PRODUCTION OF LI_2O-AL_2O_3-SIO_2 GLASS CERAMICS USING COAL BOTTOM ASH AS RAW MATERIAL

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1. INTRODUCTION

The development of glass-ceramics has enabled producing materials with differentiated microstructure: refined grain size, random orientation, differentiated amounts of residual amorphous phase and reduced porosity. Glass-ceramics (GC) are obtained starting from the melting and crystallization of glasses[1]. The glass-ceramics production process allows producing materials with superior properties compared with those made by conventional processes. Briefly, glass ceramic materials could be described as crystalline materials generated from a glass. The selection of the glass depends on the possibilities of combining its oxide components and on thermodynamic and kinetic aspects[2].

Another important characteristic of glass-ceramics production is the possibility of using by-products that are difficult to incorporate in conventional processes, such as industrial wastes: steelwork slag, fly and bottom ashes from mineral coal. The use of industrial by-products can help to solve environmental problems and reduce process costs. An important step toward the development of glass-ceramics starting from bottom ashes is the use of theories on crystallization phenomena, which allow adding value to the materials that are developed. Ashes have a market value four times less than commercial minerals, such as sand or mullite, and are found in granulated form[3]. This ash presents a great variation in its physicochemical properties.

Glass-ceramic materials based on the SiO_2 - Al_2O_3 - Li_2O system are characterized as materials that have a low thermal expansion coefficient[4]. The type of crystalline phase depends firstly on the initial composition, thermal treatment and the nucleating agents used. The most efficient nucleating agent of this system has been TiO_2 or rich phases of TiO_2[5]. Its utilization is very widespread, as a result of these properties. Some of the uses of these materials, can be cited: microwave windows, with low dielectric loss; electrical stove plaques, domestic and cooking utensils, with low thermal expansion coefficient and thermal shock resistance, and heat exchangers[6]. The aim of this work is to obtain glass-ceramic materials, with specific characteristics in the SiO_2 - Al_2O_3 - Li_2O system, from coal mineral bottom ashes.

This work proposes obtaining and characterizing vitreous and glass-ceramics materials starting from bottom ashes of coal mineral. The system SiO_2-Al_2O_3-Li_2O was used to study the development of the aluminosilicate lithium and virgilite phases.

2. EXPERIMENTAL PROCEDURE

Differential thermal analyses (DTA) were carried out on monolithic glass samples in order to determine glass transition temperature (Tg) and crystallization temperature (Tc). Tg and Tc were used to define the heat treatment temperatures. The resulting glass-ceramics were characterized by X-ray fluorescence (XRF), differential thermal analysis (DTA), dilatometric analysis (DIL), X-ray diffraction (XRD) and scanning electron microscopy (SEM).

3. RESULTS AND DISCUSSION

The chemical analysis of the bottom ash presents a composition rich in SiO_2 and Al_2O_3, favourable for making vitreous and glass-ceramic materials. The mineralogical analysis of bottom ash indicates that the major crystalline phases are Quartz (SiO_2) and Mullite ($3Al_2O_3.2SiO_2$). This by-product also presents the phases Magnetite (Fe_3O_4) and Hematite (Fe_2O_3).

The vitreous mass showed adequate viscosity during melt flowing, producing a dark glass sample. The glass transition temperature and crystallization temperature were 642 °C and 866 °C, respectively.

Table 1 summarizes the values of the linear thermal dilatation coefficients of the glass and glass-ceramic material, measured in the interval of 25 to 325 °C (α_{25-325}), among the vitreous transition temperatures (Tg) and softening temperatures (Tw).

	Glass	Glass-Ceramics
$\alpha_{(25-325)}$ (10^{-7} °C $^{-1}$)	30,1	-23.4
Tw (°C)	686.0	978
Tg (°C)	642.0	-

Table 1 - Values of the $\alpha_{(25-325)}$ Tg and Tw of the glass and glass-ceramic.

The XRD technique confirmed the vitreous state of the glass sample obtained by quenching and provided the X-ray pattern of the glass sample after heat treatment. The crystalline phases developed were identified as Lithium Aluminosilicate (Lix Al_x Si_{1-x} O_2 - JCPDS 15-527) and Virgilite (Lix Alx Si_{3-x} O_6 - JCPDS 21-503), as expected by the SiO_2-Al_2O_3-Li_2O phase diagram.

The SEM micrograph shows the tendency to bulk growth of the material, while crystallization starting from the surface could not be verified. The well-defined grains are randomly distributed in the residual vitreous matrix, with a refined microstructure formed by grains about 1.70 μm in diameter. The microstructure obtained in this study has a grain size lower than in other works[5]. The tendency to produce spherical forms of grains is in agreement with the literature[5]. In Fig 3b, the microstructure of the glass sample treated for 10 minutes at Tc shows high density of grains. The high crystallinity of the samples helps improve the mechanical and chemical properties; therefore grain random disposition influence tenacity and crack propagation[1].

4. CONCLUSIONS

The raw material, bottom coal ash, constitutes an attractive source of SiO_2 and Al_2O_3 for producing vitreous materials. Even though it has a considerable iron oxide content, it can be processed, as it is possible to convert this percentage into an acceptable one. The utilization of adequate additives with bottom coal ash decreases the viscosity of the molten material and leads to the obtainment of the desired glass-ceramic phases.

5. REFERENCES

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