

## Comparison Thermal and Hardness of CaO-MgO-Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>-B<sub>2</sub>O<sub>5</sub> Glass from Different Sources of Kaolin Minerals as Main Raw Mineral

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**ABSTRACT.** In this paper, all the raw materials used to synthesis glass specimen has been characterized to obtained elemental composition. In this work, silica, dolomite, colemanite, limestone, and 2 different sources of kaolin were characterize using XRF to obtain elemental composition analysis. The composition of each raw materials were calculated and synthesis glass specimen by XRF result. Different sources of raw materials have different chemical composition. Two sources of kaolin were obtained from Kota Tinggi Johor, Malaysia (LK) and China (KC) as raw minerals and having different chemical composition, thus it is important to modification and recalculation of composition after XRF result obtained in order to meet the standard specification of glass. The element composition in different kaolin minerals contain different impurities or traces thus promote different properties. Synthesized glass specimen was subjected to thermal analysis and hardness testing and found that the glass samples with kaolin china as raw mineral (glass KC) having same trend of DTA results and having higher weight loss compare to glass using local kaolin as raw mineral (glass LK). Furthermore, the hardness of glass KC is higher than glass LK because present of TiO<sub>2</sub>.

**Keywords:** Glass, X-ray fluorescence, Thermal analysis, Vickers hardness;

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### 1. INTRODUCTION

Many industries synthesis glass by using KC as main mineral because of consistence composition as well as dolomite, colemanite, limestone and silica. Ronget al. [1] and Memon et al. [2] investigated KC have consistence of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, MgO, CaO, K<sub>2</sub>O, TiO<sub>2</sub> and P<sub>2</sub>O<sub>5</sub> compound. The consistency of chemical composition in China caused most of the industry in Malaysia imported kaolin from China as raw material for many kind of product such as glass fiber grade, sheet glass, tiles and etc. Ceramics from China dominated the trade of ceramic findings in Malaysia. The use of LK as main mineral would result in reducing transportation cost and increase job opportunities thus increase the profit of glass industries in Malaysia. The main oxide for glass specimen predominantly contain oxides of boron, sodium, calcium, aluminium and silicon. Minerals that generally used in production of glass and ceramic product are kaolin, dolomite, colemanite, limestone and silica. Minerals from different sources contained different in oxide composition due to its impurities [3]. In order to synthesis glass specimen and meet the standard specification of glass, characterization of all minerals were carried out by XRF for composition determination. After composition determination, recalculated and modification of oxide composition of all the minerals to synthesis glass specimen. In this research, we emphasized the analysis of the differences in thermal behaviour and hardness of synthesized glasses. Thermal behaviour and hardness of both glasses were characterized using DTA/TG and Vickers hardness test.

### 2. MATERIALS AND METHODS

The starting raw minerals for glass specimen were silica, dolomite, colemanite, limestone, and 2 different sources of kaolin ((i) KC and (ii) LK). All the raw materials were supplied by Nippon Electric Glass. The glass

was produced based on standard chemical specification of glass. The composition of the raw materials used was recalculated and modified based on the standard specification of the glass specimen from the industries.

Element composition of each minerals were analysis by XRF in order to obtain elemental results. All raw minerals were analysed by X-ray Fluorescence Spectrometers (XRF): Benchtop EDXRF Spectrometer, Qualitaxto obtain elemental composition. The weight percent of minerals powder used in this present work were based on the standard specification of glass. After obtaining the weight of each minerals, all minerals were mixed together.

Wet milling method was used to mix the raw powders. Alumina ball was used to mix the powder homogeneously at 350 rpm for 8 hours and dried at 100°C overnight. The homogenized mixture of raw minerals was melted in alumina crucibles at 1400 °C for 4 hours. The molten glass was quenched in water immediately to form glass LK and glass KC. Glass frit was characterized to compare the thermal behaviour and hardness by using Vickers hardness test. A square base pyramid shaped diamond is used as indicator in this method. Test load used was 49.03N and dwell time was set for 10 s. Sample was place perpendicular to the indicator and test started. The test was done five times to get a correct reading by its average hardness value.

