EVALUATION REPORT OF HESS PUMICE

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INTRODUCTION

As contracted, the University of Utah has performed a series of experimental tests on Hess Pumice for use as natural pozzolan in concrete. Three grades of pumice labeled DS-200, DS-325 and Ultrafine pumice was provided by Hess Pumice Products and a control ASTM Type II/V portland cement, readily available in Utah and Idaho, were used for the testing.

This research was conducted to determine pozzolanic properties and complimentary cementitious capability of the above named pumice products for use in combination of portland and hydraulic cements. According to ASTM C 618, pozzolans are the siliceous and aluminous material in finely divided form and in the presence of moisture, at ordinary temperature chemically react with calcium hydroxide to form compounds possessing cementitious properties. Complimentary cementitious materials are the materials that provide micro-substrate materials or catalytic effects for the more efficient hydration of other cementitious material. Pumice is characterized by defining its base chemical and physical characteristics, hydration kinetics and the mixture design properties of concrete containing pumice. Five combinations of mixture designs with pumice blended with a Type II/V were examined. A control mixture with 100% cement, three mixtures with 20% cement replaced by DS200, DS325 and Ultrafine (different grade pumice) and one mixture with 30% replacement by DS325.

CHEMICAL AND PHYSICAL MATERIAL CHARACTERIZATION

It is necessary to understand the chemical and physical characteristics of pumice materials to predict and optimize the use of these materials. The materials were evaluated using X-Ray Diffraction (XRD) to determine the mineralogical composition and the chemical composition was determined using X-Ray Fluorescence (XRF). The particle size distribution of each of the products was determined using a laser diffractometer, providing a size spectrum from 0.02-2000μm. Particle shape was characterized by microscopic techniques using a 600x controlled optics microscope and the images from scanning electron microscope (SEM).

(a) MINERALOGICAL COMPOSITION OF PUMICE

X-Ray Diffraction was performed on sample of pumice from a range of 5 to 90 degrees 2Φ. It has been confirmed by XRD analyses that pumice tested are more than 99% amorphous by the halo shaped diffusion band. There is no peak in the signature, which indicates the pumice has no well-defined crystalline minerals. It also shows the vitreous/glassy nature of material. Whereas in cement, well defined peaks were observed along a level baseline. The amorphous nature of pumice is corroborated by XRD results of different grades of pumice. X-ray diffraction patterns of DS-200 pumice and cement is shown in Fig. 1(a) and (b) respectively.

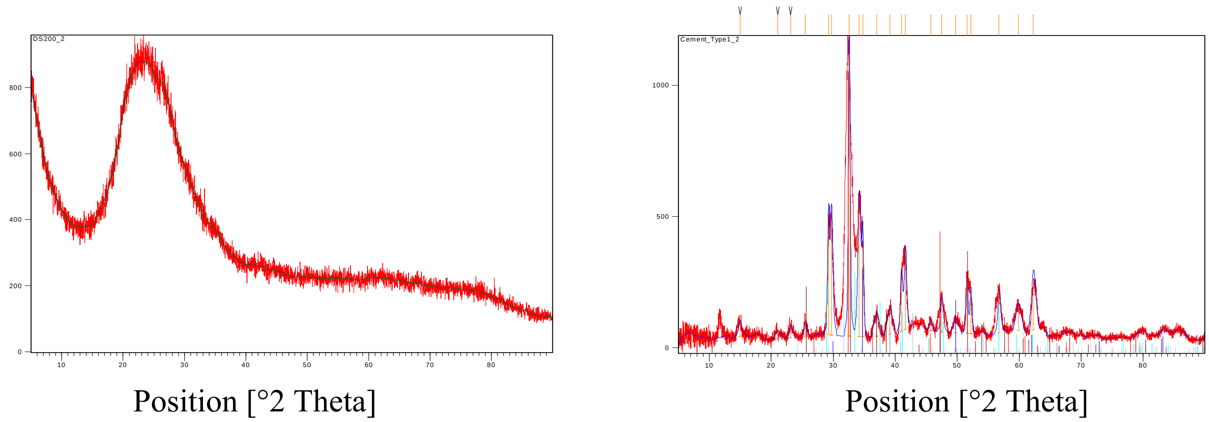


Fig. 1 (a) DS200 Pumice XRD Signature (b) Cement XRD Signature

(b) X-RAY FLUORESCENCE

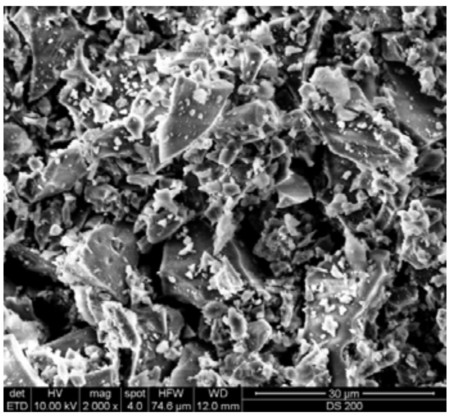
The total chemical composition of different grades of pumice and cement are given in Table 1. Chemical analysis shows that pumice is mainly composed of silica (70%) whereas cement has calcium oxide (62%). ASTM C 618 classifies pumice as a Class N pozzolan (for raw or calcined natural pozzolan) if it meets specific physical and chemical requirements. Class N pozzolan should have a minimum of SiO_2+Al_2O_3+Fe_2O_3 content of 70%, pumice has approximately 80% of these materials. Presence of siliceous and aluminous compounds is evident from chemical analysis results of pumice. From the chemical analysis result, it is evident that all grades of pumice composed of more or less same percentage of elements differ only in particle size, which can be inferred from particle size distribution analysis and scanning electron microscopy. From Table 1, it is inferred that pumice has very high silica, very low calcium, more alumina and alkali content compared to Type II cement.

Table 1: Chemical Analysis Result from XRF test

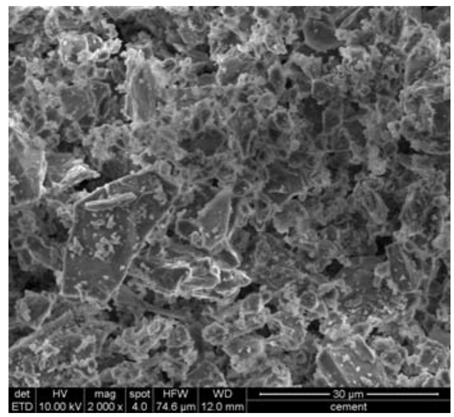
	Type II	**DS200**	**DS325**	**Ultrafine**
SiO_2	20.67	69.09	69.16	69.75
Al_2O_3	3.97	10.63	10.79	11.18
Fe_2O_3	3.65	1.01	1	1.04
CaO	63.57	0.93	0.93	0.97
MgO	1.55	0.09	0.16	0.25
SO_3	2.81	-0.04	-0.04	-0.04
Na_2O	0.06	2.49	2.13	2.34
K_2O	0.72	4.77	5.08	4.79
Cl	0.018	Nil	Nil	Nil
Total	98.43	89.12	89.33	90.42

