

United States Department of the Interior

BUREAU OF RECLAMATION PO Box 25007 Denver, CO 80225-0007

VIA ELECTRONIC MAIL ONLY

MEMORANDUM

- To: Technology Development Program Manager, Dam Safety Office Attn: 84-44000 (LKrosley)
- From: Catherine Lucero, Civil Engineer Concrete, Geotechnical, and Structural Laboratory (86-68530)
- Subject: Dam Safety Technology Development Report DSO-2017-07 Evaluating Natural Pozzolans for Mitigating Temperature Rise in Mass Concrete

A report on Evaluating Natural Pozzolans for Mitigating Temperature Rise in Mass Concrete, DSO-2017-07 from the Dam Safety Technology Development Program has been prepared by the Technical Service Center at the request of the Dam Safety Office. The report will be available in Adobe Acrobat Format on the Dam Safety website and will also be loaded into DSDAMS.

This report is one of two reports under the project "Evaluating the effectiveness of natural pozzolans for use in mitigating temperature rise in mass concrete". If you have any questions, please contact me at 303-445-2343 or at cllucero@usbr.gov.

cc (w/att):

DSDaMS Archives 86-68530 (Bartojay)



Evaluating Natural Pozzolans for Mitigating Temperature Rise in Mass Concrete

Concrete, Geotechnical, and Structural Laboratory, 86-68530, DSO-2017-07 (8530-2017-30)

Dam Safety Technology Development Program





Mission Statements

The U.S. Department of the Interior protects America's natural resources and heritage, honors our cultures and tribal communities, and supplies the energy to power our future.

The mission of the Bureau of Reclamation is to manage, develop, and protect water and related resources in an environmentally and economically sound manner in the interest of the American public.

Disclaimer:

Information in this report may not be used for advertising or promotional purposes. The data and findings should not be construed as an endorsement of any product or firm by the Bureau of Reclamation, Department of Interior, or Federal Government. The products evaluated in the report were evaluated for purposes specific to the Bureau of Reclamation mission. Reclamation gives no warranties or guarantees, expressed or implied, for the products evaluated in this report, including merchantability or fitness for a particular purpose.

	REPORT DO	CUMENTATION PA	GE		Form Approved OMB No. 0704-0188		
T1. REPORT DATE	Т	2. REPORT TYPE		T	3. DATES COVERED		
September 2017	C	am Safety Technolog	y Development	0	ctober 2015 to September 2017		
T4. TITLE AND SUE	BTITLE			5	a. CONTRACT NUMBER		
Evaluating Natural P	ozzolans for Mitigati	ng Temperature Rise	in Mass Concrete	5	D. GRANT NUMBER		
				50	C. PROGRAM ELEMENT NUMBER		
6. AUTHOR(S)				5	d. PROJECT NUMBER		
Catherine Lucero, M	SCE, EIT						
Concrete, Geotechni	ical, and Structural L	aboratory		5	e. TASK NUMBER		
Technical Service Co	enter						
Bureau of Reclamati	ion			-			
Denver, CO 80225				51	WORK UNIT NUMBER		
7. PERFORMING O	RGANIZATION NAM	E(S) AND ADDRES	S(ES)	8	PERFORMING ORGANIZATION		
U.S. Department of t	the Interior		-()	R	EPORT NUMBER		
Bureau of Reclamati	ion				8530-2017-30		
Technical Service Co	enter						
Concrete, Geotechn	ical and Structural La	aboratory					
9. SPONSORING / M	MONITORING AGEN	NCY NAME(S) AND A	DDRESS(ES)	1). SPONSOR/MONITOR'S		
U.S. Department	of the Interior			Α	ACRONYM(S)		
Bureau of Reclar	mation			D	DSO: Dam Safety Office		
Dam Safety Offic	ce in the second se			В	OR/USBR: Bureau of Reclamation		
				D	OI: Department of the Interior		
				1	1. SPONSOR/MONITOR'S REPORT		
				N	IUMBER(S)		
				D	SO-2017-07		
12. DISTRIBUTION No Restrictions	/ AVAILABILITY ST	ATEMENT					
13. SUPPLEMENTA	ARY NOTES						
14. ABSTRACT (Ma	aximum 200 words)						
The thermal behavio	or of concrete has lon	g been studied to red	uce cracking, especi	ally in massiv	e members, where heat generation is		
great. Heat dissipati	ion and restraint crea	ate volume changes th	nat can induce stress	es at an early	age, when strength and modulus are		
low. Pozzolans or of	ther supplementary of	cementitious materials	s can be used to redu	ice the tempe	erature rise in large concrete placements.		
Reclamation has rec	ently updated therma	al property data sets f	or pure ordinary Port	land cement	(OPC), OPC + Class F fly ash, and OPC		
+ slag. While Class	F fly ash has traditio	nally been specified for	or mass concrete with	nin Reclamat	ion, sources of quality ash are becoming		
increasingly scarce.	Class N natural poz	zolans can be used a	s a substitute in mas	s concrete to	decrease the temperature rise and		
mitigate early age th	ermal cracking.						
15. SUBJECT TERM	IS natural pozzolan	s, mass concrete, terr	perature rise				
16. SECURITY CLA	SSIFICATION OF:		17. LIMITATION	18. NUMBE	R 19a. NAME OF RESPONSIBLE		
			UF ABSTRACT	OF PAGES	Catherine Lucero		
	D. ABSTRACT	C. THIS PAGE			19D. IELEPHONE NUMBER		
0	U	0					

S Standard Form 298 (Rev. 8/98) P Prescribed by ANSI Std. 239-18

BUREAU OF RECLAMATION

Concrete, Geotechnical, and Structural Laboratory, 86-68530 DSO-2017-07 (8530-2017-30)

Evaluating Natural Pozzolans for Mitigating Temperature Rise in Mass Concrete

Prepared by: Catherine Lucero, MSCE, EIT Civil Engineer, Concrete, Geotechnical, and Structural Laboratory, 86-68530

Checked By / Technical Approval: Katie Bartojay, P.E. Civil Engineer, Concrete, Geotechnical, and Structural Laboratory, 86-68530

Peer Review: Veronica Madera, P.E. Civil Engineer, Concrete, Geotechnical, and Structural Laboratory, 86-68530

For Reclamation disseminated reports, a disclaimer is required for final reports and other research products, this language can be found in the peer review policy:

"This information is distributed solely for the purpose of pre-dissemination peer review under applicable information quality guidelines. It has not been formally disseminated by the Bureau of Reclamation. It does not represent and should not be construed to represent Reclamation's determination or policy."

Acronyms and Abbreviations

ACI	American Concrete Institute
HVFA	High Volume Fly Ash
OPC	ordinary portland cement
RSMC	Reinforced Structural Mass Concrete
SCM	supplementary cementitious materials
w/cm	water to cementitious material ratio
WRA	water reducing admixture

Contents

Abstract	.7
Background	.7
Natural Pozzolans in Mass Concrete	.7
Previous Reclamation Experience with Natural Pozzolans	.9
Methods1	0
Material Selection1	0
Mixture Proportioning and Fresh Properties1	1
Time of Setting1	2
Compressive Strength1	2
Adiabatic Temperature Rise1	2
Test Results1	4
Time of Setting1	4
Adiabatic Temperature Rise1	7
Conclusions1	9
References	20
Appendix A – Materials Data Sheets A-	-1
Appendix B – Adiabatic Temperature Rise CurvesB-	-1

Tables

Table 1.	Mineral admixtures and structures that used them [3]	9
Table 2.	Chemical properties of cementitious materials used	11
Table 3.	Mixture proportions of concrete	13
Table 4.	Compressive strength of concrete mixtures containing natural pozzolans	16

Figures

Figure 1. Classification of natural pozzolans	8
Figure 2. Samples of the three pozzolans used in this study.	10
Figure 3. Initial and final setting time of concrete containing natural pozzolans	
compared to Class F fly ash.	14
Figure 4. Comparison of fracture surfaces for (a) 25% zeolite and (b) 75% zeolite	15
Figure 5. Temperature rise of concrete containing 25%, 50%, and 75% Microsilex	17
Figure 6. Temperature rise of concrete containing 25%, 50%, and 75% zeolite	18
Figure 7. Temperature rise of concrete containing 25%, 50% and 75% HessPozz	18

Abstract

The thermal behavior of concrete has long been studied to reduce cracking, especially in massive members, where heat generation is great. Heat dissipation and restraint create volume changes that can induce stresses at an early age, when strength and modulus are low. Pozzolans or other supplementary cementitious materials can be used to reduce the temperature rise in large concrete placements. Reclamation has recently updated thermal property data sets for pure ordinary Portland cement (OPC), OPC + Class F fly ash, and OPC + slag as reported in DSO-2015-02. While Class F fly ash has traditionally been specified for mass concrete within Reclamation, sources of quality ash are becoming increasingly scarce. Class N natural pozzolans can be used as a substitute in mass concrete to decrease the temperature rise and mitigate early age thermal cracking.

