Chemical transformation of rice husk ash morphology for improving on early-aged characteristics of cementitious systems

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ABSTRACT

The use of agricultural by-products in cementitious systems has been shown to have significant potential for increasing the sustainability of civil engineering systems. Rice husk ash (RHA), an abundantly available by-product, is a readily available supplementary cementitious material (SCM). RHA typically contains amorphous silica. However, RHA consists of a mesoporous structure and this has high absorption characteristics. The use of RHA in cementitious systems leads to reduced workability and/or higher water requirements. Reduced workability reduces the likelihood that the RHA will be accepted by users and higher water requirements lead to lower compressive strengths. Studies using mechanical pulverization to reduce the mesoporous morphology have been performed, but these studies indicate significant energy is required. This study proposes a novel method using chemically-induced morphological transformation to achieve improved workability at lower water requirements. Results indicate this method is a viable alternative to improve the early-age characteristics of cement systems containing RHA.

Keywords. Rice Husk Ash, Cementitious Materials, Morphological Modification, Early-Age Characteristics, Sustainability

INTRODUCTION

As sustainability becomes a larger concern for our society, more industries are focusing on developing or improving existing systems to make these systems more sustainable. The need to preserve natural resources and to reduce energy consumption and greenhouse gas emissions has stimulated research in the development of alternative resources for construction materials. The construction industry is assessing a wide range of sustainable practices, with emphasis on using alternative resources and technologies, including the use of industrial by-products and agricultural wastes from energy sector. These practices can offer more sustainable systems. Among the materials and technologies being evaluated in the construction industry, RHA is a renewable bio-resource that can be introduced as an alternative and renewable resource as a pozzalanic material.

A large amount of RHA is produced annually and this product has historically been considered to be waste, as there are few commercial applications for this product. This leads

to challenges with disposal and storage at power plants (Kibriya, 2006; Naiya *et al.*, 2009). Current disposal methods include dumping into landfills. Although currently economical, this practice is not sustainable as RHA can create long-term environmental challenges. As a reusable bio-resource, broadening the application and use of RHA can increase economic value and environmental sustainability, especially as SCM, as replacing cement reduces CO_2 emissions.

Concern over the impact of man-made CO_2 emissions has increased in recent years due to growth in global warming awareness. Approximately 5% of CO_2 emissions on earth derives from the cement production (Worrell *et al.*, 2001). Although efforts are beginning to make the cement industry more sustainable, the CO_2 emissions from cement production are significant and more efforts for improved sustainability are needed. The partial substitution of RHA as a source of pozzalanic materials into concrete can significantly reduce not only the CO_2 emissions but also the cost of both producing cementitious materials and disposing the RHA. RHA has been successfully used as a SCM for many decades, but efforts have been mostly focused on laboratory studies. Zhang and Malhotra (1996) studied concrete containing various SCMs and reported that RHA is similar to silica fume regarding its pozzalanic activity.

RHA-based concrete has been reported to exhibit good early- and later-age characteristics. Chandrasekhar *et al.* (2003) reported concrete containing RHA exhibited improved resistance to segregation and increased strength. The authors reported that the pozzalanic reaction of the RHA allowed for better pore refinement, resulting in reduced segregation and the strength increase was a result of the pozzalanic reaction. RHA-based concrete also has been reported to have good resistance to chloride penetration (Azevedo *et al.*, 2001; Maeda *et al.*, 2001; Nehdi *et al.*, 2003; Gastaldini *et al.*, 2007), good freeze-thaw performance (Shuichi *et al.*, 1992), resistance to alkali-silica reaction expansion (Mehta and Folliard, 1995), and improved resistance to deicing salt scaling (Nehdi *et al.*, 2003).

RHA-based concrete can be a solution to improve sustainability and can offer ecological, economic, and energy benefits for the cement and concrete industries. However, the development of RHA-based concrete has some practical limitations and challenges. These limitations and challenges likely decrease its acceptance and use and include:

(1) Increased water requirements largely due to the mesoporous structure;

(2) Lower early-age strength development as a result of the later pozzalanic reactions (Rukzon *et al.*, 2009), and;

(3) Reduced flow and workability characteristics, thereby limiting its addition quantities in concrete (Mehta and Monteiro, 2006; Massazza, 2008).

