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THE CHARACTERIZATION OF FOAM GLASS-CERAMICS BASED ON INDUSTRIAL SOLID WASTE

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ABSTRACT

Industrial processes invariably generate waste, such as fly ash from coal combustion, basalt stone dust from crushing, aluminum dross from casting, and glass from bottle manufacturing. These residues consist of non-biodegradable solids with limited value. Hence, repurposing them into value-added products becomes imperative. This study harnessed these waste types – comprising 36% fly ash, 4.5% basalt, 49.5% glass bottle waste, and 10% aluminum dross – for crafting foam glass ceramics. Preliminary analysis deployed XRF Epsilon 4 X-RF Spectrometer to ascertain chemical composition and XRD Panalytical Xpert-3 Powder XRD for crystalline phase identification. Notably, SiO_2, CaO, Fe_2O_3, and Al_2O_3 emerged as primary compounds. SiO_2 dominated glass waste (64.697%), while aluminum dross held the least (19.940%). Glass waste had the highest CaO (22.823%); coal fly ash showed the lowest (11.609%). Basalt rock marked the peak Fe_2O_3 (18.047%), with glass waste registering the least (3.454%). Aluminum dross exhibited the highest Al_2O3 (45.625%), and glass waste was the lowest (3.736%). Material proportions stood at 36% fly ash, 4.5% basalt, 49.5% glass waste, and 10% aluminum dross. The resultant glass-ceramic foam hosted crystalline phases – wollastonite, anorthite, and diopside – with a sealed pore configuration

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INTRODUCTION

Foam glass-ceramics are a porous material that has light properties with high strength, can absorb heat, dampen sound, and has a porosity of up to 90%. Glass-ceramics foam has unique properties: lightweight, rigid, resistant to pressure, non-flammable, frost resistant, moisture resistant, non-toxic, rodents, insects, bacteria, and water resistant. Therefore, foam glass ceramics are widely used in various industrial applications. They are widely used as air filters, water treatment, and high-temperature heat retaining [1-2].

Research related to manufacturing glass-ceramics foam with various raw materials (blast furnace slag, glass, glass waste, metallurgical slag, fly ash, coal bottom ash, red mud from bauxite processing waste, diatomaceous earth) has been widely carried out and is still developing [3-9]. In manufacturing glass-ceramics foam, a foaming agent is added, an added material for adding gas or substances capable of producing gas during heating [5]. The gas will sink into the cells of the melted glass so that when the melted glass cools, there will be pores in the glass. Foaming agents such as carbon (graphite, carbon black), silicon carbide (SiC), carbonates ($CaCO_3$, $SrCO_3$), dolomite, calcite, marble, trash, water glass, MnO_2, sulfate ($CaSO_4$), sodium hydroxide (NaOH), bone pigs, and even egg shells can be used in the process of making glass-ceramic foam [7,10-13].

The choice of raw materials holds significant sway over the foam glass ceramics' quality. Currently, the production of foam glass ceramics mainly relies on one or two types of raw materials. The broader integration of more than two varieties of industrial solid waste as foundational materials is a practice that has yet to gain widespread traction [5]. However, the simultaneous utilization of industrial solid waste such as fly ash, glass bottles, basalt rock, and aluminum dross can open doors to creating foam glass ceramics[12]. To facilitate this innovative approach, it's imperative to conduct preliminary research aimed at characterizing these materials, enabling their combined use in the production of foam glass ceramics[7,11].

The core objective of this study is to harness industrial waste materials as raw inputs to manufacture foam glass ceramics. This involves tapping into diverse sources: waste from coal power plants, manifesting as fly ash; discarded beverage bottles; and byproducts from the processing of split stone, which yields basalt powder. These raw materials are readily available locally and can be harmoniously employed to fabricate foam glass ceramics imbued with attributes like buoyancy and substantial porosity. Such ceramics hold promise for multifaceted technological applications. The post-sintering phase entails an in-depth analysis of the formed crystalline phase and the surface characteristics, indicating the final product's quality and appropriateness for various intended uses.

