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How to cite this manuscript

If you make reference to this version of the manuscript, use the following information:

Mirzahosseini, M., & Riding, K. A. (2014). Effect of curing temperature and glass type on the pozzolanic reactivity of glass powder. Retrieved from http://krex.ksu.edu.

Published Version Information

Effect of Curing Temperature and Glass Type on the Pozzolanic Reactivity of Glass Powder

Mohammadreza Mirzahosseini[1], Kyle A. Riding[1]

Abstract

Glass cullet has been considered for use in concrete as a fine aggregate or a supplementary cementitious material (SCM). Glass reaction kinetics depends on both temperature and glass composition. Uniform composition, amorphous nature, and high silica content of glass make it ideal for studying effects of glass type on reactivity at different temperatures. In this study, the pozzolanicity of clear and green glass crushed smaller than 25 µm at three different temperatures (i.e. 10°C, 23°C, and 50°C) was measured using isothermal calorimetry, chemical shrinkage, and thermogravimetric analysis. Additionally, mortar compressive strength and water absorption of samples at different temperatures and ages have been used to relate reaction degree to performance. Results indicate that elevated temperatures cause glass powder to exhibit significant pozzolanic reaction, even at early ages. Moreover, glass composition was seen to have a large impact on reactivity. In this study, green glass showed higher reactivity than clear glass.

Keywords: Glass cullet; Pozzolanic reactivity; Degree of hydration; Hydration rate; Elevated temperature

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1. Introduction

Millions of tons of glass cullet are landfilled throughout the world every year. In 2005, approximately 12.8 million tons of glass cullet was landfilled in the United States [1]. When waste glass is collected, different color glass is often intermixed. Mixed color glass cannot be recycled, however, because a mixing of coloring agents results in an unpredictable and uncontrollable color in the new glass. Machines are capable of using optical sensors to efficiently sort large glass pieces by color; however, sorting small glass pieces is not economical and much of this unrecyclable glass cullet is then landfilled. For instance, every year 1.65 million tons of waste glasses are disposed in the U.K. because of unsuitability for recycling [2]. As the economic and environmental consequences of landfilling rise, the incentive to reuse glass cullet has grown. The concrete industry is one of the potential ways of reusing millions of tons of glass cullet per year either as aggregate or supplementary cementitious material (SCM).

Most research on crushed glass has focused on the use of glass as fine aggregates in concrete. However, durability concerns over alkali-silica reaction (ASR) have limited the use of glass as a fine aggregate replacement in concrete [3]. Several studies have shown that glass behaves pozzolanically if ground finely enough, with a surface area of more than 300 m^2/kg [4-9]. The pozzolanic reaction occurs between amorphous silica in the SCM, calcium hydroxide (CH) as a byproduct of the cement reaction, and water to form additional calcium silicate hydrate. Most studies on the effect of glass cullet on cementitious mixtures as SCM focused on mechanical and durability properties. Increases in long term compressive strength, flexural strength, resistance to ASR and sulfate attack, and reduction in water sorptivity of concrete containing finely ground glass powder have been found [10-11]. Moreover, some studies showed that finely ground glass powder had comparable or slightly better mechanical properties at later ages than fly ash and slag, but much less than silica fume [12-13]. Despite all aforementioned results, few studies have

aimed to connect the microstructural properties of cementitious mixture containing glass powder to performance characteristics of the glass mixtures. Federico [14] performed extensive study on the influence of glass powder on kinetic and performance properties of cementitious mixtures. However, the effect of curing temperatures on different types of glass cullet reaction kinetics and performance has not been studied.

Temperature is one of the most pivotal parameters which affect cement or cementitious material hydration. Concrete temperature can change due to weather, heat curing applied, and heat of hydration. Elevated temperatures can increase the rate of hydration, early strength gain, change hydration products formed, change density of the formed products, and accelerate activation of pozzolanic activity [15-16]. However, high temperatures can also lower ultimate strength, increase permeability and drying shrinkage, and in some cases, cause delayed ettringite formation (DEF) [17,15].

This study focuses on quantifying the kinetic reactivity of very finely ground glass particles (finer than 25 µm) in concrete as partial cement replacement at different isothermal temperatures, namely 10°C, 23°C, and 50°C with performance as measured by strength and sorptivity.

2. Experimental Program

2.1. Materials

An ordinary portland cement type I/II was utilized for the current study and distilled water was used for the mixing water. Table 1 summarizes the chemical properties of the portland cement obtained by X-ray fluorescence (XRF) analysis. Table 2 shows the potential compositions of the cement based on Bogue equations.

Table 1. Chemical components of cementitious materials

Cementitious Materials	Chemical Components							
	SiO_2 (%)	Al_2O_3 (%)	CaO (%)	Na_2O (%)	K_2O (%)	Cr_2O_3 (%)	Fe_2O_3 (%)	$CaCO_3$ (%)
OPC	19.66	4.71	62.74	0.12	0.56	-	3.26	2.2
Clear Glass	73.50	0.06	9.02	12.65	0.02	0.02	0.28	-
Green Glass	73.10	1.65	10.55	12.34	0.58	0.24	0.44	-

Table 2. Potential composition of cement based on Bogue equations

| C_3S (%)	C_2S (%)	C_3A (%)	C_4AF (%)
58	11	7	10

Clear and green glass was used in this study because previous research results showed that clear glass is the most commonly available type of glass and green glass has the highest pozzolanic reaction. Small impurities are added as coloring agent in glass production. These coloring agents change the glass composition and structure. The source of clear glass was waste window glass collected from recycling company at Kansas City, KS, and the source of green glass was bottle glass from the same bottle manufacturer and bottle type collected from recycling center in Manhattan, KS. The glass was washed and dried to remove any residues before crushing. After crushing to smaller than 1.18 mm (No. 16), glass particles were milled in a laboratory ball mill. After ball milling, the glass was wet-sieved using a sieve with 25 μm openings and isopropanol. The chemical compositions of the glass powders used for this study are shown in Table 1, while Table 3 shows glass powder density and Blaine fineness. Glass powder and cement particle size distribution is shown in Figure 1, demonstrating that the gradation of glass powder is very similar to the cement gradation. Particle shape and texture of cement grain and glass particles was investigated by scanning electronic microscopy (SEM), as shown in Figure 2.