Background

Natural Pozzolans in Mass Concrete

The use of natural pozzolans dates back to 2000 BC and they have been used extensively throughout history in Roman, Greek, Indian and Egyptian structures [1]. Natural pozzolans were first used in the USA in mass concrete projects including the Bay Bridge and the Golden Gate Bridge in San Francisco, CA [2]. Reclamation used natural pozzolans to mitigate heat rise, improve resistance to sulfate attack and mitigate alkali-silica reaction in large dams. The last Reclamation structure constructed with natural pozzolans was Glen Canyon Dam in 1964. With current concerns regarding fly ash quality and availability and the continued need to mitigate temperature rise in massive placements, there is a need to revisit and reevaluate the potential of natural pozzolans are more consistent in chemical composition from a single source. From source to source however, there can be great differences.

Natural pozzolans are siliceous and or siliceous aluminous materials that are not inherently cementitious, but will react with reaction products (calcium hydroxide, CH) of Portland cement hydration to create cementitious products [3]. Examples of natural pozzolans include volcanic ashes and tuffs, clays and shales, and diatomaceous earth as illustrated in Figure 1. It is well established that pozzolans have much lower heats of hydration than Portland cement. A general rule of thumb is that pozzolans will contribute about half of the heat of the cement that it is replacing [1]. There have been several studies investigating heat rise in mass concrete containing pozzolans, mostly Class F fly ash or slag and less so on natural pozzolans found in the USA.



Figure 1. Classification of natural pozzolans

Since the pozzolanic reaction is dependent on CH in the system, the reaction is slow and increased strength benefits are not seen in early ages. For most mass concrete applications, where required strengths are low, this is not an issue and pozzolans may be specified at very high replacements (50% or more). However, in reinforced structural mass concrete (RSMC), strength requirements are typically higher. A large amount of research has been conducted on the use of high volume fly ash (HVFA) systems for structural use, but there has been much less investigation on whether natural pozzolans can be used in high volumes for structural concrete.

Uzal et al. evaluated the performance of structural concrete with high volumes of Turkish pozzolans [4]. The study evaluated three natural pozzolans and compared them with lowcalcium fly ash (conforming to ASTM C618 Class F) and blast furnace slag (conforming to ASTM C989 Grade 100). The three pozzolans were a natural zeolite, perlite, and volcanic tuff. The natural pozzolans were ground to a fineness where 80% passed through the 45 µm sieve, but was otherwise unprocessed. The target 28-day compressive strength of the control mixture was 30 MPa (4350 ksi) which is typical for structural concrete that is not in a severe exposure condition. At early ages (3 and 7 days), the OPC concrete had the highest compressive strength values. At 28 days, the mixtures containing 50% volcanic tuff and natural zeolites surpassed the strength of the control mixture. The only pozzolan that did not surpass the control mixture at later ages (91 and 180 days) was the perlite. While this study is a good example of using natural pozzolans at high volumes, the amount of water reducer required to achieve a specified slump (3.5 to 4 inches) and the relatively low strength of the control mixture should be taken into consideration. A compressive strength of 4350 psi at 28 days is suitable for some structural applications, but if the concrete element will be exposed to high sulfates, freezing and thawing, or deicing salts, the minimum required strength is 4500 psi per ACI 318 [5]. Additionally, some applications can specify even higher 28 day strengths.

Previous Reclamation Experience with Natural Pozzolans

Several studies investigating natural pozzolans in concrete were completed by the USBR from 1934 until 1961. Several potential pozzolans were investigated and research was conducted to determine the reactivity of the material [6]–[8]. Once the basic hydration mechanisms of pozzolans were understood and potentially beneficial materials were identified, engineers at Reclamation began refining the mixture design and proportioning for concrete containing pozzolans (including fly ash). In the early years, the chemical admixtures were not as readily available, understood, or effective as today's modern admixtures.

Aside from the early research conducted, some dams were constructed using natural pozzolans for at least the interior portion of the dam. Table 1 summarizes the projects using natural pozzolans constructed by Reclamation.

		Date	
Name	Location	Completed	l ype of Pozzolan
	Boise/Elmore Counties,		
Arrowrock Dam	Idaho	1915	Granite
Lahontan Dam	Churchill County, Nevada	1915	Siliceous silt
Elephant Butte	Elephant Butte, New		
Dam	Mexico	1916	Sandstone*
Friant Dam	Fresno Counties, California	1942	Pumicite
	Greer/Kiowa Counties,		
Altus Dam	Oklahoma	1945	Pumicite
Davis Dam	Clark County, Nevada	1950	Calcined opaline shale
	Santa Barbara County,		Calcined oil-impregnated
Glen Anne Dam	California	1953	diatomaceous shale
	Santa Barbara County,		Calcined oil-impregnated
Cachuma Dam	California	1953	diatomaceous shale
	Santa Barbara County,		Calcined oil-impregnated
Tecolote Tunnel	California	1957	diatomaceous shale
Monticello Dam	Napa County, California	1957	Calcined diatomaceous clay
	San Luis Obispo County,		
Twitchell Dam	California	1958	Calcined diatomaceous clay
Flaming Gorge	Daggett County, Utah		
Dam		1963	Calcined montmorillonite shale
Glen Canyon Dam	Coconino County, Arizona	1964	Pumice

Table 1. Mineral admixtures and structures that used them [3]

* Ground sandstone was found to not contribute to the pozzolanic reaction, served mostly as a filler.

As seen in Table 1, some of the structures are have reached over 100 years of service. Others are at approximately 60 to 70 years of service. The performance of the concrete containing pozzolans has been monitored with coring reports at 5, 10, 20 or more years.

Objective

The objective of this project is to test multiple sources and types of natural pozzolans available in the Western United States for adiabatic temperature rise for use in mass concrete elements. The results are compared to a similar mixture produced with Class F fly ash.

Methods

Material Selection

For this study, three pozzolans were selected for their potential in replacing fly ash as a means to reduce temperature rise in mass concrete. The materials were selected based on their availability in the Western United States where Reclamation projects are located. The following materials were selected for this study and are shown in Figure 2:

- GCC Microsilex
- Bear River Zeolite
- HessPozz



Figure 2. Samples of the three pozzolans used in this study.

	Type I/II Cement (%)	Class F Fly Ash (%)	GCC Microsilex (%)	Bear River Zeolite (%)	HessPozz (%)
SiO ₂	19.7	57.78	89	70.32	76.2
Al ₂ O ₃	4.5	22.82	3.6	12.55	13.5
Fe ₂ O ₃	3.2	7.35	0.9	3.38	1.1
CaO	64.3	3.56	2.6	3.05	0.9
MgO	2.6	1.31	0.34	0.48	0.05
SO₃	3.6	0.29	0.4	0.02	0.0043
Loss on Ignition	2.6	0.29	1.75	4.5	

Table 2.	Chemical	properties o	f cementitious	materials used
----------	----------	--------------	----------------	----------------

GCC Microsilex is a high surface area micro-siliceous pozzolan. It is industrially manufactured by GCC which allows for a very stable, highly siliceous composition. As seen in Table 2, it has the highest percentage of silicon dioxide (SiO₂) and much less aluminum oxide (Al₂O₃) than the other two natural pozzolans or Class F fly ash.

Bear River Zeolite deposit is located in Southeast Idaho. It can be purchased in various particle sizes for other uses, but the finer material is more suitable for use in concrete. The material used in this study has a particle size from 15 to 25 microns. The material conforms to ASTM C618. It can be ordered in 50-lb bags, super sacks, pallets, and bulk truckloads.

