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Microstructural properties of glass composite material made from incinerated scheduled waste slag and soda lime silicate (SLS) waste glass

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ABSTRACT

Glass composite material (GCM) was produced from incinerated scheduled waste bottom slag (BS) and soda lime silicate (SLS) waste glass. The effect of BS waste loading on the GCM and the microstructural properties was studied. Batches of powder mixture is formulated with 30 wt.% to 70 wt.% of BS powder and SLS waste glass powder for GCM sintering. The powder mixtures of BS and SLS waste glass were compacted by uniaxial pressing method and sintered at 800 °C with heating rate of 2 °C/min and one hour soaking time. The phases identified by X-ray diffraction (XRD) method in all sintered samples are anorthite sodian, quartz, hematite and diopside. It was observed that higher BS waste loading results in higher porosity, higher water absorption and lower bulk density according to ASTM C373. In contrast, the Vickers microhardness value determined according to ASTM C1327, decreases with higher BS waste loading. This similar trend is observed for modulus of rupture (MOR) analysis which was performed according to ISO 10545-4. This physical and mechanical properties can be related to the microstructure observed during scanning electron microscope (SEM) analysis. More open pores and less dense surface are observed for higher BS waste loading samples. On the other hand, samples with lower BS waste loading consists of higher dense surface and no open pores. GCM with batch formulation of 30 wt.% BS and 70 wt.% SLS waste glass has shown the lowest water absorption percentage of 1.17%, the lowest porosity percentage of 2.2% and the highest bulk density value of 1.88 g/cm^3. It also shows the highest MOR of 70.57 MPa and 5.6 GPa for Vickers microhardness with congruent microstructure features.

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1. Introduction

At present, incineration is one of the treatment methods for scheduled waste in Malaysia (source: Kualiti Alam Sdn.Bhd). Incineration of waste is defined as the burning of a substance in controlled parameters such as temperature range, oxygen input, turbulence, atmospheric pressure and other aspects of combustion environment [1,2]. A waste incinerator typically, consists of a rotary kiln (primary combustion chamber) which can incinerate up to 1100 °C and a secondary combustion chamber which operates more than 1000 °C, meeting the EU incineration requirement (source: Kualiti Alam Sdn.Bhd). About 13,300 tons of bottom slag and 2800 tons of fly ash are produced annually by incineration [3]. According to the classification of scheduled waste by the Department of Environment Malaysia, incinerated scheduled waste BS (bottom slag) falls under the SW501 code whereby the code is defined as any residue from treatment or recovery of scheduled waste [4]. At present, the disposal of BS is done by landfilling.

The practice of landfilling creates issue of contaminated land, should the hazardous substance present in sufficient concentration, causing harm to human, animals and environment [5]. Moreover, landfilling does not provide a sustainable solution whereby more land areas are required due to increasing waste volume, hence decreases the land availability for nation's development and reducing the land capacity [6]. Utilization of incinerated scheduled waste BS into producing a beneficial product or into a GCM would be an effective effort in overcoming the disadvantage of landfilling which is the current scheduled waste treatment method. It reflects advantages in reducing disposal cost, preserving environment, and economical impact [7]. The variety of waste compositions gives advantage in producing glass ceramic by a specific processing route and conditions. Recent studies have involved sintering route to produce GCM [11,13–17]. Sintering occurs by atomic diffusion process stimulated by high temperature resulting particles to form bond and hold the mass together [24]. Sintering involving glass or amorphous material occurs through viscous flow mechanism results on the densification and shrinkage of the final sintered glass ceramic [11].

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In this work, Malaysian incinerated scheduled waste BS is incorporated into glass matrix to produce GCM as there is a lack of sufficient work being carried out on incinerated Malaysian scheduled waste BS [7,8]. The BS can be classified as a silicate waste due to high content of SiO_2 [12]. Silicate waste has been studied in other research works for the production of glass ceramic or glass ceramic tiles, whereby it has projected better mechanical, thermal and microstructural properties compared to standard ceramic products [7]. Among silicate wastes studied for producing glass ceramic or GCM are incinerated metallurgic waste, fly ash from coal production, incinerated fly ash and slag from industrial waste; municipal solid waste and incinerated scheduled waste bottom ash.

