

FORMING AND PROPERTIES OF GLASSES MADE FROM TWO THAI BOTTOM ASHES

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Received: August 11, 2023; Revised: February 06, 2024; Accepted: February 15, 2024

Abstract

Industrial wastes are often an under-utilized source of raw materials. Bottom ashes resulting from incineration processes can contain various metal oxides, including many commonly used in glass making. This research studied the forming and properties of glasses made from two bottom ashes. Lignite bottom ash (BL) from Lampang, Thailand and a municipal solid waste incinerator bottom ash (BP) from Phuket, Thailand. Both bottom ashes were found to contain significant amounts of SiO_2 , CaO , Al_2O_3 , and Fe_2O_3 . After ball milling, the ashes were melted without additions, at temperatures from 1300 to 1500°C. The resulting glasses, as confirmed by XRD, were black and fully opaque, a result of the high Fe_2O_3 content. It was found that the BP glasses were slightly more refractory, had a greater average thermal conductivity of 1.52 W/m·K and a lower coefficient of thermal expansion (CTE) of $6.97 \times 10^{-6}/^\circ\text{C}$, compared to the BL glasses, which had an average thermal conductivity of 1.39 W/m·K and a CTE of $7.98 \times 10^{-6}/^\circ\text{C}$. The BL glasses also had a higher average density of 2.88 g/cm³ and a Vickers hardness of 6.29 GPa, compared to the BP glasses, with average values of 2.74 g/cm³ and 6.06 GPa, respectively. The increase of glass melting temperature up to 1450°C tended to improve the observed properties, followed by a decrease at 1500°C. Despite the atypical glass chemistry of the two bottom ashes, they were successfully melted to produce glasses and the measured properties demonstrated suitable potential to warrant further study.

Keywords: Coal Bottom Ash; Glass; Glass Forming; MSW Bottom Ash

Introduction

Waste byproducts from various industrial processes can provide alternative resource streams with which to manufacture materials. Several silicate-based waste materials have been observed to have compositions similar to those of cement, ceramics, and glasses (Rambaldi *et al.*, 2010; Chinnam *et al.*, 2013). Examples of these silicate waste materials include agriculturally derived ashes, dredge sludges, metallurgical slags, fly ashes, and bottom ashes (Aprianti, 2017).

Bottom ashes are a byproduct of the incineration of various types of fuel sources. An electric power plant that burns lignite coal, will produce what is called lignite bottom ash or LBA, while a municipal solid waste (MSW) incinerator that burns consumer refuse, will produce MSW bottom ash. Due to their larger particle sizes, bottom ashes are often underutilized as a raw materials source, mainly being used as aggregate and backfill in construction (Kim and Lee, 2015), compared with

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DOI: <https://doi.org/10.55766/sujst-2024-01-e02100>

Suranaree J. Sci. Technol. 31(1):030169(1-13)

the much smaller fly ashes, which can find added value being used as pozzolans in the cement industry (Paris *et al.*, 2016).

A large percentage of bottom ashes go unused and must be dealt with by the producers, who often simply landfill them. The long-term storage of bottom ashes can require a considerable amount of land, with ever-increasing space requirements. As these storage sites are outdoors, weather exposure can lead to problems with the leaching of potentially toxic chemicals from the bottom ash. This therefore requires monitoring and potential remediation of the site (Al-Ghouti *et al.*, 2021). All of this adds to the operating costs for the producers.

It is obviously desirable to find potential applications that can utilize large quantities of bottom ash besides simple aggregate and backfill, which even then can still have leaching problems. Some researchers (Dou *et al.*, 2017) have explored the recovery of valuable metal elements, while others have utilized bottom ashes in small quantities in cement or ceramics (Pan *et al.*, 2008; Glymond *et al.*, 2018). While worthwhile, these applications utilize only a small portion of the waste, leaving the rest behind or using them in very limited quantities. An application that can utilize all of the bottom ash in large amounts and with improved chemical inertness to reduce leaching is therefore desirable.

Glasses can fulfill all of those requirements (Chinnam, *et al.*, 2013). Commercial glasses, such as for windows and containers, are manufactured with carefully designed and controlled compositions, using a variety of synthetic and refined natural raw materials. Their chemical compositions are intended to yield a specific combination of properties, including low cost. As an amorphous material though, glass has the capability for a very wide range of chemical compositions. Glasses can be made from a wide variety of different materials and do not require high purity, as a rule. Glasses can also be very chemically durable, having high corrosion resistance to a variety of different liquids, effectively trapping potentially toxic elements (Heuss-Aßbichler and Bayuseno, 2021).

The aim of this research was to determine the glass-forming ability of two different domestically produced bottom ash powders: a lignite bottom ash from the Mae Moh electric power plant in Lampang, Thailand, and a municipal solid waste incinerator bottom ash from Phuket, Thailand. To our knowledge, this research has not been conducted previously and may provide another utilization opportunity for these materials. The bottom ashes were characterized for chemical and phase composition and then melted at several different temperatures to observe the glass-forming ability. The resulting glasses were then characterized for

several basic physical, optical, thermal, and mechanical properties.

Materials and Methods

Materials and Glass Preparation

The bottom ashes for this study were obtained from two different sources. The first bottom ash was from a municipal solid waste incinerator of the Phuket City Municipality, Phuket province, Thailand, referred to here as BP. The second bottom ash was from a lignite coal-fired electric power plant, the Mae Moh electric power plant, Lampang province, Thailand, referred to here as BL. The BP powder was light gray in color with median particle sizes of 6.04, 48.42, and 161.94 μm (D_{10} D_{50} D_{90}). The BL powder was light black in color with median particle sizes of 11.75, 84.55, and 189.74 μm (D_{10} D_{50} D_{90}), as determined by laser diffraction particle size analysis (Malvern Mastersizer S).