HessPozz is a pure pumice product. It is an amorphous white silica product ground to a fine particle size of 45 microns with 93% passing the No. 325 sieve. The pumice is mined in Southeast Idaho and can be shipped worldwide. It is available in 44-lb bags, 1-ton super sacks or in bulk pneumatic rail cars and tanker trucks.

Mixture Proportioning and Fresh Properties

All mixtures were proportioned with 658 lb of cementitious materials, which is the equivalent of a 7-sack concrete mixture proportioned with cement only. Cement was replaced by natural pozzolans at 25, 50, and 75% by mass. Comparisons were made to concrete made with Class F fly ash from previous research [9]. Table 3 summarizes the mixture proportions.

Typical Reclamation specifications for mass concrete requires $25 \pm 10\%$ Class F fly ash and a temperature control plan to mitigate early age thermal cracking and delayed ettringite formation. Some projects require more fly ash, up to 50%, or high volumes of slag, up to 65% [10].

Time of Setting

The initial and final setting time was determined using ASTM C403. Mortar was prepared separately for each measurement with the same paste to sand volume ratios within the concrete rather than sieving from the original concrete mixture. The time of setting was measured for all natural pozzolan mixtures and compared to mortar containing Class F fly ash.

Compressive Strength

The compressive strength of the specimens was determined using 4-inch by 8-inch cylinders in accordance with ASTM C39 [11]. Strength was tested at 7, 14, 28, and 56 days. Data was not collected for the Class F fly ash mixtures as those were investigated in a previous study that did not include compressive strength.

Adiabatic Temperature Rise

The adiabatic temperature rise of the concrete was measured in accordance with USBR 4911 [12]. The individual components were pre-cooled to 50 °F overnight prior to mixing. The temperature rise was recorded for at least 30 days and up to 56 days.

Table 3. Mixture proportions of concrete

Mix ID	Description	Cement (lb/yd³)	Pozzolan (lb/yd³)	Sand (lb/yd³)	3/4" Rock (lb/yd³)	Water (lb/yd³)	WRA (oz/cwt)	w/cm
OPC-7	Control	671	0	1366	1750	260	2	0.39
FA-25	25% Class F Fly Ash	494	165	1268	1775	280	2	0.42
FA-50	50% Class F Fly Ash	329	329	1252	1750	274	2	0.42
FA-75	75% Class F Fly Ash	165	494	1258	1700	260	2	0.39
MS-25	25% GCC Microsilex	494	165	1232	1725	315	2	0.48
MS-50	50% GCC Microsilex	329	329	1207	1700	315	2	0.48
MS-75	75% GCC Microsilex	165	494	1181	1675	315	4	0.48
ZEO-25	25% Zeolite	494	165	1196	1650	350	4	0.53
ZEO-50	50% Zeolite	329	329	1153	1625	350	4	0.53
ZEO-75	75% Zeolite	165	494	1108	1600	350	4	0.53
HP-25	25% HessPozz	494	165	1213	1700	330	2	0.50
HP-50	50% HessPozz	329	329	1186	1675	330	2	0.50
HP-75	75% HessPozz	165	494	1157	1650	330	2	0.50

Test Results

Time of Setting

According to the PCA's *Design and Control of Concrete Mixtures*, Class F fly ash and most natural pozzolans tend to delay the time of setting about 15 minutes to 1 hour for initial set and 30 minutes to two hours for final set [1]. Mixtures with 25% natural pozzolan had similar initial and final setting time to the fly ash mixture. An increase to 50% natural pozzolan appeared to have little influence on the initial setting time, but increased the final setting time by approximately 1 hour. At 75% replacement, the fly ash mixtures had a delayed initial and final set (7 hours and 9.5 hours respectively). The Microsilex also had a delayed time of setting. The 75% addition of HessPozz and Bear River Zeolite did not appear to have a significant effect on the initial set, but the final set was delayed compared to the mixtures containing 25% and 50%.



Figure 3. Initial and final setting time of concrete containing natural pozzolans compared to Class F fly ash.

Compressive Strength

The compressive strength was tested at 7, 14, 28, and 56 days. The mix design was not optimized for strength, as the focus of this study was temperature rise. The proportions were based on the total cementitious content of the fly ash mixtures designed in previous studies [9]. There were minimal adjustments made to ensure the temperature rise would remain comparable with the same amount of cementitious material per cubic yard. To improve the strength, a higher cementitious content could be used. Additionally, an appropriate admixture (water reducer) formulated to use with natural pozzolans and a lower w/c could result in higher compressive strengths.

The paste-to-aggregate bond was particularly poor in the mixtures containing zeolite, as seen in Figure 4. The mixtures were much stickier and required high water to cementitious materials ratios in order to be workable. There were some issues properly consolidating the mixture containing 75% zeolite.



Figure 4. Comparison of fracture surfaces for (a) 25% zeolite and (b) 75% zeolite.

	Compressive Strength (psi)									
			7 day	1	l4 day	2	28 day	8 day 56 day		
Mix ID	% replacement	Average	Percent of OPC Mix	Average	Percent of OPC Mix	Average	Percent of OPC Mix	Average	Percent of OPC Mix	
OPC-7	0	5547	100%	6140	100%	6253	100%	7087	100%	
MS-25	25	3947	71%	4723	77%	4750	76%	5650	80%	
MS-50	50	2569	46%	3250	53%	3790	61%	4117	58%	
MS-75	75	905	16%	1195	19%	2047	33%	2713	38%	
ZEO-25	25	3573	64%	4255	69%	4557	73%	4753	67%	
ZEO-50	50	2453	44%	2967	48%	3463	55%	3523	50%	
ZEO-75	75	923	17%	1687	27%	1893	30%	2070	29%	
HP-25	25	3323	60%	3630	59%	4147	66%	4710	66%	
HP-50	50	1893	34%	2450	40%	2897	46%	3150	44%	
HP-75	75	557	10%	760	12%	1193	19%	1467	21%	

Table 4. Compressive strength of concrete mixtures containing natural pozzolans

Adiabatic Temperature Rise

The adiabatic temperature rise of nine concrete mixtures was tested in the laboratory in accordance with USBR 4911, Temperature Rise of Concrete. In mass concrete placements, there is very little heat dissipation; therefore, adiabatic temperature rise testing is the most accurate laboratory measurement for determining the maximum temperature in the center of the placement in the field.

The temperature rise of Microsilex, Bear River Zeolite, and HessPozz are shown in Figure 5 through Figure 7. The temperature rise was compared to a similar mixture containing fly ash (dashed line) and a straight cement mixture (black line). The Microsilex had a higher temperature compared to the fly ash mixture, especially within the first 7 days. The mixtures containing the zeolite appeared to reach a plateau in temperature rise earlier than that of concrete containing fly ash. The HessPozz performed the most similarly to Class F fly ash in the 25% and 50% mixtures, but had a lower temperature rise at 75%.



Figure 5. Temperature rise of concrete containing 25%, 50%, and 75% Microsilex



Figure 6. Temperature rise of concrete containing 25%, 50%, and 75% zeolite



Figure 7. Temperature rise of concrete containing 25%, 50% and 75% HessPozz

Conclusions

Class N pozzolans can be used in mass concrete to mitigate temperature rise similarly to Class F fly ash. There are several sources available in the Western US and could be suitable for Reclamation projects as long as they conform to ASTM C618 and test batches are made to confirm strength gain.

The mixtures containing 75% pozzolan were difficult to mix and required a high w/c or a lot of admixtures in order to make a workable mixture. Fly ash particles are spherical and improve the workability, whereas natural pozzolans tend to make a mixture more stiff since they are ground to a powder and have an irregular particle shape. At this time, it is not recommended to use more than 50% pozzolan by mass.

Further research should be done to develop recommendations for mixture proportioning and design for achieving high strengths in concrete containing natural pozzolans. It may open opportunities to allow the use of natural pozzolans in RSMC by maintaining a low temperature but achieving a high long-term (56+ day) strength.