METHOD

1. Material Collection and Preparation

The research utilized raw materials including was, te from basalt stone crushers in Sukadana, East Lampung, fly ash residue from coal burning at PLTU Sebalang, South Lampung, clear colored glass bottles gathered from collectors of used goods in Bandar Lampung, and aluminum casting waste (aluminum dross) sourced from a foundry enterprise in Tanjung Bintang, South Lampung. In the preliminary processing stages, glass bottle waste, basalt rock, and aluminum dross underwent crushing using a jaw crusher, resulting in materials smaller than 2 cm. Subsequently, the material from the jaw crusher underwent a 24-hour grinding procedure in a ball mill during the grinding process, yielding a product that could traverse through a 200-mesh screen. The fly ash was also sieved to acquire a substantial material capable of passing through a 200-mesh sieve.

2. Powder Preparation and Sieving

The initial phase of the process involves grinding the raw materials using a ball mill until they achieve a smooth consistency. Subsequently, the resulting powder undergoes sieving through an ASTM sieve with a mesh size 325. This sieving process reduces the impact of mineralogical and grain size variations, ensuring precise XRD (X-ray diffraction) and XRF (X-ray fluorescence) readings. In terms of sample preparation, test samples are meticulously extracted, each with a minimum weight of 2 grams, to be subjected to thorough XRF and XRD analyses. This rigorous methodology ensures the accuracy of the subsequent analytical results.

3. XRF and XRD Analysis

The analysis process employed advanced instrumentation, notably the XRF Spectrometer Epsilon 4 XRF from Malvern Panalytical, for comprehensive chemical composition evaluation. Additionally, the Panalytical Xpert 3 Powder XRD apparatus was utilized to discern the crystalline nature of minerals. Specific parameters were adhered to during the evaluation: XRF testing was conducted within the angular range of $10° < 2\theta < 80°$ for the initial raw materials and the ultimate foam glass-ceramic products. These cutting-edge techniques and carefully defined parameters ensured a thorough and accurate understanding of the elemental composition and mineral crystallinity across the investigated materials.

4. Foam Glass-Ceramic Production

The composition of the raw materials utilized in producing foam glass-ceramics consisted of 36% fly ash by weight, 4.5% basalt by weight, 49.5% glass bottle waste by weight, and 10% aluminum trash by weight. These materials were shaped into pellets using a hydraulic press machine to initiate manufacturing. This machine employed a cylindrical mold measuring 20 mm in diameter and 2 mm in thickness, with a 100 kg/cm² compression force. Subsequently, the formed pellets underwent a controlled heating procedure. The heating was carried out within a muffle furnace, with the process temperature at 1,000 °C. The temperature was increased gradually, at a rate of 10 °C per minute, and maintained for 30 minutes. Following this, a deliberate slow cooling process was initiated. This meticulous series of steps ensured the transformation of the initial material composition into foam glass-ceramic products of the desired quality and characteristics

RESULTS AND DISCUSSION

1. Chemical Composition Results

The results of the analysis using XRF are presented in Table 1. It can be seen in Table 1 that the raw materials (basalt rock, fly ash, glass bottle waste, and aluminum dross) contain the main compounds SiO_2, CaO, Fe_2O_3, and Al_2O_3. K_2O, TiO_2, P_2O_5, MgO, and MnO are minor compounds in the four raw materials.

Table 1. The chemical composition of the raw material to produce foam glass-ceramics.

Chemical Comp.	Basalt (wt%)	Fly Ash (wt%)	Glass Bottle (wt%)	Al.Dross (wt%)
SiO_2	40.792	45.392	64.697	19.940
Fe_2O_3	18.047	14.362	3.454	7.302
Al_2O_3	12.204	21.757	3.736	45.625
CaO	22.463	11.609	22.823	11.917
K_2O	1.113	1.123	0.817	3.110
TiO_2	1.703	1.580	0.553	0.404
P_2O_5	-	0.888	0.912	1.109
MgO	2.30	0.753	0.597	2.785
MnO	0.618	0.146	0.106	0.549
ZrO_2	-	0.107	-	-
PbO	-	-	0.238	0.270
Eu_2O_3	0.119	-	-	0.122

a. Basalt Stone

Based on Table 1, The chemical composition of basalt rock is dominated by the main content of SiO_2 at 40.792%, CaO at 22.463%, Fe_2O_3 at 18.047%, and Al_2O_3 at 12.204%. In principle, basalt is a rock containing SiO_2 [14], while the CaO content contained in basalt rocks can act as a foaming agent [15]. The basalt used has a high iron oxide content compared to basalt from other countries [16]. The presence of this Fe significantly affects the crystallization mechanism. When the silicate (Si) bonded to Fe is subjected to high heat treatment in an oxidizing environment, the cations diffuse from the inside of the glass to the surface [17]. The iron oxide in glass ceramics has a marked effect on increasing uniform crystallization at relatively lower temperatures. The magnetite core acts as a nucleating agent when heated to temperatures between 650 and 800ºC. This oxide will also affect the color of the final product [18].