Besides incinerated scheduled waste BS, SLS (soda lime silicate) glass is also incorporated in this work. SLS glass is obtained from urban waste (used glass bottle and household glass containers). This glass contributes a portion in the domestic waste category. Through recycling SLS glass, the raw material consumption is reduced yielding economical and environmental benefits [9]. Thus, by incorporating two waste streams (SLS glass and BS), the initiative to promote recycling can be achieved. GCM produced by incorporating industrial waste is a low investment approach and has shown good ability to accommodate complicated articles [10]. It has projected desirable properties such as higher mechanical strength and low porosity for the application of floor and roof in industrial and public building, wall covering panels, road surfacing, light weight aggregates and bricks [10,13].

Ceramic, glass ceramic and GCM performance are evaluated by their mechanical, physical and thermal properties whereby these properties are governed by the microstructural characteristics [23]. Microstructural characteristics of crystalline phases, elemental composition and surface attributes influenced the performance and properties of a ceramic product [7]. Existence of crystalline phases and high amount of crystals in the glass ceramic contributes to greater mechanical and physical properties of glass ceramic [10]. Apart from crystalline phases, the porous microstructural characteristic is utilized in producing humidity controlling porous ceramic to prevent retention of moisture in building walls [14] and production of highly porous glass ceramic from metallurgic slag, fly ash and waste glass to be applied as air diffuser for waste water aeration, tiles and wall bricks [15].

The properties of GCM can be tailored based on selection of suitable glass powder, volume fractions, powder processing and firing schedule gives higher possibility to achieve the objective of this research work [11]. Thus, this paper reports on the effect of BS waste loading and incorporation of SLS waste glass to produce GCM by analyzing the physical, mechanical and microstructural properties of the GCM.

2. Experimental procedure

Incinerated scheduled waste BS, was obtained from Kualiti Alam Sdn.Bhd. The collected BS was ready to be landfilled. The BS is than pulverized and sieved for a particle size of <75 μm. Transparent SLS waste glass (bottles) was obtained from urban waste. The bottles are pulverized and sieved for particle size of <75 μm. X-ray fluorescence (XRF) analysis was conducted on both powders using wavelength dispersive XRF spectrometer (S4 Pioneer); capable of measuring all elements from beryllium, Be to uranium, U with trace level below one part per million and up to 100%. The analytical error measurement during analysis is estimated <5% upon considering the sample preparation handling and equipment accuracy. The chemical composition of the SLS waste glass and bottom slag (BS) is tabulated in Table 1.

Table 1
Chemical composition of soda lime silicate (SLS) waste glass and bottom slag (BS). Analytical error is lower than 5%.

Oxides	SLS waste glass [wt.%]	BS [wt.%]
SiO_2	72.60	52.60
Al_2O_3	2.20	24.50
Fe_2O_3	0.28	10.10
CaO	19.30	3.98
BaO	–	2.99
Na_2O	2.70	–
TiO_2	0.17	2.42
P_2O_5	0.07	2.10
K_2O	0.33	1.11
MgO	1.06	–
SO_3	0.31	–
ZnO	–	0.24
MnO	0.01	–

Loss of ignition (LOI) for SLS glass = 0.96%.

2.1. Glass composite material (GCM) sintering

Batch of powder mixtures was formulated according to weight percentage of the BS and SLS waste glass powder. The sieved BS and SLS waste glass powders were then mixed to obtain a mixture of BS and SLS waste glass as shown in Table 2. The batch mixture is formulated based on the attempt to utilize more BS through higher waste loading percentage. Maximum utilization of BS waste is a significant effort to reduce the amount of BS subjected to land filling. Thus waste loading ranging from 30 to 70 wt.% BS waste is studied in reference of previous research work carried out on GCM from wastes [15,22].

