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Development of Magnesium Phosphate Cement (MPC) Concrete Mixture Proportioning for Airfield Pavements

Laboratory and Field Validation MPC Test Report

Monica A. Ramsey, Dylan A. Scott, Charles A. Weiss Jr.,
and Jeb S. Tingle

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Development of Magnesium Phosphate Cement (MPC) Concrete Mixture Proportioning for Airfield Pavements

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Abstract

Magnesium phosphate cements (MPCs) have been used in proprietary products for pavement repairs for over 30 years. However, these products generally are intended for small repair sections less than 0.5 ft³ due to high heat generations and short working times.

The objective of this research was to develop optimal mixture proportions of MPCs for use in pavement applications. Materials used in this research included three types of magnesium oxide (MgO), one phosphate salt, two types of fly ash, a retarder, two coarse aggregate gradations, and one sand. From these materials, 24 mixture proportions were batched with variations including water content, retarder, and fly ash to determine the effects on physical and mechanical properties.

Laboratory testing at various scales was conducted on a mixture consisting of a dead-burned MgO, potassium dihydrogen phosphate, Class C fly ash, boric acid, and local aggregates. Four variations were developed and commercially blended in bulk super sacks. These concrete mixtures met the workability, setting time, and strength requirements. A volumetric mixer was used to batch 2 yd³ field placements in test sections 8.5 ft wide x 8.5 ft long x 8 in. deep. This was noteworthy because no literature was discovered for MPC concrete placements at this large volume.

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Preface

This study was conducted for the Air Force Civil Engineer Center (AFCEC) located at Tyndall Air Force Base in Panama City, FL, under MIPR F4ATA48226JW01. The AFCEC technical manager of this project was Dr. Craig Rutland.

The work was performed by the Airfields and Pavements Branch (GMA) and the Concrete and Materials Branch (GMC) of the Engineering Systems and Materials Division (GM), U.S. Army Engineer Research and Development Center, Geotechnical and Structures Laboratory (ERDC-GSL). At the time of publication, Dr. Timothy W. Rushing was GMA Chief; Mr. Christopher M. Moore was GMC Chief; Mr. Justin S. Strickler was GM Chief; and Mr. R. Nicholas Boone was the Technical Director for Force Projection and Maneuver Support. The Deputy Director of the ERDC GSL was Mr. Charles W. Ertle II, and the Director was Mr. Bartley P. Durst.

COL Teresa A. Schlosser was the Commander of ERDC, and Dr. David W. Pittman was the Director.

1 Introduction

Magnesium phosphate cement (MPC)-based products are increasingly being used as a rapid-repair material for highway and airfield pavements. There are many advantages of MPCs over conventional ordinary portland cements (OPCs) including heat resistance, quick-setting time even at low temperatures, high early strength, good bond to existing concretes, acid resistance, chemical and mold resistance, durability with respect to freezing and thawing, low-drying shrinkage, low coefficient of thermal expansion, and reduction of CO₂ emissions. Because of these favorable properties, MPCs are used on many small-scale concrete repair projects for heavily trafficked roads and airfield damage repair.

Engineering Technical Letter 97-5, *Proportioning Concrete Mixtures with Graded Aggregates – A Handbook for Rigid Airfield Pavements*, is a document that guides the production of concrete for use in airfield construction (AFCEC 1997). However, this document does not include guidance on the MPCs. This research is intended to assist the Air Force Civil Engineering Center (AFCEC) in the development of guideline tools to better understand the exothermic acid-based reaction mechanism of the hydration of MPCs and to control the setting time to provide sufficient time for casting and finishing. These guidelines will help engineers and scientists in material selection, mixture proportioning, and construction techniques that will ensure long-term performance and durability for airfield pavement repairs.

1.1 Problem statement

MPCs have several limitations for use in pavement repairs. The driving reaction is an acid-base reaction that is highly exothermic and can result in short working times. Other disadvantages of MPCs include the emission of ammonia gas when an ammonium phosphate is used, lower strengths when set-retarders are used, and a reduction in the 28-day material strength when water curing is used (Qiao 2012, Qiao et al. 2014).

