The Effects of Resonant Acoustic Mixing on the Microstructure of UHPC

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Abstract:

As interest grows to produce ultra-high performance cementitious (UHPC) materials that are more durable and economical, the mixing process becomes the essence of producing high quality UHPC. Exploring other mixing technologies could be a way to increase the quality of production. Consequently, with any new mixing technology, adequate research is needed to explore the effects a new mixing technology could have on UHPC. In this research study, we present a novel type of UHPC mixing technology called Resonant Acoustic® Mixing (RAM) Technology. RAM combines the principles of reciprocating movement agitation and acoustic streaming micro-mixing by applying a high frequency acoustic pressure wave to induce mixing. We study the effects of RAM on the microstructure of a designated UHPC mix through back-scattering electron scanning microscopy (BSE-SEM) and mercury intrusion porosimetry (MIP). Our results show that RAM mixing produces a dense UHPC matrix with low porosity.

Keywords: UHPC, Resonant Acoustic Mixing, SEM, microstructure, mixing

1. Introduction

One of the most critical aspects to assembling multi-phase and multi-scale materials is the mixing process, which includes the type of mixing device and the mixing energy that this device brings to the system. Current mixing technologies for the concrete industry have not changed much in the last century relying heavily on planetary type mixers with shearing tools that provide low mixing energies to the system (Dils, De Schutter and Boel 1-11; Rupnow et al. 1-96). High-intensive mixers that provide high energy input to the system and have power consumption instrumental capabilities have advanced the field of high performance concretes and concretes with supplementary cementitious materials (Chopin, de Larrard and Cazacliu 2237-2243; Chopin et al. 897-907); yet they still rely on tool agitation as a means for mixing. These types of mixing instruments may not be able to handle the mixing energy demands of the next generation of cementing agents and construction solutions. Exploring mixing technologies used in other fields(Paul, Atiemo-Obeng and Kresta) and building upon these ideas to create new mixing technologies with smart or intelligent functionalities(Moreno Juez, Artoni and Cazacliu 477-487; Wenzel and Górak ; Danilevskii, Korobko and Terekhov 338-345) built-in is one possible avenue to take. This is especially important for creating smart construction materials with carbon nanoparticle inclusions [14-16], whose material properties require high energy mixing for proper dispersion.

As interest grows to produce ultra-high performance cementitious (UHPC) materials that are more durable and economical, the mixing process becomes the essence of producing high quality UHPC. Consequently, with any new mixing technology, adequate research is needed to explore the effects a new mixing technology could have on a UHPC.

This research study presents a novel type of UHPC mixing technology called Resonant Acoustic® Mixing (RAM) Technology. The effects of RAM on the microstructure of a designated UHPC mix through back-scattering electron scanning microscopy (BSE-SEM) and mercury intrusion porosimetry (MIP) are studied and compared to UHPC samples mixed with a conventional table top paddle mixer. Conclusions are then made.

2. Background

The cement and concrete industry mainly rely on impeller agitation mixers where the main mixing mechanisms are shear and convection (Lacey 257-268) via a blade or paddle tool. More in-depth reviews of these types of concrete mixing technologies are found in (Dils, De Schutter and Boel 1-11; Saleh and Teodoriu 388-401). However, a recent study by Remus et al. (Remus, Roessler and Ludwig) used ultrasound-assisted mixing to assess spread and compressive strength properties. They found that the compressive strength increased, while the spread decreased depending on the strength of the ultrasound mixing.

The resonant acoustic mixer (RAM) used in the experimental program is presented in Figure 1. RAM does use an impeller tool to mix its material, but instead uses reciprocal vertical movement that induces bulk and micro-mixing zones to mix. A more detailed explanation about the system is found in (Vandenberg and Wille 716-730).