References

- M. L. Wilson and S. H. Kosmatka, "Supplementary Cementitious Materials," in *Design* and Control of Concrete Mixtures, 5th ed., Skokie, Ill: Portland Cement Association, 2011, pp. 58–72.
- [2] H. S. Meissner, "Pozzolans Used in Mass Concrete," in *First Pacific Area National Meeting of the American Society for Testing Materials*, 1949.
- [3] ACI 232.1R-12, "Report on the Use of Raw or Processed Natural Pozzolans in Concrete," in *ACI Manual of Concrete Practice*, 2012.
- [4] B. Uzal, L. Turanli, and P. K. Mehta, "High-Volume Natural Pozzolan Concrete for Structural Applications," *ACI Mater. J.*, vol. 104, no. 5, pp. 535–538, 2007.
- [5] ACI 318-11, "Building Code Requirements for Structural Concrete," in *ACI Manual of Concrete Practice*, 2011.
- [6] E. J. Benton, "Investigation of the Correlation of Mineralogy of Pozzolans With Performance in Concrete." PET-128, Bureau of Reclamation, Denver, CO, 1961.
- [7] H. S. Meissner, "Laboratory Procedure for Determining the Relative Cementitious Value of Pozzolanic Material." Technical Memorandum No. 500, Bureau of Reclamation, Denver, CO, 1935.
- [8] R. C. Mielenz, "Effect of Calcination on Natural Pozzolans," in *First Pacific Area National Meeting of the American Society for Testing Materials*, 1949.
- [9] K. Bartojay and C. Lucero, "Updating Thermal Data Sets to Better Evaluate Thermal Effects of Concrete." DSO-2015-02, Dam Safety Technology Development Program, Bureau of Reclamation, 2016.
- [10] D. P. Bentz, C. J. Bognacki, K. A. Riding, and V. H. Villareal, "Hotter Cements, Cooler Concretes," Concr. Int., no. Bentz, pp. 41–48, 2011.
- [11] ASTM Standard C39/c39M-14a, "Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens." ASTM International, West Conshohocken, PA, 2014.
- [12] USBR 4911-92, "Temperature Rise of Concrete," in *Concrete Manual, Part 2*, 9th ed., Denver, CO: Bureau of Reclamation, 1992.

Appendix A – Materials Data Sheets

microSilex

200 nm

14m





Contents

1)	Abstr	act 1
2)	Introc	luction/Background1
3)	Chara	cterization of powders
4)	Portla	nd-pozzolan cement paste6
	4.1)	Material used
	4.2)	Pozzolanic Activity Index with Portland cement
	4.3)	Setting time
5)	Morta	ar paste
	5.1)	Heat released
	5.2)	Strength
	5.3)	Durability issues
6)	Conce	rete
	6.1)	Material used 14
	6.2)	Plasticizer consumption15
	6.3)	Rheology
	6.4)	Compressive and flexural strengths
	6.5)	Durability
7)	Conc	lusions

Characterization and Effects of Microsilex and Silica Fume on Cement Paste, Mortar and Concrete properties

1) Abstract

Microsilex is a high surface area microsiliceous pozzolan that combined with portland cement and water improves the physical-chemical properties of the plain binder. When used in combination with portland cement, **Microsilex** increases the mechanical properties in terms of strength, as well as, the durability with respect to sulfate resistance, chloride-ion penetration and mitigation of alkali-aggregate reaction. Furthermore, due to its unique particle size distribution, **Microsilex** is easier to handle and operations such as truck unloading can be performed faster. **Microsilex** is an environmentally responsible product since the energy consumption to produce **Microsilex** is much lower than to produce common binders.

2) Introduction/Background

Pozzolans are used to enhance mechanical and physical-chemical properties of cementbased materials. According to the norm ACI-116R "pozzolans are siliceous or siliceous and aluminous materials which in itself possess little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperature to form compounds possessing cementitious properties". This norm states that there are two different families of pozzolans: siliceous and siliceous-aluminous. The resistance to chemical aggression and the properties obtained in blending cement with these two families of pozzolans are different.

Pozzolans are generally derived from naturally occurring materials or by-products of industrial processes, such as steel, silicon and coal-based power production. In recent years, due to their good properties and their lower environmental impact, pozzolans have gained popularity so rapidly that a shortage of premium materials is occurring in the market. Premium pozzolans are often difficult to supply, despite the availability of large quantities of by-products usually classified under the general names of fly ash, silica fume and ground granulated blast furnace slag (GGBFS). For example, fly ashes can have very different characteristics from batch-to-batch and not always able to be adapted for use in the concrete and cement industries. Furthermore fly ash is a by-product and consequently its process is not optimized and controlled to obtain a premium pozzolan. For these reasons GCC have decided to develop and produce **Microsilex**, a new siliceous pozzolan with stable characteristics and flexible rates of production so that GCC clients can be assured that the effect of Microsilex in their mixes will always be consistent in terms of strength, durability and color. **Microsilex** is industrially manufactured using a very precise process that allows for a very stable composition and is a patented product protected by the international laws (WO2006079875).



3) Characterization of powders

In order to understand the properties of **Microsilex** and two different types of silica fume, a series of fundamental tests were conducted on the plain powders. Among the features that characterize a cementitious material are: chemical composition, morphology and particle size distribution.

A very fine powder might have a very good reactivity but might increase the water demand that the extra water will negate any benefit from its reactivity.

Since **Microsilex** is an industrially manufactured compound, its composition is very stable and controlled. Pozzolans derived from by-products might have large variations in composition because they are waste-products of other processes. Their composition can be affected by changes in the principal process resulting in subsequent changes to their physical-chemical properties.

Chemical composition of the powders has been determined also using X-Ray Fluorescence (see Table 1).

 Table 1: Oxide composition of Microsilex, Silica Fume 1 and Silica Fume 2, measured by X-Ray Fluorescence, in %

	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	K_2O	TiO ₂	Na ₂ O
Microsilex	2.6	89.0	3.6	0.9	0.34	0.09	0.27	0.07
Silica Fume 1	0.5	93.0	0.6	0.7	0.66	0.85	0	0.26
Silica Fume 2	0.4	89.9	0.1	0.2	0.34	0.60	-	0.06

Both powders can be classified as siliceous pozzolan since they met the limits imposed by the ASTM norms (see Table 2) and XRF analysis shows that the main component is silicon oxide.

Table 2: Comparative chemical properties of Microsilex, Silica Fume 1, Silica Fume 2 and
standard requirements of ASTM C618 and ASTM C1240

	SiO ₂ +Al ₂ O ₃ +Fe ₂ O ₃ (%)	SO ₃ (%)	Moisture (%)	LOI (%)	Alkalis (%)	
ASTM-C618	70.0 min.	4.0 max.	3.0 max.	10.0 max.	1.5 max.	
Microsilex	93.5	0.4	0.11	1.75	0.16	
Silica Fume 1	94.3	0.3	0.16	2.06	1.11	
Silica Fume 2	90.2	0.2	1.50	4.00	0.66	
ASTM-C1240	85.0 SiO ₂ min		3.0 max	6.0 max		
C618: specifications for raw or calcined Natural pozzolan, C1240: specifications for Silica Fume						

The chemical reactivity of a pozzolan is related to the overall amorphous content, even though this parameter is not the only parameter that influences the Supplementary Cementitious Materials (SCM) effect on concrete performances. Amorphous content has been evaluated by XRD and Rietveld method. Estimation of crystalline and amorphous contents is presented in Table 3.

 Table 3: Quantitative analysis of crystalline and amorphous content of Microsilex and Silica Fume 1 by XRD and Rietveld method

Material	Crystalline, %	Amorphous, %
Microsilex	23.5	76.5
Silica Fume 1	0.4	99.6

Particle Size Distribution (PSD) is very important with respect to packing density of binders, rheology and safety concerns. **Microsilex** is coarser than Silica Fume 1 (see Table 4) and this implies a good workability (easier to disperse) while Silica Fume 1 might have a slightly larger packing density assumed that there is enough plasticizer and mixing power to disperse the powder in the mix.

 Table 4: Median diameter of Microsilex and Silica Fume 1 particles, measured by laser diffraction granulometer

Material	D _{ν50} (μm)
Microsilex	8.13
Silica Fume 1	0.34

It may be difficult to adequately disperse very fine particles of silicon, such as, silica fume, (see Figure 1) which could led to the possibility of localized alkali-silica reaction. In order to avoid this problem, **Microsilex** is milled at an optimum particle size distribution. This particle size distribution, in conjunction with other factors, also allows for much easier transfer of **Microsilex** from trucks to silos, without the occurrence of electrostatic charge formation problems that sometimes arise during transferring other types of pozzolans which slow the transfer rate.