In order to improve the glass melting and homogenization, the BP and BL bottom ash powders were ball milled and sieved to obtain a fine powder with a maximum particle size of 45 μm . Glass samples were prepared directly from the milled BP and BL powders without any other additions. For each glass sample, 100 g of milled bottom ash powder was melted in an open alumina crucible at a temperature of 1300, 1350, 1400, 1450, or 1500°C for 2 hours, using a heating rate of 10°C/min. The glass melts were cast into a rectangular brass mold (25×75 mm) that was pre-heated to 450°C, and the glass was allowed to solidify for approximately 1 minute before removal. The still hot glass was then placed into a 500°C annealing furnace for 2 hours, and then cooled to room temperature inside of the annealing furnace.

Characterization and Testing

For the various characterization methods, the annealed glass samples were cut using a low-speed saw (Buehler IsoMet) with a diamond blade and then mounted in epoxy resin. The cut samples were polished with silicon carbide paper in the order of 150, 200, 600, 1000, and 1200 grit and then the mirror was finished using 1 μm colloidal alumina powder.

The chemical composition and phase composition of the bottom ash powders and glass samples were analyzed using an X-ray fluorescence (XRF) spectrometer (Bruker AXS S4 Pioneer) and an X-ray diffractometer (XRD) (Bruker AXS D8 Discover). For XRD, samples were analyzed from 20 to 80° with a step size of 0.02° using the standard Cu K α radiation with a wavelength of 1.541 Å.

The optical transmittance of the glasses from 200 to 1000 μm was measured using a spectrophotometer (Varian Cary 50 UV-Vis Spectrophotometer). Cut and polished samples had dimensions of $10 \times 10 \times 20$ mm. The refractive index of the glasses was measured using a refractometer (GIA Duplex II). A 5 mm thick polished sample of each glass was coupled to the refractometer using refractive index liquid. A standard monochromatic yellow light source ($\lambda \sim 589.3$ μm) was used, corresponding to the refractive index n_D of the sodium D line.

The viscosity behavior of the glasses was measured using the fiber elongation method (BAHR-Thermoanalyse GmbH model VIS402), according to the standard ISO 7884-3 (ISO, 1987). An approximately 5 cm long fiber with spherical ends was prepared from each glass sample. The low-temperature standard viscosity reference points up to and including the Littleton softening point ($\eta = 10^{6.6}$ Pa·s) were measured, and curve fitting with the provided software was used to determine the high-temperature reference points. Thermal conductivity of the glasses was measured using the transient laser flash method (NETZSCH LFA447) from 25 to 250°C. A cut and polished sample with dimensions of $12 \times 12 \times 3$ mm was used. The thermal expansion behavior of the glasses was characterized using a dilatometer (NETZSCH DIL 402 PC) in the temperature range of 25 to 800°C. The coefficient of thermal expansion (CTE) in the range of 50 to 300°C was determined, as well as the dilatometric softening temperature T_d . A cylindrical glass specimen 5 cm long with a 5 mm diameter was used.

The glass density was measured using the Archimedes method (A&D Weighing AD-1653). A cut and polished sample was weighed in air and then weighed again while submerged under distilled

water, to an accuracy of 0.0001 g. The density of the glass ρ_G was calculated according to equation (1).

$$\rho_G = \frac{W_1}{W_1 - W_2} \times \rho_L \quad (1)$$

Where W_1 is the sample mass in air, W_2 is the sample mass submerged under water and ρ_L is the density of the water for a given temperature.

The Vickers hardness of the glasses was measured using a microhardness tester (SHIMADZU HMV2 TDA), according to ASTM C1327 (ASTM, 2015). A cut and polished sample was indented with a load of 980.7 mN ($H_v0.1 = 100$ g) for an indentation time of 10 seconds. Five indents were made per sample. The diagonal lengths of the indents were measured using an optical microscope, and the hardness H_v was calculated according to equation (2).

$$H_v = 1.8544 \frac{P}{d^2} \quad (2)$$

Where P is the load in kg and d is the average length of the indent diagonals in mm.

Results and Discussion

Bottom Ash Characterization

The chemical compositions in weight percent (wt.%) of the BP and BL powders, as determined by XRF, are shown in Table 1. The results are reported as the stoichiometric oxide of the detected metal element. It was found that both BP and BL powders contained a large amount of CaO and SiO_2 . The BP powder contained a greater amount of CaO than SiO_2 , while the BL powder contained a greater amount of SiO_2 than CaO. From a glass-forming

Table 1. XRF chemical composition of the BL and BP powders. The results are reported as the stoichiometric oxides

Chemical composition	BL Powder (wt.%)	BL Ref. ^a (wt.%)	BP Powder (wt.%)	BP Ref. ^b (wt.%)
SiO_2	31.00	38.33	11.4	48.26
CaO	23.24	18.01	41.0	19.07
Fe_2O_3	16.47	15.78	5.81	4.44
SO_3	4.51	w/ LOI	5.44	1.06
Al_2O_3	16.87	16.98	4.78	4.04
Cl	-	-	3.04	0.01
P_2O_5	0.17	-	2.85	-
ZnO	-	-	2.68	-
Na_2O	1.07	-	1.83	2.41
K_2O	2.76	2.15	1.50	1.17
MgO	1.90	2.46	1.40	1.16
Other	0.99	0.67	1.41	-
LOI	-	5.62	-	16.56
Total	98.98	100.00	81.73	98.18

^aNamkane *et al.*, 2016, ^bKrammart *et al.*, 2004

perspective, the greater SiO_2 content of the BL powder would better favor glass formation, while the greater content of CaO in the BP powder would possibly favor crystal formation. Both BL and BP powders also contained significant amounts of Al_2O_3 , Fe_2O_3 , MgO , Na_2O , and K_2O , all common glass-making components, with the exception of Fe_2O_3 . The BP powder also contained a moderate amount of P_2O_5 and ZnO , also used in some glass compositions. The BP and BL powders contained trace amounts (<0.2 wt.%) of other elements, given as a summation under the 'Other' row in Table 1. These included TiO_2 , MnO , SrO , BaO , and ZrO_2 in the BL and BP powders, and also CuO , PbO , SnO_2 , and NiO in only the BP powder. The BP powder had a greater number of different chemical elements, likely due to the wide range of items found in the municipal waste (Heuss-Aßbichler and Bayuseno, 2021).