The flow and workability challenge can be reduced by adding increased levels of water, however, increasing the water content results in lower water-to-cementitious materials ratios (w/cm) and reduced strength. Because most specifications have strength requirements and earlier strength is more economical to contractors than later-age strengths (Reinschmidt and Trejo, 2006), the acceptance of RHA has not been widespread.

Researchers have investigated particle size effects of RHA on concrete performance and reported that reduced particle size leads to improved flow, earlier strength gain, and improved durability (Mehta and Folliard, 2002; Rangaraju and Harish, 2010). The researchers also reported that when using smaller RHA particles, less water was required to

achieve adequate flow characteristics. As the mesoporous structure of RHA is decreased, the water demand is decreased and the ability of the material to fill interstitial pores (i.e., the filling capacity) is improved (Chandrasekhar *et al.*, 2002; Bui *et al.*, 2005). These researches used mechanical grinding to reduce the RHA particle size and this process was reported to consume significant effort and energy (Lu and Wei, 1992; Datta and Rajamani, 2002). Halim (2008) reported that as the requirement for smaller particle sizes increase, larger energy requirements are necessary. In addition to the significant energy requirements, wear of mill and grinding components is very costly (Snow *et al.*, 2008). Successive reductions in particle size can lead to improved fresh and hardened characteristics; however, obtaining these smaller particles is also costly.

Additional research is needed to identify economic and efficient alternatives to mechanically pulverize RHA such that this material can become a more widely used SCM. Thus, the objective in this work is to develop a novel method to transform the size and morphology of RHA. This paper will report on a chemical treatment method used to modify the size and morphology of RHA. The early-age characteristics including ion concentration kinetics, setting, flowability, and early-age compressive strength of chemically transformed RHA (t-RHA) systems are characterized in this study.

MATERIAL AND METHODS

Material. ASTM C150 Type I/II portland cement was used for all mixtures in this study. The chemical composition of the cement and RHA obtained from Agrilectric Companies, Lake Charles, LA are shown in Table 1. Fine aggregate, used for compressive strength specimens, was procured from a local source in Corvallis, OR. Graded sand meeting ASTM C778, *Standard Specification for Standard Sand*, was used for the flowability study. ASTM type II de-ionized water was used for all mixtures and experiment. Pellet sodium hydroxide (NaOH) was an American Chemical Society (ACS) grade (>98% purity). Polyvinyl alcohol (PVA) powder (molecular weight of 1750 ± 50) was procured and used as a phase-stabilizing agent and microfiber reinforcement.

Preparation of morphological transformation method. Recent studies on chemical treatment of RHA have found that its structure transforms into soluble silicates by alkali extraction (Kalapathy *et al.*, 2000). This alkali extraction is a technique to extract amorphous silica from RHA. This is done because the solubility of amorphous silica significantly increases at higher pH levels (Kalapathy *et al.*, 2000). The alkali extraction mechanism of silica from RHA by using NaOH to produce sodium silicate was proposed by Kamath and Proctor (1998).

$$xSiO_2 + 2NaOH \rightarrow Na_2O \bullet xSiO_2 + H_2O \qquad (1)$$
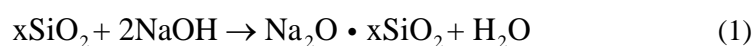
where x is a variable that allows the SiO_2 to Na_2O ratio in sodium silicate solution.

RESEARCH METHODOLOGY

Methods used to transform the as-received (AR) RHA and to test the RHA systems are described in the following sections.

Preparation of t-RHA. The t-RHA was prepared by mixing AR RHA with 2M NaOH solution using a magnetic stirrer at a mixing rate of 400 rpm. Mixing was performed at approximately 23˚C. No heat was used to accelerate the transformation process due to

concerns regarding energy-consumption. The t-RHA was mixed continuously for 3 hours. PVA was added at 1% by weight of NaOH prior to mixing. The mean particle size of AR RHA, the t-RHA, and the t-RHA + PVA are shown in Figure 1. Particle size was measured using a Malvern Mastersizer 2000 particle size analyzer, based on light scattering. The size of t-RHA and t-RHA + PVA is about 8 and 5 times smaller than AR RHA, respectively.