b.Fly Ash

Fly ash from PLTU contains a dominant composition of 45.392% SiO_2, 14.362% Fe_2O_3, and 21.757% Al_2O_3. When viewed from the dominant composition, fly ash is a type of class F with a total of SiO_2, Al_2O_3, and Fe_2O_3 of 81.5% [19]. In contrast to type C, type F is widely used to synthesize MgO-Al_2O_3-SiO_2 fly ash coal in glass-ceramics systems. This glass-ceramics system is commonly employed, especially in high-temperature fields requiring superior thermal shock resistance [20].

***c.* Glass Bottle**

The chemical composition of glass bottle waste is dominated by the main content of SiO_2 at 64.697%, CaO at 22.823%, Fe_2O_3 at 3.454%, and Al_2O_3 at 3.736%. SiO_2 compounds dominate the chemical composition of glass bottle waste, indicating that the glass bottle waste used is clear glass based on silica and no other types of glass, such as phosphate glass based on phosphor oxide. Silica, in ample quantities, is the most crucial constituent of glass; 95% of industrial glass production is made of silicate materials [21].

The CaO measurement outcomes exhibit an exceptionally noteworthy value of 22.823%. This quantity stands as the most substantial oxide content following SiO_2. This considerable CaO ionic compound concentration contributes to Ca^{2+} cations and oxygen anions. The Ca^{2+} cations play a pivotal role within the silica structure by mitigating the excess negative charge present on the terminal oxygen atoms of the silicate framework. Similar to the role performed by Na^+ for Na_2O. Concurrently, the oxygen anions exert influence over the configuration of the silica covalent framework. Covalent bonds shape network structures in both crystalline silica (SiO2) and amorphous silica within glass. The heightened CaO content simultaneously augments the ratio of oxygen to silicon, disrupting the three-dimensional network and generating singly bonded oxygen that doesn't partake in the network, ultimately reducing chain length. It can be deduced that CaO ions can substitute Na_2O in transparent glass. Past researchers have similarly detailed the primary impact of this modifying agent in diminishing the melting temperature [22].

d. Aluminium Dross

The chemical composition of aluminum dross is dominated by the main content of Al_2O_3 at 45.625%, SiO_2 at 19.940%, CaO at 11.917%, and Fe_2O_3 at 7.302%. The four raw materials are generally dominated by SiO_2, CaO, MgO, and Al_2O_3 compounds, essential glass ceramics components [23-26]. According to modern theory, SiO_2 can be used as a glass-ceramic network former, MgO and CaO as glass ceramic network modifiers, Al_2O_3 as a network intermediary, and TiO_2 in small amounts is a good crystal nucleating agent so that this component can be used in the glass-ceramics production process [27].

SiO_2 has the property of increasing strength, where the high fracture strength is directly proportional to the value of thermal shock. High-purity silica is an excellent material for forming ceramics with meager expansion. The dominance of the SiO_2 content in basalt, fly ash, glass bottles, and alumina in aluminum dross can increase the mechanical properties of the resulting composite material [28].

Magnesium Oxide is one of the primary materials in the manufacture of ceramics because it has a very high melting point of around 2,000ºC [29]. Magnesium oxide has a relatively good thermal conductivity, and magnesium oxide is excellent as an electrical barrier. It is widely used as an insulating material with characteristics that tend not to change when heated. Magnesium oxide is also a safe material for the human body [30-31]. Adding Al_2O_3 to ceramics can reduce the mass of the ceramics, but the hardness value obtained increases. The hardness is very much needed in manufacturing light, solid, and stable ceramics when heated to 1,000°C [32]. Al_2O_3 is a material that is often used in various applications because of its high physical and chemical properties, such as its very high strength and hardness [33-34].