The XRF chemical compositions of the BL and BP powders, reported by other researchers (Namkane *et al.*, 2016; Krammart *et al.*, 2004), are also shown in Table 1. The chemical composition of the BL powder measured in this paper compared well to the reported composition for the same lignite bottom ash powder (Namkane *et al.*, 2016). Their powder was a bit richer in SiO_2 and had slightly less CaO . The contents of the other elements were all very similar. This slight chemical variability is to be expected from a naturally derived powder. The chemical composition of the BP powder from this work was also comparable to that reported by (Krammart *et al.*, 2004) but with two very significant differences; they reported a much greater SiO_2 content and a much lower CaO content. When XRF data is reported as a stoichiometric oxide, it can be an assumption. The sum of the chemical components should be very near to 100% for that assumption to be correct. As seen in Table 1, the sum of the chemical components of the BP powder was only 81.73%, indicating that one or more of the elements within the BP powder was not in the form

of a stoichiometric oxide. While the detected elements within the BP powder were correct, the calculated weight percentages of the components were in error.

The XRD patterns of the BP and BL powders are shown in Figure 1. It was seen that the BP powder (Figure 1(a)) was highly crystalline and the phases matched with JCPDS patterns included CaCO_3 (JCPDS 47-1743), SiO_2 (JCPDS 05-0493), Al_2O_3 (JCPDS 11-0661), Fe_2O_3 (JCPDS 32-0469) and ZnO (JCPDS 74-0534). The presence of CaCO_3 in the BP powder was responsible for the previously mentioned erroneous XRF stoichiometric oxide contents, shown in Table 1. Carbonates alter the matrix absorption of X-rays generated with XRF, which requires adjustments beyond the standard stoichiometric oxide assumption (Quye-Sawyer *et al.*, 2015; de Winter *et al.*, 2017) which were not undertaken here. As the BP powder was the product of high-temperature incineration, the standard stoichiometric oxide assumption was logical. Municipal incinerators often operate at temperatures of 1000°C or greater (Heuss-Aßbichler and Bayuseno, 2021), which is higher than the decomposition temperature of $<900^\circ\text{C}$ for CaCO_3 (Kenny *et al.*, 2012). The presence of CaCO_3 in the BP powder was likely the result of carbonation of Ca(OH)_2 , itself the result of hydration of CaO , both due to outdoor weathering of the CaO contained in the BP powder (Campo *et al.*, 2021).

The XRD pattern of the BL powder (Figure 1(b)) showed that it was much more amorphous, as indicated by the amorphous hump centered near 30° , although it also contained several crystalline phases, including $\text{Ca}_3\text{Si}_2\text{O}_7$ (JCPDS 02-0323), $\text{Ca}_2\text{Al}_2\text{SiO}_7$ (JCPDS 87-0968), $\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$ (JCPDS 70-2144), $\text{Ca}(\text{Mg},\text{Fe},\text{Al})(\text{Si},\text{Al})_2\text{O}_6$ (JCPDS 41-1483), Fe_2O_3 (JCPDS 84-0309) and SiO_2 (JCPDS 82-1560). There was no detected free CaO , CaCO_3 , or any other potentially reactive phase, indicating that outdoor weathering of the BL ash is less of a concern, unlike the BP ash.

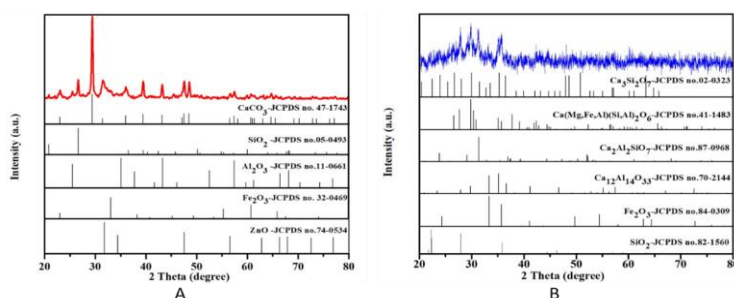


Figure 1. XRD patterns of BP and BL powders. (A) The BP powder is composed of multiple crystalline, single oxide phases. (B) The BL powder is more amorphous and contains a mixture of single oxides and various calcium aluminosilicates

Glass Characterization

Chemistry and Phase: An XRD analysis of the BP and BL powders melted at 1300 and 1450°C is shown in Figure 2(a). All of the XRD patterns showed a single hump centered near 29°. The characteristic amorphous hump of the XRD pattern indicates that the melted powder samples have an amorphous atomic structure, i.e. they are glass. The BP glass melted at 1300°C and also showed a single narrow peak in the XRD pattern, which indicated the presence of a crystalline phase. The phase was identified as an alumino-silicate phase, Al_2SiO_5 (JCPDS 72-1441), shown in Figure 2(b). Al_2SiO_5 was not previously identified in the XRD analysis of the starting BP powder (Figure 1(a)), so it was not from unmelted powder, but rather it was formed during the glass melting and cooling process. This was also reported for the melting of a different MSW bottom ash, where an unidentified crystalline phase was observed after melting at 1300°C for 1 hour, but was gone after melting at 1400°C for 2 hours (Monteiro *et al.*, 2006). As seen in Figure 2(a), Al_2SiO_5 was not observed in the BP glass melted at 1450°C or the BL glasses melted at 1300 and 1450°C.

The XRF chemical analysis of the BL glasses melted at 1300, 1400, and 1500°C is shown in Table 2. The composition of the starting BL powder is included for comparison. The high-temperature melting of the BL powder would be expected to decrease the content of typically volatile components, such as water and sulfur compounds. As the melting temperature is increased, oxides prone to volatilization, such as alkali oxides, would also be expected to experience losses in an open crucible melting (Shelby, 2005). It is seen in Table 2, that the sulfur (SO_3) content was significantly reduced at 1300°C, and was nearly zero at 1500°C. The alkali oxides, Na_2O and K_2O , also experienced some losses as the melting temperature was increased to 1500°C. The changes in the glass composition as a function of melting temperature generally showed small changes, either up or down, for the various non-volatile elements. This is likely to be expected from a naturally derived powder, in addition to the anticipated volatile losses.