### 3. RESULTS AND DISCUSSION

#### 3.1 Elemental analysis.

Elemental composition of all raw materials which were measured by XRF was tabulated in Table 1. LK contains 47.4% of SiO<sub>2</sub> and 36.8% of Al<sub>2</sub>O<sub>3</sub>. Others oxide that were present in LK is Fe<sub>2</sub>O<sub>3</sub> (0.50%), TiO<sub>2</sub> (0.50%), CaO (0.01%), MgO (0.10%), K<sub>2</sub>O (2.00%) and P<sub>2</sub>O<sub>5</sub> (0%). Amount of LOI of LK is 12.55% due to the evaporation of water content. Chemical composition of KC contain SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> are 43.0% and 36.2%. The main impurities that present in KC such as Fe<sub>2</sub>O<sub>3</sub> (0.59%), TiO<sub>2</sub> (1.02%), CaO (1.90%), MgO (0.54%), K<sub>2</sub>O (0.18%) and P<sub>2</sub>O<sub>5</sub> (0.06%). Others compound as traces that contain % in KC. KC also has 16.13% of LOI. Traces contain in KC is higher than LK. The different impurities contain in KC and LK give different properties to the synthesized glass. The elemental analysis was done to confirm each of the raw materials with its chemical composition for modification and recalculating the weight percent needed to produce glass specimen.

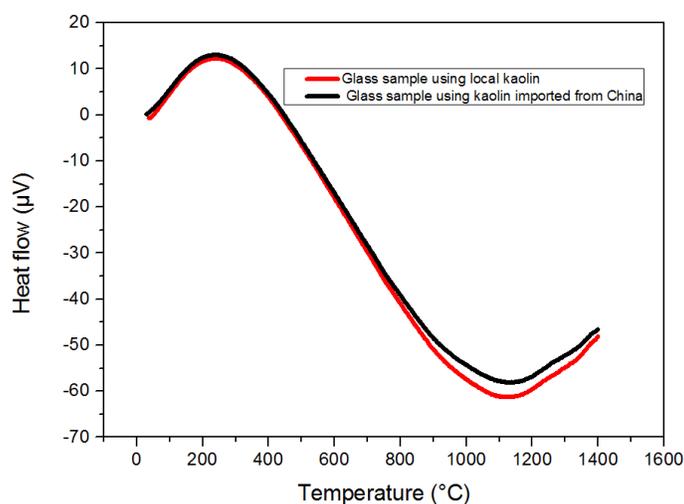
#### 3.2 Thermal Analysis.

Thermogravimetric analysis was used in the thermal analysis of synthesized glass. Fig. 1 demonstrated the DTA curve for glass LK and glass KC. The two curve is the same in trend. There are an exothermic peak for both glass at temperature about 300°C that can explain with the oxidation occur inside the glass samples. When temperature reach 1100°C, there are another peak occur that is endothermic slope. The endothermic peak demonstrated the glass melting at the temperature 1100°C. Although excess of CaO and MgO in KC as fluxes will promote to a lower melting point but in DTA curve of glass KC does not shift the endothermic peak to lower temperature but showed same thermal behaviour.

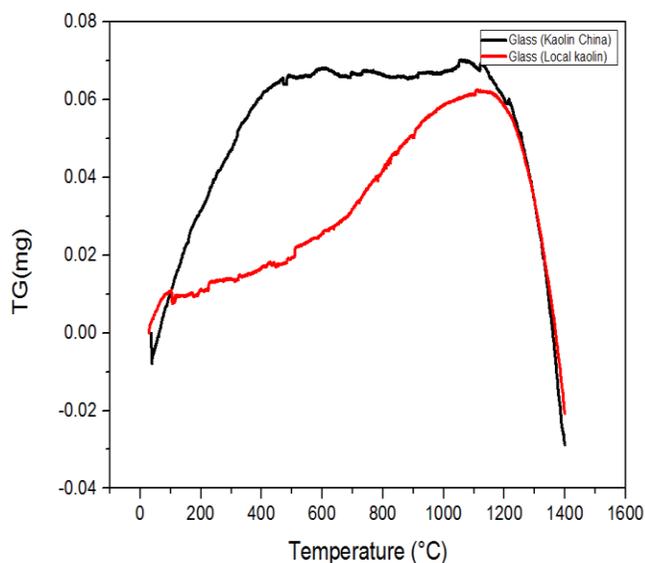
Fig. 2 shows the TGA curves of glass samples after synthesized using different raw minerals which are LK and KC. The weight gain of glass sample using KC is known as glass KC is about 0.07mg from total weight of 10.39mg, less than 0.1wt.% at room temperature until about 400°C. Attila investigated that is likely due to the oxidation reaction occur in molten glass [4]. There is a weight loss of about 0.03mg from its original weight starting at temperature about 1200°C. A similar behaviour is seen in the TGA curve of glass sample using LK (glass LK). The weight gain is increase moderately until 0.06mg from room temperature and stop at about 1200°C and undergo a weight loss of 0.02mg from its original weight of about 0.02wt.% until 1400°C. The total weight loss for both are match to the result of TGA individual for KC and LK.