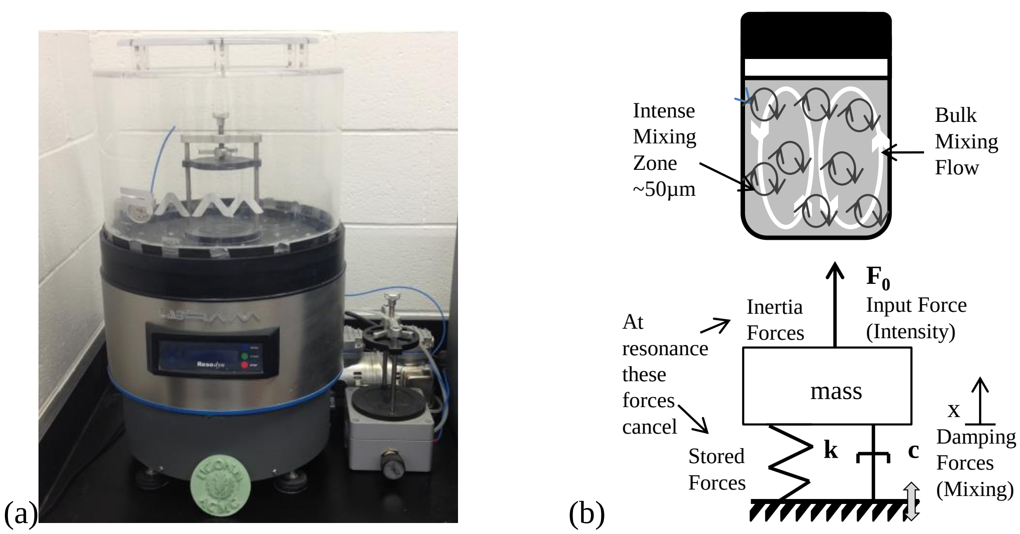

Figure 1 (a) The acoustic laboratory size mixer LabRAM and (b) the schematic of its mixing technology (S L Coguill).

3. Testing Methods

3.1. Materials

Type I white cement conforming to ASTM C150 (ASTM C150 / C150M-15, Standard Specification for Portland Cement) was used in all UHPC mixtures. A commercially available high-range water reducer conforming to ASTM C494 (ASTM C494 / C494M-15, Standard Specification for Chemical Admixtures for Concrete) Type A & F polycarboxylate (PCE) superplasticizer (SP), with specific gravity of 1.06 and solid content of 29%, was used at 1% by weight of cement (bwoc). White silica fume (SF) and quartz powder (QP) were used as secondary cementitious and filler materials, respectively. Two different sizes of aggregates were used, which consisted of fine grade quartz sand (QS1) with a medium particle size of d_{50} ~0.10 mm and a maximum size of d_{max} ~0.21 mm and a coarse grade quartz sand (QS2) with a medium particle size of d_{50} ~0.42 mm and a maximum size of d_{max} ~0.60 mm.

3.2. Mixture Compositions

The mixing proportions for the UHPC mixtures were based off the recommendations provided in (Wille, Naaman and Parra-Montesinos 46-54). The silica fume and silica powder proportions remained the same at 25 mass% of the cement. They did not replace part of the cement but were added to enhance the solid volume fraction of the UHPC. The water-to-cement (w/c) ratio was 0.21.

3.3. Mixing Methods

UHPC specimens were prepared using a four-step mixing protocol presented in Figure 2. First the dry materials were mixed together from lowest to highest bulk volume density. Then the liquid materials were added to complete the mixing. The time of wet mixing for the RAM specimens was 5 minutes and for the table top paddle mixer it was around 20 minutes.