Table 3. Density and Blaine surface area of cementitious materials

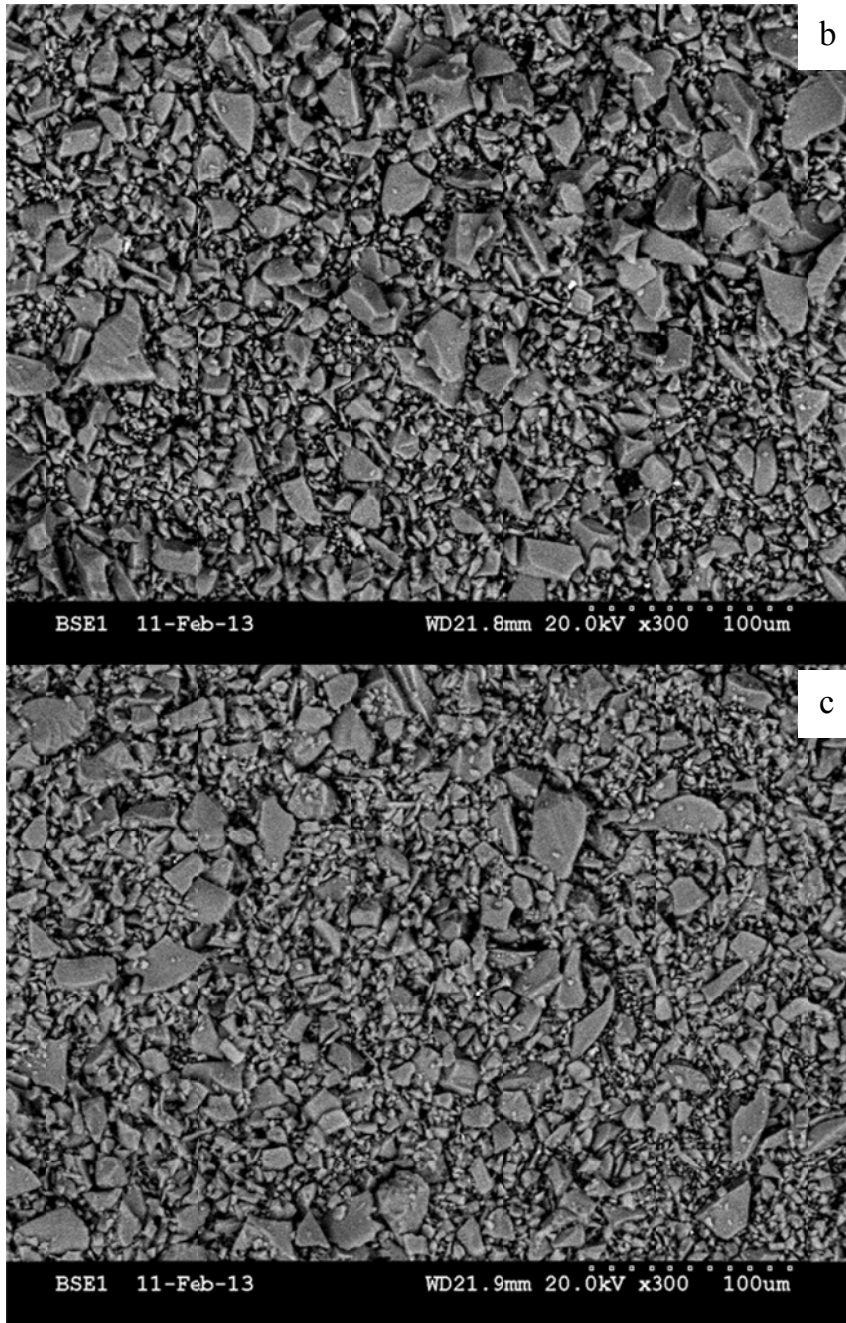| Materials	Density (Kg/m^3)	Blaine Surface Area (m^2/Kg)
OPC	3150	395
Clear Glass 0-25 μm	2477	433
Green Glass 0-25 μm	2501	476
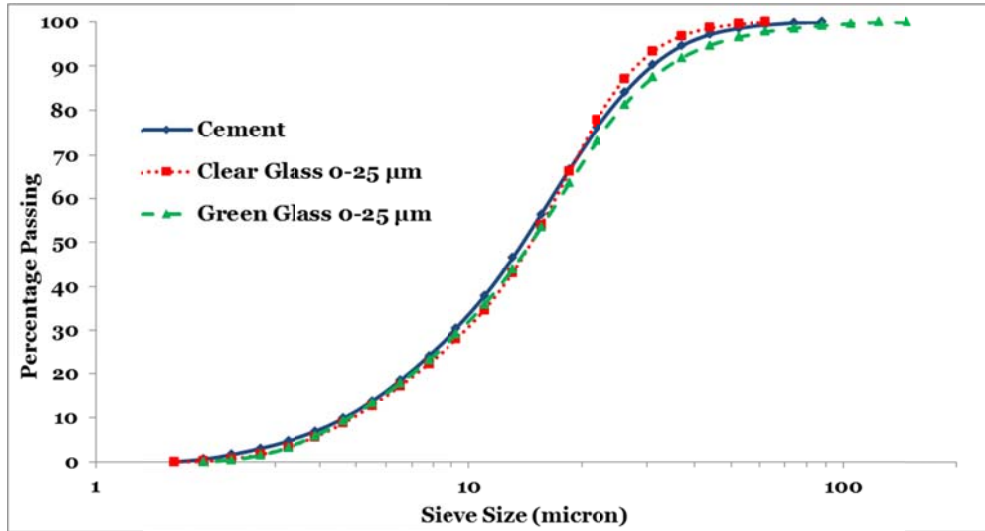
Figure 1. Gradation of cementitious materials
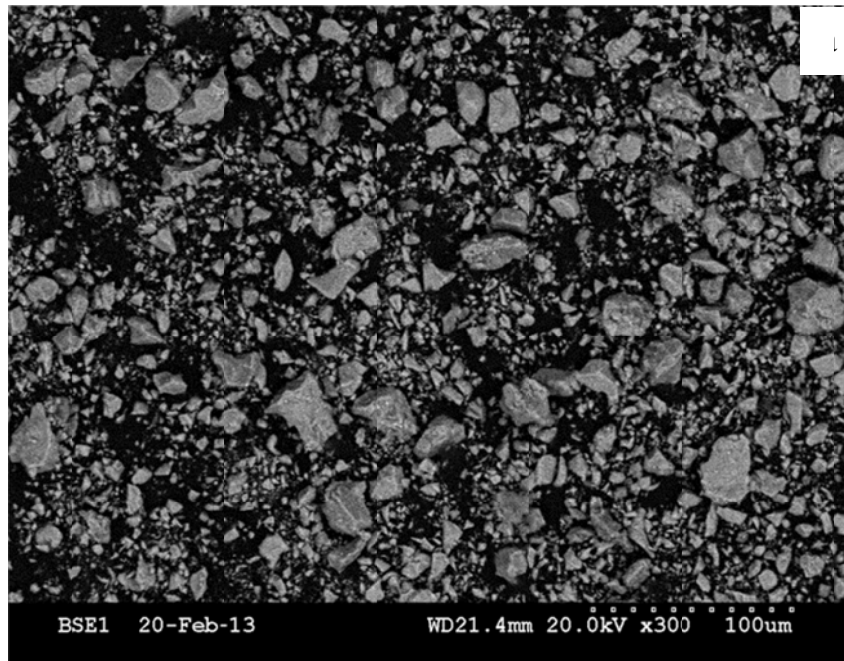
Figure 2. SEM Images; a. Cement grain, b. Clear glass 0-25 μm, c. Green glass 0-25 μm

2.2. Methodology

2.2.1. Bottle Leaching

Bottle leaching experiments were carried out to measure concentration of aluminum and silica ions that leach out into alkaline solutions at different pH values. In the bottle leaching, five

grams of glass powder with different types and sizes was poured into 50 grams of potassium hydroxide (KOH) solution with pH varying from 12 to 14.5. This variation in pH of KOH solution was obtained by adding different amounts of KOH to ultrapure water until the target pH was achieved. The solution was rotated at 30±1 rpm for 24 h and filtered. Inductively Coupled Plasma (ICP) Spectrometry was used to measure ion concentrations.

2.2.2. Cement Paste Experiments

Cement paste samples were made with a water-cementitious materials ratio of 0.35, using a 25% replacement by mass of portland cement with clear or green glass powder. All materials were preconditioned at target temperatures (10°C, 23°C, and 50°C) before mixing. The mixing procedure combined water and cementitious materials at 500 rpm for three minutes, followed by two minutes rest, and finally, mixing at 2000 rpm for two minutes. After mixing, paste samples for thermogravimetric analysis (TGA) and SEM were casted in a polystyrene vials with diameter of 17 mm and height of 50 mm. Paste samples with or without the glass powder were wet-cured beginning at six hours after casting at three curing temperatures in a temperature-controlled chamber.