Although MPCs have been used for decades as proprietary products, they are generally intended for small repair sections less than 0.5 ft³, and the mixture proportions of the binders are not completely known due to patent and trade secrets. In addition, companies frequently re-formulate

their products over the years often under the same brand name. This raises the potential of adverse reactions between the repair materials and the parent pavement surface.

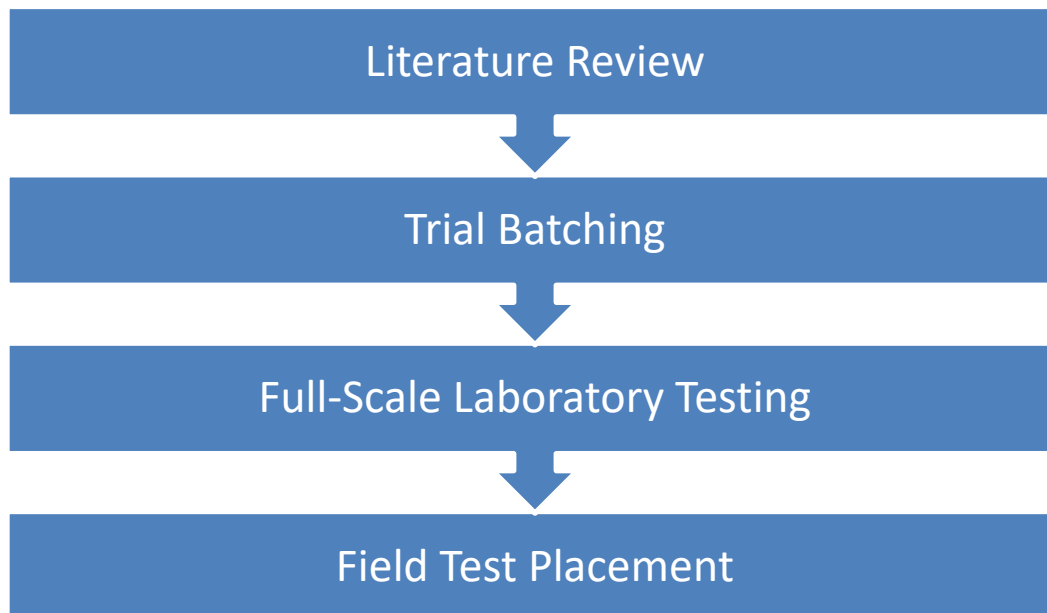
1.2 Objective

The objective of this research was to develop specifications for the use of MPCs in pavement applications and to overcome the primary unfavorable properties of MPCs, including short working times and high exothermic reactions. The goal was to develop an MPC mixture for large-volume concrete placements. The desired properties included (a) 5,000-psi compressive strength at 28 days, (b) 500-psi flexural strength at 28 days, (c) 60-min setting time, (d) good bond strength, (e) adequate workability, (f) good durability, and (g) wide availability of raw materials.

1.3 Research approach

Concrete mixes were designed by selecting the proportions of the raw material components to develop the required strength, produce a workable consistency concrete that can be handled and placed easily, and attain sufficient durability under exposure to in-service environmental conditions. Figure 1 illustrates the research approach to develop an MPC mixture design for pavements.

Figure 1. Research approach to develop an MPC mixture design for pavements.



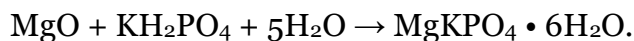
2 Literature Review

A review of literature was conducted pertaining to the use of MPC-based repair materials. Research focused on the general mixture constituents used to develop the products, test methods used to evaluate the materials, and factors that affect their properties. Only fundamental properties selected for testing in the present research are briefly summarized in this literature review.

