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An investigation into the hydration and microstructure of cement pastes modified with glass powders

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HIGHLIGHTS

- Fine glass powders were shown to improve the hydration of cement pastes.
- Electrical resistivity was significantly increased in cement pastes containing fine glass powders.
- Fine glass powders can be used as a pozzolan in cementitious materials.

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ABSTRACT

This paper examines the hydration and microstructure evaluation of cement pastes modified with two types of glass powders with a fine size distribution and a class F fly ash. Hydration was investigated by measuring the heat of hydration, chemical shrinkage and setting time at early age and non-evaporable water content at late ages. Pozzolanic behavior was studied using thermogravimetric analysis and X-ray diffraction. The microstructure was evaluated by the electrical resistivity measurement and image analysis. Based on the findings of this study for the glass powders and fly ash used, glass powders were shown to enhance the early hydration of cement and fly ash demonstrated a small delaying effect. Glass powders were found to possess pozzolanic behavior and to improve the hydration of cement paste at later ages. Microstructure evaluation indicated pore refinement in the microstructure of cement pastes modified with glass powders. It was concluded that glass powders with a microscale size distribution can be used as a pozzolan in cement-based materials.

1. Introduction

There is an increasing interest in the use of recycled materials in construction industries with the goal of promoting sustainable and green construction [1–6]. This has been motivated by an increase in the cost of local materials as well as the environmental benefits of utilizing recycled materials [1,3,4,7,8]. Use of waste glass in the concrete industry has received increased attention in recent years [9–11]. Waste glass as a replacement of aggregate in concrete was investigated and observed to increase the potential of the alkali silica reaction (ASR) and, as a result, degrade the mechanical and durability properties of concrete [12–14]. Several studies have shown that glass powders with a microscale size distribution resulted in improvements in the strength and transport properties of concrete without showing undesirable ASR cracking [9–11,15–18]. Enhancement in concrete performance was attributed to the pozzolanic behavior of glass powders improving the microstructure of concrete [11,15,19–21].

The influence of glass powders on the microstructure of cementitious materials has been investigated in the literature [15,19,21]. Shayan and Xu [15] and Nassar and Soroushian [9] showed densification in the microstructure of cementitious materials modified with glass powders using microscopic examinations. There have been a few studies regarding the effect of glass powders on the hydration characteristics of cementitious materials [19,20,22–25]. Schwarz et al. [25] and Schwarz and Neithalath [19] investigated the hydration of cement pastes modified with glass powders. Their results indicated that glass powders facilitated enhancement in the hydration of cement paste. In another study, Neithalath et al. [20] examined the hydration behavior of cement paste modified with vitreous calcium aluminosilicate, a post-industrial by-product of recycled fiber glass. It was concluded that vitreous calcium aluminosilicate improved the hydration of cement pastes until 7 days and exhibited pozzolanic reactivity after this time.

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In this study, the effect of two types of glass powders produced from two different recycling processes on the hydration and microstructure of cement paste has been investigated and compared to those of fly ash. The glass powders studied in this paper had the same size distribution with a median size less than 10 μm, but differed in chemical composition and, therefore, permitted investigating the dependence of cementitious system behavior on the chemical composition of glass powders. The early hydration was evaluated using setting time, heat of hydration, and chemical shrinkage measurements. The non-evaporable water content measurement, thermogravimetric analysis (TGA), and X-ray diffraction (XRD) were conducted to evaluate the hydration and pozzolanic reactivity of the modified cement pastes at later ages. Electrical resistivity measurement combined with image analysis was utilized to assess the microstructure of the modified cement pastes.

2. Materials and specimen preparation

In this study, specimens were prepared using Type I/II Portland cement, two types of glass powders (GP1 and GP2) and a class F fly ash (FA). GP1 is vitreous calcium aluminosilicate produced from waste glass fibers and GP2 is a post-consumer by-product of recycled glass. Both GP1 and GP2 were obtained from Vitro Minerals. The physical and chemical properties of glass powders and fly ash are given in Table 1. The scanning electron microscopic images and XRD spectra of FA and glass powders were given in one of our previous publications [26]. The median particle size of GP1 and GP2 was 8.4 μm and that of FA was 13.1 μm. The spectra of glass powders did not show any crystalline phases indicating their amorphous structure, which is one of the factors increasing the potential of these materials for the pozzolanic reactivity [27,28]. It should be noted that there is a large variability in the chemical and mineralogical properties of fly ashes and this should be considered in the comparison between the effect of fly ash and other supplementary cementitious materials. Cement paste cubes of 50 mm dimensions and with 5%, 10%, 15%, and 20% replacements of cement with FA, GP1, and GP2 at 0.5 water to binder (cement plus supplementary cementitious material, SCM) ratio were prepared according to ASTM C109. The cubes were cast in two layers; each layer was tamped with 32 strokes of a 13 mm by 25 mm tamper. Immediately after casting, the cubes were placed in a moist room with more than 95% relative humidity and at a temperature of 23 ± 2 °C for 24 h. Cubes were subsequently demolded and stored in a saturated lime solution until testing.