1.5 g mixture of each batch formulation is utilized to form pellet samples and 3 g for square shaped samples. The mixtures were compacted in a 13 mm diameter stainless steel die (for pellet samples) and 18 mm × 18 mm × 4 mm mold (for square sample) using 90 MPa uniaxial pressure. Sintering was carried out using laboratory electric furnace on the compacted green body at 800 °C with heating and cooling rate of 2 °C/min and one hour soaking of time. Sintering at 800 °C is carried out upon consideration that the glass transition temperature is ~T_g was observed around 442–592 °C with softening point around 695–730 °C determined by differential thermal analysis (DTA) [27].

2.2. Physical analysis

Physical characteristics of porosity, water absorption and bulk density of the GCM was determined according to ASTM C373 standards. 5 samples of 13 mm diameter pellet from each batch formulation were subjected to this analysis.

2.3. Mechanical testings

2.3.1. Modulus of rupture

3-Point bending test was carried out on the GCM samples according to ISO 10545-4 to determine the fracture strength. 10 samples of square shape 18 mm × 18 mm × 4 mm were subjected to the test of each batch formulation to obtain the average value.

2.3.2. Vickers microhardness test

Vickers microhardness test was conducted according to ASTM C1327 standard using Vickers microhardness HM-200 Series. Prior to the test, the GCM samples were grinded on 400, 600, 800, 1200

Table 2
Ratios of BS to waste glass powder (wt.%). Analytical error is lower than 5%.

BS powder [%]	Waste glass powder [%]
30	70
40	60
50	50
60	40
70	30

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and 2400 grid SiC paper, to obtain smooth sample surface. The grinded GCM samples were then polished using 0.1 μm alumina paste. 15 indentations on each sample were carried out with a load of 0.1 kg and the average value was reported.

2.4. Phase analysis

XRD analysis is conducted to identify the crystalline phase using PANalytical X'PERT PRO MPD Model PW 3060/60 operating at 40 kV and 30 mA with Cu Kα radiation. The obtained peaks are than compared with ICDD in order to identify the crystalline phases. Scanning is done in the range of 2θ angle from 10° to 90° with a step size of 0.017°. The data is analyzed using X'Pert Highscore software.

2.5. Microstructural analysis

Microstructural analysis was carried out using SEM EVO 50 Carl Zeiss SMT model operated at 20 kV with back scattered electron (BSE) mode attached with energy dispersive X-ray (EDX). Prior to SEM investigation, the glass ceramic samples were grinded using 400, 600, 800, 1200 and 2400 grid SiC paper and polished using 0.1 μm alumina paste. The samples are chemically etched in HF (5%) solution for 1.5 min. The etched samples are then Au-coated.

3. Results and discussion

3.1. Physical properties of sintered glass composite

Figs. 1 and 2 show the apparent porosity, bulk density and water absorption of GCM with increasing BS waste loading ranging from 30 to 70 wt.%. It was observed that the porosity [%] increases as the BS waste loading [%] increases.

A gradual increase in porosity (Fig. 1) was observed from BS waste loading of 30–60 wt.% followed by a drastic increase at 70 wt.%. The lowest porosity value was observed at 30 wt.% BS waste loading GCM samples which is 2.20%. The water absorption shows similar trend as the porosity. The minimum water absorption value was observed at 30 wt.% BS waste loading GCM samples which is 1.17%. Porosity contributes to the bulk density and water absorption characteristics of the sample [28] thus; bulk density (Fig. 2) shows a decreasing trend with increasing waste percentage. 30 wt.% BS waste loading GCM samples show the highest bulk density of 1.88 g/cm^3. Based on these physical properties, GCM samples from batch formulation of BS 30 wt.% and 70 wt.% SLS waste glass have shown the optimum performance. The water absorption of samples with 30 wt.% BS (1.17%) has met the requirements of Malaysian Standard for floor tiling application [25]. Samples of this batch have shown better water absorption results compared to 5% water absorption of ceramic tiles currently applied for floor tiling [26].