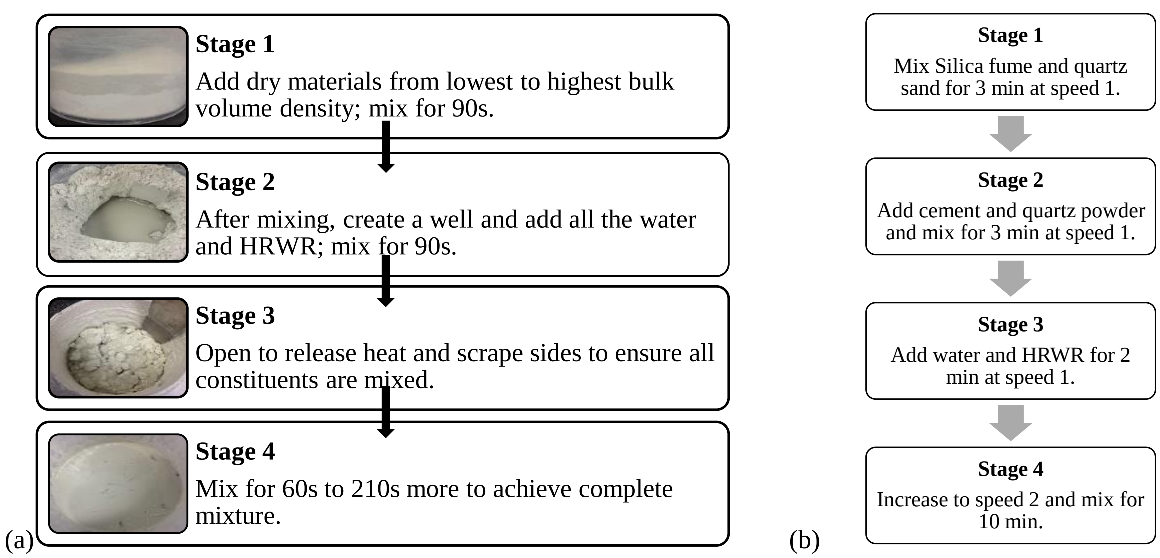

Figure 2 The 4-stage mixing protocols for a) the resonance acoustic mixer and b) the table top paddle mixer.

3.3. Microstructure Testing Methods

3.3.1. Scanning Electron Microscopy

For the back-scattering scanning electron microscopy experiments, a piece of a specimen was collected and impregnated using epoxy-based EPO-TEK® 301 resin and polished using diamond sprays of decreasing sizes (9, 3, and 1 µm) with petrol as a lubricant. The sample was coated with a 20–30 nm carbon film using a Leica EM CED030 Carbon Thread Evaporation Device. It was left an a desiccator for 24 hours before testing.

3.3.2. Mercury Intrusion Porosimetry

Mercury intrusion porosimetry (MIP) was used to characterize the pore structure of the specimens. This technique is based on the intrusion of a non-wetting mercury into the connected pore structure under increasing pressure. Samples were crushed to obtain a mass of 1g. The dried samples were placed in the dilatometer and the air was removed. The measurement was in two steps for two different populations of the pores. In the first step a pressure of 100kPa was applied to intrude the larger pores. In the second step, the pressure was increased up to 400 MPa which allowed the mercury to intrude pore entries down to 2 nm. The Washburn equation was used to relate the pressure to the entry pore size.

4. Results

The BSE-SEM results for one specimen are presented in Figure 3 and Figure 4. Figure 3 shows the matrix and the hydration regions around un-hydrated grains, while Figure 4 shows the interfacial transition zone (ITZ) between the matrix and a sand aggregate.