Preparation of t-RHA-cement systems. After mixing, portland cement was added to the t-RHA solution to assess the early-age characteristics. Mixing of cement paste and mortar followed ASTM C305, *Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency*. The mixtures were prepared with 10% and 15% by weight of the RHA components (i.e., AR RHA and t-RHA with and without PVA). A control mixture (100% portland cement) was also mixed and tested for comparison.

Characterization methods. The rate at which calcium and aluminate ions dissolute from the cementing materials can directly affect the early-age characteristics of the system. Therefore, the concentrations of calcium and aluminate ions of the control and RHA-cement systems were analyzed using flame atomic absorption spectroscopy (AAS). The w/cm of the solution assessed was 4.0. Mixing for all systems was performed using a magnetic stirrer rotating at 400 rpm throughout the test. Control and RHA-cement solutions were drawn from the mixing beaker and filtered using a vacuum pump and No. 40 filter paper at different times. Time was measured after introduction of the cementing materials to the water. The time elapsed after introducing the cementing materials to the water will be referred to here as the "hydration time." Solutions for AAS evaluation were drawn from the mixing beaker at 5, 10, 15, 30, 45, 60, 90, 120, 150, 180, 210, 240, 300, 360, and 420 minutes. At each hydration time, filtered solution was used for analyzing calcium (1 ml) and aluminate (10 ml) ion concentration. Extracted and filtered of calcium solutions were diluted with 9 ml of deionized water to get the solution within the detection range of the AAS. Because the aluminates had lower concentrations, solutions for assessing aluminate concentrations did not have to be diluted. Solutions were mixed with 1 ml of lanthanum acid solution (50 g/l lanthanum oxide (La_2O_3) in 3M hydrochloric acid (HCl)). The calcium concentrations were determined using AAnalyst 100 AAS (Perkin Elmer Instrument, Waltham, MA) using air-acetylene gas at a wavelength of 422.7 nm ignited at the temperature of 2100 to 2400˚C. The analysis of the concentration of aluminate ions was determined using the AAS with nitrous oxide-acetylene gas at a wavelength of 309.3 nm ignited at the temperature of 2600 to 2800˚C. Triplicate specimens were evaluated for all systems.

The setting time of all systems was determined following ASTM C191, *Standard Test Methods for Time of Setting of Hydraulic Cement by Vicat Needle*. The w/cm was 0.4 for all mixtures. The flowability of the different systems was determined following ASTM C1437, *Standard Test Method for Flow of Hydraulic Cement Mortar*. The w/cm of the flowability tests included mixtures with a w/cm of 0.5. The cementitious material-fine aggregate ratio was 1:2.5. The 1-, 7-, and 28-day compressive strength of the materials was evaluated using 50 mm x 50 mm cube specimens following ASTM C109/C109M, *Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)*. After casting, test specimens were kept in plastic molds for 24 hours and then demolded. Test specimens were then cured in saturated-lime water until testing.

RESULTS AND DISCUSSION

Ion concentration. The hydration process of cementitious materials generally consists of four stages: 1) the dissolution stage, 2) the nucleation stage, 3) the precipitation stage, and 4)

the continuous dissolution and precipitation stage. After water is introduced with unhydrated cement, calcium and aluminate ions are dissolved into water and rapidly reach a supersaturated stage. This stage is referred to as the dissolution stage. It is noted that gel layers are formed around the surfaces of cement grains at early-ages as there are more aluminates near the surface regions. The role of this aluminate gel phase induces the slowdown of hydration reactions (Scrivener, 1984; Scrivener and Pratt, 1984; Taylor, 1997). In addition, Begarin *et al.* (2011) reported that aluminate ions in portland cement system can induce a delay before precipitation (referred to as the "dormant period"). The higher concentration of aluminate ions in solution can be attributed to higher formation of aluminate gel layers, which can also decrease the rate of the hydration reactions at early ages. As ions are in the supersaturated stage, intermolecular reactions between these ions occur and these reactions attract other ions to form new nuclei. Because the formation of these nuclei is random, nuclei can form into various sizes. This stage is referred to as the nucleation stage. When the nuclei exceed a critical size (the minimum size that creates a new product), hydrated product begin precipitating from the solution. The concentration of ions in solution is then reduced. This stage is referred to as the precipitation stage. The concentration of ions is reduced and eventually reaches a steady state when the rates between dissolution and precipitation are equal.