2. Crystal Phase Results

The results of the characterization of the XRD structure of the raw material for making glass ceramic foam are shown in Figure 1-4.

a. Basalt Stone

Based on Figure 1, the detected phase in the basalt rock is the anorthite phase ($CaSi_2Al_2O_8$). Anorthite is a plagioclase mineral with the highest Ca composition and the lowest silica compared to other plagioclase minerals. Anorthite often occurs in alkaline affinity igneous and volcanic rocks, basalt and gabbro, with pyroxene and olivine [35]. The addition of anorthite becomes essential as a technical material in ceramics. In the presence of anorthite, the ceramic material has good physical properties such as a low coefficient of thermal expansion, an increased thermal shock value, high creep resistance at high temperatures, and a low dielectric constant [36]. Other phases formed in samples of basalt rock raw materials are diopside ($MgCaSi_2O_6$), olivine ($FeMgSiO_4$), and pyroxene ($MgSiO_3$). Diopside is widely used as a raw material for glass ceramics due to its low melting point and melting viscosity. Diopside-rich ore added to the ceramic body can improve its technical features, reducing water absorption while increasing flexural resistance for low-temperature ceramic production [37]. Diopside can be found in ultramafic igneous rocks (kimberlite and peridotite), olivine and andesite basalts, skarns (silica-rich dolomite), and mantle minerals, such as peridotite xenolith, kimberlite, and alkaline basalt [35].

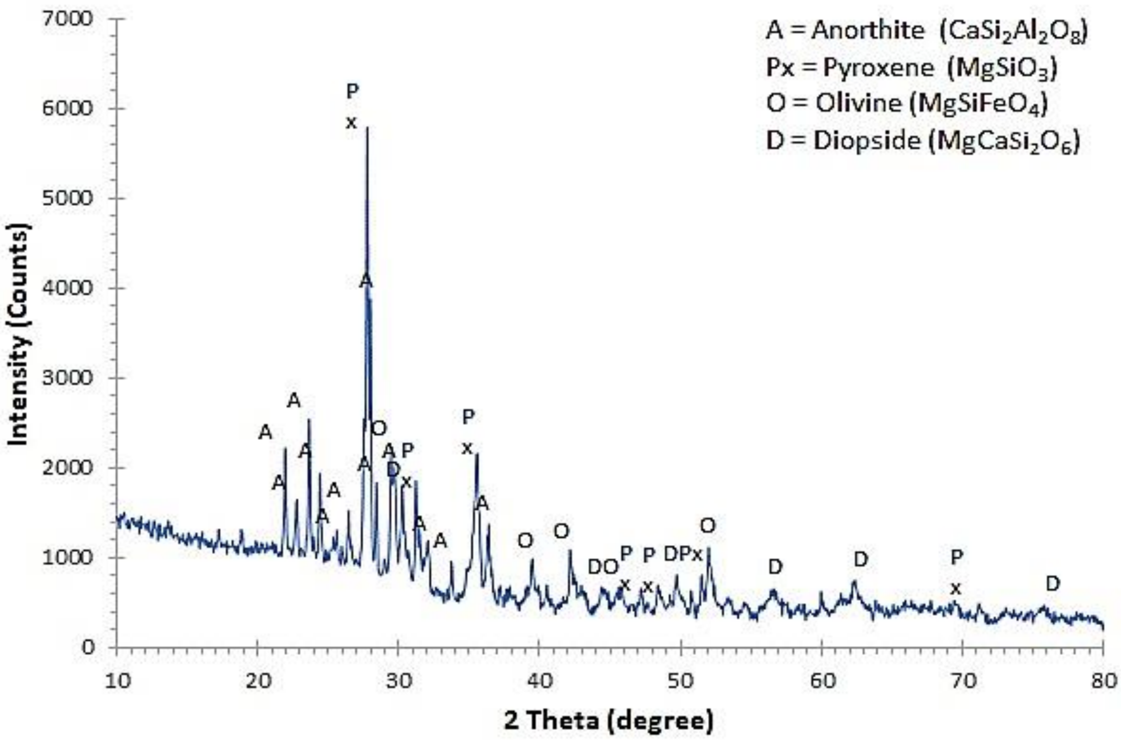

Figure 1. Crystal phase of raw basalt.

