The Microstructural Study of Cullet-Clay Ceramics

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\section*{Abstract}

A series of ceramic based on (x) cullet-(100-x) clay has successfully been made by slip casting followed by sintering at 900°C with \(40 \leq x \leq 80\) wt\%. The content of the ceramic has been measured by EDX (Energy Dispersive X-ray) analysis while the phase occurrence has been determined by XRD (X-ray Diffraction) technique. It is found that the ceramic contains largely of Si, Na and Al while the phase is dominated by the occurrence of Al\(_2\)SiO\(_5\) (Kyanite) and SiO\(_2\) as a minor phase. The microstructural morphology can be described by the well distributed of kyanite agglomerate with SiO\(_2\) dispersed in the matrix. The area density of their agglomeration is very much dependence on the cullet content, where the higher the cullet, the lower the area density.

\section*{1. Introduction}

The issues regarding to the disposal of glass waste or cullet has gained a lot of attention especially from the environmental point of view. It has been known that the use of cullet in the manufacturing process of new glass will cause less pollution effect as well as reducing the manufacturing cost of about 2.5-3.0\% [1]. Besides, it can also help to reduce the release of green house gases such as CO\(_2\), SO\(_2\) and NO\(_x\) to the minimal level [2]. Thus the search for new ceramics that incorporating cullet with other material seems to be unavoidable.

Red clay brick which contains large amount of iron oxides are very stiff and rigid [3]. Normally, the fabrication of this material would take the temperature up to 900-1150°C [4]. The major crystalline
phases of these materials are calcite, clinichlore, muscovite and quartz [5]. These bricks exhibits good physical and mechanical properties thus are normally use for construction [6-7].

However, there is not many attempt has been reported to compose new ceramic based on the mixture of cullet and clay. In our earlier study [8-9], it has been shown that these new ceramics can be formed with higher density, higher hardness, lower drying shrinkage, less water absorption and other good physical qualities [7, 10]. It is therefore the aim of this paper to report the latest development on the cullet-clay brick from recycle glass. The study will be focused on the microstructural point of view and the result will be discussed with respect to the cullet content.

2. Materials and Methods

Cullet is firstly crushed into small pieces before being grounded into a relatively fine powder by using ball milling for 4 hours. Cullet and clay are then each sieved at a size of 100μm. A proportional amount of cullet and clay are mixed together according to Table 1. The mixture is then undergoes the milling process for 15 minutes to ensure the homogeneity. 40 wt% of water is then added into the mixture to form slurry before being soaked for 1 day. After that the mixture is slip casted into a mould for 2 days at room temperature before being sintered at 900ºC for 1 hour where a solid and hard brick will be obtained.

Table 1. The nominal and actual composition of cullet-clay brick

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Nominal Composition (Wt %)</th>
<th>Actual Composition (Mol%)</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Cullet</td>
<td>Clay</td>
<td>SiO₂</td>
</tr>
<tr>
<td>S1</td>
<td>40</td>
<td>60</td>
<td>89</td>
</tr>
<tr>
<td>S2</td>
<td>50</td>
<td>50</td>
<td>94</td>
</tr>
<tr>
<td>S3</td>
<td>60</td>
<td>40</td>
<td>95</td>
</tr>
<tr>
<td>S4</td>
<td>70</td>
<td>30</td>
<td>95</td>
</tr>
<tr>
<td>S5</td>
<td>80</td>
<td>20</td>
<td>97</td>
</tr>
</tbody>
</table>

Energy Dispersive of X-ray (EDX) analysis is used to determine the actual content of the ceramic while the phase occurrence and crystal phases are investigated using X-ray Diffraction (XRD) technique. The microstructural morphology of cullet-clay brick samples is obtained by observing their flat surface under Scanning Electron Microscope (SEM).

3. Results and Discussions

From Table 1, it can be seen that as the cullet content is increased the color of brick obtained is changing from dark brown to whitish. The changing in color of the brick might indicate that there are lots of transition elements in the mixture [9]. However, it should be noted out that the individual percentage of these elements are relatively very small. The actual compositions of the ceramics as evaluated by EDX are shown in Table 1. From Table 1 it can be seen that the brick is dominated by the existence of SiO₂ embedded in the matrix. It is believed that SiO₂ might play a major role in most of the properties reported earlier [11]. From Table 1, it can also be observed that as the cullet level is increased, the SiO₂ content increases. This is expected since the major content of cullet is SiO₂. The presence of Al₂O₃ in this
ceramics is very important since it would weaken the interatomic bonding of Si-O [12] thus producing ceramics with required properties.

Figure 1 shows a typical result of X-ray diffraction for some samples at different cullet level after being sintered at 900 °C. From Figure 1, there are three phases may be seen. At low cullet level of about 40 -50 wt%, the major phase occurrence is dominated by the existence of quartz (α-SiO₂). However, as the cullet level is increased up to more than 50wt%, the phase of Tridymite (β-SiO₂) starts to be seen. But, as the amount of cullet is further increased to 70 wt%, the phase of aluminum oxide, α-SiO₂ and β-SiO₂ can simultaneously be seen. This result indicates that the brick contain more SiO₂ than any other phases. It can also be observed that the phase of α-SiO₂ exists in all composition. Thus, it can be said that α-SiO₂ could be the major phase that make up the structure of this ceramic.

Figure 2 shows the microstructural morphology of the cullet-clay brick at different cullet level after being sintered at 900°C as observed under Scanning Electron Microscope. From Figure 2, it can be seen that the morphological surface of the ceramic with lower cullet level exhibits granulated surface texture. This is perhaps due to the incomplete phase transformation of cullet-clay within the ceramic matrix. However as the cullet level is further increased up to more than 70 wt%, the crystallization process has almost been completed and all phases are completely embedded in the ceramic matrix. Further on examining the surface morphology, a clear phase boundary may be seen to lie in the vitrified glass matrix. This is why a smoother morphological surface may be obtained.
Fig. 2. The microstructural morphology of cullet-clay ceramic at different cullet level after being sintered at 900°C.

4. Conclusion

Based on the above discussions, some conclusion may be summarized as follows. It is found that the cullet-clay brick contain of about 89-97% of SiO$_2$ and 11-3% of Al$_2$O$_3$. The analysis shows that the major phases of the ceramic are identified as quartz, tridymite and alumina. It is also found that the microstructural morphology of the ceramic is very much depending on the cullet level. For low cullet level, the granulated morphology is obtained while for the higher cullet level the phase boundary may be seen, apparently due to the complete cullet-clay crystallization process.

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