Because calcium, alumina, and silica are the main chemical constituents in cement, the concentration of these constituents should be assessed. However, the concentration of silica was not evaluated in this study due to equipment unavailability. Figure *2a* and *2b* show the concentrations of aluminate and calcium ions for the control, 10% AR RHA, and 15% AR RHA systems, respectively. The AR RHA systems exhibit higher ion concentrations of aluminate but lower ion concentrations of calcium compared to the control system. Higher aluminate ions of the AR RHA systems possibly correspond to higher amounts of aluminate gel phase formation. This gel could slow the transport of water to the unhydrated cement grains. Thus, the dormant period or nucleation stage can be longer when the system contains AR RHA. On the other hand, the presence of the RHA seems to reduce the concentration of calcium at early ages which suggests that the cement hydration reactions would decrease, resulting in slower strength gain – this has been observed in the field for other SCMs.

As the AR RHA content increases, the concentration of calcium ions decreases, indicating that increasing the AR RHA content would result in further decreases in early strength gain. Figure *3a and 3b* show the effects of t-RHA+PVA on the concentration of calcium and aluminate ions. The 10% and 15% t-RHA systems exhibit higher concentrations of aluminate and lower concentrations of calcium ions than the control systems, similar to the AR RHA systems. The results of t-RHA systems are expected to be the same as AR RHA systems. However, it should be noted that the t-RHA has a smaller mean particle size (see Figure 1), and these smaller particles are likely to be more reactive than larger particles, irrespective of ion concentration. This may lead to faster setting and higher early-age strengths.

Setting time. For cementitious systems to be useful to contractors, the setting time of cementitious system must not be too early or too late. Systems that set too early could require removal as there would not be sufficient time to consolidate and finish. Systems that take too long to set are costly as crews have to wait to consolidate and finish the concrete. Thus, being able to control this characteristic is critical in getting RHA more readily accepted in the study. Figure *4a)* and *4b)* show the initial and final setting times, respectively, of the control and RHA-cement mixtures as a function of RHA content. The results show that both initial and final setting times for the AR RHA increase with increasing

AR RHA contents. This is what is commonly found with other SCMs and is why contractors are generally reluctant to use SCMs. However, the t-RHA and t-RHA+PVA systems show a significant reduction in setting time. This decrease in setting time corresponds to the effect of the alkali and sodium silicate compounds that can accelerate the hydration kinetics of the cementitious material. Sodium silicate can be a rapid setting accelerator for cementitious systems (Schutz, 1981). In addition, it is believed that the smaller particles in the t-RHA lead to larger interfacial surfaces, providing more location for hydration to occur, thereby accelerating the set. The 1% addition of PVA of the composites shows no significant effect on both initial and final setting time.

Flowability. The flowability of mixtures with different contents of RHA is shown in Figure 5*a)* and 5*b)*. It is shown that substituting portland cement with either AR RHA or t-RHA reduces the flowability of mortar. Increasing RHA contents results in decreasing flow values. However, the t-RHA exhibits less reduction (about 19% reduction for 10% RHA systems and about 22% reduction for 15% RHA systems) in the flow compared to the AR RHA. The flow of mixtures with and without the addition of PVA does not seem to significantly affect the flowability of the system.

Compressive strength. Figure 6*a)* shows the effects of AR RHA content on the 1-day, 7-day, and 28-day compressive strength. Results indicate that when adding the AR RHA in cement systems, the compressive strength development is retarded. Increased contents of AR RHA lead to more delay of early-age compressive strength development. The increased contents of AR RHA from 0% to 10% and 15% can reduce the 28-day compressive strength by 37% and 42%, respectively. The effects of replacing cement with t-RHA and t-RHA+PVA are shown in Figure 6*b)* and 6*c)*. Results indicate that increased contents the t-RHA from 10% to 15% in the cement systems exhibits no significantly effect on compressive strength development. The 1- and 7-day compressive strength of t-RHA (+PVA) exhibits significantly higher than the AR AHA, and similar or higher that the control. The 28-day compressive strength of t-RHA (+PVA) systems shows no significant improvement compared with the AR RHA systems. The results can be concluded that the chemical transformation of RHA morphology has significantly enhanced compressive strengths at 1 day and 7 days, but exhibits less effect on 28-day compressive strength. The challenge of adding RHA into cement systems due to delayed early-age strength development is reduced by using this method.