The cementitious system heat of hydration rate was measured using an eight-channel isothermal calorimeter. Approximately 30 gr of paste samples were placed in calorimetry cups, and the cups were immediately placed into calorimetry channels. The time between initial contact of water and cementitious materials and placing samples into the calorimeter was less than 15 min in all cases. The influences of glass type and curing temperatures on hydration kinetics could be observed as the change in heat of hydration when the calorimetry results were normalized by the mass of dry portland cement used in the paste. This change in the heat of hydration in the mixtures containing glass compared to the control mixture shows changes in system reaction [18-19]. The apparent activation energy concept describes the temperature

sensitivity of physical and chemical properties of different cementitious mixtures [13]. For cementitious systems where multiple chemical reactions occur simultaneously, the Arrhenius concept does not yield true activation energy but provides a parameter for temperature sensitivity of overall reaction rate useful for modeling [20]. Eq. (1) shows the mathematical form of the apparent activation energy [21]:

$$K(T) = A\ e^{-Ea/RT}$$ Eq. (1)

Where K(T) is the rate constant, T is absolute temperature, A is proportionality constant, E_a is apparent activation energy of concrete (J/mol), and R is universal gas constant (8.314 J/mol K). The hydration rate can then be presented by Eq. (2) [21]:

$$\frac{d\alpha}{dt} = g(\alpha) \times K(T)$$ Eq. (2)

Where α is degree of hydration, g(α) is the function of the degree of hydration. The apparent activation energy was also calculated based on the Arrhenius plot according to the procedure described by Poole et al. [20].

Chemical shrinkage was used as a complementary method to calorimetry for measuring the cementitious system reaction rate. Chemical shrinkage is especially suited for measuring reaction rate after the heat of hydration drops too low to be measured by the isothermal calorimeter. Chemical shrinkage measurements in the current study were performed according to ASTM C 1608 [22]. This method involved placing cement paste in a vial, covering it with water, tightly inserting a rubber stopper into the vial, and placing a drop of red oil on top of the water raised through a pipette. The cementitious system volume change was measured through image analysis (IA) of pictures automatically taken of a drop of colored petrol on top of the water in the filled capillary tube. The software for image analysis was developed by Bishnoi [23].

A pozzolanic reaction between CH and glass cullet can be responsible for the strength increase in concrete. The CH content of cement paste samples was determined by thermogravimetric analysis (TGA) using the approach outlined by Marsh [24]. Paste samples with or without the glass powder were wet-cured beginning at 6 h after casting at three curing temperatures. At 1, 7, 28, and 91 days after casting, hydration was stopped by solvent exchange with isopropanol. Paste samples were cut from the 17-mm diameter samples into 2 mm thick discs and placed in isopropanol for seven days. Samples were then dried in a vacuum for four to five days, ground to fine powder, and heated in a nitrogen rich atmosphere in a TGA at 20°C/min up to 1000°C.

Scanning electron microscopy (SEM) was performed on cut and polished paste discs, after solvent exchange and drying in a vacuum to examine any differences in microstructure. Thirty SEM images were taken in backscattered mode (BSE) from each paste disc containing glass powder at a given curing age and curing temperatures. In backscattered mode (BSE) of SEM, image brightness is a function of the material average atomic number and can be used to differentiate between different phases in the image [25]. In particular, C-S-H relative brightness was used to measure change in C-S-H relative density and effect of temperature on C-S-H density [26]. C-S-H density was calculated using the approach described by Zhang [27].

2.2.3. Cement Mortar Samples

All mortar mixtures used in this study were made with a water-to-cementitious material ratio (w/cm) of 0.485 and sand to cementitious material (s/cm) of 2.75 specified by ASTM C 109 [28]. The w/cm of mortar samples is different from that for paste samples because of bleeding issues found in paste samples at high w/cm ratio and workability problems in mortar samples with lower w/cm. Mortar samples were made with and without glass powders using a 25% cement replacement by mass. Mortar samples were cured at 10°C, 23°C, and 50°C.

Fifty mm mortar cubes were made and tested at 1, 7, 28, and 91 days after mixing for compressive strength. Compressive strength of mortar cubes was performed according to ASTM C 109 [28] as a measure of pozzolanic reaction of the glass particle with time. Compressive strength at each age is reported as the average of compressive strength results of three mortar cubes.

Durability of concrete is strongly impacted by concrete water and ion transport properties. A comparison between strength and water sorptivity of mortar provides information in regards to pore connectivity and tortuosity because compressive strength is a function of pore volume [29]. Water sorptivity is also dependent on pore volume but additionally dependent on pore size distribution, connectivity, and tortuosity. Cylinders with 75 mm diameter by 150 mm height were casted for water sorptivity tests. Water sorptivity was measured using the procedure outlined by ASTM C 1585 and modified by Zhang [30, 27]. Samples were sawcut at the same ages as compressive strength testing to 30 mm thick for use in a water sorptivity test. After sawcutting, three samples for each age were placed in isopropanol to stop hydration by solvent exchange. Following seven days in isopropanol, the samples were dried in a vacuum for seven days and weighed. The top and sides of the disc samples were sealed with plastic and waterproof tape, leaving the bottom side exposed to water. The samples were then placed in water so that 2 ± 1 mm from the bottom of the disc was submerged. The mortar discs were taken out of the water; the sample bottom was blotted with a paper towel to remove free surface water and weighed. The procedure was repeated at regular intervals until eight days [27].

3. Results and Discussions

3.1. Leaching

Based on the bottle leaching test (Figure 3), green glass has more silica and aluminum ion in solution at higher pH (i.e. more than 12.5), meaning that changes in green glass microstructure,

which was caused by adding impurities to give color to glass, as well as breakdown of silica framework by high pH resulted in higher dissolution rate of green glass. Thus, green glass may react faster because aluminum and silica of the green glass are more easily and quickly dissolved in cement matrix [31].
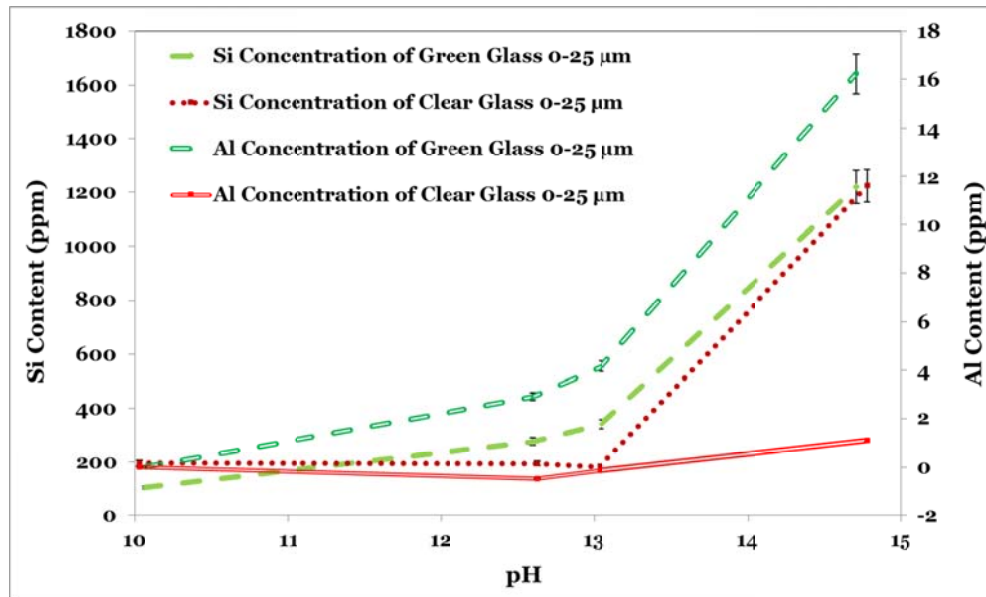

Figure 3. Al and Si concentrations at different pH (ICP test)