2.1 Introduction

MPC is a type of cementitious binder formed from an acid-base chemical reaction between a type of magnesia and a soluble acid phosphate, such as ammonium or potassium phosphate, accompanied by hydration. MPCs can be classified as a chemically bonded ceramic (CBC), which refers to the bonding that takes place in a chemical reaction at low temperature as opposed to fusion or sintering at elevated temperature (Roy 1987). Bonds in CBC bond are predominately ionic and covalent bonds with some van der Waals bonds. Traditional cement hydration products are predominately van der Waals and hydrogen bonds.

The chemical acid-base reaction of the magnesium oxide and monopotassium dihydrogen phosphate in solution is frequently cited as



The main hydration product of MPC paste is considered to be struvite ($\text{MgKPO}_4 \cdot 6\text{H}_2\text{O}$), which has both crystalline and amorphous phases (Wilson and Nicholson 1993; Park et. al 2016; Soudee and Pera 2000). The amount of unhydrated MgO is most dominant in either early or late hydration (Yang and Wu 1999). Besides struvite, other minerals can form, which makes the quantitative analysis of this material complicated. The dissolution kinetics of MgO is complicated and depends on factors including the extent of dissolution, pH, surface structure, hydration, species in solution, and impurity and dopant concentrations. Because of the substantially different chemistry, one cannot directly compare MgO-based cements to conventional or alternative cements.

2.2 Mixture constituents of MPC

This section briefly describes the mixture constituents identified in the reviewed literature including magnesium oxide (MgO), phosphates, supplementary cementitious materials (SCM), retarders, aggregates, and water.

2.2.1 Magnesium oxide

Magnesium oxide (MgO), also known as magnesia, is produced either naturally from the calcination of mined magnesium-based minerals or synthetically through treatment of seawater, well and lake brines, and briniters. Magnesium is the eighth most abundant element and constitutes about 2% of the Earth's crust (USGS 2016). Although magnesium is an element in more than 60 minerals, the main source for MgO worldwide is magnesite, or magnesium carbonate (MgCO_3). Other mineral sources include dolomite ($\text{CaCO}_3 \cdot \text{MgCO}_3$), brucite ($\text{Mg}(\text{OH})_2$), and olivine ($\text{Mg}_2\text{Fe}_2\text{SiO}_4$). Magnesia produced from magnesite can contain between 88 and 98% magnesia, with varying quantities of alumina, calcium, iron, and silica impurities. Synthetic magnesia normally is purer than natural magnesia, containing between 92 and 99.5% magnesia, with smaller quantities of other compounds (Kramer 2001).

In 2019, seawater and natural brines accounted for about 57% of U.S. magnesium compound production (USGS 2019). Magnesium oxide and other compounds were recovered from well and lake brines in California, Delaware, Michigan, and Utah and from seawater. Magnesite was mined by one company in Nevada. One company in Washington processed olivine that had been previously mined.

MgO is typically classified by calcination temperature, which has resulted in varying definitions. Canterford (1985) defined three forms of magnesia and calcination temperatures: caustic-calcined magnesia, 600 to 1300°C; dead-burned magnesia, 1600 to 2200°C; and fused magnesia, >2800°C. The U.S. Geological Survey (USGS) uses the same terminology to reference magnesia products (USGS 2016). In contrast, a producer of MgO commonly cited in much of the technical literature uses the following grades and calcination temperatures: light-burned (LB), 700 to 1000°C; hard-burned (HB), 1000 to 1500°C; and dead-burned (DB), 1500 to 2000°C.

The reactivity of MgO plays a significant role in the effectiveness of the concrete. Reactivity is a measure of how quickly MgO reacts with the acid phosphate. The reactivity of MgO is a result of its chemical composition, temperature of calcination, particle size distribution, surface area, and loss on ignition specific gravity. Lower reactivity means more MgO passes through the system without having much effect. In general, the reactivity of MgO decreases with increasing calcination temperature.

2.2.2 Phosphates

Phosphates commonly used in reaction with MgO include ammonium dihydrogen phosphate ($\text{NH}_4\text{H}_2\text{PO}_4$ or ADP), potassium dihydrogen phosphate (KH_2PO_4 or KDP), dipotassium hydrogen phosphate (K_2HPO_4 or DHP), and sodium dihydrogen phosphate (NaH_2PO_4 or SDP). The ADP and KDP salts were used most frequently among researchers, as shown in Table 1. A commonly reported problem with ADP is the release of ammonium gas during the hydration process, which creates an unpleasant odor and may lead to corrosion.

