

Aging of glass powder surface



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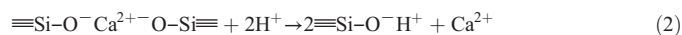
ABSTRACT

Glass powder (GP) obtained by grinding waste bottle glass has been recently introduced in construction materials as a supplementary cementing material (SCM). The behavior of SCMs strongly depends on surface chemistry, which governs their reactivity. The freshly ground glass (FGP) was analyzed by X-ray photoelectron spectroscopy (XPS) and changes in the chemical composition of its surface were monitored over time up to one year. The carbonation level that could affect the long-term reactivity of GP was also assessed. The results indicate a higher sodium content of the surface of the early milled glass. Surficial sodium and calcium contents tend to decrease over time with a negligible level of carbonation after one year. Furthermore, in order to assess the reactivity of GP in cementitious media over time, the fresh and hardened properties of mortars made with 20% FGP and two-year-old GP (OGP) as partial replacement of cement were characterized. The FGP mortar had reactivity slightly higher than that of the OGP with early setting due to surficial alkalis. Both GPs, however, had similar pozzolanic properties, particularly with a high water-to-binder (w/b), suggesting that age does not affect GP reactivity, at least in a cementitious medium.

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1. Introduction

Glass powder obtained by milling mixed waste soda–lime–silica glass has been shown to possess pozzolanic properties, giving it potential for use as a supplementary cementing material (SCM) [1,2]. A material's reactivity, especially in a cement system, depends significantly on its fineness and surface composition, since reactions occur on or begin from the surface of particles. The surface reaction of glass requires particular attention because of its high alkali (13% Na₂O) and alkaline-earth (11% CaO) contents, since these ions are highly mobile and readily react with their environment, particularly in the presence of water. It has been assumed that, even in the absence of water, a film of water could form on the surface of glass as the result of fog or condensation of air moisture, absorbing gases such as CO₂, SO₂, and NO_x from the ambient atmosphere [3,4]. This induces a decrease in pH promoting ion exchange according to Eqs. (1) and (2).



Such a phenomenon leads to the formation of a so-called leached or gel layer on the glass, which is depleted in alkali and alkaline-earth elements and enriched in hydrogen-bearing species [5]. Enhancing the temperature or the evaporation of the water film (or when the solubility products are exceeded) allows the formation of crystalline products such as sulfates, carbonates, hydrocarbonates, chlorides, and nitrates [6].

Since reducing the particle size of glass yields greater surface area (i.e., larger reaction surface), glass powder might be exposed to more carbonation or other reactions, thereby lowering its long-term reactivity after the milling process. This could compromise its use as SCM when significant time has elapsed since milling. Therefore, research is needed on the change in the chemical composition of the surface of glass powder over time. X-ray photoelectron spectroscopy (XPS) is one of the most widely used surface-analysis techniques, providing valuable information about the relative chemical-element concentration of the outermost atomic layers close to the surface (approximately several 10 Å) of the analyzed sample [7]. This technique has proved useful in determining the structure of oxide glasses, since it provides information about bridging (BO) and non-bridging (NBO) oxygen atoms [8,9]. XPS can also serve in investigating the chemical state of all other elements, excluding hydrogen. Many studies used XPS to investigate alkali [10–13] and alkali–lime–silicate glasses [14,15] as well as other oxide glasses [13,14,16,17]. Using high-resolution XPS and NMR instruments on sodium–silicate glasses, Nesbitt and Dalby [13] obtained similar BO/NBO values that

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were consistent with other experimental studies [18,19] depending on glass composition. XPS has also been used to study the effect of electron-beam irradiation [11,20,21]. Irradiation with X-rays seems to cause the migration of mobile ions such as alkali ions [22,23] depending on the irradiation dose and duration of exposure [23].

XPS can also be used to determine the level of carbonation in a material. Black et al. [24] monitored the carbonation of calcium silicate hydrate (C-S-H) gel over time. El-Turki et al. [25] characterized the carbonation level of hydraulic lime in the presence of moisture. These authors assumed that XPS was ideally suited to investigate surface phenomena such as carbonation.

Even though XPS has been widely used to characterize the surface composition of alkali-lime-silicate glasses, such studies have generally focused on the depth profile analysis [15], the differentiation of BO and NBO [8,9], and the effect of irradiation [20,21].

In this study, high-resolution XPS was used to monitor changes in the chemical composition of the surface of glass powder over time. The objective was to determine if the reaction between the powder surface and the environment could drastically affect the reactivity of the glass powder long after the milling process.

To assess the effect of aging on the fresh and hardened properties of mortar incorporating 20% of glass powder as a cement replacement, two glass powders of different ages were used: a freshly ground glass (FGP) and two-year-old glass powder (OGP). The fresh properties considered were flow, initial and final setting time, and heat flow. The hardened properties were characterized by mortar compressive strength after 1, 7, 28, and 91 days of curing.