(c) PARTICLE SIZE DISTRIBUTION AND SCANNING ELECTRON MICROSCOPY

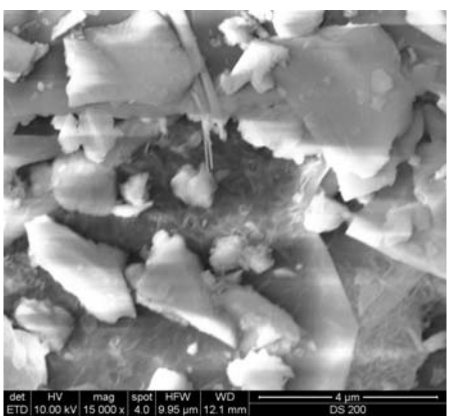
Scanning electron micrographs for DS200 and cement with two different magnifications are shown in Fig. 2. From the image, the glassy nature of pumice is evident and it also illustrates the crushed nature of the material. The particle analysis results for different grades of pumice are shown in Fig. 3(a), (b), (c) and Table 2, was determined using Horiba LA-930 with a size spectra of 0.02-2000 μm. It is clear from the mean diameter of particle, the finest is ultrafine and coarsest is DS200. Ultrafine pumice is approximately four times finer than portland cement.



DS200



Cement



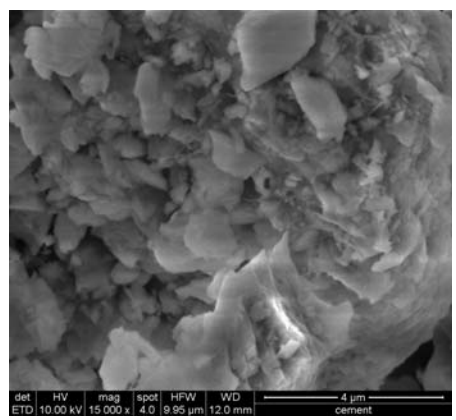


Fig. 2 Scanning Electron Micrograph for DS200 and Cement @ 2000X and 1500X

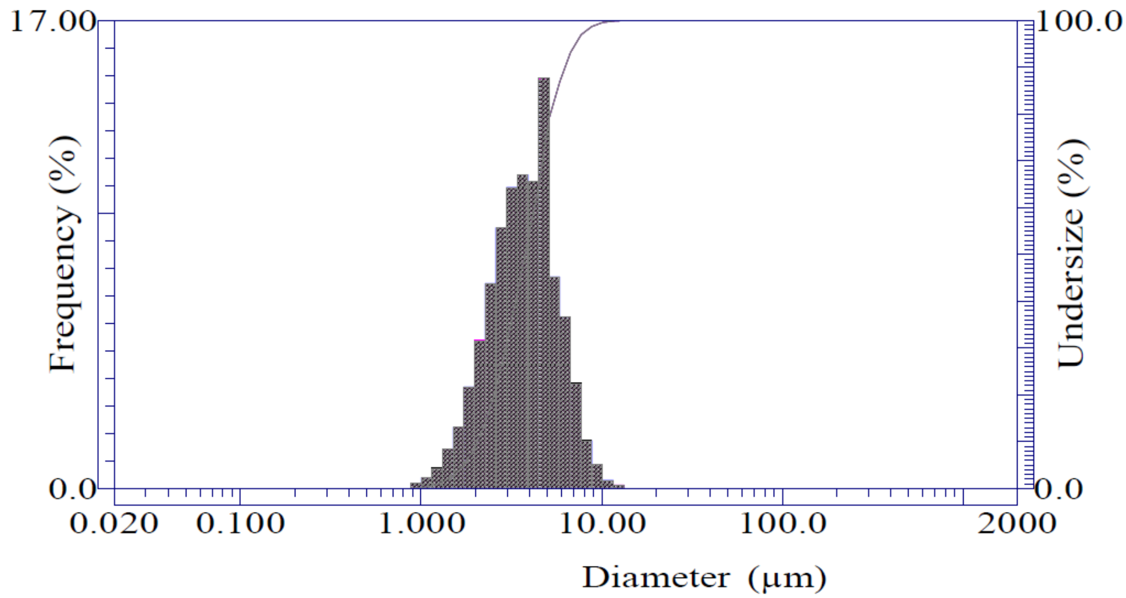


Fig. 3(a) Particle Size Distribution of "Ultrafine" Pumice

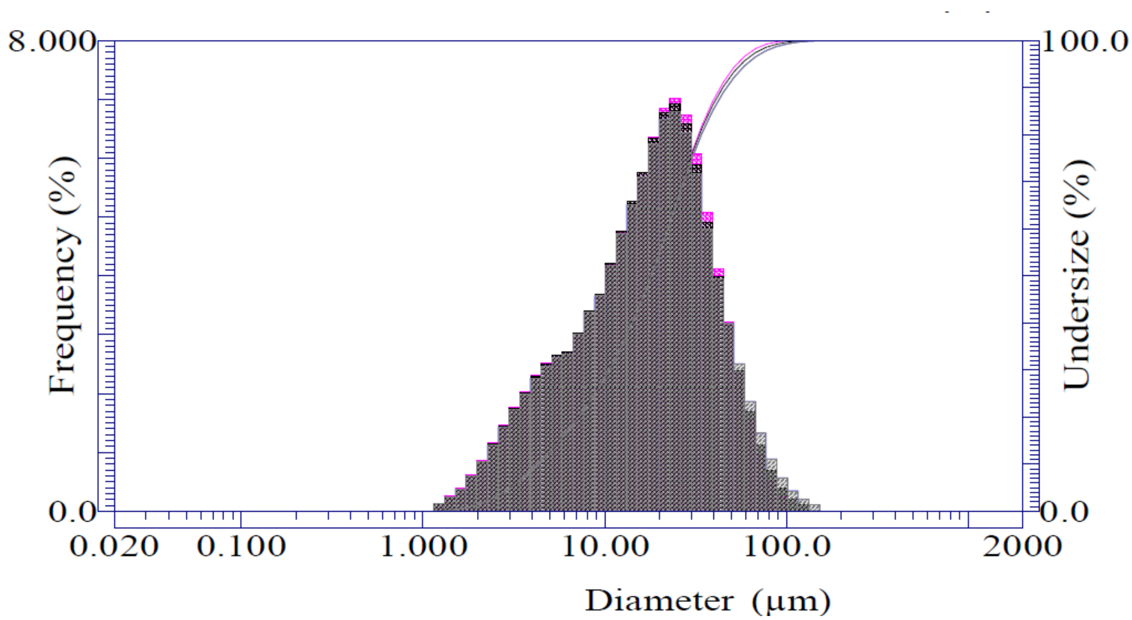


Fig. 3(b) Particle Size Distribution of "DS325" Pumice

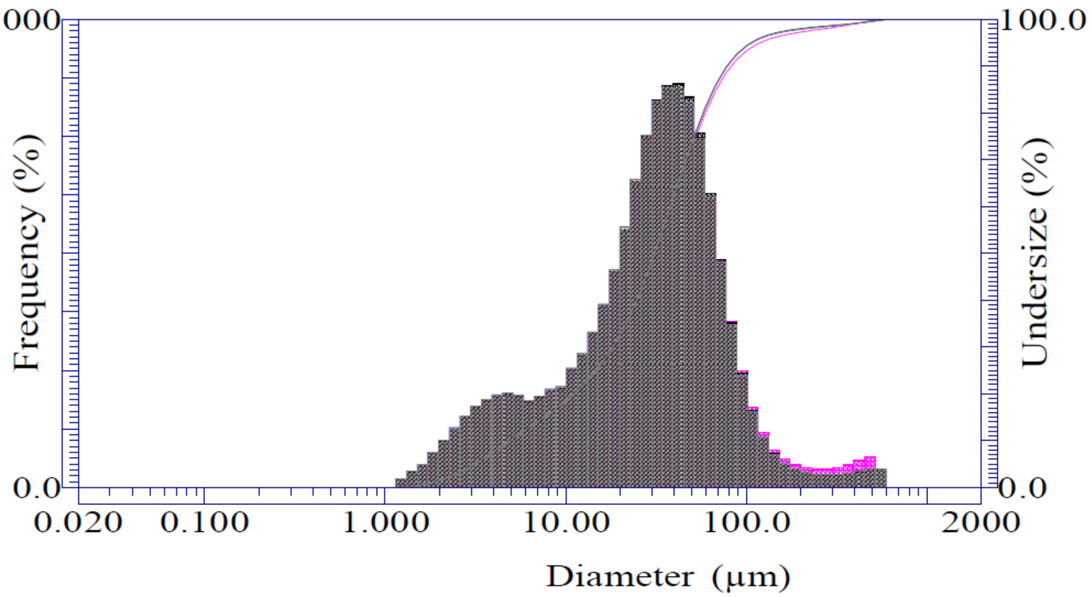


Fig. 3(c) Particle Size Distribution of "DS200" Pumice

Table 2: Particle Size Details of Pumice

Sample	**Ultrafine**	**DS325**	**DS200**
S.P. Area (cm^2/cm^3)	18093	5921.2	4375.4
Median (μm)	3.755	17.788	31.725
Mean (μm)	3.995	21.292	45.369
S.D. (μm)	1.695	16.158	60.756
Mode (μm)	4.711	24.373	41.895
R.R. Index	1.5	1.5	1.5

HYDRATION KINETICS OF PUMICE BLENDED CEMENTS

Cementitious materials generate heat through exothermic hydration reaction. The kinetics of pozzolanic and cementitious reaction can be measured with an isothermal heat conduction calorimeter. A TAM air isolated heat conduction calorimeter was used to analyze 8 pumice combinations with a control cement. The 8 combinations used were 100% portland cement, ASTM Type II/V; 20 and 30% DS200; 10, 20, 30% DS325; 20 and 30% ultrafine pumice. Each of the tests was conducted at 21° C (70° F) for 30 days, and replacement percentages are done by mass.

A sample of 10 g per ampoule with a reference of 10 g per ampoule was used with w/cm ratio of 0.5. Eight combinations were tested at ambient temperatures for 30 days and the results are shown for the first 225 hours in Fig. 4 and 5.