Table 1. Phosphate salts used in various studies.

Phosphate Salt	Reference
Ammonium Dihydrogen Phosphate ($\text{NH}_4\text{H}_2\text{PO}_4$ or ADP)	Abdelrazig et al. (1988; 1989); Seehra et al. (1993); Yang and Wu (1999); Hall et al. (2001); Ding and Li (2005); Nim et al. (2013); Shijian and Bing (2014)
Potassium Dihydrogen Phosphate (KH_2PO_4 or KDP)	Qiao et al. (2010); Ma et al. (2014); Li and Chen (2013); Park et al. (2016); Nicu et al. (2016)
Dipotassium Hydrogen Phosphate (K_2HPO_4 or DHP)	Shijian and Bing (2014); Gardner et al. (2015); Li et al. (2016)
Sodium Dihydrogen Phosphate (NaH_2PO_4 or SDP)	Seehra et al. (1993); Nim et al. (2013)

Shijian and Bing (2014) compared MPC mortars made with both ADP and KDP and found the exothermic reaction of ADP was stronger than that of KDP. Faster setting times and higher temperatures were attributed to solubility. The solubilities (mol./100 g H_2O) of ADP and KDP are 0.2484 and 0.147, respectively. Hence, the higher solubility of ADP is responsible for the faster reaction. Likewise, Ding and Li (2005) documented that

KDP has a lower dissociation content and molar solubility that contributes to reducing the reaction rate.

2.2.3 Supplementary cementitious materials

Supplementary cementitious materials (SCM) such as fly ash are commonly used as fillers with MPCs to control the rate of reaction, reduce water demand, enhance physical and mechanical properties, and possibly reduce production costs. The ASTM C618 (2017a) defines two classes of fly ash for use in concrete: (1) Class F, usually derived from the burning of anthracite or bituminous coal, and (2) Class C, usually derived from the burning of lignite or subbituminous coal. ASTM C618 also delineates requirements for the physical, chemical, and mechanical properties for these two classes of fly ash. Class F fly ash is pozzolanic, with little or no cementing ability on its own. Class C fly ash is principally used as a pozzolan in cement-based composites but also has self-cementing properties. Generally, a Class C fly ash has more than 10% by weight CaO, and a Class F has less than 10% by weight of CaO.

Yang et al. (2000) investigated the effects of fly ash replacement (up to 30 wt%) of MPC and found at the same water to binder (W/B) ratio the fluidity is enhanced with increasing fly ash replacement, but the compressive and flexural strengths are reduced. However, at similar fluidity, the strengths increase with fly ash replacement. On the contrary, Ding and Li (2005) reported up to 40% of a Class F fly ash can be added to the system without degrading the mechanical properties.

Another study by Wagh et al. (1997) compared the effects of both a Class F and a Class C fly ash on compressive strength at loadings up to 80 wt% and found the Class C fly ash produced the highest strengths at a 60 wt% optimal loading. The authors postulate Class C fly ashes are more compatible with MPC materials due to higher levels of calcium that may react with remaining phosphates in the system. This leads to a denser, stronger, less-permeable material.

Although fly ash was the most common SCM discovered in the reviewed literature, other SCMs used with MPC include slag cement, red mud, silica fume, and metakaolin. (Nicu et al. 2016; Gardner et al. 2015; Unluer and Al-Tabbaa 2015).

2.2.4 Retarders

Cements based on MgO are fast-setting materials that generate high exothermic heat. The addition of a retarder is critical to slow the reaction rate for sufficient casting and finishing time. Setting times can be extended by using suitable retarders such as boric acid (H_3BO_3), borax ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$), or sodium triphosphate ($\text{Na}_5\text{P}_3\text{O}_{10}$ or STP). Borax was the most commonly used retarder identified in the reviewed publications presented in Table 2. However, a study by Park et al. (2016) found that borax did not always reduce the rate of the hardening process and cautioned care be taken using borax to increase the setting time. Research using boric acid primarily focused on microstructural characterization, and mixture proportioning using boric acid as a retarder appeared insufficiently explored.

