulting pores formed from CO_2 bubbles trapped more evenly on the glass-ceramic structure formed. For samples C and D, the condition of incomplete fusion is still visible with the particles that collect not fused well. The equilibrium point of the Ca-Al-Si fusion diagram has shifted above 1300°C, so that the cellular glass-ceramic structure is not formed.

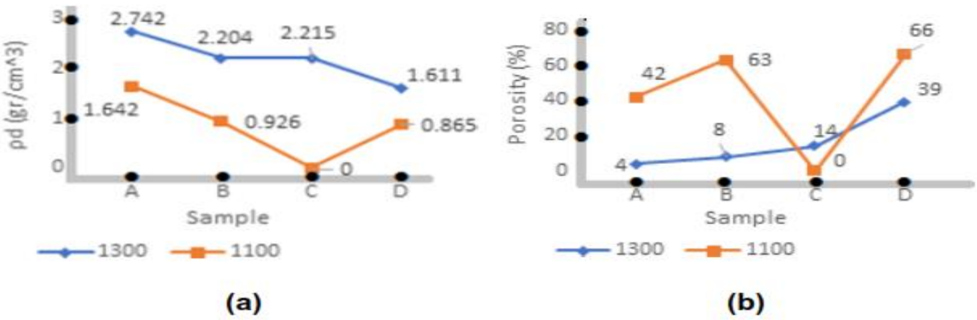

Figure 5. Comparison of the Results of Sample Analysis at Temperatures of 1100°C and 1300°C, (a). Density test, (b). Porosity Test

Based on Figure 5, the samples that formed the glass-ceramic structure at temperatures of 1100°C and 1300°C, namely sample B, had density and porosity values of 2,204 g/cm^3 and 8% at 1300°C and 0,92 g/cm^3 and 63% respectively.

Discussion

Basalt rock as raw material for glass ceramic was chosen because of its ability to form ceramic formations that have a phase structure formed in it. The advantage of phase formation is the improvement of the mechanical properties of the material. Moreover, basalt rock as a glass ceramic material has the ability to modify its mechanical characteristics by applying a cooling treatment and holding time during the combustion process (Deng et al., 2022; Fiore et al., 2015). The process of making lightweight glass ceramics is to apply the foam method into the glass ceramic body to form cellular formations like a honeycomb in the end result (Fernandes et al., 2009b; Zhang et al., 2022). The use of raw limestone as an initiator of gas formation is applied to a mixture of basalt samples. The priority to select raw lime so that the gas produced is large enough when it passes through the lime sintering point at a temperature of 900°C. The melting temperature of Lampung scoria basalt is between 1200°C to 1300°C, so the temperature point is ideal for the formation of cellular structures because the trapped gas is below the melting temperature. When burning at 1100°C, only sample B formed a glass ceramic structure where the other samples only underwent sintering and only happened a gathered granular particles.

Normally, apart from being a gas-producing initiator, limestone has a role as an additive component to lower the melting temperature of basalt. So that the stage of softening of basalt rock can be obtained far below the normal melting temperature of basalt around 1200°C to 1300°C. With a CaO composition of up to 73%, the softening phase of the basalt can be achieved so that the glass ceramic structure is formed at a lower temperature (Abd Rashid et al., 2014; Rasmussen et al., 2003). Hypothetically, with the high composition of limestone, the possibility of gas formation in the softening phase of the basalt should be more likely to form, but because the difference between the normal melting temperature of limestone and the combustion temperature of 1100°C is quite large, the phenomenon of loss of CO_2 gas occurs during the combustion process to the target temperature. This phenomenon also occurs during combustion at a temperature of 1300°C, because CO_2 gas has been formed before the temperature reaches 1300°C, the gases that occur tend to disappear to the surface and only leave traces of trapped gas that are irregular to resemble cavity defects.

Based on the XRD results, both at 1100°C and 1300°C have the same trend of results. For sample A, pyroxene and albite phases were detected which are usually found in scoria basalt rock samples. Because sample A which consists of 100% basalt only undergoes a combustion process without any mixture of elements, the tendency of the phase formed due to the characteristics of the basalt rock itself, the phase that occurs will mostly return to its origin (Candra et al., 2020; Manfrinetti et al., 2000). For samples B and C, a pyroxene phase was detected followed by lime as a result of precipitation from the addition of a fairly large limestone composition, while for sample D, due to the high iron concentration and due to the influence of the addition of limestone, silica precipitated to form quartz and partly associated form olivine.

Based on the results of the porosity and density tests carried out, referring to the results of other analyzes where only a glass ceramic structure can be formed, the optimal result of sample B is at a temperature of 1100°C with a density value of 0.92 g/cm^3 and a porosity of 63%. Based on Fernandez research, with utilizing carbonates to produce CO_2 gas to form cellular structure which is the concept mostly same with the occurance of CO_2 in lime sintering, the density which can be obtain is 0.36 g/cm3 at 850°C. It more than half from the result obtained in this reseach, where fernandez use cullet glass as source of his glass ceramic structure (Fernandes et al., 2009a; Markov, 2017). If we only look at the magnitude of the values obtained, the lowest density is owned by sample C with a temperature of 1100°C at 0 g/cm^3 and the highest porosity in sample D with a temperature of 1100°C at 66%. However, if we take into account the results of the SEM analysis of the sample, the surface morphology does not fuse to form a glass-ceramic structure where only agglomeration occurs between the particles so that the gases formed cannot be maintained during the cooling process.

Several studies on cellular foam glass generally utilize industrial solid waste where the process of forming glass is carried out at a low softening temperature where the average is 1000°C. In Marangoni's research, in an effort to utilize industrial waste, namely used glass, basalt was chosen as a component of the ceramic structure and lime-based glass waste was positioned as a foaming agent. With a composition of 50%-50% between cullet and basalt, it gives optimal results in morphology, cellular structure and mechanical strength (Dhir et al., 2018; Marangoni et al., 2014). However, from a review study conducted by Dhir, the self-forming that occurred in the sample from the Marangoni study was less stable so that the formation of the cellular structure was sometimes less than optimal.

The use of raw limestone as an additive component of foaming agent can increase the chances of gas formation which is the raw material for components forming cellular structures. However, due to the large enough distance between the softening temperature and the CO_2 gas temperature that is formed, it gives too much time for the gas to escape to the surface, so in the future it is necessary to add other additive components that are able to lower the melting basalt temperature again so the gas which is formed can be maintained. But overall by adding raw lime in basalt mixture up to 70%, cellular structure can be formed in body glass ceramic although heated at the sintering temperature far below the melting basalt temperature, which is about 1300°C.

4. CONCLUSION

Raw lime can be used as a foaming agent in the manufacture of lightweight materials made from basalt rock through the foaming method in basalt softening phase. The optimal composition to form foam formation which has low specific gravity and large porosity occurs in the addition of 70% lime. Based on the characterization results, the best glass-ceramic sample formed with pores structure formation. Basalt cellular ceramic is obtained in optimal condition with low density and higher percentage porosity.

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