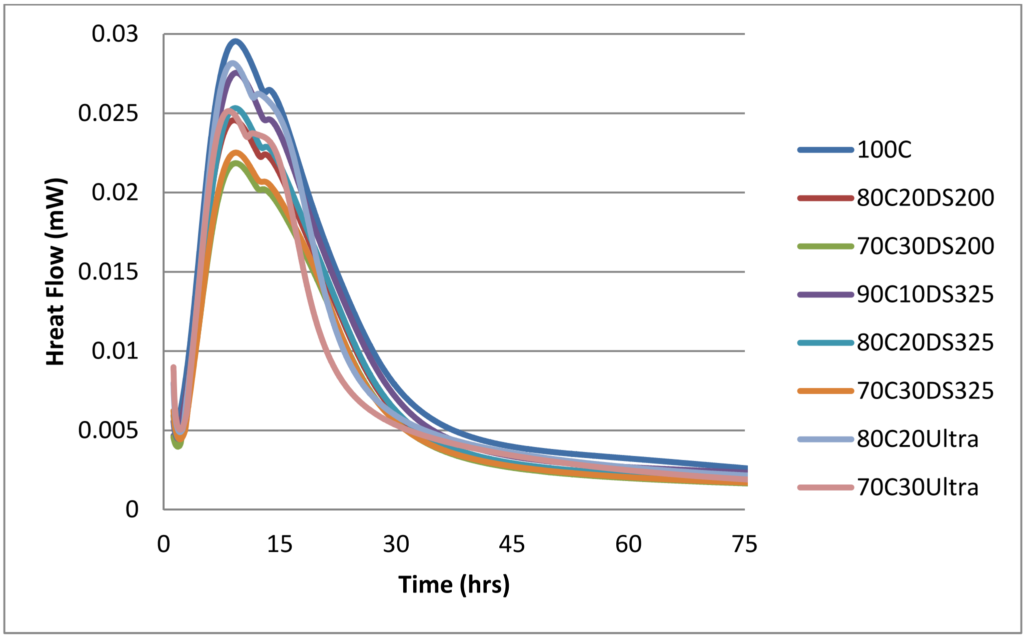


Fig. 4 Heat Flow for different mixture combinations during first 75 hours

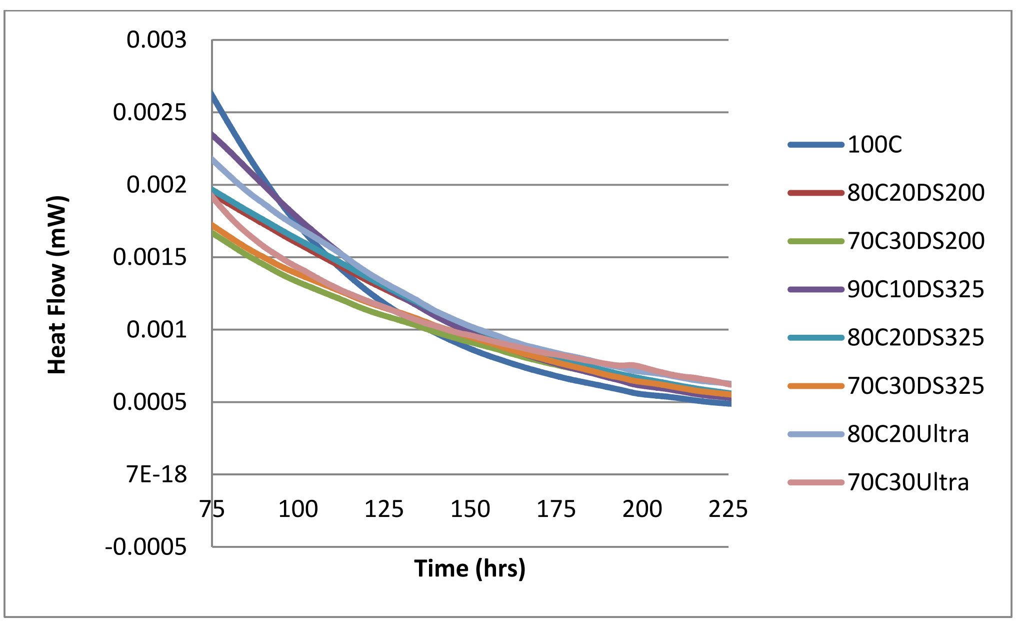


Fig. 5 Heat Flow for different mixture combinations during 75 -225 hours

Fig. 4 shows the heat flow for eight combinations during first seventy five hours and Fig. 5 shows the heat flow between 75 to 225 hours at 70° F. 100% cement mixture produces more heat as compared to the mixtures containing pumice. As the pozzolanic content increases, the main peak of heat flow decreases. Depend on the grades of pumice; the height of main peak varies for same percentage combination of cement and pozzolanic material. 70% cement and 30% DS200 & DS325 produces lowest heat flow respectively among the 8 mixtures whereas 70% cement and 30% ultra produces heat comparable to 80% cement and 20% DS200 & DS325 mixtures. 90% cement and 10% DS325 produces the heat flow comparable to 80% cement and 20% ultra. The calorimeter testing shows that there is limited pozzolanic activity in the first 100 hours for DS200 or DS325. However the ultrafine pumice impacts the early age hydration characteristics and clearly yields more hydration than that of the remaining portland cement. Figure 5 shows that after 100 hours, hydration of 100% cement mixture starts declining whereas for pumice containing mixtures hydrate at greater than the portland cement reactions. This shows the pozzolanic reaction and complimentary effects of different grades of pumice.

CONCRETE MIXTURE DESIGNS WITH PUMICE

By replacing a portion of cement with pumice, many properties of the cementitious system can be influenced, some by physical effects associated with small particles, finer particle size distribution than portland cement and others by pozzolanic and cementitious reactions. The tests described in the next section were conducted under standard laboratory conditions and according to the respective ASTM standards in two phases.

CONCRETE AND MORTAR TESTING - PHASE I & II

1. WORKABILITY

Following ASTM C143, five mixture designs were tested for workability. Five mixtures include 100% cement (ASTM Type II/V), 20% of DS200, DS325, ultrafine pumice with 80% cement and 30% DS325 with 70% cement with a w/cm ratio of 0.485. ASTM Type II/V cement along with different grades of pumice is used as cementitious materials and a polycarboxylate-based mid-range water reducer admixture (Glenium 3030) is used in the mixtures to maintain the slump of 3-5 in. For Mix-3 and Mix-4, it was difficult to control slump loss between 