Table 2. Retarders used in various studies.

Retarder	Reference
Borax ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$)	Seehra et al. (1993); Yang and Wu (1999); Hall et al. (2001); Ding and Li (2005); Li et al. (2016); Yang et al. (2000); Qiao et al. (2010); Yang and Qian (2010); Yang et al. (2014); Shijian and Bing (2014); Park et al. (2016)
Boric Acid (H_3BO_3)	Nicu et al. (2016); Soudee and Pera (2000); Hall et al. (2001); Ding and Li (2005); Ribeiro and Morelli (2009); Gardner et al. (2015)
Sodium Triphosphate ($\text{Na}_5\text{P}_3\text{O}_{10}$ or STP)	Abdelrazig et al. (1988, 1989); Seehra et al. (1993); Nicu et al. (2016); Hall et al. (2001); Ding and Li (2005); Ribeiro and Morelli (2009)

Hall et al. (2001) investigated the effect of all three retarders and found that successive additions of boric acid and borax led to a cumulative increase in setting time to a maximum of 1 hr. Interestingly, the fractional mass of boron in boric acid is approximately 0.175, while in borax it is 0.113. Therefore, if the effectiveness of these retarders was solely dependent on the boron content, then the mass of borax added to MPC would need to be a factor of 1.55 greater than that of boric acid in order to produce an equivalent retardation effect. The effect of STP was quite different and exhibited poor workability and a maximum set time of only 15 min. The authors postulated that the retarding action is limited by the solubility of STP and that the stiffening of the wet mix resulted in premature setting.

In contrast, Abdelrazig et al. (1988) reported a beneficial effect, when STP was incorporated, of improving the workability. However, the authors' primary focus investigation on the STP was not on the time of set but rather the influence of the morphology of the hydration products formed and porosity and compressive strength of the mortar.

2.2.5 Aggregates

Common fine aggregates identified in previous research include natural river sand, magnesia sand, granite sand, and alumina sand. Yang et al. (2000) tested five types of fine aggregates for the MPC mortar and found the order of strength was quartz sand > granite sand > river sand > slag > limestone. This indicates sands containing minerals with high CaO such as CaCO₃ are not suitable for MPC mortars. A similar study by Chong et al. (2017) found that the addition of limestone aggregate into the MPC matrix accelerated the setting time, decreased the total heat evolution, and degraded the strengths. The authors concluded the poor water stability of MPC with limestone might be caused by the poor crystallinity and crystal morphologies of hydration products, worsened pore structure, and increased dissolution of hydration products.

Coarse aggregates can be used with MPCs but literature revealed limited research. A pea gravel with a 3/8-in. nominal maximum size aggregate (NMSA) was used in a laboratory and field study using proprietary MPC-based products (Priddy 2011). Limestone aggregates were eliminated from this study based on compatibility and potential expansion.

2.2.6 Water

The cementing process does not proceed without the presence of water. As with traditional OPC, increasing the W/B ratio improves the workability and increases the setting time, but also decreases the strengths of the materials. Ding and Li (2005) reported that water acts as both solvent and component in the formation of the cements. First, water is a solvent when chemical reaction takes place, and the solvent is not merely a passive medium in which relevant molecules perform; the solvent itself makes an essential contribution to the reaction. Second, water acts as an important component of the hardened cement. After setting and hardening, part of the water becomes one component of the reaction products, which are usually salt with crystallized water (Wilson and Nicholson 1993). Potable water was the most used source in literature reviewed.