The XRF analysis of the BP glasses melted at 1300, 1400, and 1500°C is shown in Table 3. The starting BP powder composition is included for comparison. It is seen that while the compositions of the three glasses were generally similar, there was a

Table 2. XRF chemical composition of BL powder and glass samples. The results are reported as stoichiometric oxides

Chemical Composition	BL Powder (wt. %)	BL 1300 (wt. %)	BL 1400 (wt. %)	BL 1500 (wt. %)	Container Glass ^a (wt. %)
SiO_2	31.00	31.00	32.07	27.27	74
CaO	23.24	25.73	24.66	26.12	3
Fe_2O_3	16.47	19.98	18.46	20.83	0.04
SO_3	4.51	0.49	0.11	0.02	0.2
Al_2O_3	16.87	16.10	18.14	19.72	1.5
Cl	-	-	-	-	-
P_2O_5	0.17	0.18	0.19	0.15	-
ZnO	-	-	-	-	-
Na_2O	1.07	0.90	0.88	0.67	16
K_2O	2.76	2.47	2.33	2.35	1
MgO	1.90	1.98	2.10	1.70	4
Other	0.99	1.13	1.05	1.13	-
Total	98.98	99.96	99.99	99.96	99.74

^aShelby, 2005

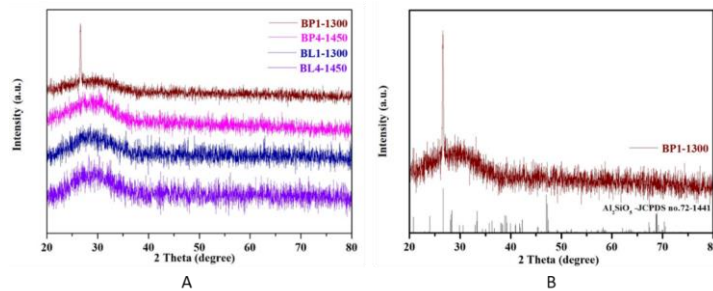


Figure 2. (a) XRD patterns of BP and BL glasses melted at 1300 and 1450°C. (b) XRD pattern of BP glass melted at 1300°C, identifying the Al_2SiO_5 phase

large difference between them and the starting BP powder. The BP powder was assumed to be composed entirely of stoichiometric oxides. However, this was not the case because the calcium was not in the form of a simple oxide, but instead a carbonate. This was confirmed in the XRD pattern, seen in Figure 1(a). At the melting temperatures used in this research, the CaCO_3 in the BP powder decomposed to CaO and the volatile CO_2 gas. Due to this, the compositions of the glasses in this study were more comparable to the composition of the BP powder reported by (Krammart *et al.*, 2004), previously shown in Table 1.

Also included in Tables 2 and 3 are the compositions of a typical soda lime container glass and rock wool, which is a fiberglass product made from volcanic rocks such as basalt and rhyolite (Kleinholz *et al.*, 2012). It is apparent that the BL and BP glass compositions are very different from a typical soda lime composition. They generally contain much less SiO_2 and Na_2O and much more CaO , Al_2O_3 , and Fe_2O_3 . Both the BL and BP glasses compare more favorably to the rock wool composition. Rockwools typically contain less SiO_2 and alkali but more alkaline earth, Al_2O_3 , and Fe_2O_3 than regular soda-lime glasses. The discontinuous

rockwools are typically used as thermal and sound insulation (Yue *et al.*, 2021), while the compositionally similar, continuous basalt fibers are used as reinforcement in concrete and other composites (Mahlting, 2018).

Optical Properties

Images of the as-cast and annealed samples of BP and BL glasses melted at 1300°C are shown in Figure 3(a) and (b), respectively. The samples were deep black in color and exhibited a glossy surface finish, free of visible cracks. The glasses resembled obsidian, a natural volcanic glass (De Campos *et al.*, 2021). Among the many reasons that the common glass-making materials are used, e.g., Na_2O , CaO , and SiO_2 , is that they produce an optically transparent, colorless glass. The black color of the BP and BL glasses is due to the significant amount of the glass colorant Fe_2O_3 in the starting bottom ash powders (Shelby, 2005; Varshneya *et al.*, 2019). In low concentrations (<0.1 wt.%), iron can produce green-blue or yellow-brown colors, depending on the valence state. This is largely independent of the glass structure, and depends more on the glass composition and the processing conditions. However, with higher colorant concentrations, more

Table 3. XRF chemical composition of BP powder and glass samples. The results are reported as stoichiometric oxides

Chemical Composition	BP Powder (wt. %)	BP 1300 (wt. %)	BP 1400 (wt. %)	BP 1500 (wt. %)	Rockwool ^a (wt. %)
SiO_2	11.4	49.32	47.47	46.94	46.3
CaO	41.0	22.86	24.17	24.56	10.9
Fe_2O_3	5.81	11.24	11.87	11.03	11.9 ^b
SO_3	5.44	0.05	0.03	0.01	-
Al_2O_3	4.78	6.32	6.46	7.46	13.3
Cl	3.04	0.00	0.00	0.00	-
P_2O_5	2.85	1.91	1.90	1.99	-
ZnO	2.68	0.48	0.56	0.74	-
Na_2O	1.83	4.10	3.61	3.27	2.6
K_2O	1.50	1.11	1.07	1.13	1.3
MgO	1.40	1.33	1.30	1.24	9.7
Other	1.41	1.24	1.54	1.59	3.1
Total	81.73	99.96	99.98	99.96	99.1

^aKleinholz *et al.*, 2012, ^b $\text{Fe}_2\text{O}_3 + \text{FeO}$



Figure 3. Images of the cast (A) BP glass and (B) BL glass, both melted at 1300°C . (A) The glass shows large regions of grey discolorations. (B) The glass is free of discolorations

light is absorbed by the glass, and in the case of iron, results in a black, opaque glass.