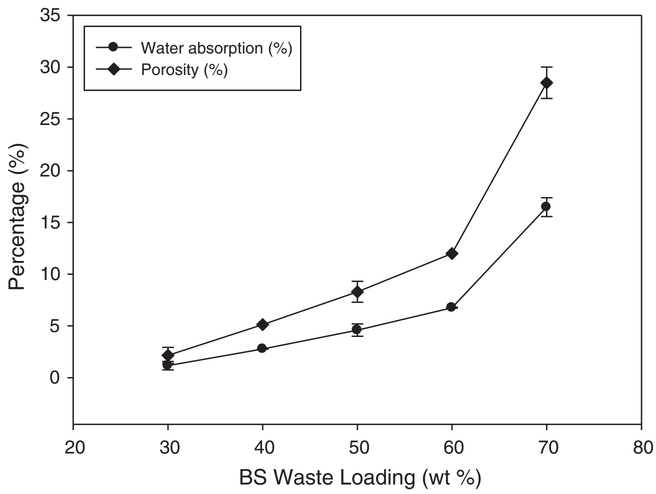

Fig. 1. Apparent porosity (%) and water absorption (%) of glass composite material according to BS waste loading (wt.%).

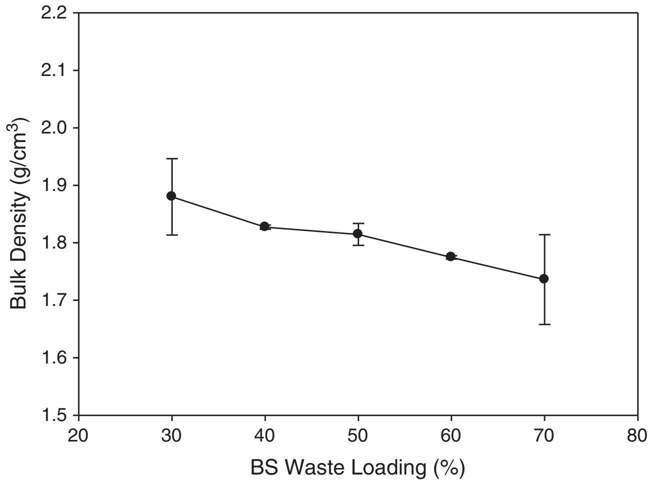

Fig. 2. Bulk density of glass composite material according to BS waste loading (wt.%).

Based on the sintering theory, the volume of glass powders which varies according to BS waste loading in the samples influences the rate of pore percentage and isolated pore formation through production of viscous flow [14]. Porosity decreases when there is a good flow of viscous liquid phase towards the open pores. Here, the formation of liquid phase is influenced by the firing effect and glass powder volume in the mixture resulting in volume and viscosity of the phase. The flow mechanism of the viscous flow towards the open pores is influenced by the surface energy created by fine pores on the glass composite body [16]. The drastic increase at 70 wt.% BS waste loading in apparent porosity and water absorption measurements is due to significant insufficient glassy phase to form viscous flow [14]. Water absorption was influenced by the porosity percentage in the GCM which can be observed from the similar trend of the both results. Also, the bulk density was influenced by partial crystallization of the amorphous phase and the rate of porosity percentage in the GCM [15].