3. Experimental methods

The effect of glass powders GP1 and GP2 and fly ash FA on the hydration and microstructure of cement pastes were investigated in this study. The hydration characteristics were studied by evaluating the setting time, chemical shrinkage, heat of hydration, degree of hydration, and calcium hydroxide (CH) content in the modified cement pastes. The microstructure of cement pastes was investigated via scanning electron microscopic examination and electrical resistivity measurement to elucidate pore structure in the modified cement pastes.

Table 1
Chemical and physical properties of cement, glass powders and fly ash.

Composition (% by mass)	Cement	FA	GP1	GP2
Silica (SiO_2)	20.6	54	57.5	63.3
Alumina (Al_2O_3)	4.8	28	12.7	6.4
Iron oxide (Fe_2O_3)	3.5	7	0.06	0.31
Calcium oxide (CaO)	64	1.4	22.7	17.1
Magnesium oxide (MgO)	0.9	1	3.6	4.5
Sodium oxide (Na_2O)	0.1	0.3	0.62	6.1
Potassium oxide (K_2O)	0.3	2.4	0.06	0.07
Sulfur trioxide (SO_3)	3.4	0.1	0.22	0.19
Titanium dioxide (TiO_2)	0.3		0.98	0.44
Boron trioxide (B_2O_3)			0–6	0–5
Loss on ignition (%)	2.75	3.4	0.5	1
Specific gravity		2.31	2.6	2.5
Passing sieve #325 (%)		81	98	>99
Median particle size (μm)		13.1	8.4	8.4

3.1. Hydration

3.1.1. Heat of hydration

The hydration temperature of the control cement paste and cement pastes with 20% FA, 20% GP1, and 20% GP2 were measured using a four cell Grace AdiaCal semi-adiabatic calorimeter. Cement pastes in an amount of 300 g were mixed according to ASTM C305 at a water to binder ratio of 0.4 and placed in the calorimeter within 5 min after the initial contact of binder and water. Temperature was logged every one minute for about 24 h.

3.1.2. Chemical shrinkage

Chemical shrinkage is a measure of the early hydration of cement pastes. The control paste and the pastes with 20% FA, 20% GP1, and 20% GP2 were prepared at a water to binder ratio of 0.4 according to ASTM C1608. After mixing for 5 min, the pastes were placed into small glass vials to fill about 5–10 mm of the vial. The vials were filled with de-aerated water and sealed with rubber stoppers with capillary tubes passing through. The water level in the capillary tubes was adjusted by adding de-aerated water. A drop of oil was added on the water surface in the tube to prevent evaporation. The change in water level was measured every hour for eight hours and also after 24 h. Chemical shrinkage at 24 h was calculated using the following equation:

$$CS = \frac{[h_{24} - h_1]}{m_c M_{binder}} \quad (1)$$

where CS is the chemical shrinkage at 24 h (mL/g cement), h_1 and h_{24} are the water level in the capillary tube at one and 24 h, respectively, m_c is the mass fraction of cement, and M_{binder} is the mass of binder in the vial (g). Two samples of each mix were prepared and the average chemical shrinkage was reported.

3.1.3. Setting time

Setting time is indicative of cement paste solidification in the early age [29]. In this study, the setting time of cement pastes was evaluated using a Vicat setup per ASTM C191. The control cement paste and the modified cement pastes with 20% FA, 20% GP1, and 20% GP2 were mixed at a water to binder ratio of 0.35. A 1 mm diameter needle attached to a 300 ± 0.5 g rod was allowed to penetrate into the pastes every 15 min starting 30 min after molding, and penetration depth was measured. The time between initial contact of cement and water, and the penetration depth of 25 mm of the needle calculated by interpolation, was referred to as "Vicat initial time of setting". The time from the initial contact of cement and water until the penetration of the needle did not leave any indent on the cement paste surface was measured as the "Vicat final time of setting".

3.1.4. Non-evaporable water content

The non-evaporable water content of cement pastes with 20% FA, 20% GP1, and 20% GP2 were measured at 28 days and 91 days of curing. This method is commonly used to evaluate the degree of hydration of cementitious materials [20,25,30,31]. A small piece of cement pastes from the center of the cube was ground and passed through the sieve #60 to achieve about 6 g of cement paste powder. The powder was dried at 105 °C for about 24 h and then ignited for three hours at 1050 °C. The non-evaporable water content was calculated using the following equation:

$$W_n = \frac{m_{105} - m_{1050}}{m_{1050}} - (m_c \cdot LOI_c + m_{GP,FA} \cdot LOI_{GP,FA}) \quad (2)$$

where W_n is the non-evaporable water content of the paste (g/g binder), m_{105} is the mass of cement paste powder after drying at 105 °C (g), m_{1050} is the mass of powder after ignition at 105 °C (g), m_c is the mass fraction of the cement in binder, $m_{GP,FA}$ is the mass fraction of