3.2. Isothermal calorimetry

Figure 4 shows total heat of hydration for control samples and samples containing clear and green glass of 0-25 μm hydrated at 10°C, 23°C, and 50°C. As seen in the figure, the total heat of hydration at 96 hours at 10°C and 23°C temperature is similar for both types of glass, meaning the degree of cement hydration at 96 hours is the similar for clear and green glass samples. At 50°C, green glass showed 10% higher total heat of hydration compared to clear glass, reflecting the higher reactivity of green glass at elevated temperatures. However, glass samples show slightly higher total hydration heats than control samples at all temperatures (i.e. 12%, 13.5%, and 20% at 10°C, 23°C, and 50°C, respectively). At lower temperatures, i.e. 10°C and 23°C, the increase in heat of hydration rate of samples containing glass particles is most likely the result of

greater space availability from increased effective w/cm. While at 50°C, the increase in hydration rate is mostly caused by combination of higher effective w/cm and glass particle pozzolanic reaction, which was evidenced by higher reaction rate of green glass compared to clear glass at 50°C.
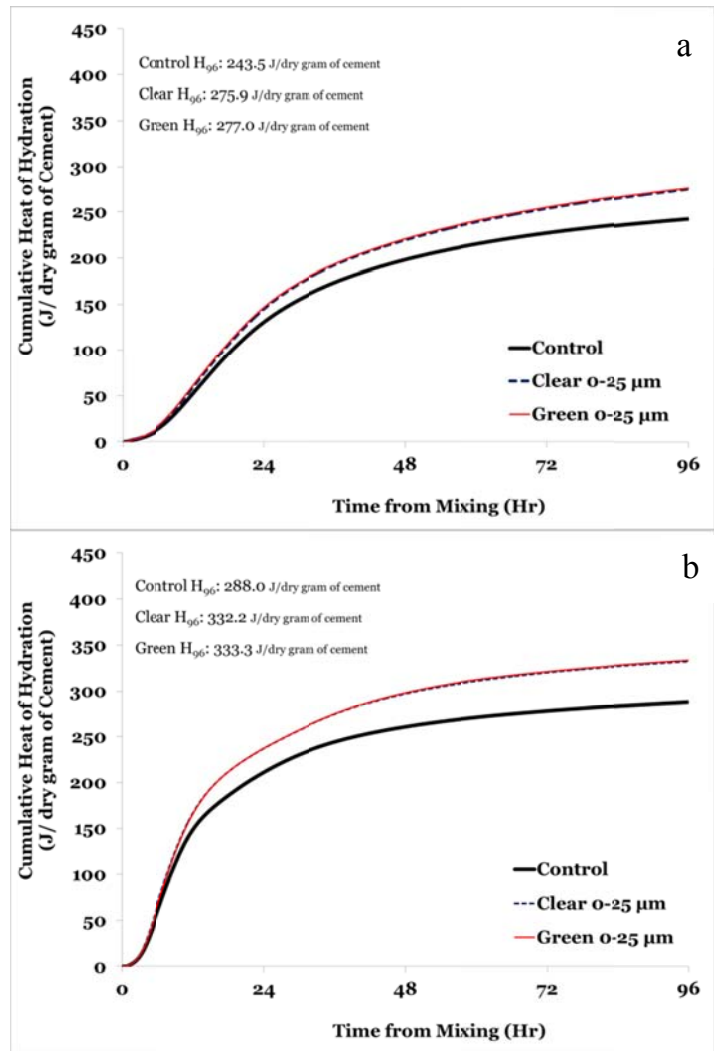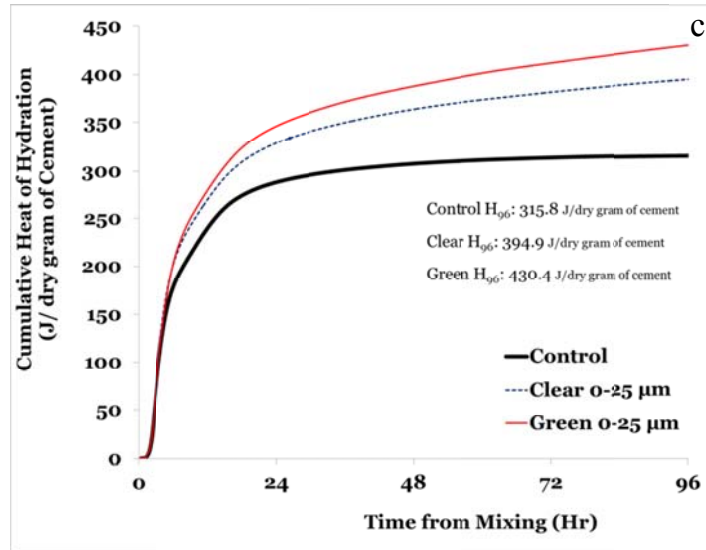
Figure 4. Total heat of hydration of paste samples; a. at 10°C, b. at 23°C, c. at 50°C

The Arrhenius plot of control samples and samples containing glass powder is shown in Figure 5. While control samples and clear glass samples have similar E_a (i.e. 38.2 and 37.0, respectively), the green glass sample has slightly higher apparent activation energy (i.e. 9%–12%) than the other two mixtures [20], indicating that green glass is more sensitive to temperature and more reactive at elevated temperatures than clear glass.
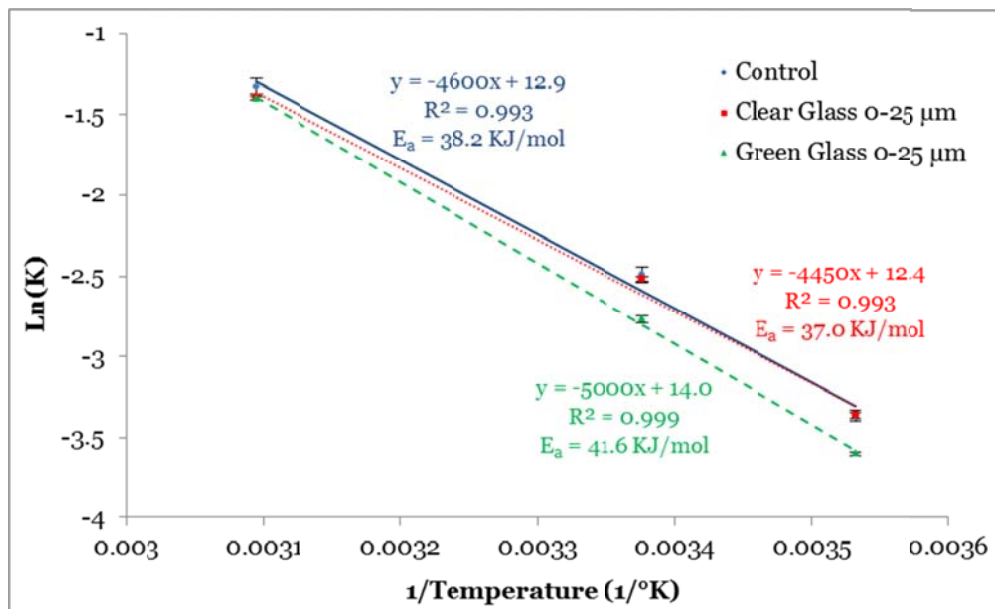