3.2. Mechanical properties of sintered glass composite

Fig. 3 shows the results of modulus of rupture (MOR) of the GCM samples. The MOR decreases as the BS waste loading increases. The

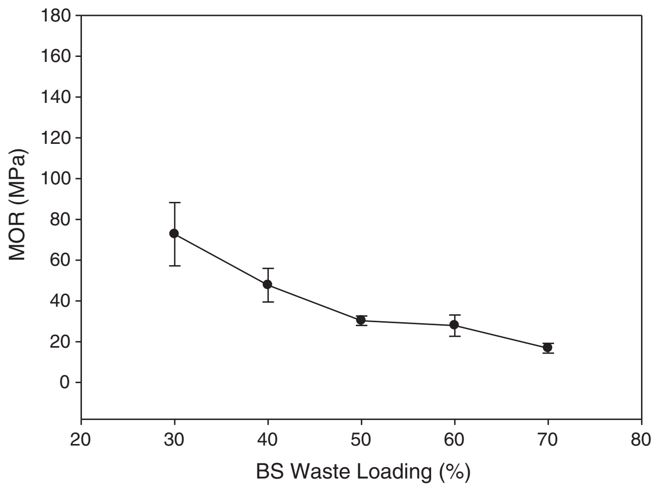

Fig. 3. Modulus of rupture of the final sintered ceramic glass composite material.

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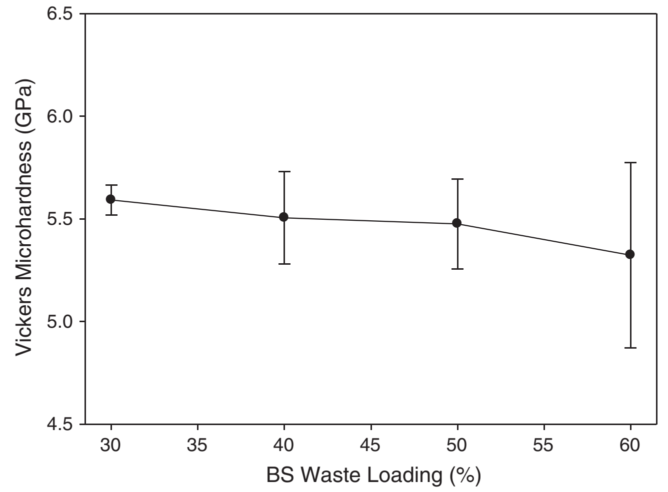

Fig. 4. Vickers microhardness measurement of the final sintered glass composite material.

highest MOR of 70.57 MPa was observed in sintered samples of 30 wt.% BS waste loading. The lowest MOR is (16.79 MPa) is observed at GCM samples of BS 70 wt.% waste loading. 30 wt.% BS batch samples have shown higher MOR compared to ceramic tiles currently applied for floor tiling which is 40 MPa and have met the Malaysian Standard requirements [25,26]. In Fig. 4, the Vickers microhardness measurements show a decrease as the BS waste loading percentage increases. Samples of 30 wt.% BS waste show the highest value of 5.6 GPa. Microhardness for samples of 70 wt.% BS waste could not be reported as there is no reading obtained due to failure of the sample to resist the indentations. It is determined that samples from batch formulation 30 wt.% BS waste shows optimized mechanical properties. The mechanical properties results indicate that an increase in BS waste content gives lower value of material microhardness and MOR. It shows that higher glass volume content contributes to improved mechanical properties. The volume of SLS glass in the sample would induce crystallization, thus by increasing the SLS glass weight percentage higher hardness values can be obtained due to crystal phase presence. The presence of crystals in the sintered sample will affect the mechanical properties of the glass ceramic whereby higher crystallization results in better mechanical performance [17].

It is also observed that values of MOR and microhardness decrease in correlation with increasing porosity. The 70 wt.% BS waste sample of the highest porosity is unable to withstand the indentation due to porous surface and the microhardness value could not be obtained. Level of porosity influences the mechanical properties of a glass ceramic sample as well. It can be seen that values of MOR and microhardness decrease in correlation with the presence of higher porosity percentage. The porous attribute influences the densification and congruency of the sintered glass ceramic sample [14].