3 and 5 in. Hence the closest result is reported for Mix-3 and Mix-4. The slump loss of five different mixtures in 15 minutes interval is recorded and shown in Table 3 and Fig. 6. Addition of different grades of pumice as cementitious material does not affect the workability of concrete much. After 30 minutes of mixing, slump was around 1 in for four mixtures and 0.6 in for Mix-3 (80C20DS325).

Table 3: Slump Loss Variation versus Time Interval

Time (min)	Slump Loss (in)				
	C	Mix-2	Mix-3	Mix-4	Mix-5
0	3.25	5	2.5	2.7	3
15	1.87	2.37	1.1	1.5	2.25
30	0.87	1.62	0.6	0.9	1.65
45	0.37	0.5	0.2	0.37	1.1

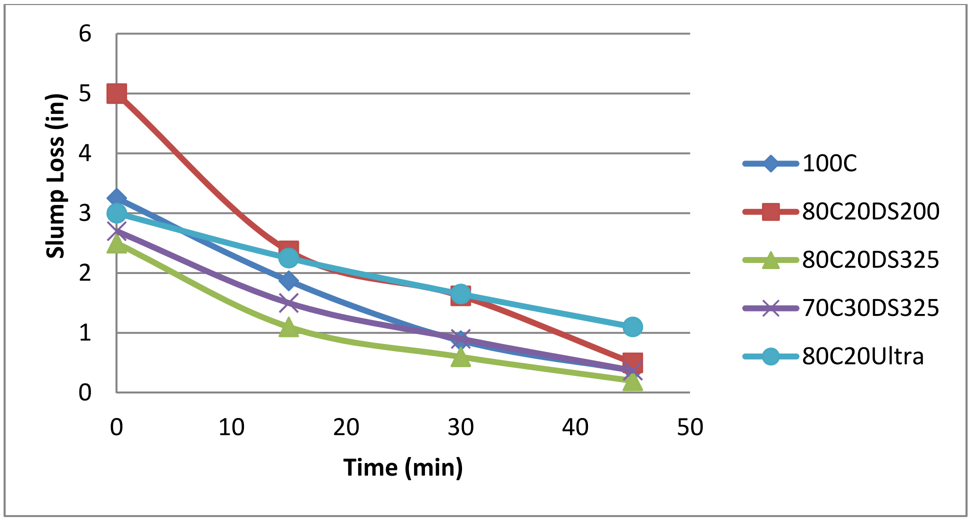


Fig. 6: Slump Loss versus Time Interval

2. BLEEDING

Following ASTM C232 method A, five mixture designs were tested for bleeding and the mixture designs used were same as for workability. In this test method, samples were consolidated by rodding only and then it was tested without any further disturbances. Bleeding of concrete for different mixtures is reported in Fig. 7. Addition of pumice as cementitious material does not produce bleed from the sample of freshly mixed concrete for three mixtures. Mix-4 (70C30DS325) produced a bleeding of around 15 ml which may be due to the addition of more porous material (pumice) in the mixture design.

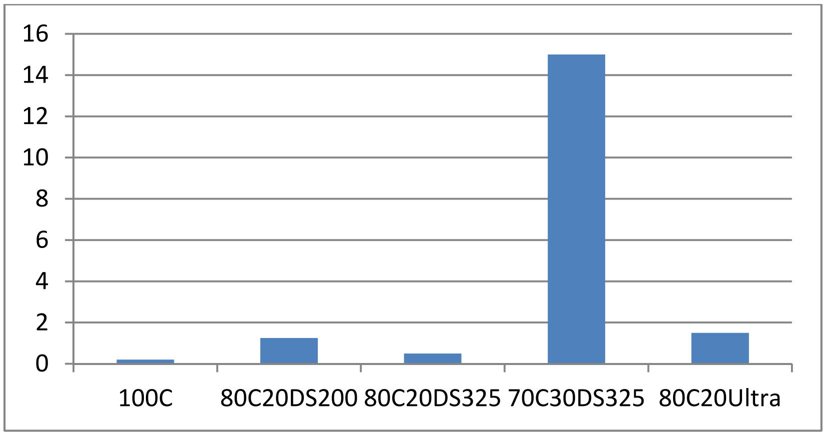


Fig. 7: Bleeding for different mixture designs

3. SETTING TIME

Setting times of five mixtures were determined by Vicat needle test method ASTM C191. The variation of setting time and water requirement for different mixture are presented in Table 4 and Fig. 8. There is an increase in initial and final setting time for the mixtures containing pumice compared to 100% cement (ASTM Type II/V) when tested at a constant flow without admixtures. The increases are well within the limits of ASTM C595 specification for blended hydraulic cement is likely attributed to the increased water demand. Water demand is greater for mixture containing pumice compared to 100% cement. The percentage increase in water demand is shown in Table 4. The grade of pumice which has very small particles (Ultra) has greater water demand compared to grades with comparatively larger particles (DS200 and DS325). Also the mixture which has 30% pumice by mass or total cementitious material had greater water demand due to increase in surface area and also due to porous nature of pumice. The increase in water demand is not a major detriment in concrete production, as it can be addressed by addition of common water reducing admixtures.