The BL glass melted at 1300°C (Figure 3(b)) appeared visually uniform, and an increase in the melting temperature to 1500°C produced no noticeable visual changes to the glass samples. The BP glass melted at 1300°C (Figure 3(a)) had several noticeable regions of light grey discoloration. The discolorations were most apparent in the glass melted at 1300°C and decreased in size and number as the melting temperature was increased. They were generally limited to the surface. These discolorations were previously identified by XRD as an Al_2SiO_5 phase, as shown in Figure 2(b). The regions of Al_2SiO_5 remained in the cast BP glasses at a melting temperature of up to 1400°C but were absent in glasses melted at 1450°C, as seen in Figure 4(a) and (b), respectively.

The average % transmittance of the BL and BP glasses melted at 1300, 1400, and 1500°C are shown in Table 4. The % transmittance was measured between 200 and 1000 nm, which corresponds to the middle ultraviolet (UV, 200-380 nm), the visible (VIS, 380-750 nm), and the near-infrared (IR, 750-1000 nm) parts of the electromagnetic spectrum. The % transmittance measurements confirm that the BL and BP samples were optically opaque in the visible portion of the spectrum, regardless of the melting temperature. The results also found that the glasses were opaque into the near IR and the middle UV. The absorption of IR radiation by the BP and BL glasses shows a potential for usage in heat shielding applications (Chinnam *et al.*, 2013). The BL glasses had a slightly lower % transmittance of approximately 0.007%, while the BP glass averaged approximately 0.016%, likely due to the difference in iron content of the glasses. The BP glass showed slightly more variation in the transmittance as a function of melting temperature.

The measured refractive index n_D of the BP and BL glasses melted at 1300 to 1500°C are shown in Figure 5. The refractive index of the BL glasses ranged from 1.63 to 1.64, with an average of 1.64, while the refractive index of the BP glasses ranged from 1.58 to 1.60, with an average of 1.59, across the melting temperatures. For comparison, a transparent soda lime silicate glass, commonly used for windows or containers, has an average refractive index of ~ 1.52 . The refractive index curves of the BP and BL glasses were relatively flat as a function of melting temperature, with both of the curves slightly decreased at the maximum melting temperature of 1500°C. The decrease in refractive index at 1500°C was possibly due to volatilization of select oxides, as the glasses were melted in an open crucible. The BL glasses, for all melting temperatures, had a higher refractive index than the

BP glasses. This was likely due to the greater iron content of the BL glasses. The refractive index of a glass is increased by elements of high atomic number and iron has a high atomic number and is in the BL glass in a larger amount (Shelby, 2005; Varshneya *et al.*, 2019).

Thermal Properties

In discussing further results, a brief explanation of glass structure is relevant. Due to the random atomic structure of glass, structure-property relationships are often approached by using the composition of the glass. Each glass-making material is understood to play a particular role in the structure of the glass, and this is related to an observed property change. The main chemical components of an oxide glass may be divided into three categories, based on how they contribute to the

Table 4. Percent transmittance from 200 to 1000 nm of BL and BP glasses melted at 1300, 1400 and 1500°C. The average is reported

Melting Temperature	BL Glass %Trans.	BP Glass %Trans.
1300°C	0.006	0.023
1400°C	0.008	0.007
1500°C	0.008	0.017

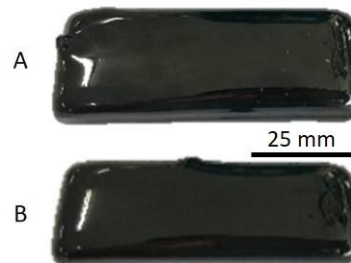


Figure 4. Images of the cast (A) BP glass and (B) BL glass, both melted at 1300°C. (A) The glass shows large regions of grey discolorations. (B) The glass is free of discolorations

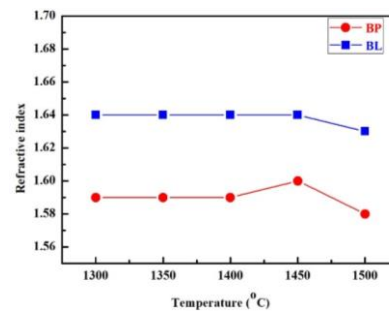


Figure 5. Refractive index n_D of BP and BL glasses melted at 1300 to 1500°C

structure and properties of the glass (Shelby, 2005; Varshneya *et al.*, 2019). The three categories are the network formers, the network modifiers and the property modifiers.

The majority of a glass structure is usually comprised of the network former, an example being SiO_2 . The network former contributes greatly to the baseline thermal and mechanical properties of the glass. The network modifiers, such as Na_2O and K_2O , replace stronger network former bonds, termed bridging oxygens (BO), with weaker non-bridging oxygens (NBO). In a silicate glass, an oxygen bonding with two silicon atoms, such as Si-O-Si , is a bridging oxygen, while if it bonds with just one silicon atom, as well as the modifying atom, it is a non-bridging oxygen, such as Na-O-Si . The presence of NBOs in a glass can decrease the overall network strength and connectivity. Network modifiers are primarily used to decrease the temperature required to achieve a desired glass viscosity, but in doing so, tend to negatively impact many other properties, such as corrosion resistance and strength. Thusly, the property modifiers are used to recover the properties that are negatively impacted by the network modifiers. Examples, such as CaO and MgO , also technically create NBOs, but they are more moderate in bond strength and improve the overall network connectivity.

Some materials, such as Al_2O_3 , can function as either network formers or property modifiers depending on the glass composition (Shelby, 2005; Varshneya *et al.*, 2019). The Al_2O_3 content in the BL and BP glasses (Tables 2 and 3) are higher than what is found in common soda lime glasses and should be addressed. When the Al_2O_3 content is less than the total alkaline (M_2O) and alkaline earth (MO) content ($\text{Al}_2\text{O}_3 < \text{M}_2\text{O} + \text{MO}$), the Al^{3+} ions are tetrahedrally coordinated and can form $[\text{AlO}_4]^{-1}$ tetrahedra that are incorporated into the silicon-oxygen tetrahedra (SiO_4) network. Thusly, Al_2O_3 is acting as a network former. The AlO_4 tetrahedron has a charge of -1, which must be balanced by one alkali ion (+1) or a pair of AlO_4 tetrahedra are balanced by one alkaline earth ion (+2). It is seen that the addition of Al_2O_3 reduces NBO content in this case. When the content of Al_2O_3 exceeds the total alkali and alkaline earth content ($\text{Al}_2\text{O}_3 > \text{M}_2\text{O} + \text{MO}$), the Al^{3+} ion can become octahedrally coordinated with the result of 3 BO and 3 NBO being created. The Al_2O_3 then acts as a property modifier and still provides general improvements to the properties. Considering only the Al_2O_3 content of the BP and BL glasses, it is lower than the combination of CaO , Na_2O , and K_2O , and it would, therefore, likely act as a network former in the BP and BL glasses.