Olivine is a group of silicate minerals; this mineral is formed at the highest temperature and pressure in the crystallization process of magma. Olivine generally consists of three main types found in plutonic, volcanic, and secondary rock groups [35]. The pyroxene mineral group is a single series of inosilicate minerals with a composition of Si_2O_6. The cation that binds it consists of magnesium (Mg), iron (Fe), and calcium (Ca), which will later determine the elemental composition of the bond. This mineral's growth consists of two major groups: orthopyroxene (poor Ca) and clinopyroxene (affluent Ca). Minerals are insufficient in Ca, meaning they are rich in Fe and Mg, while minerals rich in Ca can contain Fe or Mg [35].

b.Fly Ash

In Figure 2, the mineral phases detected in coal fly ash are anorthite ($CaSi_2Al_2O_8$), quartz (SiO_2), mullite ($Al_{4.5}Si_{1.5}O_{9.74}$), and magnetite (Fe_3O_4). Quartz (SiO_2) is known to prevent the shrinkage of heated materials. However, its presence can harm the mechanical behavior of ceramics [38]. Mullite is an alumina-silica compound used for various refractory applications [39-40]. Mullite has several superior properties, such as high heat strength, good creep resistance, low dielectric constant and thermal conductivity, good thermal shock resistance, and resistance to radiation damage [41].