3.3. Microstructural analysis

Fig. 5 shows XRD pattern of every GCM based on BS waste loading (wt.%). Anorthite sodian (ref: 01-085-0878, $Na_{0.48}Ca_{0.52}Al_{1.52}Si_{2.48}O_8$), quartz (ref: 01-089-1961, SiO_2), diopside (ref: 01-075-1092, $CaMgO_6Si_2$) and hematite (ref: 01-085-0599, Fe_2O_3) are identified in every waste loading batch. Formation of anorthite and diopside phase at 800 °C sintering temperature has been observed in other research work on studying the relationship between crystallization and sintering behavior for glass ceramic glazes [18]. Anorthite exists as brown crystals which can be observed from the brownish color of the sintered sample and its application is mainly in brick making [19]. Quartz phase is identified in corresponds to the major composition of SiO_2 in both of BS and SLS glass. Quartz is categorized as glass network formers [20]. It is also known as silicate mineral mainly applied for brick manufacturing or construction material [19]. The formation of diopside is an advantage to sintered glass composite as it exhibits excellent mechanical properties whereby it is highly recommended for construction material application [21]. Hematite phase presence is due to the existence of Fe_2O_3 oxide in the BS and SLS glass chemical compositions. The crystalline phases identified are in agreement with the major oxide composition of SiO_2, Fe_2O_3, Al_2O_3 and CaO from BS and SLS glass XRF results.

3.4. SEM & EDX analysis

Fig. 6 illustrates the SEM micrograph of sample cross section of 30–70 wt.% BS waste loading. Pores and dense surface attributes are observed. The micrographs show the increase in pores and decrease in dense surface as BS waste loading increases. Micrograph of 30 wt.% BS waste loading sample cross section (Fig. 6a) shows least pores and higher presence of dense even surface compared to other GCM samples. The observed pores are closed pores (verified by EDX analysis) indicating high congruency of the sample. It is observed that the presence of dense surface reduces as the BS waste loading increases (Fig. 6b–e). Open pores can be observed in 50 wt.% (Fig. 6c) and 60 wt.% (Fig. 6d) BS waste loading but the size of the pores varies. Samples of 60 wt.% waste loading have bigger open pores ~30 μm (Fig. 6d) compared to 50 wt.% waste loading sample ~6 μm pore size (Fig. 6c). 70 wt.% BS waste loading sample (Fig. 6e) depicts

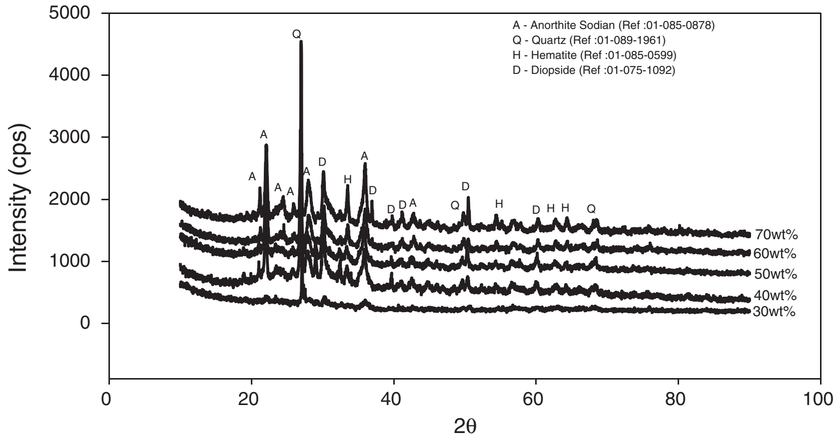

Fig. 5. X-ray diffraction pattern of final sintered glass composite for every BS waste loading (wt.%).