Figure 5. Arrhenius plot of cementitious samples

3.3. Chemical shrinkage

Figure 6 depicts chemical shrinkage results through 28 days of hydration for control samples and samples containing glass powder at 10°C, 23°C, and 50°C. As was seen in heat of hydration measurements, increased temperature greatly increased reaction rate, as measured by chemical shrinkage. Furthermore, pastes made with clear and green glass show different reactivity at all temperatures, unlike the calorimetry results. At 10°C, clear glass sample showed 31% higher chemical shrinkage than control samples at 28 days, while green glass powder showed an increase in reactivity at 28 days of 48% compared to the control samples. Increases in reactivity at 50°C compared to 10°C are 14.5%, 46%, and 22.5% for control, clear glass, and green glass samples, respectively. These results not only reveal that green glass is likely more reactive than clear glass, but the effect of curing temperature could also be taken into account. Higher chemical shrinkage measurements which show a higher amount of reaction at 50°C shows the great influence of elevated temperatures on cement and glass hydration.
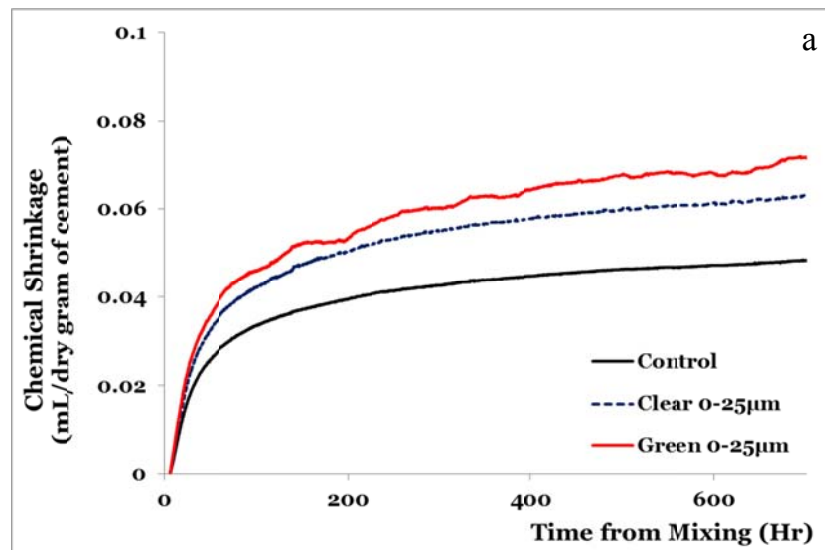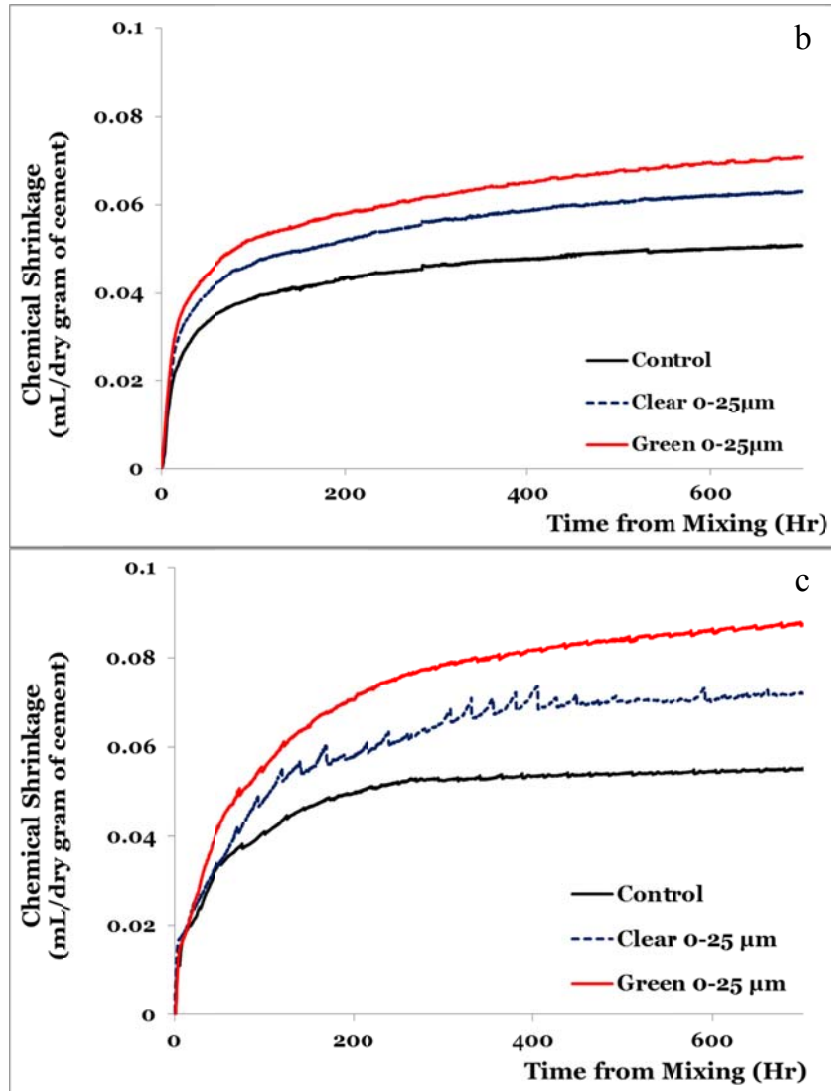
Figure 6. Chemical shrinkage of paste samples; a. at 10°C, b. at 23°C, c. at 50°C

3.4. Thermogravimetric Analysis (TGA)

Change in CH content with time and curing temperature in cement paste samples is shown in Figure 7. At the low temperature (10°C), the decrease in CH seen with time as an indication of the pozzolanic reaction was not seen to be significant. However, at 23°C, samples with green glass powder had about 13%, 18%, and 19% lower CH than control samples at 7, 28, and 91 days, respectively. Glass powder showed significant long-term CH reaction at 50°C: samples with clear and green glass of 0-25 μm showed 9% reduction in CH even at one day. At seven

days, clear and green glasses showed CH reduction of 13% and 23%, respectively. At 28 and 91 days, clear and green glass samples behaved similarly since both can lower CH content by approximately 34%, confirming that elevated temperatures can accelerate the glass hydration reaction.
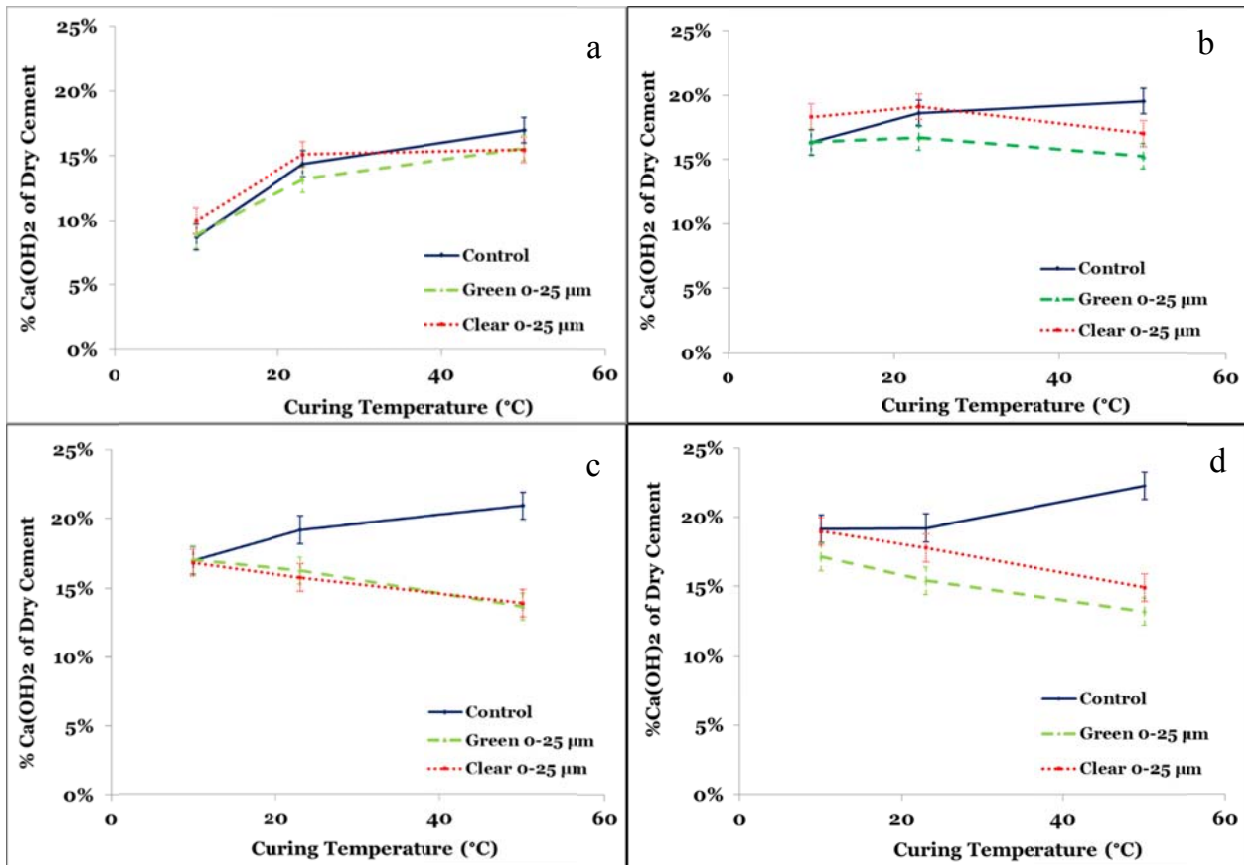