Penetration resistance for different mixture combinations over a time period is shown in Fig. 9. Penetration resistance indicates the setting characteristic of cement mixture paste. From Fig. 9 it is clear that 100% cement (100C) mixture has setting characteristic that are slightly sooner as compared to other mixtures and 80% cement with 20% Ultrafine (80C20Ultra) mixture setting characteristic is closer to 100C compared to other pumice mixtures. This indicates 80C20Ultra mixture has the ability to set faster than other pumice mixture combinations.

Table 4: Effect of Pumice on Setting Time and Water Demand

Mixture	Setting Time (min)		Water Used (in g)	% Increase in water
	Initial	Final		
100C	117	242	173	
80C20DS200	143	286	181	4.6
80C20DS325	148	271	195	12.7
70C30DS325	159	315	201	16.2
80C20Ultra	129	323	199	15

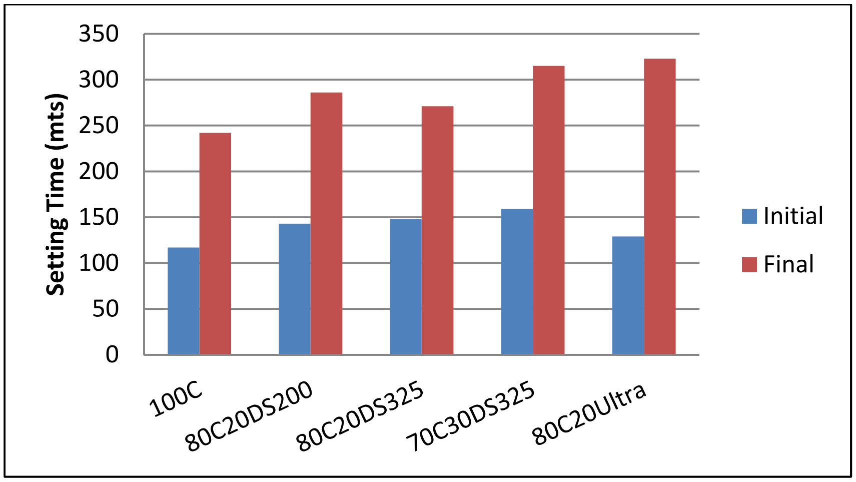


Fig. 8: Effect of Pumice on Setting Time

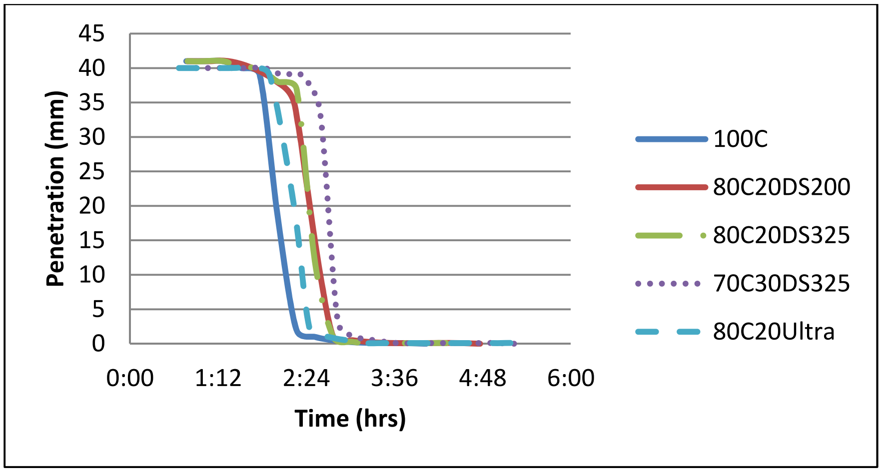


Fig. 9: Penetration resistance of different mixtures

4. STRENGTH DEVELOPMENT

The compressive strength of concrete is one of the primary considerations in concrete mixture design. Following ASTM C39, compressive strength of 4"x8" cylinders were tested with different grades of pumice for five mixtures commonly used for 4 ksi specification. Five mixtures include 100% cement (ASTM Type II/V), 20% of DS200, DS325, ultrafine pumice with 80% cement and 30% DS325 with 70% cement with a w/cm ratio of 0.485. ASTM C192 is followed for preparing concrete test specimens and the basic mixture design used to produce 1 cubic feet concrete is shown in Table 5. ASTM Type II/V cement along with different grades of pumice is used as cementitious materials and a polycarboxylate-based mid-range water reducer admixture is used in the mixtures to maintain the slump of 3-5 in. Compressive strength results were shown in Table 6 and Fig. 10.

The mixtures containing pumice reached the compressive strength later than control mixture. However, the minimum strength at age 7 days is greater than 3000 psi and at age 28 days is greater than 4500 psi. Concrete with slightly slower strength gain qualities is less likely to be subject to early age cracking and has long term strength capability. Mixture containing ultrafine pumice reaches higher early strength compared to mixture containing DS200 and DS325. This trend is supported by the results from hydration behavior of blended cements, in which ultrafine pumice mixture showed rapid hydration characteristics. 80C20DS200 mixture reaches higher strength at 7 and 28 days compared to 80CDS325 mixture which shows the difference in hydration behavior exhibited by different grades of pumice.

Table 5: Basic Mixture Design to Produce 1 Cubic Foot of Concrete

Ingredients	**Mix-1**	**Mix-2**	**Mix-3**	**Mix-4**	**Mix-5**
Cement (lb)	20.9	16.7	16.7	14.6	16.7
Pumice (lb)	0	4.2	4.2	6.3	4.2
Coarse Aggregate (lb)	67	67	67	67	67
Fine Aggregate (lb)	54	53	53	52	53
Water Content (lb)	10	10	10	10	10

Table 6: Compressive strength of 4x8 cylinders

Mixture Design	**Strength at age 7 (psi)**	**Strength at age 28 (psi)**
Cement (C)	5636	7400
80%C+20%DS200 (Mix-2)	4214	5749
80%C+20%DS325 (Mix-3)	3343	4860
70%C+30%DS325 (Mix-4)	3398	5359
80%C+20%Ultrafine (Mix-5)	4648	7083