2. Experiments

2.1. Materials

Three materials consisting essentially of ordinary Portland cement (OPC) and glass powder of two different ages: freshly ground glass and 2-year-old glass powder were considered in this study. The glass powder was obtained by milling mixed recycled bottle glass from a sorting center. It is important to note that, for XPS analysis, the chemical composition of the surface was assessed from $t = 0$ up to one year. The cement-based materials were, however, made with freshly ground glass or with glass powder two years after grinding (OGP).

2.2. Mortar preparation

Three mixtures of mortars with w/b of 0.485 and 0.350 were prepared according to ASTM C305. The different mixtures were the reference (100% OPC), and 20% FGP or 20% OGP as a cement replacement. A polynaphthalene-based superplasticizer was used to enhance the fluidity of the mortars made with a w/b of 0.35. Samples were cured at room temperature and 100% relative humidity in a saturated lime solution until their compressive strength was measured at 1, 7, 28, and 91 days.

2.3. Methods

2.3.1. Granulometry test

The particle size distribution of materials was measured using Mastersizer 2000 laser diffraction granulometer associated with a dispersion unit which was equipped with a centrifugal pump, a stirrer and an ultrasonic probe. This device measures particle size distributions in the 0.02 to 2000 μm range. It consists in measuring the angular variation in intensity of light scattered as a laser beam passes through a dispersed particulate sample. The angular scattering intensity data is then analyzed to calculate the size of the particles responsible for creating the scattering pattern, using the Mie theory of light scattering. About 6 to 10 mg of the sample was introduced in the dispersion unit filled with an appropriate solvent (alcohol) and dispersed under a continuous

stirring by the ultrasonic probe during measurement. The particle size is reported as a volume equivalent sphere diameter and the cumulative volume was deducted.

2.3.2. Density and Blaine fineness

The density of materials was determined by means of a helium pycnometer (multipycnometer). Samples were first weighed to 0.0001 g on an electronic balance that was calibrated prior to use. The pycnometer measures the volume of gas displaced by the sample by expanding a known reference volume of helium gas at a known initial pressure into a sample cell of known volume that contains the unknown sample. Assuming an ideal gas equation of state, the change in pressure upon expansion of the gas into the sample cell in relation with the volume displaced by the sample was calculated. Five sequential determinations of the volume were made for each sample. Densities were calculated using the determined volumes and the measured mass. The resulting density of the material was averaged nearest 0.01 and the standard deviation of the measurements (or errors) calculated.

The fineness of materials was determined using the Blaine air-permeability apparatus in accordance with ASTM C204. This device allows an indirect measurement of the total surface area per unit of mass (m^2/kg or cm^2/g) of the material. It consists essentially of a means of drawing a definite quantity of air through a prepared bed test sample of definite porosity and density. Two measurements were performed and the average fineness calculated within 1 m^2/kg .

2.3.3. X-ray fluorescence analysis (XRF)

The chemical composition of materials was determined by means of Axios-Advanced X-ray fluorescence spectrometer. The principle consists in radiating the material by an X-ray beam inducing its excitation. The excited sample in turn emits X-rays along a spectrum of wavelengths characteristic of the types of atoms present in the sample. The technique allows the analysis of bulk chemical composition of both major and trace elements of the analyzed material.

Samples were prepared by mixing about 6.3 g of the material with 0.7 g of a binder (Licowax). The mixture was homogenized by grinding during 2–3 min and then pressed using a hydraulic pellet press (sample pelletizing) prior to XRF measurements. Results are reported in oxide percentage within 0.01%.

2.3.4. XPS analysis

The reactivity of any material, especially in an aqueous medium, is based on its surface chemistry. XPS is the most suitable technique for determining the chemical composition of a powder's surface.

After milling, about 50 g of the glass powder was put into a plastic bottle, sealed, and kept at room temperature until XPS analysis to simulate real storage conditions, since cementitious materials are generally stored in sealed bags or drums. The different ages of XPS characterization were 0, 7, 28, 90, and 365 days. For age 0, the powder was milled and immediately transferred to the XPS analysis chamber.

Experiments were conducted using a Kratos Axis Ultra DLD X-ray spectrometer with an AlK α 1 (1486.6 eV) source operating at 140 W for the survey scans and 225 W for the high-resolution spectra. The pass energy of the hemispheric analyzer was adjusted to a constant value of 20 eV for the high-resolution spectra and 120-eV for survey scans. The spectrometer work function was calibrated using the binding energy of gold orbital Au 4f $_{7/2}$ (83.98 eV) and the spectrometer dispersion adjusted with the Cu 2p $_{3/2}$ (932.67 eV) photoelectron line. The charge neutralizing of the company was used for each sample to avoid the charge effect. Thereafter, the energy shift was corrected, keeping the C 1s peak at 284.8 eV. The analysis area was an oval measuring 300 \times 700 μm with a depth of 5 to 7 nm. The detection limit varied from 0.1 to 0.5 at.% (atomic%), depending on the element. All XPS spectral peaks were fitted with CasaXPS software (version 2.3.16 Pre-rel 1.4). This software was used to calculate atomic concentration taking

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