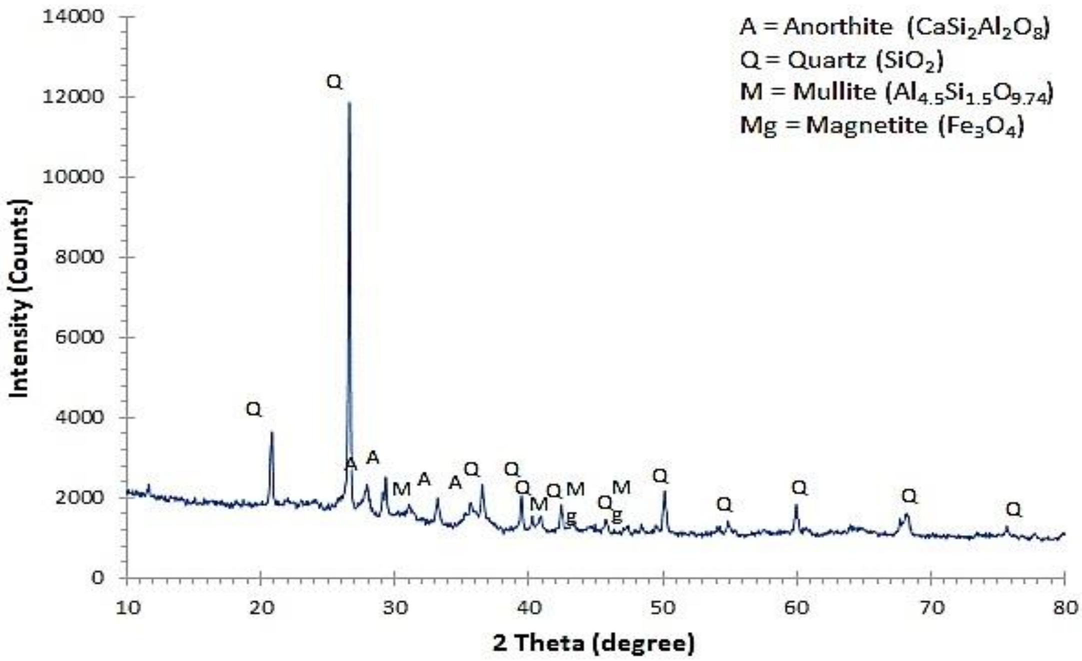

Figure 2. Crystal phase of fly ash

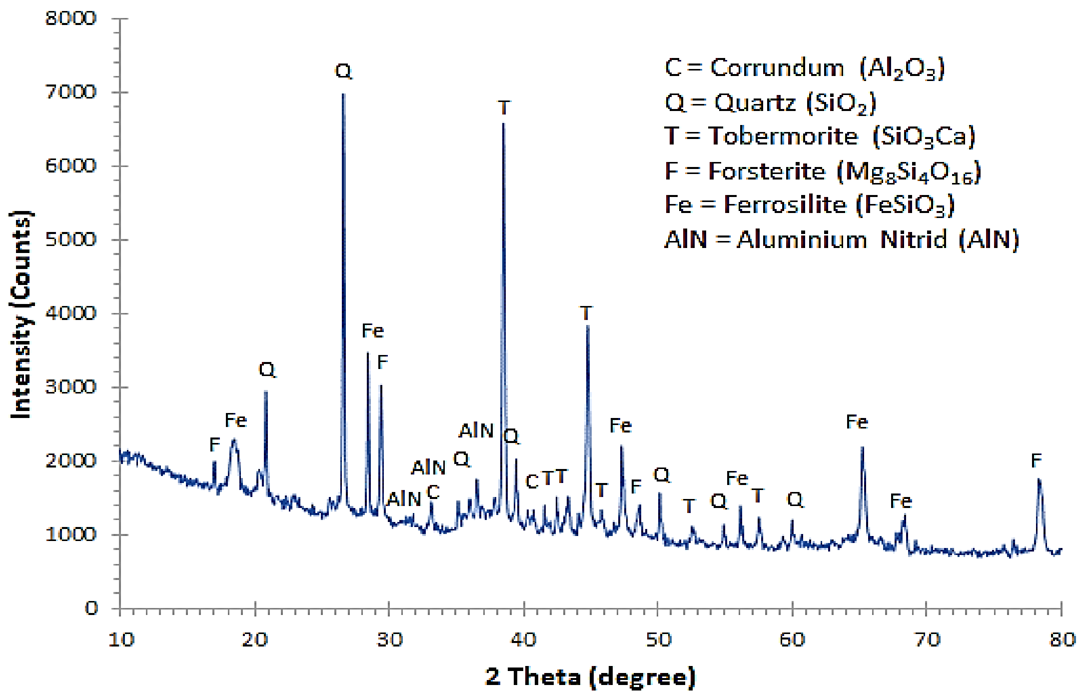

Figure 3. Crystal Phase of Aluminum Dross

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c. Aluminium Dross

In Figure 3, the mineral phases detected in aluminum dross are corundum (Al_2O_3), quartz (SiO_2), tobermorite (SiO_2Ca), forsterite ($Mg_8Si_4O_{16}$), ferrosilite ($FeSiO_3$), and aluminum nitride (AlN). In aluminum dross, a corundum phase is instrumental in ceramic materials.

Ceramics containing corundum have excellent properties, such as high mechanical strength, melting point, chemical stability, and thermal shock resistance [42-44]. This material can be widely used in abrasives, refractories, porcelain, electrical, and optical applications because of its excellent performance [45-47]. Forsterite (Mg_2SiO_4) has many applications in the electronics, communications, and refractory industries due to its low electrical conductivity and melting temperature [48-49]. Recently, forsterite has been used as a bioceramic material with good mechanical properties [50-52]. Forsterite exhibits better mechanical properties and biocompatibility than calcium phosphate ceramics such as hydroxyapatite [50]. The elements magnesium and silicon in forsterite are essential elements in the human body that have the potential to develop bone implant materials [53].

d. Glass Bottle

No peaks indicate a particular phase in the X-ray diffraction data in Figure 4. Thus, the characterization results using XRD of the glass waste raw material show an amorphous structure.