2.3 Test methods and factors influencing properties of MPC

The purpose of this research was to determine whether MPC-binder materials could be used to develop a concrete for pavement repairs with properties superior to those of portland cement concrete (PCC). A number of sources were reviewed to establish the important properties to consider for a successful repair material. In addition to the factors affecting the raw material proportioning, there are many parameters such as temperature, curing conditions, etc., that have significant influence on a repair material. One recurring theme in publications was the need for compatibility between the repair material and the parent substrate to form a composite system. Guidance provided in testing criteria for evaluating cementitious pavement repair materials (Priddy 2011; Ramsey and Tingle 2018¹) recommends considering the material's compressive strength, flexural strength, bond strength, modulus of elasticity, setting time, length change, and coefficient of thermal expansion when choosing a repair material. Keeping this in mind, an effort was made to find studies that had tested MPC materials in an attempt to identify appropriate laboratory and field tests for the given properties.

Several test methods for MPCs were investigated by Popovics and Rajendran (1987) including mixing, flow, setting time, air content, compressive strength, flexural strength, shear bond strength, length change, specific gravity, absorption, and voids content. It was found that strengths were reduced when wet curing was used; therefore, air curing was recommended with these materials. It was also discovered the epoxy bond system did not work well with bond strength specimens due to the rapid heat generation during setting. Therefore, composite cylinders were prepared without any epoxy bond and tested for shear bond strength. The dry curing and non-epoxy preferences of the MPC materials can be seen as a significant advantage for field repair application. Often, it is difficult to ensure adequate access to water and equipment for moist curing conventional concrete. In addition, more time is required to prepare the existing concrete substrate with epoxy before applying a repair material.

¹ Ramsey, M. A., and J. S. Tingle. 2018. *Evaluation of rapid-setting cementitious materials and testing protocol for airfield spall repair*. ERDC/GSL TR (Draft). Vicksburg, MS: U.S. Army Engineer Research and Development Center.

Less site preparation and monitoring is required, saving both time and money without sacrificing the material's performance.

Only a few works in literature demonstrate field applications. Yang et al. (2000) experimentally used MPC mortars for repairs including potholes, cracks, surface scaling, and edge spalling on main municipal roads. The MPC with a phosphate to magnesia (P/M) ratio of 1:3, sand/MPC ratio of 1:1, about 15% fly ash, between 5 to 15% borax, and 0.16 W/B ratio had an operable time of about 25 min. The volume of the MPC mortar or batching sequence used with the materials was not documented in the report. Field observations on the repaired works during three years showed 100%, 75.4%, and 49.3%, respectively, successful rehabilitations. The authors emphasized defective concrete and chipping must be removed, leaving a sound, clean surface for a successful MPC mortar repair.

Field investigations by Seehra et al. (1993) used an MPC mortar to seal cracks, fill potholes, and repair edge spalls in concrete pavements. Surfaces were first cleaned out to remove any foreign and loose materials. To achieve better bonding with the concrete substrate, all ingredients except the sand were mixed and the paste was applied as primer before the application of the MPC mortar. Repair patches were air cured for 4-5 hr before being opened to traffic. The successful repairs were attributed to good bonding characteristics of the MPC mortar and similar thermal coefficient of thermal expansion behavior between the MPC mortar and the concrete substrate.

A study by Qiao et al. (2010) concluded that the properties of MPC are affected mainly by the molar ratio of the magnesium/phosphate, the addition of retarders, and the water content, as well as the reactivity of the magnesia. Similar findings were reported by Yang and Wu (1999), who found the setting time and early age strength were mainly controlled by the amount of retarder, the fineness of magnesia, and the temperature. The authors reported the retarding action is directed towards the MgO and not the phosphate.