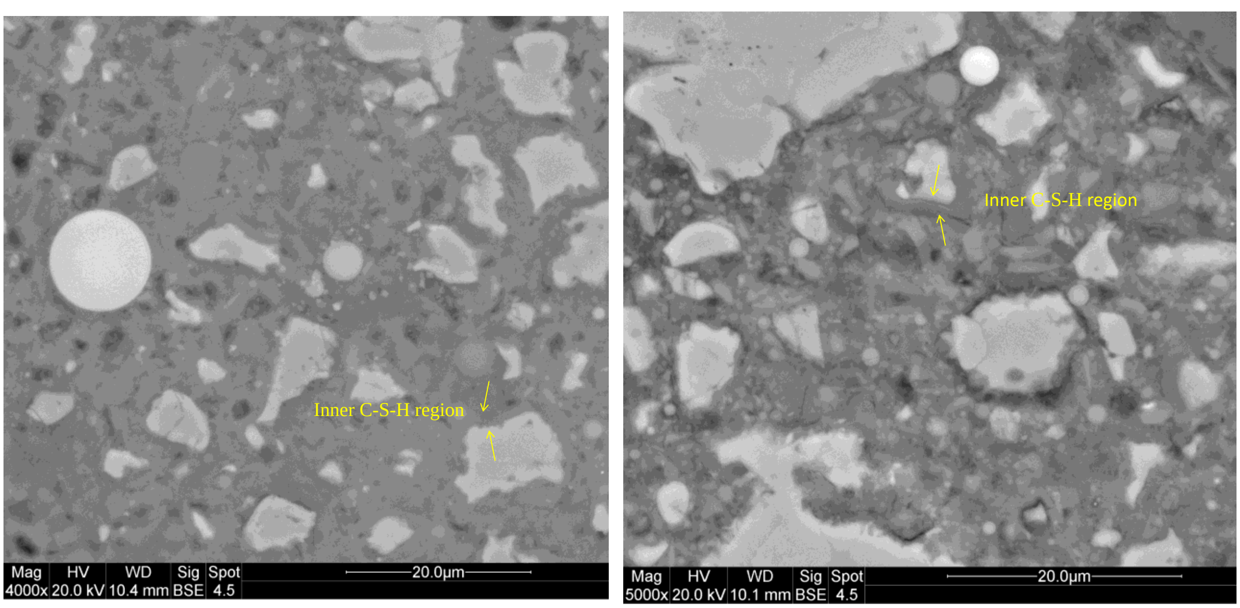

Figure 3 Inner C-S-H region is lighter around the Hobart (left) than the RAM (right) for the samples.

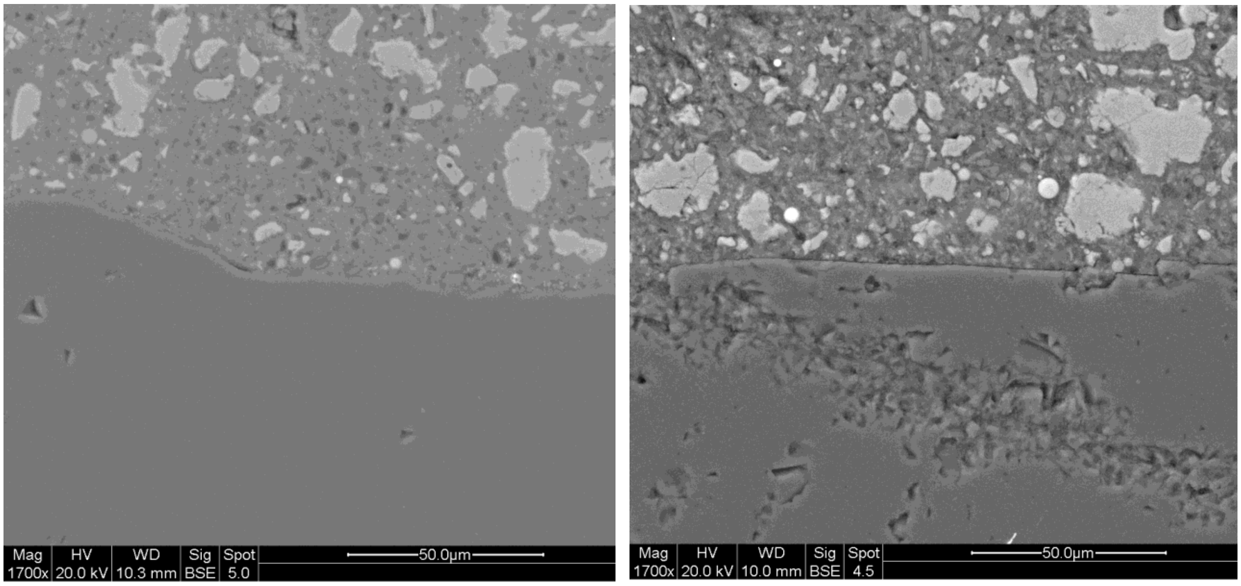

Figure 4 The interfacial transition zone for a Hobart (left) and a RAM (right) specimen.

The results for the MIP are shown in Figure 5. The area underneath the curve relates to the volume of pore entries in the specimen. From the data, it appears that the RAM has a larger area than the paddle mixed samples. This implies that the RAM mixed specimens contain higher volumes of smaller pore sizes than the paddle mixed specimens.