Figure 7. CH content of paste samples; a. at 1 day, b. at 7 days, c. at 28 days, d. at 91 days
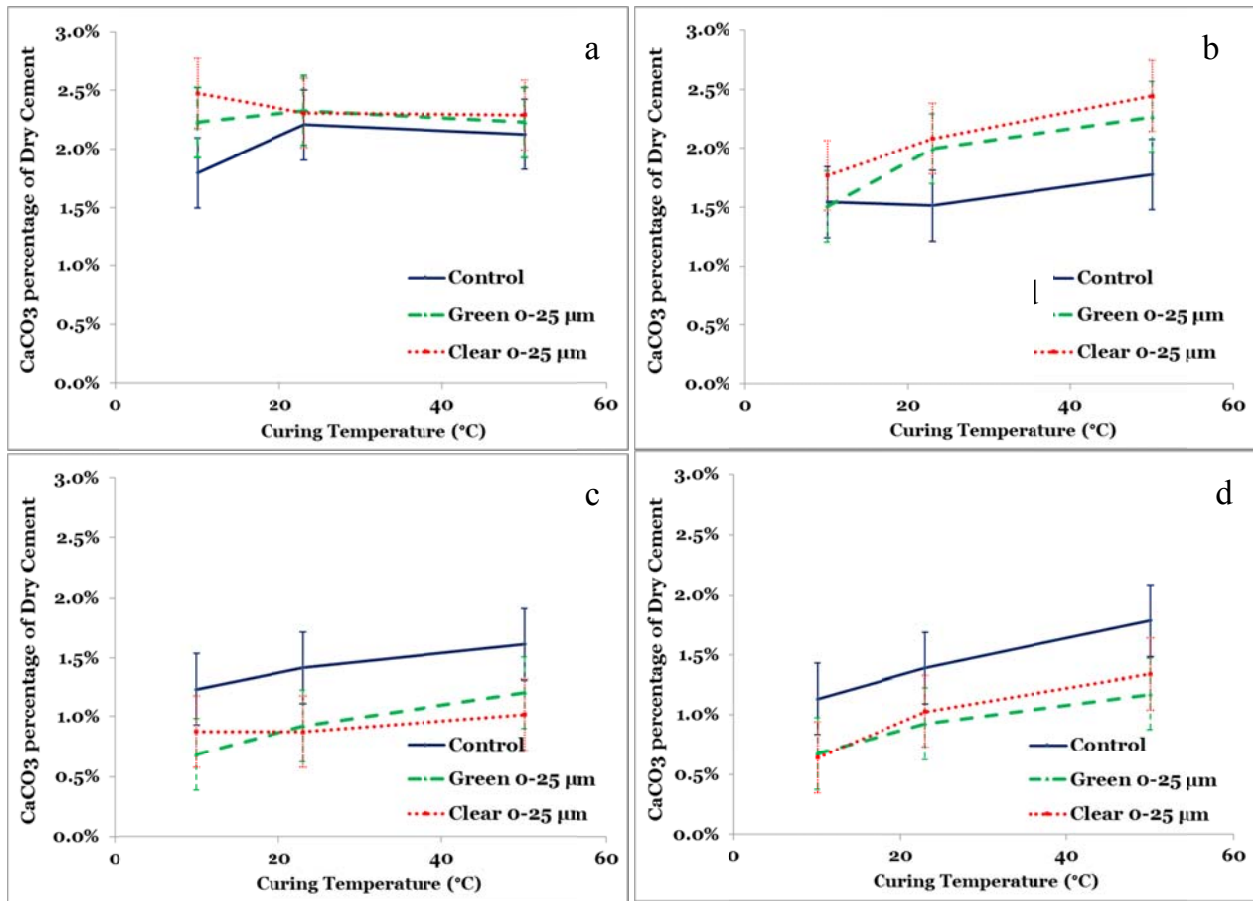
Figure 8. Calcium Carbonate content of paste samples; a. at 1 day, b. at 7 days, c. at 28 days, d. at 91 days