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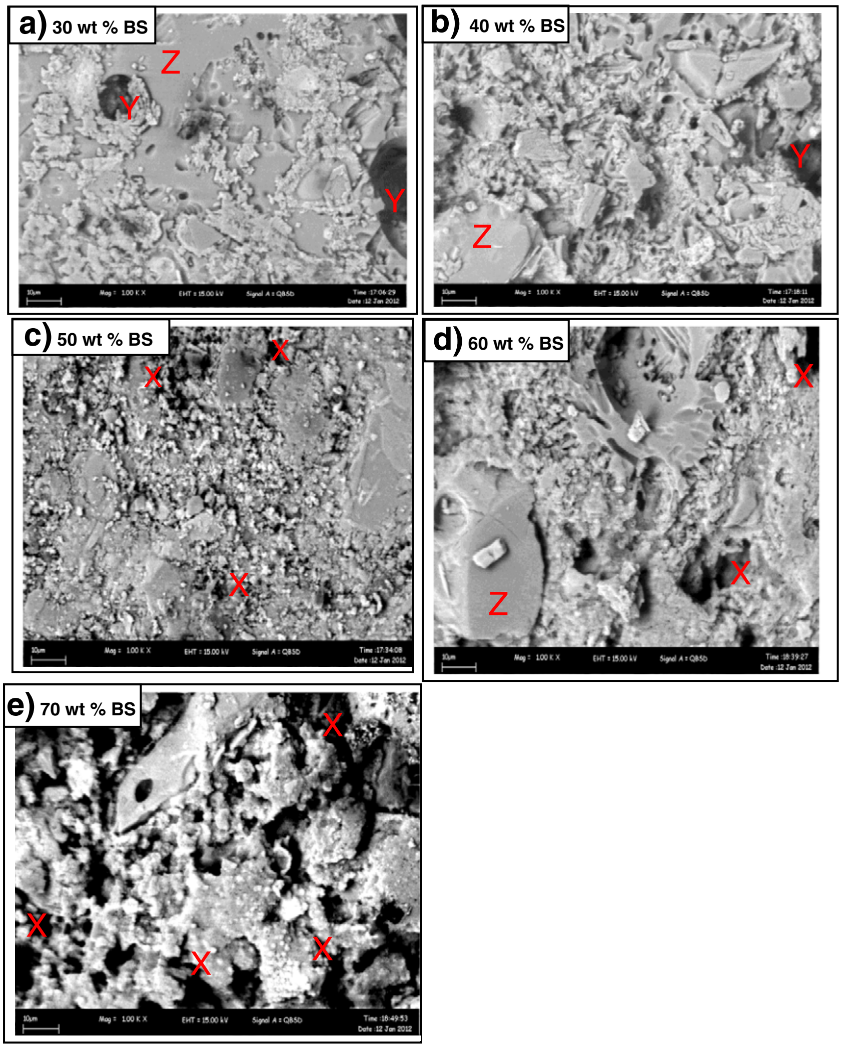

Fig. 6. SEM micrograph cross section of BS waste loading 30 wt.% BS waste loading (a), 40 wt.% BS waste loading (b), 50 wt.% BS waste loading (c), 60 wt.% BS waste loading (d) and 70 wt.% BS waste loading (e) sintered at 800 °C with 1 hour soaking time. (x = open pores, y = closed pores, z = dense surface).

highest open pores without dense surface compared to other samples. It can be deduced that, microstructural attributes are resulted by viscous sintering through its shrinkage and densification process [24]. Low BS waste loading produces denser surface and less pores with no open pores structure. The congruent microstructure of 30 wt.% BS waste loading has shown the highest performance in reference with the physical and mechanical results.