There are two other minor categories of chemical components used in glass, the colorants and the fining agents. Colorants are used to give glasses different colors, while fining agents are used to help remove gas bubbles during the melting process. Common colorants are often one of the transition metals elements, such as copper or iron. Fining agents include arsenic and antimony oxides. The colorants and fining agents are normally used in such small quantities, that their contribution to the glass structure and most properties is often ignored.

The significant amount of iron in the BL and BP glasses (Tables 2 and 3) is well in excess of what would normally be used as a typical glass colorant (< 0.1 wt.%) and the potential contribution to the glass structure cannot be ignored. Like with aluminum, how iron is incorporated into a glass depends on the coordination number (CN). The Fe^{2+} ion (FeO) is classed as a property modifier, while the Fe^{3+} ion (Fe_2O_3) is classed as either a property modifier when $\text{CN}=6$ or as a network former when $\text{CN}=4$. It was found (Belova *et al.*, 2015) that increased iron content up to 17 wt.% in a soda silicate glass changed the coordination number of Fe^{3+} and its role from a property modifier to a network former. It was also noted that charge compensation to the Fe^{3+} was by Na^+ , which may impact the glass transition temperature. Another researcher (Zawada, 2020) observed the change of the CN of iron from 6 to 4 with up to 9 wt.% of iron in a soda lime glass. While the compositions of the BL and BP glasses in this research were not identical to those studied in the aforementioned papers, the amount of iron that the BL and BP glasses contain would indicate that it would likely be acting as a network former, like the Al_2O_3 , with both being charged balanced by the large amounts of CaO in the glasses, thus enabling them to contribute to the improvement of several of the reported properties.

Table 5 shows the measured temperatures corresponding to standard viscosity reference points of the BL and BP glasses melted at 1450°C . Reported temperature values for a soda-lime glass (SRM 710) are included for comparison. SRM 710 is a historical standard reference material used for glass viscosity measurements (NIST, 1962). It was apparent that the BL and BP glasses have temperatures for the viscosity reference points that are well above those of SRM 710. The BL and BP glasses required higher temperatures to reach the standard viscosity values, meaning that they were both more refractory than SRM 710. The measured temperature values for the BL glass were also all greater than the BP glass, indicating that the BL glass was slightly more refractory.

The reported composition of SRM 710, in weight percent (wt.%), is 70.5 SiO_2 , 8.7 Na_2O ,

7.7 K₂O, 11.6 CaO, and 1.1 Sb₂O₃. The sum of the network former and property modifiers in SRM 710 is 82.1 wt.% (SiO₂, CaO) and the sum of the network modifiers is 16.4 wt.% (Na₂O, K₂O). For the BL glass melted at 1400°C the sum of the network former and property modifiers is 95.43 wt.% (SiO₂, Al₂O₃, Fe₂O₃, CaO, MgO) and the sum of the network modifier is 3.21 wt.% (Na₂O, K₂O), while for the BP glass melted at 1400°C it is 91.27 wt.% (SiO₂, Al₂O₃, Fe₂O₃, CaO, MgO) and 4.68 wt.% (Na₂O, K₂O). The combination of network former and property modifiers can be thought of as contributing to the improvement of many properties of a glass, while the sum of the network modifiers degrades many of those same properties.

SRM 710 has a greater amount of the main network former SiO₂ than either BL or BP glasses, however the BL and BP glasses incorporate significantly more Al₂O₃, CaO, as well as the addition of Fe₂O₃, which as discussed previously, could be incorporated into the glass network. Additionally, SRM 710 also has a much greater total network modifier content than the BL and BP glasses. The smaller total amount of network former and property modifiers, combined with the greater total amount of network modifiers, creates a significant number of NBOs in SRM 710 and decreases the temperatures for the viscosity reference points.

In comparing the viscosity reference point temperature for BL and B glasses (Table 5), the BL glass had a slightly greater amount of network

former and property modifiers than the BP glass (95.43 wt.% BL vs. 91.27 wt.% BP) and a slightly lower amount of network modifier than the BP glass (3.21 wt.% BL vs. 4.68 wt.% BP). Subsequently, the BL glass should contain a slightly smaller number of NBOs than the BP glass and therefore should be slightly more refractory.

It is also noted that the Littleton softening temperatures for both BL and BP glasses (Table 5) were over 800°C. This temperature represents the onset of noticeable fiber deformation due to gravity. This may be thought of as a temperature limit for thermal fiber applications. These temperatures are comparable to the maximum use temperatures for several rock and slag wools, which are reported to be in the range of 700 to 750°C (Kleinholz *et al.*, 2012).

A graph of the measured thermal conductivities between 25 and 250°C for BL and BP glasses melted at 1400 and 1450°C is shown in Figure 6. The thermal conductivities of the four samples all decreased as a function of testing temperature. Typically, silicate glasses show the opposite behavior in that the thermal conductivity increases with temperature. This is the result of the contribution of photon transmission in an optically transparent glass (Hofmeister *et al.*, 2021). However, the BL and BP glasses are optically opaque as previously shown in Table 4, and lack the contribution from photon transmission. Thus, they behave as opaque ceramic might, with decreasing thermal conductivity for increasing temperature, as

Table 5. Measured temperatures of standard processing points for BP and BL glasses melted at 1450°C.