Another significant parameter for reactivity of SCM is the Specific Surface Area (SSA); since the larger is the surface area, the more contacts between chemically reactive surfaces and reactants occur. A common method for measuring SSA is called BET (named after Brunauer, Emmett and Teller), which measures SSA on the basis of nitrogen adsorption on particle surfaces. For this study the BET values were cross-checked with morphological studies using Scanning Electron Microscope (SEM) images. Values of BET for **Microsilex** and Silica Fume 1 (see Table 5) show that **Microsilex** has a larger specific surface area even though Silica Fume 1 is smaller in size; this is because **Microsilex** has a porous surface as it is possible to see in SEM micrographs (see Figure 2). Density of the powders has been determined as well (see Table 5).





Figure 1: Photomicrograph to illustrate densified silica fume that did not brake up during mixing. Note that the silica fume particle has undergone ASR (Black Arrow). Note other silica fume agglomerates that have not yet reacted (Red Arrows).

Tuble 5. Opcomo Cunace Area and denoity of Micrositex and Cinou Fame F					
Material	SSA (m²/g)	d (g/cm ³)			
Microsilex	25.68	2.31			
Silica Fume 1	20.18	2.27			

Table 5: Specific Surface Area and density of Microsilex and Silica Fume 1



Figure 2: SEM technique, low and high magnification: (a), (b) micrographs of Microsilex; (c), (d) micrographs of Silica Fume 1

Color of SCM is very important because it has an impact on the color of the final product. **Microsilex** (see Figure 3) doesn't affect the color of final product.



Figure 3: Colors of Microsilex (left) and Silica Fume 1 (right)



4) Portland-pozzolan cement paste

4.1) Material used

Tests on pastes made with SCM and different types of cements have been carried out in order to check the effect of pozzolans (**Microsilex** and Silica Fume) when combined with cement. Cements used in the tests are all commercially available and produced by different companies. In particular, limestone cement and an ordinary portland cement manufactured by Holcim were used, as well as, an ordinary portland cement manufactured by GCC.

The two Holcim cements (Normo 4 and Fluvio 4) are respectively an ordinary portland cement and limestone cement conforming to the European norms; GCC OPC is a Type II portland cement. Holcim limestone cement is characterized by a limestone content of about 15%.

Oxide compositions for the three cements used are available in Table 6. The purpose for using cements available in different markets and from different sources was to further verify if **Microsilex** and Silica Fume 1 are efficient with different types of cements and not just with a single source.

Compound name	Weight fractions (%)				
	Holcim Normo 4	GCC OPC	Holcim Fluvio 4		
Al ₂ O ₃	4.85	4.65	4.24		
SiO ₂	19.45	20.15	17.50		
Fe ₂ O ₃	3.05	3.20	2.69		
CaO	61.91	64.13	60.20		
MgO	2.31	2.18	1.98		
K ₂ O	0.82	0.37	0.76		
Na ₂ O	0.26	0.08	0.29		
SO ₃	2.80	2.98	2.56		
Density, measured by Helium pychometer is from 3.05 to 3.15 q/cm^3					

 Table 6: Oxide composition of cement Holcim Normo 4, Fluvio 4 and GCC OPC measured by X-Ray Fluorescence, in %

Pozzolans are ingredients used in concrete to enhance performance, both mechanical and long-term durability. Of course, the rate of pozzolan that will give a specific level of performance has to be determined. For this reason, all cements were intimately blended at a rate of 5, 10 and 20% of pozzolan.

4.2) Pozzolanic Activity Index with Portland cement

A typical test used to determine if a pozzolan is chemically active when blended with cement is the pozzolanic activity test. Several protocols are available; two of the most used are the ASTM standards C311 and C595. This protocol has been applied to all cement-pozzolan blends as it is possible to see in Tables 7 and 8.

Microsilex has the best pozzolanic activity for all the cements tested, which demonstrates the reactivity of this pozzolanic compound.

 Table 7: Pozzolanic activity index (ASTM C311) for Holcim Normo 4 (HN4), GCC Chihuahua (CHI) and Holcim Fluvio 4 (FLU) blended with Microsilex and Silica Fume 1 at 7 and 28 days

Activity index	HN4		СНІ		FLU	
ASTM C311	7d	28d	7d	28d	7d	28d
Microsilex	105%	118%	91%	98%	85%	104%
Silica Fume 1	73%	95%	63%	85%	69%	86%

 Table 8: Pozzolanic activity index (ASTM C595) for Holcim Normo 4 (HN4), GCC Chihuahua (CHI) and Holcim Fluvio 4 (FLU) blended with Microsilex and Silica Fume 1 at 28 days

Activity index	HN4	СНІ	FLU
Microsilex	105%	91%	85%
Silica Fume 1	73%	63%	69%

4.3) Setting time

Setting time is a critical parameter in relation to the batching, transportation, pouring and vibration of concrete. It is important that the addition of a pozzolan does not modify too much the setting time.

Testing conditions are very important for the reproducibility of the results. To avoid operator errors or temperature and humidity variations, an automatic Vicat apparatus equipped with a temperature and humidity control was used for this study. Figure 4 shows that final setting time does not vary too much when any rate between 5% and 20% of Microsilex and Silica Fume 1 are used.



Figure 4: Final setting times of pastes blended with Microsilex and Silica Fume 1



5) Mortar paste

5.1) Heat released

When constructing thick concrete elements, it is of importance to limit the maximum temperature between core and external surfaces. Pozzolans can be useful because they moderate the maximum peak temperature of concrete. Figure 5 presents a semi-adiabatic test conduced on composite portland cement (Holcim Fluvio 4) blended with 10% pozzolans in 4:1 mortar. The peak temperatures of the mixtures are reduced with the incorporation of pozzolans. It is however important to see that this decrease of temperature is not excessive.



Figure 5: Semi-adiabatic test in mortars based on mix 350L (description of the mix 350L in Table 9, section 6.1)

5.2) Strength

Strength is the most well-known characteristics for every binder. According to the ASTM standard C109, a mortar is made using enough water to reach a specified consistency. The nominal strength of a portland cement/pozzolan blend is thus a function of its composition and the quantity of water that is required to achieve the specified consistency. Finer binder is typically associated with higher strength than that of an equally (chemically) composed, but coarser cement. The water requirement of a portland cement blended with a very fine pozzolan may be so high that it can negatively affect the optimum strength. The higher water demand is an indicator that a plasticizer is required to obtain a concrete of a desired workability without increasing the w/c ratio.

Compressive strengths were evaluated according to ASTM C109 procedures for mortars using binders made by blending Holcim Normo 4 cement and pozzolans from 5% to 20% substitution.

Microsilex

Water required to reach standard consistency for every mix is shown in Figure 6. **Microsilex** requires less water than Silica Fume 1 at all substitution rates.



Figure 6: Water requirement in order to keep standard consistency for mortars with Silica Fume 1 and Microsilex

Compressive strengths at 1, 7, 28 and 60 days show that the mixture that incorporates **Microsilex** has an excellent strength enhancing capability and does not significantly impact the early age strength development (see Figure 7). The mixture that incorporates Silica Fume 1, because the high water demand, does not outperform the reference which suggests that large quantities of plasticizer are likely to be necessary if Silica Fume 1 is used.

Based on the results of paste and mortar tests, 10% of **Microsilex** is an optimum value for most applications since it gives excellent mechanical strength and good protection against harmful environment.







5.3) Durability issues

Sulfate attack

Sulfate attack, especially in conjunction with other environmental conditions such as cold temperature, can be devastating for concrete structures. If sulfate reacts after hardening and forms Thaumasite or Ettringite, structural safety could be compromised and structures might have to be demolished. An idea of how sulfate attack develops is shown in Figure 8. Sulfate ions combine with Portlandite (CH) (Eq. 1) to form gypsum. Furthermore, gypsum in presence of water reacts with calcium aluminate hydrates to form delayed ettringite (Eq. 2) and in presence of calcium carbonate, low temperature and high humidity reacts with calcium silicate hydrates to form thaumasite (Eq. 3).



Figure 8: The mechanisms of delayed gypsum, ettringite and thaumasite formation

Portlandite is produced along with CSH during the ordinary portland cement hydration. It is clear that by reducing portlandite, there would be less possibility for sulfate to start harmful reactions.