CONCLUSIONS

A novel method to eliminate the effect of the mesoporous morphology of RHA systems using chemical transformation method of RHA morphology was developed in this study. The results indicate that mean particle size of RHA is reduced after processing (i.e., transforming). After transformation, the mean particle size is reduced, increasing its surface areas, which allows for tailoring replacement levels for specific construction applications. The concentration of calcium ions is lowered but the concentration of aluminate ions is higher as the content of RHA increases. Faster setting, improved flow, and faster compressive strength development at early-ages for the t-RHA-cement systems was observed. Research is on-going to further develop the method.

ACKNOWLEDGEMENTS

REFERENCES

Azevedo, A. A., M. L. C. Martins, and D. C. D. Molin (2001). A Study of the Penetration of Chloride Ions in Rice-Husk Ash Concrete. *Third CANMET/ACI Intern. Symp. on Sustainable Development of Cement and Concrete*. V. M. Malhotra. San Francisco, CA, ACI: 379-396.

Begarin, F., S. Garrault, A. Nonat, and L. Nicoleau (2011). "Hydration of Alite Containing Aluminum." *Adv. Applied Ceram.* 110(3): 127-130.

Bui, D. D., J. Hu, and P. Stroeven (2005). "Particle Size Effect on the strength of Rice Husk Ash Blended Gap-Graded Portland Cement Concrete." *Cem. & Con. Comp.* 27(3): 357-366.

Chandrasekhar, S., P. N. Pramada, P. Raghavan, K. G. Satyanarayana, and T. N. Gupta (2002). "Microsilica from Rice Husk as a Possible Substitute for Condensed Silica Fume for High Performance Concrete." *J. Mater. Sci. Letters* 21(16): 1245-1247.

Chandrasekhar, S., K. G. Satyanarayana, N. Pramada, and P. Raghavan (2003). "Review Processing, Properties and Applications of Reactive Silica from Rice Husk—An Overview." *J. Mater. Sci.* 38(15): 3159-3168.

Datta, A., and R. K. Rajamani (2002). "A Direct Approach of Modeling Batch Grinding in Ball Mills Using Population Balance Principles and Impact Energy Distribution." *Inter. J. Mineral Processing* 64(4): 181-200.

Gastaldini, A. L. G., G. C. Isaia, N. S. Gomes, and J. E. K. Sperb (2007). "Chloride Penetration and Carbonation in Concrete with Rice Husk Ash and Chemical Activators." *Cem. Con. Comp.* 29: 176-180.

Halim, S. C. (2008). Application of Reactive and Partly Soluble Nanomaterials. *Department of Chemistry and Applied Biosciences*. Zurich, ETH Doctor of Science.

Kalapathy, U., A. Proctor, and J. Shultz (2000). "Silica Xerogels from Rice Hull Ash: Structure, Density and Mechanical Strength as Affected by Gelation pH and Silica Concentration." *J. Chem. Tech. & Biotech.* 75(6): 464-468.

Kalapathy, U., A. Proctor, and J. Shultz (2000). "A Simple Method for Production of Pure Silica from Rice Hull Ash." *Bioresor. Tech.* 73: 257-262.

Kamath, S. R., and A. Proctor (1998). "Silica Gel from Rice Hull Ash: Preparation and Characterization." *Cereal Chem.* 75(4): 484-487.

Kibriya, T. (2006). *Performance of Blended Cement in High Strength Self Compacting Concrete*. 17th Analysis and Computation Specialty Conf. at Structures, St. Louis, MI, American Society of Civil Engineering.

Lu, D., and S. Wei (1992). "Effect of Grinding Aids on Producing Ultrafine Particles." *Adv. Powder Tech.* 3(1): 47-53.

Maeda, N., I. Wada, M. Kawakami, T. Ueda, and G. K. D. Pushpalal (2001). "Chloride Diffusivity of Concrete Incorporating Rice Husk Ash." *ACI Spec. Pub.* 200: 291-308.