Reference Point	Viscosity (Pa·s)	BL 1450 (°C)	BP 1450 (°C)	SRM 710 ^a (°C)
Strain Point	10 ^{13.5}	635.7	601.1	504
Annealing Point	10 ¹²	671.6	631.5	546
Dilatometric Softening Point	10 ^{9.3}	756.6	703.8	620
Littleton Softening Point	10 ^{6.6}	884.9	813.9	724
Working Point	10 ³	1210.4	1098.2	1019
Gob Point	10 ²	1370.3	1240.7	1181
Melting Point	10	1594.2	1443.5	1434

^aNIST, 1962

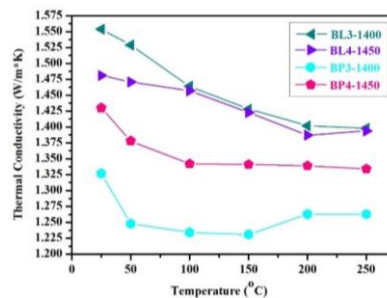


Figure 6. Thermal conductivity behavior between 25 and 250°C for BP and BL glasses melted at 1400 and 1450°C

scattering of thermal vibrations is increased. The BL glasses had a higher thermal conductivity than the BP glasses across the temperature test range. This was possibly due to the improved glass network strength as provided by the greater Al_2O_3 content. The different melting temperatures had little impact on the thermal conductivity of the BL glass but was more significant for the BP glass, possibly due to improved structural homogeneity with the higher melt temperature.

The room temperature (RT) thermal conductivity of fused silica (SiO_2) is $\sim 1.39 \text{ W/m}\cdot\text{K}$ and $\sim 1.1 \text{ W/m}\cdot\text{K}$ for ordinary soda lime glass (De Jong *et al.*, 2011). The creation of NBOs by the addition of network and property modifiers decreases the thermal conductivity of fused silica. The addition of heavier elements, such as iron, is also expected to decrease the thermal conductivity, yet both the BL glass ($\sim 1.52 \text{ W/m}\cdot\text{K}$) and the BP glass ($\sim 1.39 \text{ W/m}\cdot\text{K}$) glasses had similar RT thermal conductivities to SiO_2 and still yet the iron-rich BL glass was more conductive than the BP glass. The resemblance of the BL and BP glasses to natural obsidian glass may extend beyond just their appearance. It has been reported that even a small fraction of crystallites in obsidians, only 1 to 2 volume %, can significantly increase the thermal diffusivity which is proportional to the thermal conductivity (Romine *et al.*, 2012; Hofmeister *et al.*, 2014). Such a small volume fraction of crystallites would likely be undetectable by normal XRD and would be unlikely to noticeably impact the other properties. The formation of Al_2SiO_5 crystals, while apparently limited to the surface, was already shown for the BP glass. So, it is feasible that BL and BP glasses could contain a small fraction of crystallites, either from undissolved powder or self-nucleation.

The coefficient of thermal expansion (CTE) and the dilatometric softening temperature T_d for the BL and BP glasses melted at 1300 and 1500°C are shown in Table 6. The BL glass had a lower average CTE of $6.97 \times 10^{-6}/^\circ\text{C}$ compared to the BP glass with an average CTE of $7.98 \times 10^{-6}/^\circ\text{C}$. These values are slightly lower than an ordinary soda lime glass which is approximately $9 \times 10^{-6}/^\circ\text{C}$ (De Jong *et al.*, 2011). The average dilatometric softening temperature T_d of the BL glass was 758°C, and for the BP glass, it was 729°C. These values compared favorably to those measured by fiber extension, as reported in Table 5.

The CTE of a silicate glass is increased with additional amounts of network modifiers, such as Na_2O , as well as certain property modifiers, such as CaO , due to the creation of NBOs. Al_2O_3 additions tend to decrease the CTE, and it appears that Fe_2O_3 may function similarly in lowering the CTE (De Jong *et al.*, 2011). Comparing the compositions of the BL and BP glasses (Tables 2 and 3, respectively), it is seen that they had similar amounts of CaO , but the BL glasses had slightly lower total alkali content and an increased amount of Al_2O_3 and Fe_2O_3 , which likely explained the slightly lower CTE of the BL glasses.

Physical and Mechanical Properties

The measured bulk densities of the BP and BL glasses melted at 1300 to 1500°C are shown in Figure 7. The BL glass bulk density ranged from 2.866 to 2.889 g/cm^3 with an average of 2.88 g/cm^3 , while the BP glass bulk density ranged from 2.721 to 2.764 g/cm^3 with an average of 2.74 g/cm^3 , across all melting temperatures. The BL glass had a greater density than the BP glass for all melting temperatures. The BL glass had a slight upward

Table 6. The coefficient of thermal expansion (CTE) and dilatometric softening temperature T_d of BP and BL glasses melted at 1300 and 1500°C

Glass Melting Temperature	BL Glass		BP Glass	
	CTE ($10^{-6}/^\circ\text{C}$)	Dilatometric Softening Temp. ($^\circ\text{C}$)	CTE ($10^{-6}/^\circ\text{C}$)	Dilatometric Softening Temp. ($^\circ\text{C}$)
1300°C	7.04	743.4	8.51	724.4
1500°C	6.90	772.7	7.44	734.2

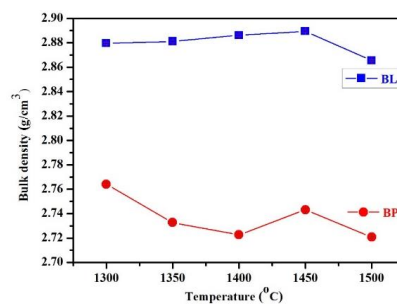


Figure 7. Bulk densities of the BP and BL glasses melted at 1300 to 1500°C

trend of increasing bulk density from a melting temperature of 1300 to 1450°C, but had a noticeable decrease at 1500°C. The BP glass had nearly the opposite trend in bulk density, with a decreasing density from a melting temperature of 1300 to 1400°C, a slight increase at 1450°C, before another decrease in density at 1500°C.