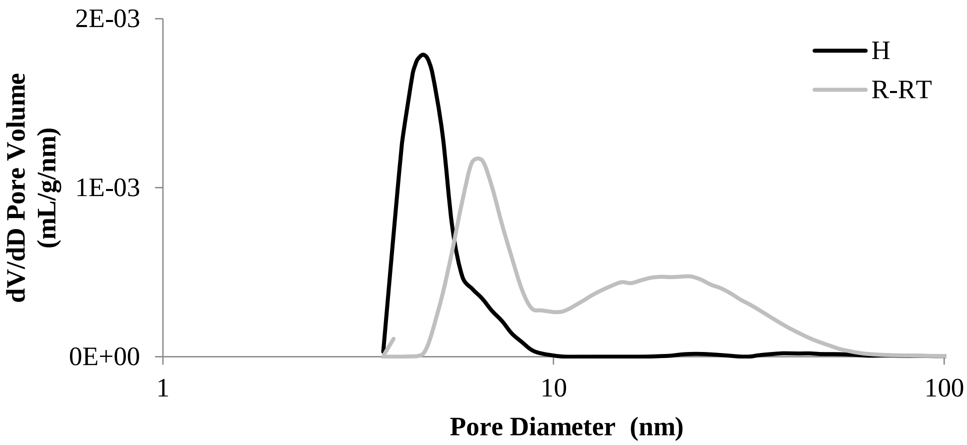

Figure 5 MIP results for UHPC mixed with a paddle mixer (H) and with RAM at room temperature (R-RT).

Visual inspections of the porosity of a RAM sample and a paddle mixed sample are given in Figure 6 as complimentary data to the MIP results. The paddle mixed sample shows large air bubbles, while the RAM sample shows very little.

(a) Hobart (b) Resodyn LabRAM

Figure 6 A visual inspection of the hardened state.

5. Discussion

Previous research by (Vandenberg and Wille 716-730) established that while the principle of RAM is not shear mixing, results from intensive shear mixing can still be applied to better understand and discuss the nature of the microstructural development of the UHPC in this study.

It is known that high mixing intensity accelerates the hydration kinetics and changes the physical and chemical nature of cement paste (Han and Ferron 95-106; Han and Ferron 278-288; Juilland et al. 1175-1188). These changes result in an increase in rheological properties, a decrease in workability, and increase in thixotropy, and sometimes an increase in mechanical strength. Thus, while RAM does not incorporate shear mixing by impeller action into the mixing material, it does introduce a velocity profile of micro-mixing and bulk-mixing zones. Initially, this profile is from frictional forces between particles, but as the system evolves into a paste and then a suspension, the velocity profile becomes a moving fluid, moving at an acceleration up to 100 times gravitational acceleration. This has a large impact on the porosity as vibration is known to reduce air bubbles of a highly viscous fluid (Zhan et al. 76-83). It is also established in the literature that the color of a specimen's matrix can be related to the density of the calcium-silicate-hydrates (C-S-H) and thus its mechanical properties (Scrivener et al. 375).

From the RAM microstructural results presented in this research there appears to be an increase in abrasion changes to the sand aggregates indicating stronger frictional forces are experienced during the mixing process. Additionally, both a reduction in the air bubbles in the hardened matrix, and a darkening of the C-S-H around unhydrated grains were found for the RAM specimens compared to a conventional concrete paddle mixer. These results suggest that RAM mixing changes the hydration kinetics and the physical and chemical microstructure.

6. Conclusions

In this research, the effects of RAM on the microstructure of a designated UHPC mix through back-scattering electron scanning microscopy (BSE-SEM) and mercury intrusion porosimetry (MIP) were presented. A table top paddle mixer was used as a comparison tool. The results show that RAM mixing produces a dense UHPC matrix with low porosity and suggest RAM mixing changes the hydration kinetics and the physical and chemical microstructure.

7. References

Standard Specification for Portland Cement, ASTM C150, ASTM International, Volume 04.01, West Conshohocken, PA, 2015.