3.5. Scanning Electron microscope (SEM)

Figure 9 shows SEM images of paste sample containing clear glass powder at seven and 180 days. To investigate the effects of glass powder and curing temperatures on reactivity, relative density of inner C-S-H around cement grains was calculated. At seven days and 23°C, inner C-S-H relative density around the cement grain in paste samples containing green glass was 2.48, while higher density was noted at seven days and 50°C (inner C-S-H density of 2.50), meaning hydration products are produced more significantly at elevated temperatures. At 180 days and 23°C, the calculated density was 2.51 and at 50°C was 2.54, reflecting that the C-S-H density at initial ages and lower curing temperatures is less than later ages and elevated temperatures; as density of C-S-H is increased while time passed and temperature is increased.
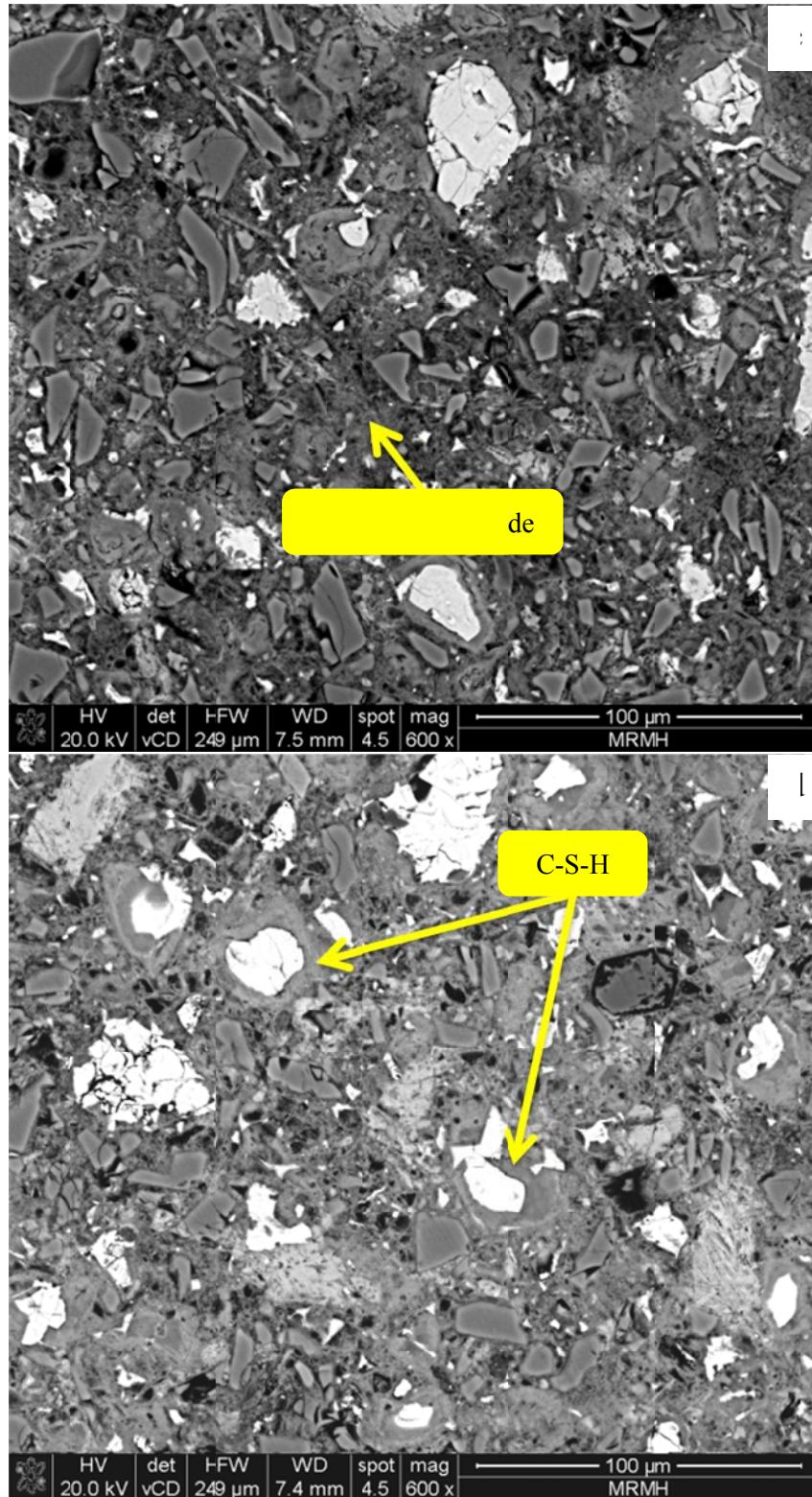
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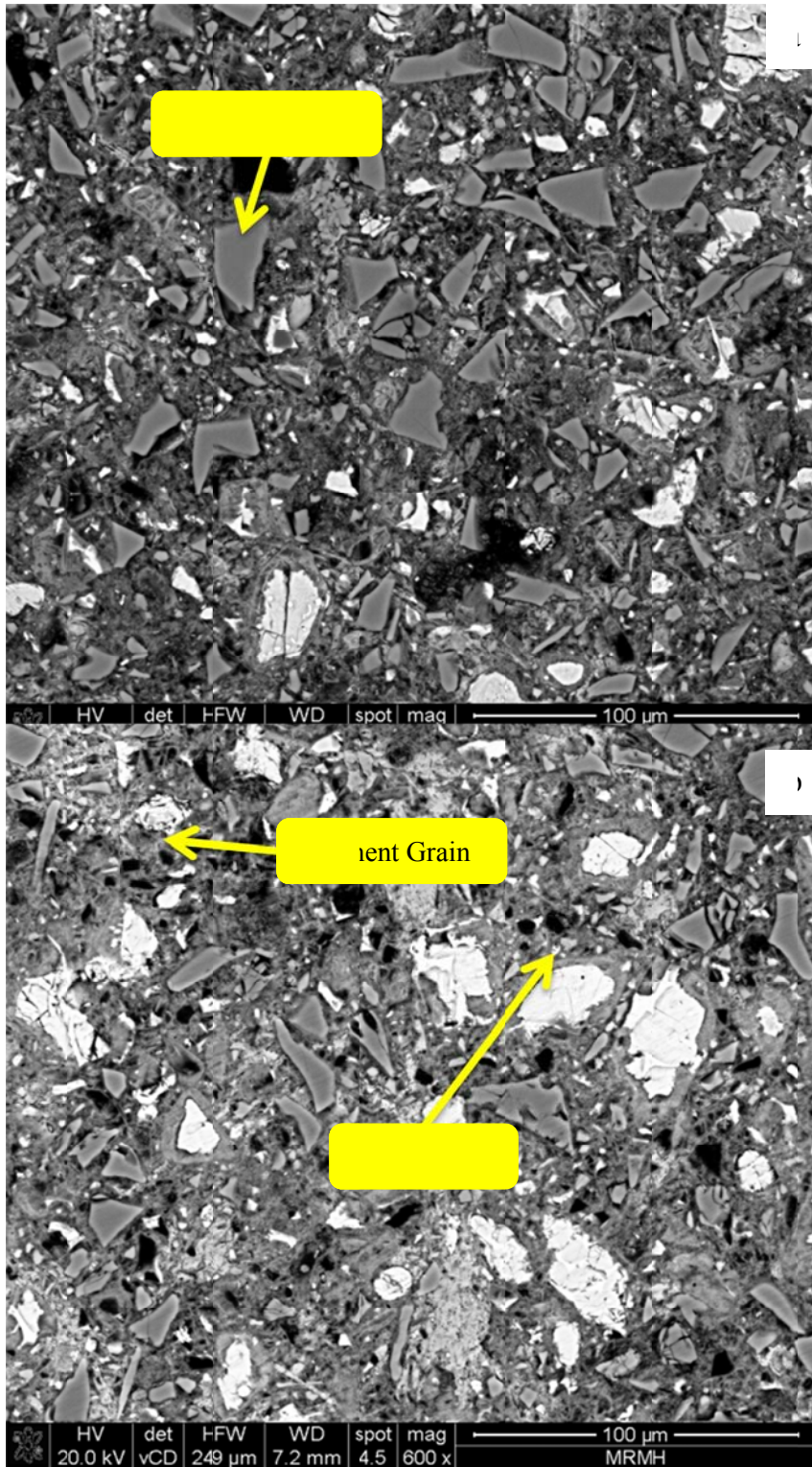
Figure 9. SEM Images of paste samples containing clear glass cullet; a. at 23°C at 7 days, b. at 50°C at 7 days, c. at 23°C at 180 days, d. at 50°C at 180 days