The density of glass is a more complicated topic than crystalline materials, not only due to the influence of composition, which is complicated by itself, but also due to the effects of molar volume and processing temperatures. A glass network former has the shape of a polyhedron in the network, e.g. the SiO_4 tetrahedron in the case of SiO_2 . The tetrahedra are connected at their corners and randomly arranged in 3D space, forming the glass network and, at the same time, creating large interstitial spaces between them. The glass former network also has a particular equilibrium volume as a function of the processing temperature conditions. The addition of network and property-modifying atoms to the network may not disrupt the equilibrium volume as they can occupy the available interstitial space. This would be observed as an increase in density. However, further additions of modifiers could hinder the equilibrium network creation, increasing the volume and resulting in a decrease in density (Shelby, 2005; Varshneya *et al.*, 2019).

The greater BL glass density was likely due to the slightly greater content of iron and calcium. The slight upward trend of the BL glass density was small, increasing from 2.880 g/cm³ at 1300°C to 2.889 g/cm³ at 1450°C, an increase of only 0.009 g/cm³. This minor difference could be due to the small changes in the composition, as seen in Table 2, or possibly the result of the differences in cooling rate experienced by the glasses from the different melting temperatures. A glass that is cooled from a higher melting temperature at the same cooling rate, would take longer to reach the same lower temperature. This would result in a lower equilibrium volume of the glass and thus a higher density, assuming the same mass. The abrupt decrease in density at 1500°C could be explained again by the volatilization of the glass, although the SiO_2 appeared to be the only element that significantly decreased, which is unlikely. Another possible explanation is the reactivity of the glass with the alumina crucible used for melting. That possibility was not explored in the current research.

The downward density trend of the BP glasses as a function of temperature was larger in magnitude, going from 2.764 g/cm³ at 1300°C to 2.743 g/cm³ at 1450°C, a decrease of 0.021 g/cm³. Again, this could be due to changes in the composition due to volatilization, particularly of the

Na_2O , as seen in Table 3. However, the SiO_2 content decreased much more, possibly as a result of a crucible reaction. There was also the possibility of sample-to-sample chemical variability of the melted powder itself. Again, that possibility was not explored in this research, but is a potential concern, as research (Heuss-Aßbichler and Bayuseno, 2021) has shown the presence of large inhomogeneities in municipal solid waste ashes.

The measured Vickers indentation hardness for the BP and BL glasses melted at 1300 to 1500°C are shown in Figure 8. The Vickers hardness curves of the BL and BP glasses exhibited a slight increase from 1300 to 1450°C, followed by a small drop at 1500°C. The Vickers hardness of the BL glasses ranged from 6.17 to 6.41 GPa with an average hardness of 6.29 GPa, while the hardness of the BP glasses ranged from 5.98 to 6.14 GPa, with an average of 6.06 GPa, across all melting temperatures.

The BL glass had a higher Vickers hardness than the BP glass for all melting temperatures. The hardness values for the BL and BP glasses are generally between that of fused silica and soda lime glass, with the reported hardness values for fused silica in the range of 7.0 to 7.5 GPa, and soda lime glass in the range is 5.4 to 6.6 GPa, but typically towards the lower end (De Jong *et al.*, 2011). The baseline hardness of the glass depends on the network former, e.g. SiO_2 . The hardness of a glass is typically decreased by the addition of network modifiers, e.g., Na_2O , and increased by property modifiers, e.g., CaO (Varshneya *et al.*, 2019). The BL glass likely had the greater hardness due to the increased amount of network former and property modifiers and the lesser amount of network modifiers, compared with the BP glass.

The increased hardness with melting temperature of the BL glasses appeared to track with the density increases, as seen in Figure 7. In addition to the contributions of network strength and connectivity, the density of the network itself, i.e.

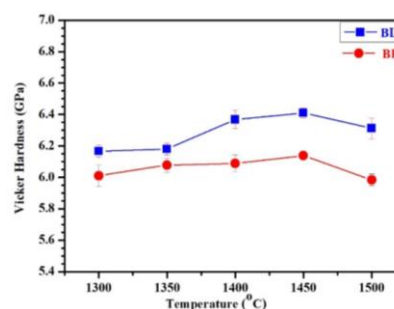


Figure 8. Vickers indentation hardness of the BP and BL glasses melted at 1300 to 1500°C

the number of atomic bonds in a given volume, would also be expected to increase the hardness. For the BP glasses however, the density was observed to decrease with increasing melt temperature. It is perhaps possible that even with a decrease in equilibrium volume provided by slower cooling, the mass, particularly from the modifiers, was decreased more so, such as to still enable a decrease of density and a slight increase in hardness. However, this interpretation would obviously require further research to verify.

Conclusions

Two domestically produced bottom ash powders, a lignite bottom ash from Lampang (BL) and a municipal solid waste incinerator bottom ash from Phuket (BP), were studied for glass-forming ability in the temperature range of 1300 to 1500°C. It was found that both powders could be successfully melted to form glasses, as confirmed by XRD. The BL glasses could be formed within the entire temperature range studied. The BP glasses were only fully amorphous at 1450°C and above, as the crystalline Al_2SiO_5 phase formed in small amounts at the lower melting temperatures. All of the glasses produced were fully opaque and black in color, due to the large amount of iron in both powders. The increase of glass melting temperature tended to improve the various properties, such as conductivity and hardness, until 1450°C, with a further increase to 1500°C degrading the properties slightly. The reason for this is not entirely clear. The BL glasses produced from the lignite bottom ash tended to have slightly better properties than the BP glasses produced from the municipal solid waste. This was likely the result of the BL composition having slightly higher property modifier content and slightly lower network modifier content. The large amount of iron in the glasses appears to function as a network former as well as a colorant. The observed refractory nature and high hardness of both BL and BP glasses suggest that further research is merited. The chemistry and thermal behavior of these glasses are comparable to rockwools and basalt fibers, which find significant use as thermal insulation and fiber reinforcement.

Acknowledgement

The authors thank the Faculty of Engineering, King Mongkut's University of Technology Thonburi, for funding this research. We would also like to thank the Mae Moh power plant operators, Lampang province, Thailand, and the municipal solid waste

incinerator, Phuket City Municipality, Phuket province, for supplying the bottom ash powders used in this research. Finally, we thank the Department of Materials Science, Faculty of Science, Srinakharinwirot University, and the Department of Physics and Materials Science, Faculty of Science, Chiang Mai University, for providing access to their research facilities.

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