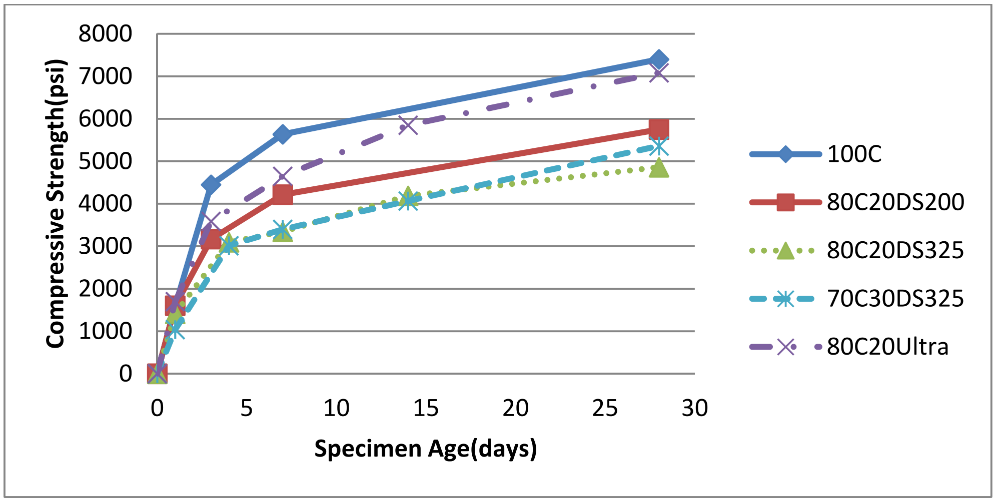


Fig. 10 Compressive strength for 4X8 cylinders

5. SULFATE MITIGATION

Following the procedures of ASTM C1012, five mortar mixture designs were tested for sulfate resistance. The cementitious combinations were same as used in the compressive strength testing. The specimens were tested through 6 months and the percentage length change of mortar specimens for different mixtures is shown in Fig. 11 and Table 7. Test values below 0.05% at 6 months indicate high sulfate resistance and test values below 0.10% at 6 months indicate moderate sulfate resistance. All the pozzolanic mixtures are within the limit of 0.1%, hence qualified to be MS (Moderate Sulfate resistance). Out of five mixtures tested, the four mixtures containing pumice are classified as HS (High sulfate resistant cement) since the length change is less than 0.05% after 26 weeks. Improved performance was seen in the mixtures which have pumice as part of cementitious blend.

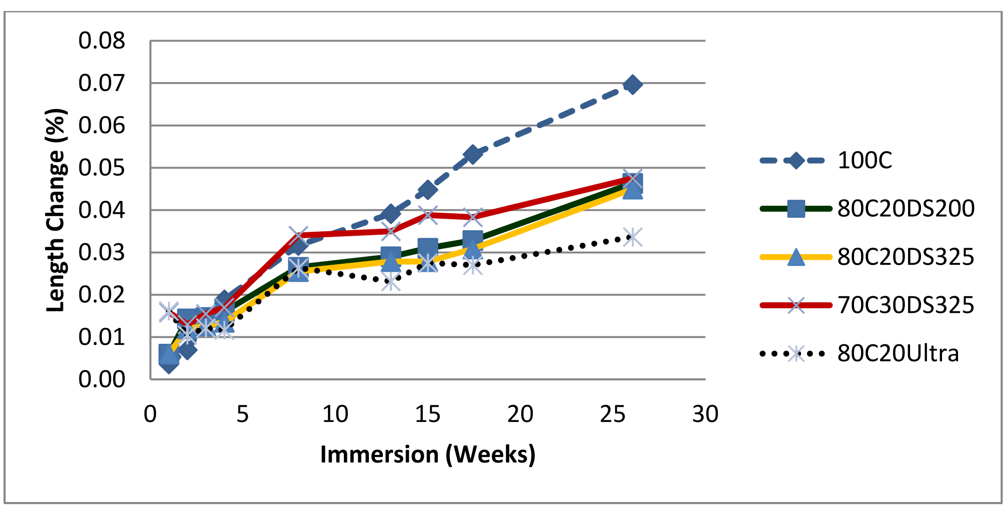


Fig. 11 Length Change (%) of Mortar Specimen due to sulfate

Table 7: Length Change (%) of Mortar Specimen due to sulfate

	Length change (%)				
Weeks	**C**	**Mix-2**	**Mix-3**	**Mix-4**	**Mix-5**
1	0.004	0.006	0.006	0.016	0.016
2	0.007	0.014	0.012	0.013	0.011
3	0.014	0.015	0.013	0.016	0.012
4	0.019	0.016	0.014	0.017	0.012
8	0.032	0.027	0.026	0.034	0.026
13	0.039	0.029	0.028	0.035	0.023
15	0.045	0.031	0.028	0.039	0.028
Month 4	0.053	0.033	0.031	0.038	0.027
Month 6	0.070	0.046	0.045	0.048	0.034

6. RESISTIVITY

Five mixture designs were tested for the resistivity of concrete specimens. Mixture proportions were same as used in the compressive strength testing. ASTM C192 procedure was followed to make 6”x12” cylinders and moist cured according to ASTM C511. Specimens were removed from the mold at an age of 23.5±0.5 hours and four marks are placed at 0, 90, 180, 270 degrees around the circumference of top of the cylinder. The cylinders are then placed back in the curing room until the time of testing. The Wenner resistivity probe, with 2 inch probe spacing, is then placed with its handle parallel with the center of the cylinder to note the reading. Readings were taken below the 0, 90, 180 and 270 degree marks. In the same way readings have been collected for all three specimens for each mixture and then it is averaged to obtain the average resistivity of the mixture. Table 8 and Fig. 12 show the resistivity reading for each mixture at different time intervals. Resistivity increases over time for the mixture with pozzolans whereas it remains relatively constant for the mixture with 100% portland cement. Mixture containing ultrafine pumice shows a considerable increase in resistivity over other four mixtures at 58 days. Mixtures containing 20% DS200 and DS325 shows a reasonable increase over 100 cement mixture but still less than 30% DS325 mixture. Addition of pumice helps in increasing the resistivity of specimens

Table 8: Resistivity at different time interval in kΩ-cm

Mixture	**14 days**	**24 days**	**58 days**
100C	4.1	4.6	6.8
80C20DS200	4.7	5.6	14.6
80C20DS325	4.3	5.5	15.7
70C30DS325	4.3	6.3	19.2
80C20Ultra	5.1	10.5	33.8