One of the methods to reduce and avoid sulfate attack in concrete is to decrease the quantity of portlandite (CH) by reducing the portland cement content in the binder and replacing it by pozzolans because pozzolans react with portlandite to form CSH which can not be attacked by sulfates if delayed gypsum has not been formed.

Both silica fumes and **Microsilex** are silicon oxide-based compounds (see Table 1) and thus there is no concern with respect to reactivity with sulfates as in silicon-aluminum compounds. Tests of sulfate expansion have been carried out according to ASTM C1012 for binder composed by Holcim Normo 4 cement and 5% to 20% substitution of **Microsilex** and Silica Fume 1. Figure 9 shows that **Microsilex** is more efficient than Silica Fume 1 in reducing the expansion due to the sulfate.

The same results (see Figure 10) are obtained when Silica Fume 2 has been used at a rate of 10%. **Microsilex** always outperforms with respect to silica fume.





Figure 9: Sulfate expansion tests on specimens prepared with and w/o Silica Fume 1 (SF1) and Microsilex (MS) up to 105 days



In order to meet specifications for concrete in aggressive sulfate environments, a blend of cement and pozzolan could be used instead of Type V cement.

Length changes in water

Length change in water was tested in accordance with ASTM C157 for Holcim Normo 4 cement blended with silica fume 1 and **Microsilex** and in all cases the requirements are met (see Figure 11).



Figure 11: Length change in mortars with and w/o Silica Fume 1 (SF1) and Microsilex (MS) up to 84 days

Based on the tests on pastes composed by different cements and different rates of **Microsilex**, Silica Fume 1 and Silica Fume 2, it is possible to conclude that **Microsilex** is an ingredient that improves the quality of the binder by mitigating sulfate expansion, water expansion and improving the pozzolanic activity index. **Microsilex** performs better than silica fume and does not have drawbacks. Furthermore, being a silicon oxide based pozzolan; **Microsilex** reduces concerns with respect to alumina-sulfate reactions.



6) Concrete

6.1) Material used

Four different mix designs were used to characterize pozzolans. In these mixes two different silica fumes were used, both commercially available in bags and bulk and both among the most popular silica fumes in the market (see oxides composition in Table 1) Pozzolans were substituted for cement in mix design 1-3 (see Table 9) and were added in mix 4 (see Table 10). Cements used for mixes 1-3 are presented in Table 6. A Type I-II cement from GCC was used for mix 4. Plasticizers were the same for mixes 1-3 (a common high range water reducer) and a blend of a mid-range and high-range water reducer for mix 4. Aggregates were all natural aggregate (lake for mixes 1-3 and river sand for mix 4), with the exception of the coarser aggregate in mix 4 (a crushed limestone aggregate). All materials are commercially available, largely used and well known in the technical literature. Cements in mixes 1-3 have been substituted with **Microsilex** and Silica Fume 1 from 5 to 20%. Mix 4 was prepared by adding Silica Fume 2 and **Microsilex** at a rate of 10%. Mix designs are detailed in Tables 9 and 10.

Kg/m ³	Base 350 P	Base 350 L	Base 250
Sand 04	735.6	735.6	843.3
Sand 48	198.8	198.8	210
Gravel 816	457.2	457.2	560
Gravel 1632	596.4	596.4	496.7
Cement Type II HN4	350	-	250
Cement Type I HF4	-	350	-
Pozzolan (Microsilex/Silica Fume 1)	variable	variable	variable
Water (effective)	133	133	136.7
Plastcizer Glenium 51 adapted to reach a fix slump	1.225	1.225	0.875

Table 9: Mix design 1 (350 P), Mix design 2 (350 L) and Mix design 3 (250)

Table 10: Mix design 4 (D1)

Ŭ (
Kg/m ³	Base D1
Coarse aggregate 3/4 limestone	1059.3
Sand River	759.6
Type I/II cement	295.5
Pozzolan (Microsilex/Silica Fume 2)	variable
Water	232.6
Plasticizer Rheobuild 1000	0.81
Plasticizer MBVR MBT	0.05

Mixes 350 P, 350L and 250 have been designed using four sizes of aggregates; they are the same mixes except for the type of cement used (Type II for 350P and 250, Type I for 350L). Mix 250 has been made using the same materials as 350P but the cement content is different (350 kg/m³ in 350P and 250 kg/m³ for 250). Water-to-cementitious ratio of mix 250 is higher than 350P.

Mix D1 is a common structural concrete produced in GCC Ready-mix plants.

6.2) Plasticizer consumption

The quantity of plasticizer added to the mixes 1-3 (at all rates of pozzolan substitution) was adapted to reach the same workability in term of slump (16-20 cm). This slump class is the most commonly used in the market. Figure 12 shows the plasticizer added for each mix.



Figure 12: Rate of plasticizer added in order to reach the target slump value

Microsilex behaves in a systematic way for all mixes, showing a lower plasticizer demand than Silica Fume 1 to maintain the workability. Silica Fume 1 behaves unsystematically with respect of the plasticizer to be added and the consumption tends to increase dramatically when the rate of substitution increases. The lower binder content mix (test 250) represents an exception because plasticizer consumption tends to decrease when **Microsilex** rate increase. This is probably due to the type of mixture.

Each mix that contains **Microsilex** tends to provide very good fluidity without stickiness while in the Silica Fume 1 concrete the target slump is reached (even though more superplasticizer has to be added); however the mixture tend to become sticky and viscous. Stickiness of the mix negatively impacts the finishing of the exposed surfaces.



6.3) Rheology

Fresh concrete behavior is characterized by many parameters. A good slump, generally obtained by adding a plasticizer, might not be sufficient to have a workable concrete. This is because some concrete tends to be sticky and to augment their stickiness with the increase of plasticizer and because they might be difficult to mix and homogenize with common batching equipments. To check these properties, rheology tests are made using equipments called Rheometers. This equipment is similar to a consistometer but more sophisticate because it is able to measure the torque force that is necessary to input on the shaft in order to shear the concrete and thus to keep constant the beater speed. Tests were conducted using five constant speeds from 50 to 250 rpm, sequences of programmed speed used are presented on the Figure 13.



Figure 13: Target speed of the rheometer as a function of time

The mixtures tested were based on the mix 350L where the concrete was modified with the method MBE (Mortar Equivalent Concrete) to reproduce the same rheology of real concrete and avoid noises on the signal due to the gravel impact on the beater. Figure 14 shows the torque force that the machine has to input on the shaft for keeping the speed constant. The base mixture 350L was plasticized with 0.7% of HRWR and then cement was substituted with 10% of **Microsilex** and Silica Fume 1. Torque force evolution is presented in Figure 14.

Microsilex



Figure 14: Torque force measured at the shaft against time

The first shearing of the concrete at an imposed speed of 250 rpm (see Figure 13) speaks for a large force on the shaft required in order to move the beater immersed in the Silica Fume 1 mix. Immediately after the first shearing at 250 rpm, the speed is first lowered to 5 rpm, then brought up step by step to 50, 100, 150, 200, 250 rpm and then lowered to 200, 150, 100, 50 rpm. Also in this sequence Silica Fume 1 mix requiring more torque force to be sheared by the beater and present a pronounced shear thinning.

The plot of the stress strain-curve (average torque force-average speed for each speed step, see Figure 15) shows that Silica Fume 1 mix has an higher yield stress (critical soil of shearing) and presents a large hysteretic loop, typical of a pronounced thixotropic behavior. When values of yield stress and the dimension of the hysteretic loop are large the concrete is likely to be "sticky". Sticky mixes with low plasticizer content tend to stick to the wall of the mixer and promote lumps formation. When the dosage of the plasticizer is increased, the mix does not stick to the wall of the mixer, but is likely to have a reduced mobility with the typical honey-like consistency and tends to stick to the trowel when finished. This was the case of the silica fume 1 mix of Figure 15.





Figure 15: Stress-strain behavior of concrete with and w/o Microsilex (MS) and Silica Fume 1 (SF1)

6.4) Compressive and flexural strengths

Compressive strength and flexural strength are two fundamental characteristics of concrete. **Microsilex** has been tested and used in several situations as strength enhancing ingredient by several GCC clients, as well as to design many special, tailored made products such as colloidal suspension, ultra high performance self leveling concretes, light-weight concretes, etc. Tests were conducted for all mixes presented in Tables 9 and 10 in standard cylinders for compressive strength and standard beams for flexural strength (see Figures 16 - 19).