3.6. Compressive strength

Evolution of compressive strength of mortar samples with and without 25% cement replacement by glass powder is illustrated in Figure 10. The control samples were seen to have higher strength than those containing glass powders at all curing ages tested up to 90 days and at 10°C and 23°C. The lower strength of samples containing glass can be attributed to the increase in effective w/c ratio and from the lower glass reaction level at lower temperatures. However, as glass reaction increased at later ages, compressive strength of mortar increased and approached the strength of the control samples. At 50°C and after seven days, control and clear glass samples exhibited lower strength than green glass samples. After 28 days, clear glass showed some pozzolanic reaction, reflected by slightly higher compressive strength than control sample. Results indicate that green glass is significantly more reactive than clear glass because high pH cementitious matrix breaks down the silica structure of the glass and silica and aluminum could dissolve more and participate in the pozzolanic reaction. Higher silica and aluminum dissolution seen in the green glass during the bottle leaching experiment correlate to the higher pozzolanic reaction and lower CH and higher compressive strength. This is likely a result of change in the glass structure from the addition of coloring agent. Additionally, the effect of elevated temperature on strength gain is observed in Figure 10. At 28 days, neither clear nor green glass powders showed significant pozzolanic reaction. Although TGA results showed some pozzolanic reaction at 23°C, that pozzolanic reaction was not enough to overcome a decrease in compressive strength at 28 days caused by the higher effective water-to-cement ratio. However, at 50°C compressive strength increased 30% at 28 days, which was in well agreement in TGA results.
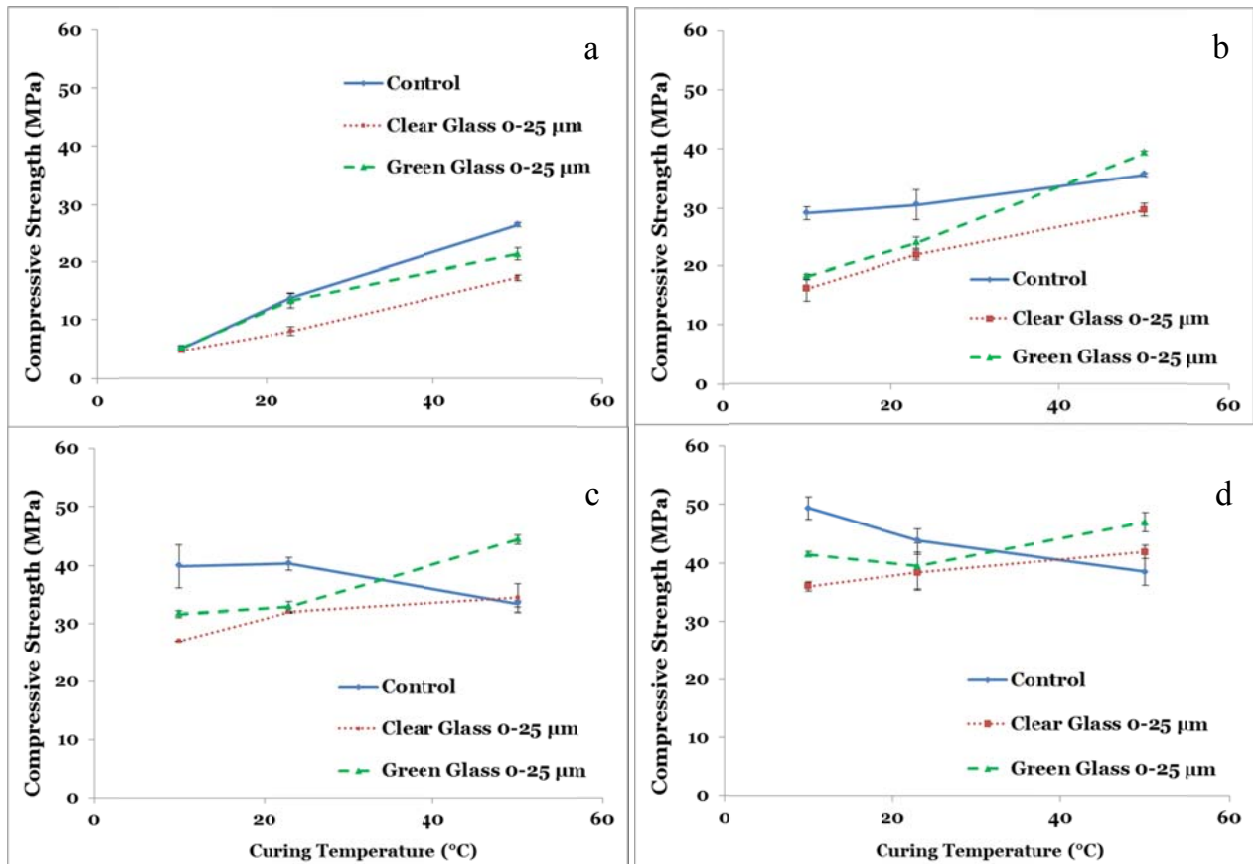
Figure 10. Compressive strength of mortar samples; a. at 1 day, b. at 7 days, c. at 28 days, d. at 91 days

3.7. Water Sorptivity

Figure 11 shows water sorptivity of mortar discs with and without glass powder. All samples had considerable absorption at early ages (i.e., one day), especially at 10°C. At 50°C, water absorption was reduced up to 30% because of accelerated hydration and the creation of more hydration product. This phenomenon indicates that at early ages, elevated temperatures decrease concrete porosity by producing denser hydration products, already observed in inner C-S-H relative density results from SEM images, especially, due to the existence of adequate space to accommodate quickly-formed hydration products. At later ages, water sorptivity of the control sample at 50°C was much higher than that at 10°C (cross-over effect). Although reaction products of control samples are denser at later ages at 50°C, there is more porosity which is the

reason for higher water sorptivity of control samples. On the other hand, glass cullet which reacts slower than the cement, is better able to fill in higher porosity from denser cement hydration products at later ages at elevated temperatures eliminating the cross-over effect as seen in Figure 11. An explanation may be because no additional space for hydration products existed (diffusion controlled stage) and the overall structure was already set. In addition, green glass is able to reduce water absorption to some extent more than clear glass, as green glass has a higher tendency to participate in the pozzolanic reaction.
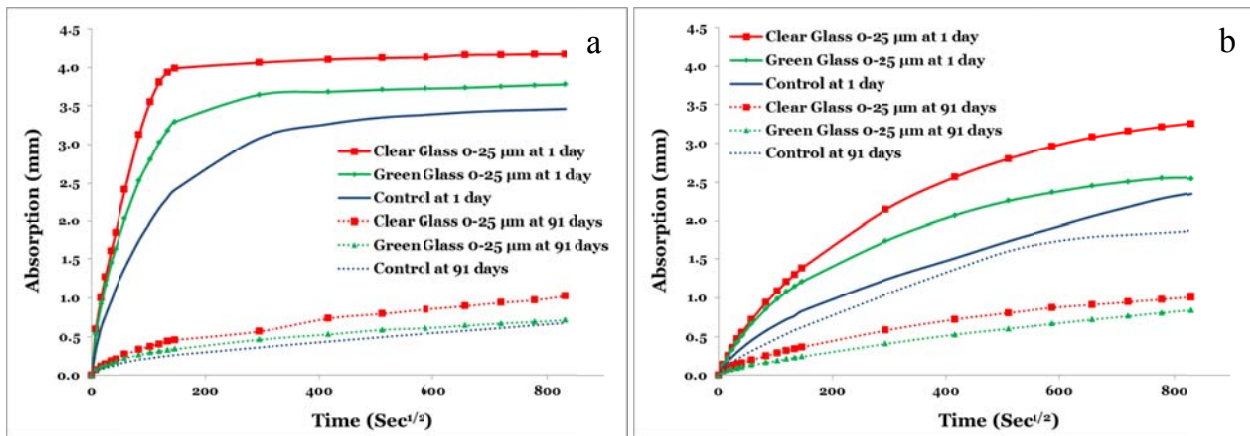

Figure 11. Water absorption of mortar samples; a. at 10°C, b. 50°C

4. Conclusion

The current study investigated the effect of curing temperatures and the type of glass powder on pozzolanic reactivity of glass cullet in concrete. Based on experimental results, the following conclusions can be made:

1- At low temperature, finely ground glass (finer than 25 µm) does not show pozzolanic behavior. At 10°C, no significant reduction in CH content was observed until 91 days. Additionally, mortar samples containing glass cullet have lower compressive strength and higher water sorptivity than control samples, even at 91 days.

2- Finely ground glass cullet (finer than 25 µm) shows significant pozzolanic behavior at elevated temperatures (i.e. 50°C). The reduction in CH content of samples containing glass with an increased heat of hydration at 1 day evidences this. At later ages – i.e. after seven days – CH content noticeably decreases, showing more pozzolanic reaction, which correlates well with results of SEM imaging, compressive strength, and water sorptivity tests.

3- Curing temperature is one of the foremost factors affecting pozzolanic behavior of glass cullet. The results of apparent activation energy calculation showed that glass – especially green glass – is temperature sensitive and has a higher tendency to participate in pozzolanic reaction. This was also evidenced by TGA and compressive strength results.

4- Part of the reactivity at low temperatures can be attributed to calcium carbonate; as AF_m phases containing calcium carbonate could react faster at lower temperatures, potentially giving a larger driving force for more dissolution of $CaCO_3$.

5- At pH ranges similar to that seen in concrete (i.e. 12.5–13.5), aluminum content of green glass – which is 27 times higher than clear glass – and silica can more easily dissolve in pore solution, evidenced by bottle leaching test results, and may be the reason for the higher reactivity seen than clear glass.

5. Acknowledgement

6. References

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