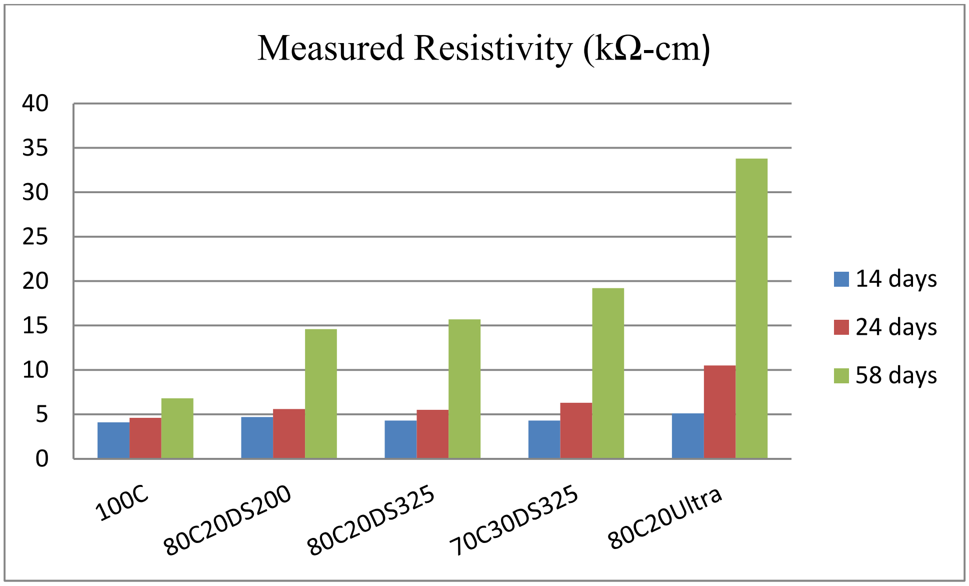


Fig. 12 Measured Resistivity of Concrete Specimens

7. SHRINKAGE

Five mixture designs were tested for length change of concrete specimens. Mixture proportions and specimen preparation were same as for electrical conductance test. Specimens were removed from the mold at an age of 23.5±0.5 hours and four marks are placed at 0, 90, 180, 270 degrees around the circumference of the cylinder. DEMEC buttons are pasted at 200 mm (approx. 8 in) intervals, one at top and other at bottom, on the four marks of cylinder. The cylinders are then placed back in the curing room for 28 days. After curing for 28 days, initial readings were taken with DEMEC digital strain gage at 0, 90, 180 and 270 degrees for each cylinder. Then the specimens are placed in air storage for next 28 days. During the period of air storage, consequent reading were taken at different intervals with DEMEC strain gage. Three concrete 6”x12” cylinders were tested for each mixture at different intervals for 28 days and the average length change is reported for each mixture design in Fig. 13. Fig. 13 shows that the addition of ultrafine pumice reduced the length change (shrinkage) compared to 100% cement, whereas the addition of DS200 and DS325 increased the length change (shrinkage) but not much. This phenomenon should be studied further. If it is repeatable, this lower shrinkage cement combination has multiple applications.

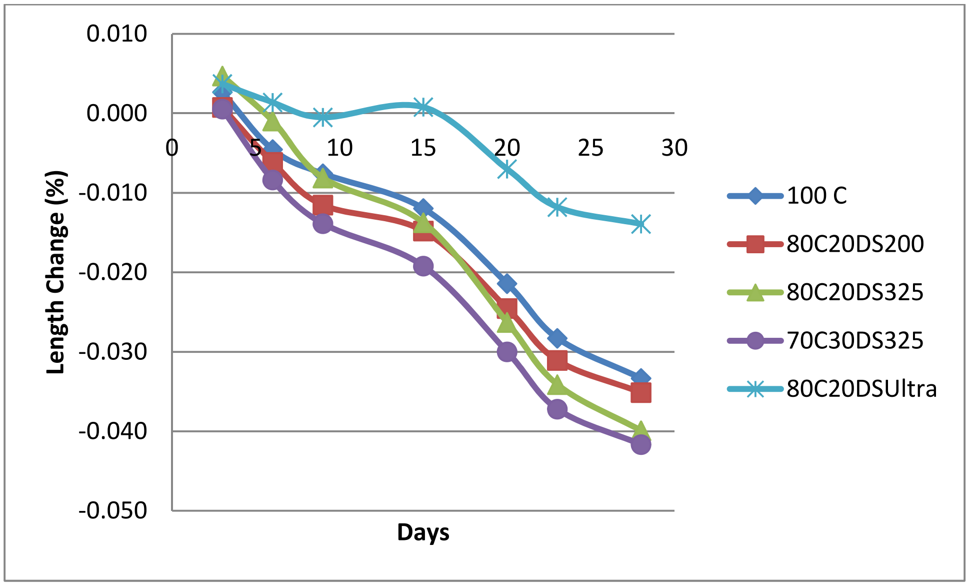


Fig. 13 Length Change (%) of Concrete Specimen due to shrinkage

8. ALKALI SILICA REACTION

Five mortar mixture designs were tested for alkali silica reaction according to a modified ASTM C1567 procedure. Mixture proportions used were same as for sulfate resistance except the ASTM Type I cement is used instead of Type II/V cement, along with 25% replacement of fine aggregate with ground cullet glass; ASTM C1567 aggregates gradation was used. The percent length change of mortar specimens for different mixtures was shown in Fig. 14 and the summary is given in Table 9. The percent length change for "acceptable expansion" is less than 0.10% at fourteen days with reactive aggregates. Any percent length changeover 0.10% is considered "deleterious expansion". From the result, it is very clear that the usage of pumice is very effective in mitigating the alkali-aggregate expansion.

Table 9: Summary of ASR Resistance

Mixture	ASR % Length Change	Rating
100C 25%Glass	0.699	Deleterious Expansion
80C20DS200 25%Glass	0.027	Acceptable Expansion
80C20DS325 25%Glass	0.029	Acceptable Expansion
70C30DS325 25%Glass	0.011	Acceptable Expansion
80C20Ultra 25%Glass	0.017	Acceptable Expansion

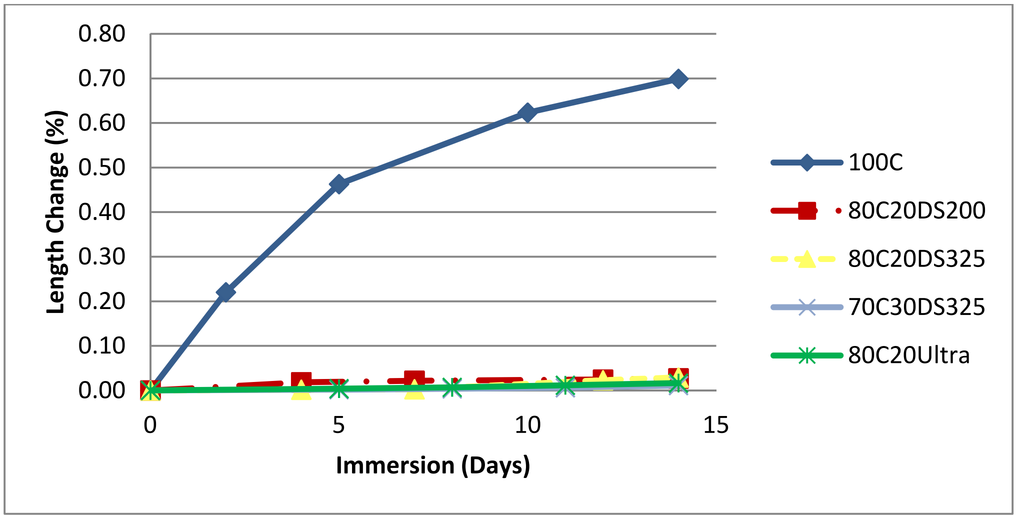


Fig. 14 Length Change (%) of Mortar Specimen due to ASR

9. ACTIVITY INDEX

Activity index was determined in accordance with C595 Annex A1. The eight mixture combinations used were 100% portland cement, ASTM Type II/V; 20 and 30% DS200; 10, 20, 30% DS325; 20 and 30% ultrafine pumice. Three mortar cubes were made for each mixture in accordance with C109 and water used for each mixture varied based on the flow requirement of 100 to 115. The specimens were demolded after 24 h storage in moist room. Then the specimens were placed in air tight glass container and stored at 38±1.7°C for 27 days. Compressive strength of the specimens were determined at the age of 28 days in accordance with C109 after allowing the specimens to cool to 23±1.7°C. Activity Index is calculated by dividing the average compressive strength of test mixture cubes with average compressive strength of control mixture cubes and reported in Table 10. Fig. 15 shows the activity Index of ultrafine mixture (80C20Ultra) is higher compared to all other mixtures and also the addition of more amount of ultrafine pumice (70C30Ultra) decreases the same. Hence, optimum proportion of cementitious material should be used for each mixture design to obtain the desired properties.