Standard Specification for Chemical Admixtures for Concrete, ASTM C494, ASTM International, Volume 04.02, West Conshohocken, PA, 2015.

Chopin, David, et al. "Monitoring of Concrete Homogenisation with the Power Consumption Curve." *Materials and Structures*, Vol. 40, No. 9, February 2007, pp. 897-907.

Danilevskii, L. N., et al. "Vibroacoustic Monitoring of the Homogeneity and Workability of Concrete Mixes by their Hydromechanical State in the Process of Mixing." *Journal of Engineering Physics and Thermophysics*, Vol. 82, No. 2, March 2009, pp. 338–345.

Dils, J., et al. "Influence of Mixing Procedure and Mixer Type on Fresh and Hardened Properties of Concrete: A Review." *Materials and Structures*, Vol. 45, No. 11, November 2012, pp. 1673–1683.

Han, Dongyeop, and Ferron, Raissa Douglas. "Effect of Mixing Method on Microstructure and Rheology of Cement Paste." *Construction and Building Materials*, Vol. 93, No. 9, September 2012, pp. 278-288.

Han, Dongyeop, and Ferron, Raissa Douglas. "Influence of High Mixing Intensity on Rheology, Hydration, and Microstructure of Fresh State Cement Paste." *Cement and Concrete Research*, Vol. 84, No. 6, June 2016, pp. 95-106.

Juilland, Patrick, et al. "Effect of Mixing on the Early Hydration of Alite and OPC Systems." *Cement and Concrete Research*, Vol. 42, No. 9, September 2012, pp. 1175-88.

Lacey, P. M. C. "Developments in the Theory of Particle Mixing." *Journal of Applied Chemistry*, Vol. 4, No.5, May 1954, pp. 257-68.

Moreno Juez, Jaime, et al. “Monitoring of Concrete Mixing Evolution using Image Analysis.” *Powder Technology*, Vol. 305, No. 1, January 2017, pp. 477-487.

Paul, E. L., et al. *Handbook of Industrial Mixing: Science and Practice*. Wiley, 2004.

Remus, Ricardo et al. "Power Ultrasound-Assisted Concrete Production." Tromsø, Norway, March 6-8, 2017.

Rupnow, Tyson D., et al. *Improving Portland Cement Concrete Mix Consistency and Production Rate through Two-Stage Mixing, TR-505*. The Department of Transportation, 2007.

Coguill, S.L., and Martineau, Z.R. *Vessel Geometry and Fluid Properties Influencing Mix Behavior for ResonantAcoustic ® Mixing Processes*. 38th International Pyrotechnics Seminar, Denver, CO, June 2012.

Saleh, Fatemeh K., and Teodoriu, Catalin. "The Mechanism of Mixing and Mixing Energy for Oil and Gas Wells Cement Slurries: A Literature Review and Benchmarking of the Findings." *Journal of Natural Gas Science and Engineering*, Vol. 38, No. 2, February 2017, pp. 388-401.

Scrivener, Karen, et al. *A Practical Guide to Microstructural Analysis of Cementitious Materials*. Taylor & Francis, 2016.

Vandenberg, Aileen, and Kay Wille. "Evaluation of Resonance Acoustic Mixing Technology using Ultra High Performance Concrete." *Construction and Building Materials*, Vol. 164, No. 3, March 2018, pp. 716-30.

Wenzel, Dennis, and Andrzej Górak. "Review and Analysis of Micromixing in Rotating Packed Beds." *Chemical Engineering Journal*, Vol. 345, No. 8, 2018, pp. 492-506.

Wille, Kay, et al. "Ultra-High Performance Concrete with Compressive Strength Exceeding 150 MPa (22 ksi): A Simpler Way." *ACI Structural and Materials Journal*, Vol. 108, No. 1, January 2011, pp. 46-54.

Zhan, Xiaobin, et al. “Removal of Gas Bubbles from Highly Viscous Non-Newtonian Fluids using Controlled Vibration.”, Vol. 185, No. 11, November 2018, pp. 76-83.

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