Figure 16: Compressive strength of concretes (350P, 350L and 250) at 3, 7 and 28 days with and w/o Microsilex (MS) and Silica Fume 1 (SF1)













Figure 19: Flexural strength of concrete (250 and 350L) at 3, 7 and 28 days with and w/o Microsilex (MS) and Silica Fume 1 (SF1)



At early ages, **Microsilex** enhanced the compressive strength of concrete unlikely silica fume which sometimes underperforms. This trend is confirmed at later age; in particular at 28 days, the strength of every concrete is enhanced by **Microsilex** and by silica fume at the same level.

Flexural strength is very much dependant on coarse aggregate content and type. SCM might enhance the bond strength but the main driving force for flexural strength remains the coarse aggregate characteristics. As it is possible to see in Figures 18 and 19, the flexural strength is improved by **Microsilex** and silica fumes but not in a systematic way. Whatever silica fume is used, the performance of **Microsilex** at 28 days is comparable in magnitude. At early ages, **Microsilex** outperforms the reference.

6.5) Durability

Durability could be equated to sustainability with respect to concrete constructions. A durable concrete is a concrete that will have a long service life without major maintenance and repair interventions. Aggressive environments such as seaside, high traffic areas, freezing-thawing and industrial plants require the use of a carefully tested concrete in order to meet the service life requirements. **Microsilex** has often proved to be the key ingredient to enhance the durability of an ordinary concrete at levels that meet strict durability specifications.

Concrete's ability of resist Ability to resist chloride ion penetration

Mixture 4 was tested in accordance with ASTM C1202 rapid-chloride penetration test (RCP) (see Figure 20). The results show that **Microsilex** can reduce by more than half the permeability of the reference concrete, reaching a very good permeability value, which was comparable to the Silica Fume 2 mixture. This characteristic indicates an improved durability of concrete in aggressive environments.





The RCP test is often criticized because of its precision, even though it is the reference test and is widely used. However, in this case, it is possible to observe that without any doubts, the chloride-ion permeability of both mixtures incorporating **Microsilex** and Silica Fume 2 are drastically reduced ensuring protection against corrosion and the ingress of aggressive salts. It is not possible to discriminate whether or not Silica Fume 2 and **Microsilex** bring the concrete on the low or very low permeability zone due to the high variability of this test.

<u>Standard Test Method for Potential Alkali Reactivity of Cement-Aggregate</u> <u>Combinations (Mortar-Bar Method)</u>

Alkali-silica reaction potential is a major concern in regions where the local aggregates are susceptible to alkali-aggregate reaction. Alkali-silica reaction is particularly dangerous because might become evident only after many years and often results in a demolition of the structure attacked. Remediation measures for concrete attacked by this particular reaction are few, expensive and not always efficient. It is thus of capital importance to exclude from the beginning the possibility of alkali silica reaction. ASTM C227 and C1260 test should be used to check the reactivity of specific aggregate in concrete when an aggregate is suspected of being alkali-reactive.

Results of test ASTM C227 test (see Figure 21) show that mixtures incorporating **Microsilex** drastically reduces the potential for alkali-silica reaction with respect to the reference. **Microsilex** and Silica Fume 2 at 10% substitution are comparable.



Figure 21: Alkali-aggregate expansion based on mix D1 and tested according to the standard ASTM C227



ASTM C1260 testing was also performed using a reactive aggregate that was provided by GCC customer. It is possible to see in Figure 22 that **Microsilex** provides good mitigation of ASR in this case as well.



Figure 22: Alkali-aggregate expansion based on mix D1 and tested according to the standard ASTM C1260 using two rates of **Microsilex** addition

Freeze and Thaw

Damage due to repeated cycles of freezing and thawing occurs when water penetrates into concrete and expands when freezing. Expansion-contraction of water inside concrete loads the matrix with a fatigue-like mechanism. It is clear that in environments where freeze and thaw cycles occur many times during the winter season, concrete integrity might be at risk. To reduce potential for damage due to this mechanism, less permeable matrix can be used in conjunction with a proper air void system.

Results of test ASTM C666 in mix D1 (see Figure 23) show that the freeze-thaw resistance is slightly enhanced by the presence of **Microsilex** at any rates (10% and 20%): **Microsilex** is able from the very early age to close the porosity (giving a denser matrix) of concrete. A visual inspection was also done after at the completion of testing indicating that the specimens are freeze-thaw durable.



Figure 23: Freeze-thaw test for mix D1 with 10 and 20% Microsilex addition

A series of tests on sorptivity conducted using ASTM C1585 tests, as well as a research protocol employed for determining the early age sorptivity of concrete, corroborate the freeze/thaw findings (see Figure 24).





Figure 24: Sorptivity tests for concrete mix 250 with and w/o Silica Fume 1 and Microsilex

7) Conclusions

Tests show that **Microsilex** is a very effective pozzolan with no adverse effects on fresh cementitious properties. When compared with silica fume, **Microsilex** has similar or better properties especially at early ages. The performance of mixtures incorporating **Microsilex** is excellent and the durability is to a large extent improved.

Even if **Microsilex** has larger mean particle size than silica fume, it is more reactive than silica fume. This is due to its high specific surface area that compensates for the higher percentage of amorphous phase and higher SiO_2 content of silica fumes.

The internal porosity observed on **Microsilex** particle explains the high reactivity of **Microsilex** and its excellent effect on physical and mechanical properties of hardened cementitious materials. **Microsilex** particle size distribution allows for a better handling when pumped from trucks to silos.

The stable composition of **Microsilex** and flexible rates of production ensure clients uniformity of behavior in concrete properties.

Testing on mortars and concrete confirms that **Microsilex** is an excellent ingredient for enhanced strength and durability properties when used as a replacement of or an addition to portland cement. Furthermore **Microsilex** does not have the negative effect of silica fumes on fresh concrete properties.

Safety packages according the USA protocols are available to ensure clients against any health concerns for their staff.

microGilex

Dr. Armando García Luna

Research & Development Director Phone 41 (24) 423 5382 Fax 41 (24) 423 5388 argarcia@gcc.com SWITZERLAND

Hank Hearon

Phone 1 (303) 739 5911 Fax 1 (303) 739 5939 hhearon@gcc.com USA

Ernesto Moreno Mona

Innovative Products Manager New Markets & Product Development Manager Phone 52 (656) 227 1600 Fax 52 (656) 227 1581 emorenom@gcc.com MEXICO



www.gcc.com

Microsilex is a high surface area microsiliceous pozzolan that combined with Portland cement and water improves the physical-chemical properties of the plain binder. When used in combination with Portland cement, Microsilex allows the increase of the mechanical properties in term of strength as well as the durability in term of resistance to sulfate attack, chloride penetration and by reducing the risk of alkaliaggregate reactivity. Furthermore, due to its carefully studied particle size distribution, Microsilex is easier to handle and operations such as truck unloading can be performed faster. Microsilex is an environmentally responsible product since the energy consumption to produce Microsilex is much lower than to produce common binders.

COMMERCIAL TESTING LABORATORIES



A DIVISION OF CTL/THOMPSON, INC.

Chemical and Physical Analysis of Fly Ash

	Developed For:	Zeolite Co. P.O. Box 64 Tompson Fa	3 alls, MT 5	59873		
Ticket: 3602 Job: 11749 Report Date: 01/22/200	Plant of Origin: Sample ID: Docket:	Bear River		Sample Dat Date I	te Range: 12/04/20 to: Received: 12/04/20	03 03
Chemica	I Composition (%)			ASTM C 618-03	Specifications Class C	
Tot	al Silica, Aluminum, Iron:	85.9		70.0 Min	50.0 Min	
	Silicon Dioxide: Aluminum Oxide:		71.6 11.3			
	Iron Oxide:		2.9			
	Sulfur Trioxide: Calcium Oxide:	0.0 2.8		5.0 Max	5.0 Max	
	Moisture Content: Loss on Ignition:	5.2 4.5		3.0 Max 6.0 Max	3.0 Max 6.0 Max	
Phy	sical Test Results			ASTM C 618-03	Specifications Class C	
Fineness, R	etained on #325 Sieve (%):	3.9		34 Max	34 Max	
:	Strength Activity Index (%)					
R	Ratio to Control @ 7 Days: atio to Control @ 28 Days:	76.2 111.6		75 Min	75 Min	
Water R	equirement, % of Control:	116.5		105 Max	105 Max	
Soundness,	Autoclave Expansion (%):	0.01		0.8 Max	0.8 Max	
	Density:	2.22			DO RECO	

Comments:

Commercial Testing Laboratories

with A Nener



Orville R. Werner II, P.E.