Massazza, F. (2008). Pozzolana and Pozzolanic Cements. *Lea's Chemistry of Cement and Concrete*. P. C. Hewlett. Oxford, UK, Butterworth-Heinemann: 471-635.

Mehta, P. K., and K. J. Folliard (1995). "Rice Husk Ash--a Unique Supplementary Cementing Material: Durability Aspects." *ACI Spec. Pub.* 154: 531-542.

Mehta, P. K., and K. J. Folliard (2002). "Rice Husk Ash--a Unique Supplementary Cementing Material: Durability Aspects." *Adv. Conc. Tech.* 154-28: 531-541.

Mehta, P. K., and P. J. M. Monteiro (2006). *Concrete: Structure, Propertries, and Materials*. New York, McGraw-Hill.

Naiya, T. K., A. K. Bhattacharya, S. Mandal, and S. K. Das (2009). "The Sorption of Lead (II) Ions on Rice Husk Ash." *J. Hazard. Mater.* 163(2-3): 1254-1264.

Nehdi, M., J. Duqette, and A. El Damatty (2003). "Performance of Rice Husk Ash Produced Using a New Technology as a Mineral Admixture in Concrete." *Cem. Con. Res.* 33(8).

Rangaraju, P., and K. C. Harish (2010). Optimization of Use of Rice Husk Ash for Use as SCM in Cementitious Mortars. *Concrete Sustainability Conference*. Tempe, AZ, National Ready Mixed Concrete Association.

Reinschmidt, K., and D. Trejo (2006). "Economic Value of Building Faster." *J. Construc. Eng. Management* 132(7): 759-766.

Rukzon, S., P. Chindaprasirt, and R. Mahachai (2009). "Effect of Grinding on Chemical and Physical Properties of Rice Husk Ash." *Inter. J. Minerals, Metall. and Mater.* 16(2): 242-247.

Schutz, R. J. (1981). Admixtures and Method for Accelerating the Setting of Portland Cement Compositions. U.S., Sika Chemical Corp. 4264367.

Scrivener, K. L. (1984). The Development of Microstructure During the Hydration of Portland Cement. . London, Imperial College London PhD.

Scrivener, K. L., and P. L. Pratt (1984). *Microstructural Studies of the Hydration of C_3A and C_4AF Independently and in Cement Paste* Brit. Ceram. Proc.

Shuichi, S., M. Shoya, and H. Tokuda (1992). "Evaluation of Pozzolanic Activity of Rice Husk Ash." *ACI Spec. Pub.* 132: 495-512.

Snow, R. H., E. Allen, B. J. Ennis, and J. D. Litster (2008). Size Reduction and Size Enlargement. *Perry's Chemical Engineers' Handbook*. R. H. Perry and D. W. Green. New York, McGraw-Hill.

Taylor, H. F. W. (1997). *Cement Chemistry*, Thomas Telford Publishing.

Worrell, E., L. Price, N. Martin, C. A. Hendricks, and L. Ozawa Meida (2001). "Annual Review of Energy and Environment." *Annual Reviews of Energy and the Environment* 26: 303-329.

Zhang, M. H., and V. M. Malhotra (1996). "High-Performance Concrete Incorporating Rice Husk Ash as a Supplementary Cementing Material." *ACI Mater. J.* 93(6): 629-636.

Table 1. Chemical composition of portland cement and RHA

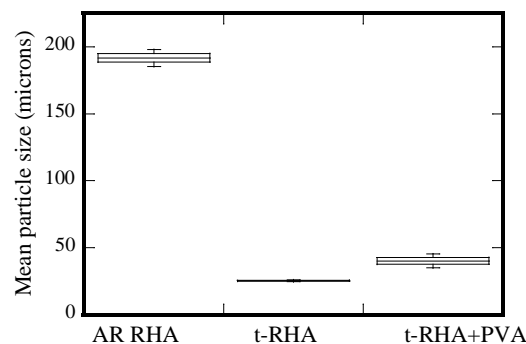| Composition (%)	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	CaO	LOI	IR	CO_2	Limestone	Na_{eq}
portland cement	20.3	4.8	3.5	0.7	2.8	63.9	2.6	0.11	1.8	3.2	0.54
RHA	89.65-96.90	0.006-0.039	0.006-0.052	0.13-0.53	0.018-0.24	0.48-0.81	-	-	-	-	-