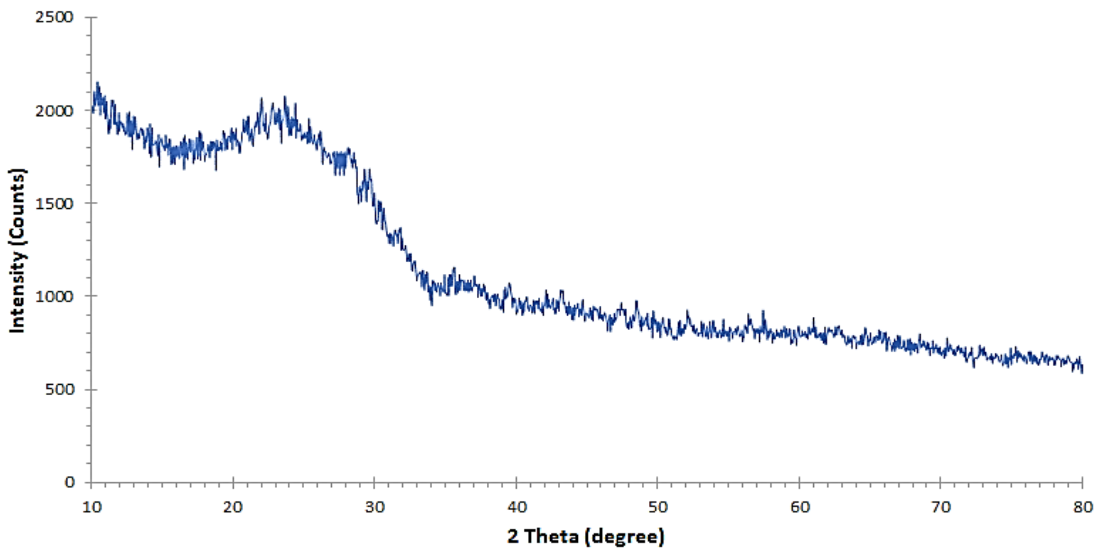

Figure 4. The amorphous phase of a glass bottle.

e. Foam Glass-Ceramics

Figure 5 shows a glass-ceramics foam product with wollastonite, anorthite, and diopside phases. The results of the crystal phase are similar to previous studies, which stated that wollastonite, anorthite, and diopside phases could be formed during the manufacture of foam glass ceramic [54-56].

Wollastonite ($CaSiO_3$) is a calcium silicate ceramic obtained as a result of the reaction between calcium carbonate ($CaCO_3$) and silica (SiO_2). The wollastonite phase occurs because CaO molecules bind to SiO_2, where both molecules are present in the raw materials used. The energy provided during the heating reaction results in a large amount of CO_2 being released and more CaO-SiO_2 reactions being produced. Heat treatment at 1,000 °C will have a higher $CaSiO_3$ peak, thanks to more energy for the interaction between CaO and SiO_2 [57]. Wollastonite forms upon heating to temperatures between 850 and 900 °C [58].

Wollastonite formation reaction occurs as follows:

$$CaCO_{3(s)} + SiO_{2(s)} \longrightarrow CaSiO_3 + CO_{2(g)}$$

Previous research studied the characteristics of Wollastonite glass ceramics from FA and glass cullet raw materials. Wollastonite is dominant at 1,050 ºC and becomes easier to form because it is influenced by augite crystal variations, such as diopside [59]. Based on previous studies, the selection of the sintering temperature in this study was appropriate. Wollastonite at a process temperature of 1,000 °C will provide glass-ceramics with good mechanical properties [59]. Wollastonite glass ceramics are widely used as bioceramics in dental implant applications [60] and prosthetic applications [61].

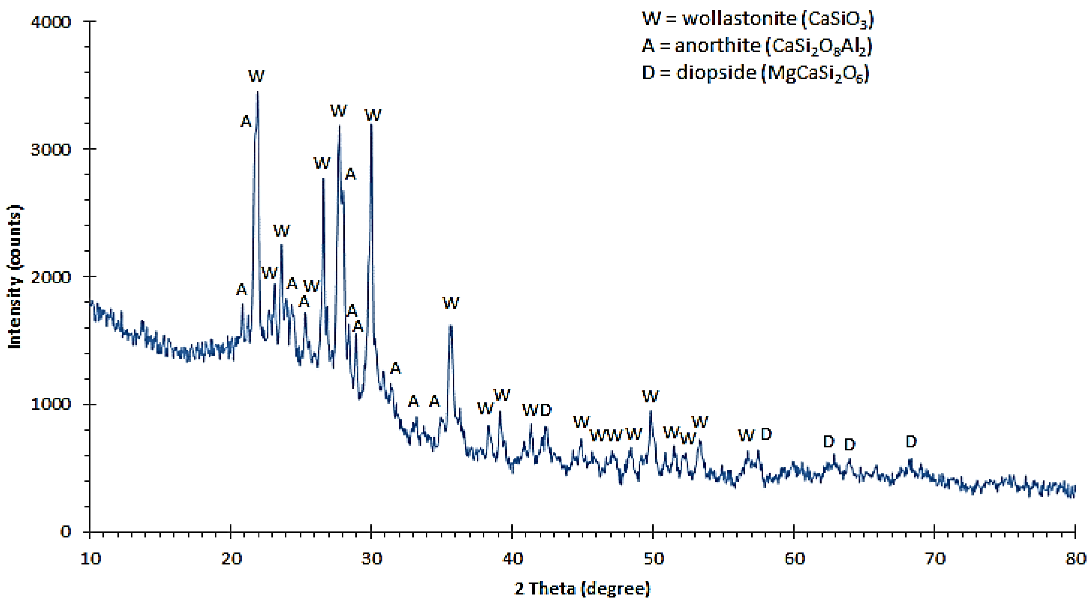

Figure 5. The crystal phase of foam glass-ceramics.