Pumice Pozzolan: The Original Pozz

SOCIETY WIDELY "REDISCOVERED" THE BENEFITS of pozzolanic-charged concrete when the coal-fired power generation industry was looking for a market to use the fly ash they were scrubbing from their stacks. Yes, fly ash works as a replacement pozzolan, but the original pozz—pumice—is consistently better.

Anciently, the Romans used fine-grained pumice to greatly enhance the strength and durability of their concrete—and the evidence of their concrete wisdom still stands some 2000 years later. The amazing benefits of the pumice-ignited pozzolanic charge have been reaffirmed by modern research, detailing how HessPozz significantly improves concrete density, strength, and durability. Additionally, thanks to modern refining processes, those benefits are consistent and predictable, pour after pour.

HessPozz is made by precisely refining a clean, pure pumice—an amorphous white silica created by volcanic events millennia ago. Pumice pozzolan is not a by-product of pollution control processes. It contains no hazardous materials. It is a natural pozzolan, born in the bowels of Nature's most fearsome monuments: volcanoes.

Amping the Performance of Standard Concrete. Almost as soon as standard concrete is placed, the process of degradation begins. Recent studies suggest that only about 75% of the cement powder is converted to Calcium Silicate Hydrate (CSH), the binder that glues concrete together. Most of the remaining 25% is converted to Calcium Hydroxide (CH), a by-product of the hydration reaction between water and cement—a by-product that has a tendency to create a host of problems that have a frustrating effect on the long-term performance, even appearance, of concrete. Adding a high-purity natural pumice pozzolan to the concrete formulation mitigates or completely eliminates the CH problem. In fact, the pozzolanic reaction ignited by the pumice converts the

deleterious CH into additional CSH, strengthening and densifying the concrete—essentially *consuming the problem and repurposing it* to amplify and enhance desirable performance.

Benefits of Pumice Pozzolan

Specifically, ASTM-standards research documents that using Hess StandardPozz and UltraPozz in concrete formulations—

- Enhances Compressive Strength
- Fortifies Against Chloride Attack
- Increases Resistance to Sulfate Attack
- Significantly Mitigates or Eliminates ASR
- Reduces Heat of Hydration Cracking
- Augments Abrasion Resistance
- Greatly Reduces Permeability
- Improves Durability and Appearance

Strength and Beauty. Not only is HessPozz ideal for projects that call for high-performance, long-lasting concrete, but our high-quality, white pumice pozzolan (84 GE Brightness) is also used for all forms of precast concrete whenever consistent color and visual appeal is important—including, but not limited toCompleted in 126 AD, the enduring Pantheon in Rome, and in particular the dome, was constructed with pumice aggregate and pumice pozzolan-enhanced concrete. Almost 2000 years later, the Pantheon still boasts the world's largest unreinforced concrete dome.



For over 2000 years, the sprawling Roman Empire of Concrete—the piers, aqueducts, temples, coliseums, roads and statuary—has withstood the relentless assault of time. The secret: Pumice Pozzolan.

HESS POZZ GRADES

Hess StandardPozz DS-325

FARTICLES	IZE OF LCIFICATION	
Dx	Micron Size	
D50	14-16	

Hess UltraPozz NCS-3

PARTICLE SIZE SPECIFICATION				
Dx	Micron Size			
D50	2 - 4			

CHEMICAL COMPOSITION

Common Name: Pumice Chemical Name: Amorphous Aluminum Silicate Silicon Dioxide - 76.2% Aluminum Oxide - 13.5% Ferric Oxide - 1.1% Ferrous Oxide - 0.1% Sodium Oxide - 0.1% Sodium Oxide - 1.6% Potassium Oxide - 1.6% Calcium Oxide - 0.9% Calcium Oxide - 0.9% Sulfate - 0.0043% Magnesium Oxide - 0.05%

Have specific questions? Want to do in-house testing in your own lab? Contact us.



Hess Pumice Products Post Office Box 209; 100 Hess Drive Malad City, Idaho 83252 1.800.767.4701 x 111 pozzinfo@hesspumice.com www.hesspozz.com



cast statuary, GFRC panels, tilt-up panels, architectural elements, and manufactured stone veneer.

Performance Research

These benefits have been documented via extensive (and on-going) studies by the University of Utah, UT-Austin, Washington University, Clemson University and others. Check out the information found on our website at www.hesspozz.com

Environmental, Health and Safety

Recent studies corroborated previous test data indicating that natural pumice pozzolans are free of Crystalline Silica and other hazardous materials. Hess pozzolans are so safe they are also used as a mild abrasive to clean teeth and as an exfoliation agent in skin creams. While by-product pozzolans struggle with regulated contaminants, Hess Natural Pozzolan is an environmentally safe, health-friendly choice.

Green Stuff. Naturally calcined, pumice pozz is effective, abundant and green! In some applications, HessPozz can replace up to 40% of the Portland cement typically needed, thus reducing, on a pound for pound basis, the colossal carbon footprint standard cement leaves behind while, *at the same time*, amplifying concrete performance in terms of durability, strength, and appearance.

Availability

Mined and refined in Southeast Idaho, USA, we offer two pozz grades: Hess StandardPozz (DS-325) and Hess UltraPozz (NCS-3). Our extensive logistical expertise means we can then ship it anywhere on the planet—packaged in 44 lb/20kg bags, 1-ton super sacks, or in bulk pneumatic rail cars and tanker trucks.

Reliable Supply. Hess has been a family-owned company delivering reliable service for more than five decades. The company promise of "On-spec, On-time" has given customers confidence for equally as long.

MITIGATING ALKALI SILICA REACTION

Mortar mix designs tested according to a modified ASTM C1567 procedure using Type 1 cement and 25% replacement of fine aggregate with ground cullet glass. The percent length change for "acceptable expansion" is less than 0.10% at fourteen days with reactive aggregates. (U of Utah Study)

MIXTURE	ASR % LENGTH CHANGE	RATING
100C 25%Glass	0.699	Deleterious Expansion
80C20DS325 25%G	ilass 0.029	Acceptable Expansion
70C30DS325 25%G	ilass 0.011	Acceptable Expansion
80C20Ultra 25%Gla	ass 0.017	Acceptable Expansion





1.800.767.4701 x 111 • www.hesspozz.com pozzinfo@hesspumice.com

ACTIVITY INDEX

Determined in accordance with C595 Annex A1. Activity Index is calculated by dividing the average compressive strength of test mixture cubes with average compressive strength of control mixture cubes. (U of Utah Study)

MIXTURE	ACTIVITY INDEX
100C	
80C20Ultra	131.2
70C30DS325	92.4
80C20DS325	69.8

SHRINKAGE

Mixture designs were tested for length change in 6"x12"cylinder concrete specimens. The addition of ultrafine pumice reduced the length change (shrinkage) compared to 100% cement. (U of Utah Study)



SULFATE MITIGATION

Per ASTM C1012, mortar mixture designs were tested for sulfate resistance through 6 months. Mixtures containing pumice are classified as HS (High sulfate resistant cement) as the length change is less than 0.05% after 26 weeks. (U of Utah Study)



RESISTIVITY AT DIFFERENT TIME INTERVAL IN k Ω -cm

ASTM C192 procedure was followed to make 6"x12" cylinders and moist cured according to ASTM C511. Resistivity increases over time for the mixture with pozzolans whereas it remains relatively constant for the mixture with 100% portland cement. (U of Utah Study)

MIXTURE	14 DAYS	24 DAYS	58 DAYS
100C	4.1	4.6	6.8
80C20DS325	4.3	5.5	15.7
70C30DS325	4.3	6.3	19.2
80C20Ultra	5.1	10.5	33.8



Appendix B – Adiabatic Temperature Rise Curves

GCC Microsilex





Bear River Zeolite







HessPozz