Table 10: Activity Index of Designated Combinations

Mixture	Activity Index
100C	
90C10DS325	85.3
80C20DS200	77.2
80C20DS325	69.8
80C20Ultra	131.2
70C30DS200	71.5
70C30DS325	92.4
70C30Ultra	122.2

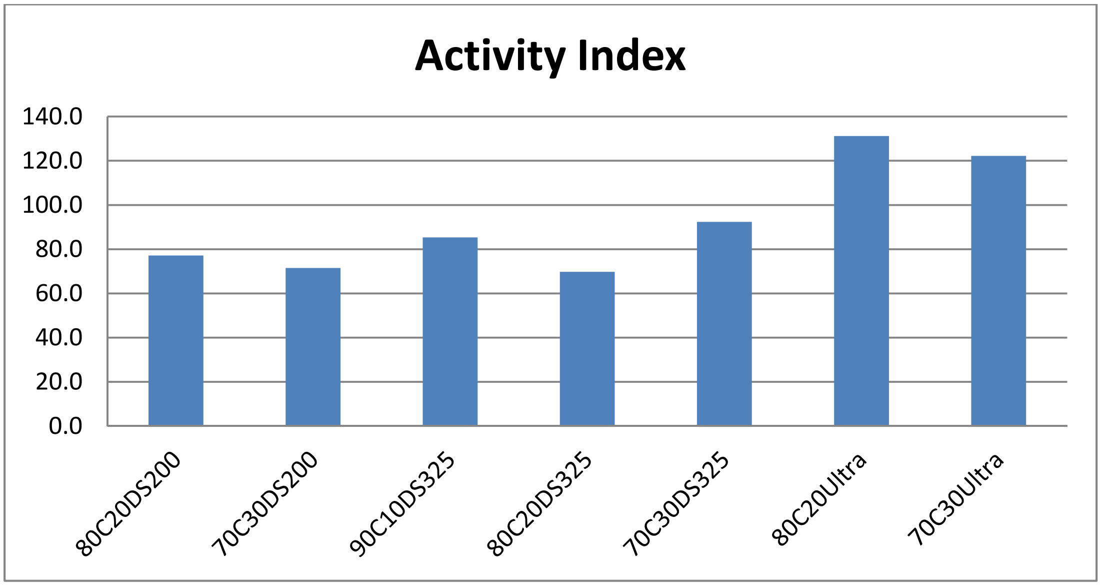


Fig. 15 Activity Index of Designated Combinations

10. MATURITY METER DATA

Fig. 16 shows the strength maturity relationship obtained according to ASTM C1074. Mixture combinations were same as for compressive strength test. Five mixture designs strength were determined by means of maturity method. Trend of different mixture combinations were same as compressive strength method (in accordance with C39).

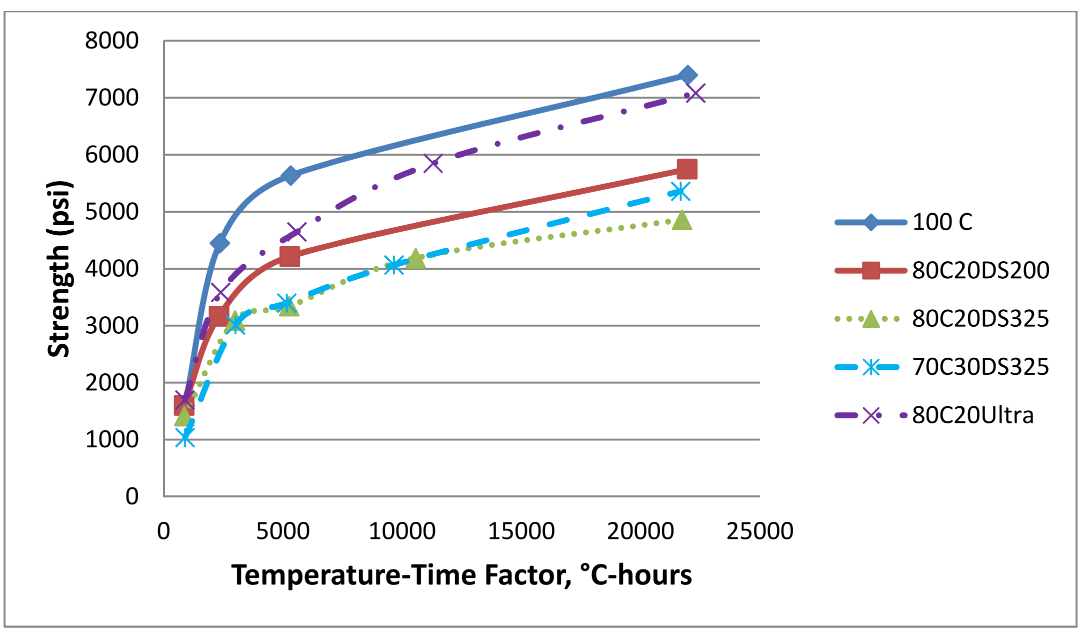


Fig. 16 Strength-Maturity Relationship

CONCLUSION

Pumice tested in this research was determined to be pozzolanic and potentially complementary in its reactions in portland cement concrete. The various grades of pumice behave differently in the hydration characteristic even with the same chemical composition, which may be due to varying particle size distribution. Ultrafine pumice showed improved performance over other grades of pumice in hydration, strength and in durability characteristics such as sulfate resistance and alkali silica reaction (ASR). The greater hydration characteristics of ultrafine pumice are also supported by the compressive strength and the penetration resistance results of the same. Though the water demand is high for the mixtures containing pumice, mid-range water reducer can be used to reduce the water demand which may help in reducing the setting time and makes it probably same as 100% cement mixture.

DS200 and DS325 pumice showed improved performance over cement in durability characteristics. If the application requires primarily durability characteristic i.e. high sulfate resistance and high ASR resistant then DS200 and DS325 pumice can be used as a part of cementitious material. If the requirement is both strength and durability, then ultrafine pumice can be used. The heat produced from mixtures containing pumice is less than that of mixtures with 100 % cement which makes it advantageous in mass concrete placements. To maintain higher strength, improved durability characteristics and reduced the potential for thermal cracking, ultrafine pumice can be used as a part of cementitious material.