Fig. 7 shows SEM surface micrograph of optimized GCM sample of 30 wt.% BS waste loading and the elements detected through EDX analysis in each features are tabulated in Table 3. It is observed that the microstructure consists of 3 main features which is even dense surface (A), uneven dense surface (B), coarse feature (C), closed pores (D & E). High content of Si, O, Ca and Fe is observed in most of the features. Even dense surface (A) shows a smooth appearance and it consists of Si and O possibly representing quartz phase (detected in the XRD analysis). Potassium is detected in uneven dense surface (B) and closed pores (D & E) contributing to the brownish color of the sintered sample [19]. The mixture of elements present in each phase, are in agreement with the SLS waste glass and BS chemical composition indicating reactions has taken place between the viscous glass and BS waste during sintering. Moreover, elements identified in EDX analysis (Table 3) are in agreement with the elements in phases identified by XRD analysis. It should be noted that, EDX analysis on pores represented by D and E reveals element presence indicating that it is a closed pores. It justifies the observation of the lowest porosity and water absorption as well as the highest bulk density determined from the physical analysis on this batch formulation.

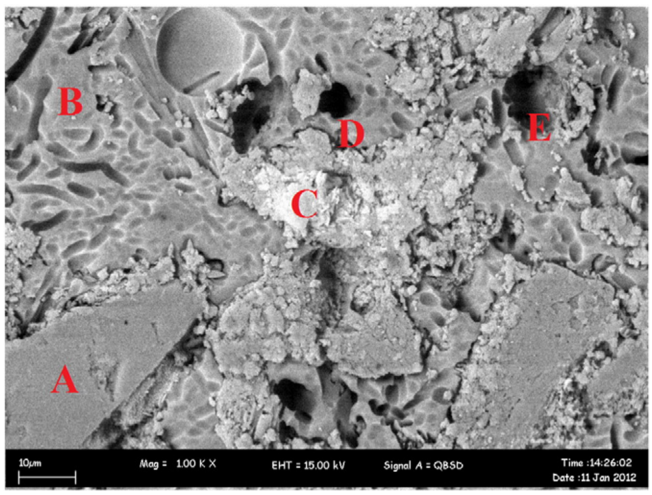

Fig. 7. EDX analysis on GCM from 30 wt.% SLS waste glass and 70 wt.% BS waste loading.

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Table 3
Element detected in EDX spectrum from sintered glass composite material of 30 wt.% BS waste loading and 70 wt.% SLS waste glass. Analytical error is lower than 25%.

Spectrum	Element (wt.%)						
	Si	O	Fe	Na	Al	K	Ca
A	53.4	46.6	–	–	–	–	–
B	62.4	–	–	9.59	1.69	1.52	11.07
C	–	–	57.65	–	2.55	–	1.9
D	54.27	7.43	9.75	1.97	1.62	2.47	22.49
E	17.69	–	51.54	2.82	1.73	3.33	22.89

4. Conclusion

Glass composite material incorporating incinerated scheduled waste (BS) and SLS waste glass has been produced by sintering at 800 °C at a rate of 2 °C/min and 1 hour soaking time. The effect of BS waste loading on the physical, mechanical and microstructural properties on the GCM is studied. The GCM porosity and water absorption increases as the BS waste loading increases and bulk density decreases on the other hand. BS waste loading influences the GCM mechanical properties whereby Vickers microhardness and modulus of rupture (MOR) values decrease as the BS waste loading increases. Microstructural analysis of GCM from every BS waste loading reveals 4 types of crystalline phases which is anorthite sodian, quartz, diopside and hematite. The optimized properties are shown by GCM produced from BS 30 wt.%. It has shown the water absorption of 1.17%, porosity percentage of 2.2% with the highest bulk density of 1.88 g/cm^3. It also has shown good mechanical properties of 5.6 GPa Vickers microhardness and MOR of 70.57 MPa. The microstructure of optimized sample consist of dense even surface with no open pores. According to Malaysian Standard Ceramic Tiles Classification, GCM produced from BS 30 wt.% is classified as ceramic tiles with low water absorption suitable for floor tiling, fulfilling the requirement of 0.5% to 3% water absorption range and above the minimum MOR value of 30 N/mm^2 or 30 MPa [25]. Moreover, it has shown higher MOR value and lower water absorption compared to current domestic ceramic tiles for floor tiling application as compared in the physical and mechanical analysis.

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