Anorthite ($CaSi_2O_8Al_2$) and diopside ($MgCaSi_2O_6$) are formed because the raw materials with a high content of SiO_2 and CaO in basalt and glass bottles bind to Al_2O_3 from aluminum dross to produce the anorthite phase. Likewise, MgO binds to CaO and SiO_2 to form the diopside phase. However, the diopside phase is in a small part at position $57^o<2\theta<69^o$. This phenomenon is understandable due to the low MgO content of the four raw materials.

The anorthite formation reaction occurs as follows:

$$CaO_{(s)} + Al_2O_{3(s)} + 2SiO_{2(s)} \longrightarrow CaAl_2SiO_{8(s)}$$

The crystallization of anorthite occurs at a temperature of 1,000 °C, at which point solid anorthite ceramics are obtained, consisting of well-crystallized anorthite with only a tiny amount of the glassy phase. Anorthite ($CaSi_2O_8Al_2$) ceramics fabricated at low temperatures can be dielectric materials [62]. Ceramic-based anorthite made from fly ash discovered that the crystallization of the anorthite phase could significantly improve the mechanical properties of the ceramics [63].

Meanwhile, the diopside phase ($CaMgSi_2O_6$) can be formed on heating to a temperature of 850–920 °C [64].

The formation of the diopside phase occurs between $CaCO_3$ and MgO with SiO_2. The reaction for the formation of diopside is as follows:

$CaCO_{3(s)} + MgO_{(s)} + 2SiO_{2(s)} \rightarrow CaMgSi_2O_{6(s)}$

Excellent mechanical qualities make diopside and anorthite useful as building decorative materials [63].

3. The Results Of Morphological Analysis

FE-SEM analysis shows that the materials used can form hollow materials with pores in the glass ceramic material, as shown in Figure 6. The pore structure formed is a closed pore. Foam glass ceramic with coal gangue content has a high proportion of closed porosity and a distribution of smaller pores between adjacent cells. It is very homogeneous and uniform [65]. The low viscosity is one of the reasons the pores that are formed are closed pores. Closed pore foam glass-ceramic is preferred for structural components, has excellent insulating properties, is easy to form, and has a cohesive structure that makes it useful for high-temperature insulation applications.

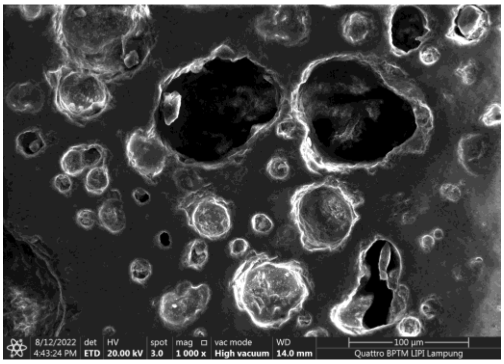

Figure 6. The surface topography of foam glass-ceramics.

CONCLUSION

The results of the analysis of raw materials originating from industrial solid waste in the form of fly ash, basalt, glass bottles, and aluminum dross obtained the dominant chemical composition in the form of SiO_2, CaO, Fe_2O_3, and Al_2O_3 compounds, where these chemical compounds have an essential role in the manufacture of foam glass ceramic. The four industrial solid wastes can be used as raw materials to manufacture foam glass ceramics. The dominant chemical element, iron oxide from basalt rocks, will affect the crystallization temperature and allow the formation of glass ceramics without a nucleating agent.

The dominance of these oxides' four chemical constituents forms glass ceramic foam with wollastonite, anorthite, and diopside crystalline phases after heat treatment at 1,000ºC. These three crystals will provide superior mechanical strength and enable their use as raw materials for bioceramics and other composite materials. The formation of a pore structure in glass ceramics makes this material light but maintains its mechanical strength. Further research is needed to determine the effect of composition and sintering temperature variations on the mechanical properties of foam glass ceramics. Foam glass ceramics, which are light in weight but retain mechanical strength, will be the most widely used material.

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