Figure 1. Mean particle size of AR RHA, t-RHA transformed with 2M NaOH for 3 hours, and t-RHA transformed with 2M NaOH + 1% PVA for 3 hours.
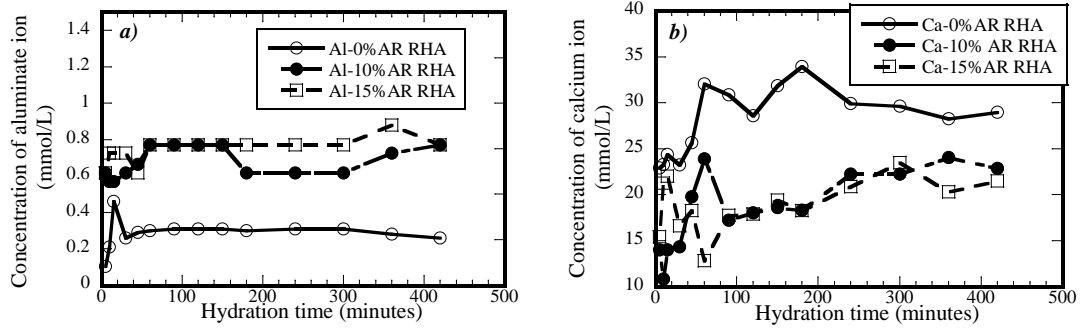
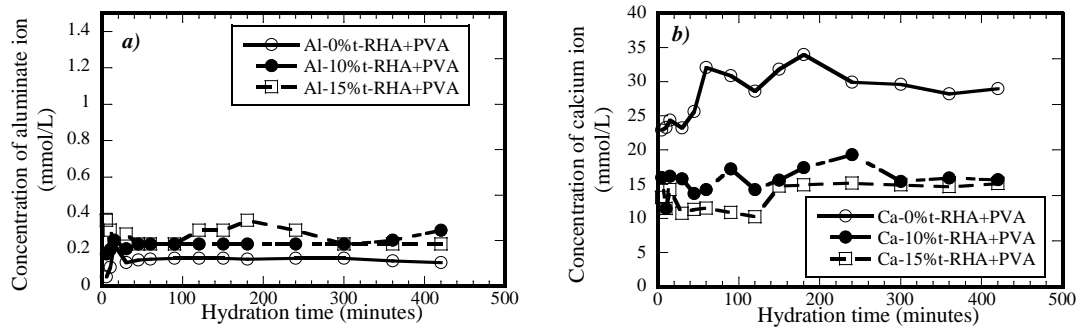**Figure 2. Concentration of *a)* aluminate ion and *b)* calcium ion in AR RHA-cement systems.**

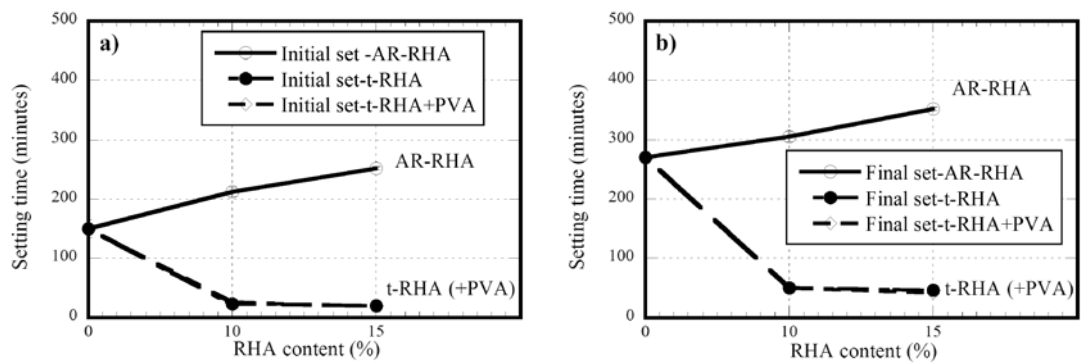**Figure 3. Concentration of *a)* aluminate ion and *b)* calcium ion in t-RHA+PVA/cement systems.**

