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Nucleation and crystallization of Ca doped basaltic glass for the production of a glass-ceramic material

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Sewage sludge from wastewater treatment plants is a waste with a composition roughly similar to that of a basalt. It may contain potentially toxic elements that can be inertized by vitrification. Using a glass-ceramic process, these elements will be emplaced in newly formed mineral phases. Glass-ceramic production requires an accurate knowledge of the temperatures of nucleation (T_N) and crystal growth of the corresponding minerals. This work arises from the study of the addition of ions to a basaltic matrix in order to establish a model of vitrification of sewage sludge.

In this case a glass-ceramic is obtained from a glass made with a basalt that has been doped with 16% CaO. Two glasses which underwent different cooling processes have been produced and compared. The first was annealed at 650^{o}C (AG) and the second was quenched (QG). The chemical composition of the glasses is SiO_2 36.11 wt%, $Al_2O_3$12.19 wt%, CaO 24.44 wt%, FeO 10.06 wt%, MgO 9.19 wt%, Na_2O 2.28 wt%, TiO_2 2.02 wt%, K_2O 1.12 wt%, P_2O_5 0.46 wt%. Glass transition temperature obtained by dilatometry varies from 640 ^{o}C (AG) to 700 ^{o}C (QG). The temperatures of nucleation and crystal growth of the glass have been determined by Differential Thermal Analysis (DTA). The phases formed after these treatments were identified by X-Ray Diffraction. The temperatures of exothermic and endothermic peaks measured in the quenched glass are, in average, 10 ^{o}C higher than those found for the annealed glass. The exothermic peaks provide crystallization temperatures for different phases: a first event at 857 ^{o}C corresponds to the growth of magnetite, pyroxene and nepheline, whereas a second event at 1030 ^{o}C is due to the crystallization of melilite from the reaction between previous minerals and a remaining amorphous phase. The complete melting of this system occurs at 1201 ^{o}C.

This glass has been nucleated inside the DTA furnace (500-850°C/3 hours) and then heated up to 1300 ^{o}C using the fraction between 400-500μm. T_N can be obtained by analysing the exothermic peak temperature (Tex) which is related to the number of nuclei per unit volume that is formed on the glass. If we represent (1000/Tex) versus T_N, we obtain the range of nucleation and the temperature of maximum nucleation rate of glass. Three different nucleation events were observed for both glasses. The first event, assigned to the nucleation of magnetite starts between 550 (AG) and 600 ^{o}C (QG); the second event is linked to the start of nucleation of pyroxene at 655 ^{o}C (AG and QG); the third event is the nucleation of nepheline between 700 (QG) and 730 ^{o}C (AG). These thermal events are more evident in QG, hence it has been chosen to make the glass-ceramic. Its production would divided in 4 steps at a heating rate of 15 ^{o}C/min: is starts by heating the original glass up to 550 ^{o}C; further heating up to 655 ^{o}C; then up to 700 ^{o}C and a final heating up to 1050 ^{o}C. A 4h isothermal step was performed after each heating.