**Table 1** Chemical compositions of KC and LK (wt.%)

Compound	KC	LK
SiO <sub>2</sub>	43.0	47.4
Al <sub>2</sub> O <sub>3</sub>	36.2	36.8
Fe <sub>2</sub> O <sub>3</sub>	0.59	0.50
MgO	0.54	0.10
CaO	1.90	0.01
K <sub>2</sub> O	0.18	2.00
TiO <sub>2</sub>	1.02	0.51
P <sub>2</sub> O <sub>5</sub>	0.06	0.00
Traces	0.38	0.13
LOI	16.13	12.55



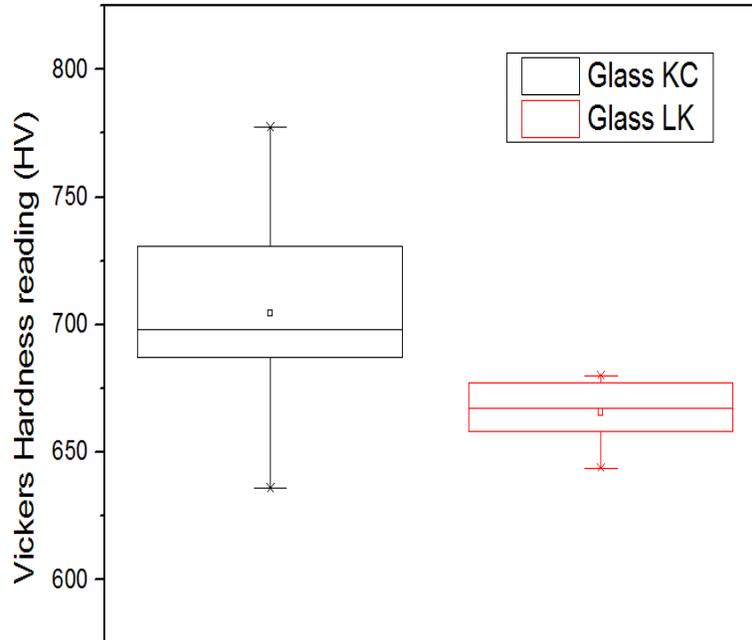
**Fig. 1** DTA curve for glass LK and glass KC



**Fig. 2** TGA results for glass samples synthesized by different raw minerals, glass LK and glass KC.

### 3.3 Vickers Hardness Analysis.

Vickers hardness analysis has been done on two types of glass fiber grade specimen using 2 different kaolin sources. The applied load used was 49.03N. The hardness value obtained were shown in Fig. 3 boxplot and tabulated in Table 4.8. Fig. 3 shows that the hardness of the glass LK and glass KC. The hardness of glass KC is higher than glass LK. The average hardness of glass KC is 704.6HV and the glass LK is 665.6HV.



**Fig. 3** Vickers hardness results for glass KC and glass LK

The higher value of hardness is due to the excess impurities content in the raw materials. XRF result demonstrated in the kaolin china content of excess  $\text{TiO}_2$ ,  $\text{NiO}_2$ ,  $\text{ZrO}_2$ , and  $\text{CuO}$  that promote excess hardness to the fiber glass grade specimen. Beside that the alkalis such as  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$  and  $\text{Li}_2\text{O}$  in glass composition will decrease the hardness of glass [5]. Result from XRF can proved that the traces in the glass LK content excess alkalis such as potassium oxide  $\text{K}_2\text{O}$  that showed 0.78wt.% compare to glass KC that contained only 0.09wt.%. The higher percentage of  $\text{K}_2\text{O}$  in glass give a lower hardness and it is matched with the result of Vickers hardness test.

**Table 2** Vickers hardness of two different types of fiber glass

Vickers hardness reading (HV)	Glass KC	Glass LK
Reading 1	635.9	677.3
Reading 2	691.4	668.9
Reading 3	687.3	643.9
Reading 4	730.7	658.1
Reading 5	777.5	680.2
Average	704.6	665.6

## 4. SUMMARY

From the comparison of glass KC and glass LK, it can be conclude that the glass KC have the same trend on thermal behaviour and only small weight loss of 0.02mg of glass KC compare to glass LK. For the present

of TiO<sub>2</sub> compound in KC, the glass KC having higher hardness compare to glass LK. Thus, this present study substitution of LK to synthesis glass is in same thermal properties but lower in hardness.

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