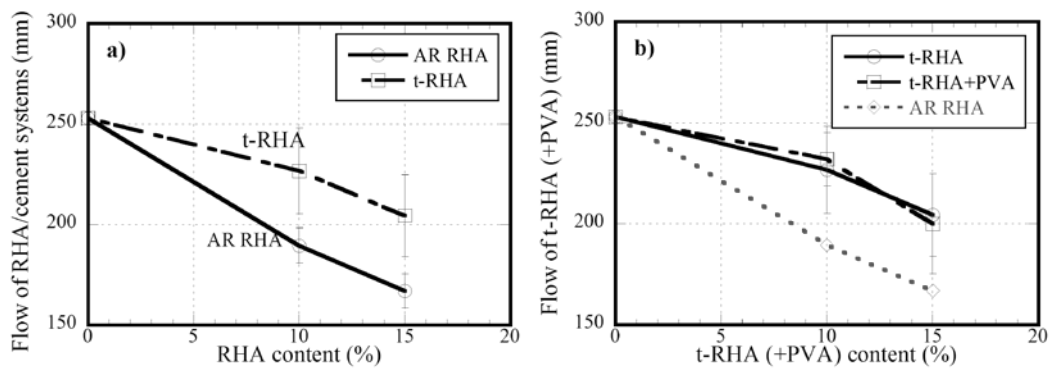**Figure 4. *a)* Effect of RHA content on initial setting time and *b)* final setting time.**

Figure 5. *a)* **Effect of AR RHA and t-RHA and** *b)* **t-RHA (+PVA) content on flowability.**
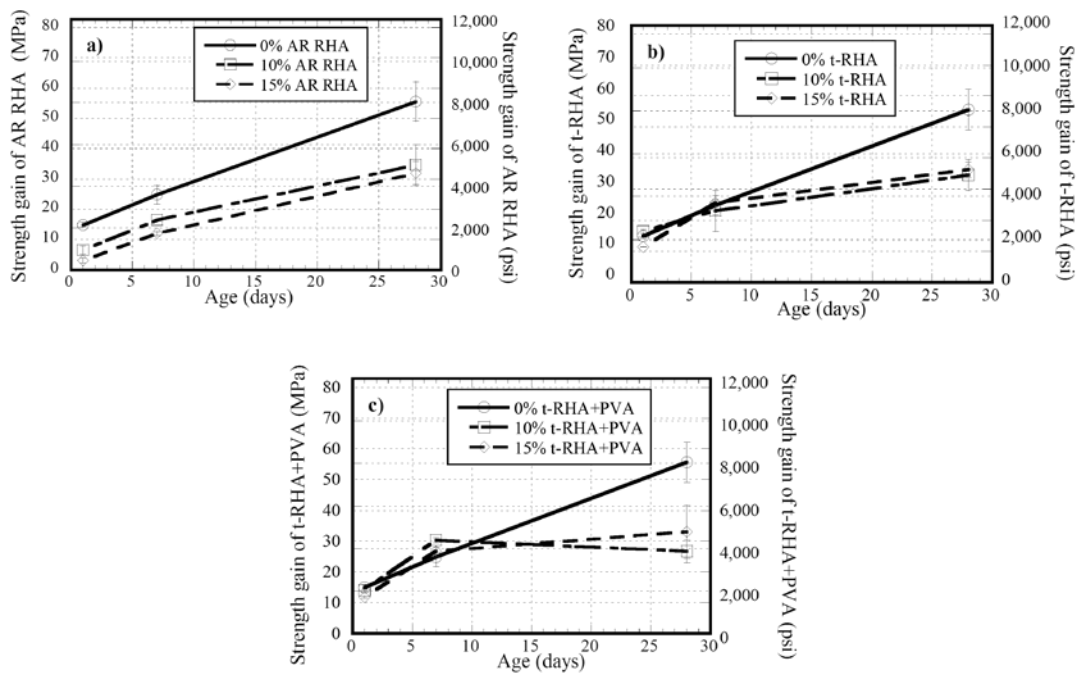

Figure 6. *a)* **Effect of AR RHA,** *b)* **t-RHA, and** *c)* **t-RHA+PVA content on 1-, 7-, and 28-day compressive strength.**