2.4 Conclusions

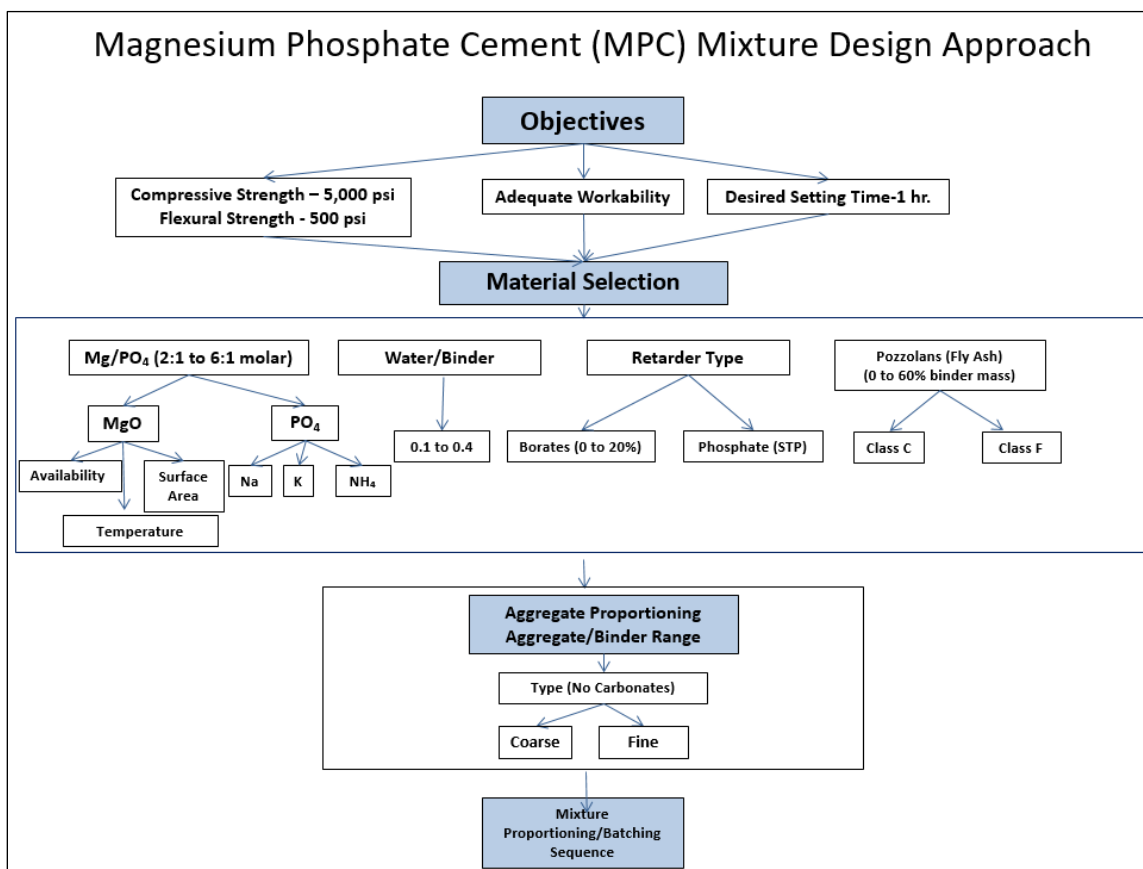
Literature research showed a good fundamental concept of the interaction of the binders with MPCs. One generalization inferred from the research is that if the MgO content is low (i.e., higher phosphate content) an unstable matrix is formed due to the unreacted phosphates. Furthermore, an

increase in the MgO raises the pH of the reaction environment and accelerates the reaction between the MgO and the phosphate. This is proven through mechanical testing and verified with the heat generation studies of calorimetry. The setting time and decrease of heat generations are mitigated best with the addition of SCMs (namely Class C fly ash) and set retarders (namely borax or boric acid). However, additional research is recommended in the areas of coarse aggregate guidelines and further understanding of developing delayed setting time and continued understanding of the microstructural characteristics. It is concluded that, due to their tremendous advantages, MPCs will continue to be used in concrete repair materials for heavily trafficked roads and for airfield damage repair.

3 Materials

A literature review indicated that most MPC-based binders consist of a magnesium oxide (MgO), a phosphate salt, a retarder, and one or more of some type of SCM. The flow chart in Figure 2 illustrates the MPC mixture design approach with material selection for trial batching for our study.

Figure 2. Flow chart for development of MPC mixture design.



For this research, the materials listed in Table 3 were proportioned by making trial batches based on ranges identified in the literature and were verified through the collection of experimental data. The W/B ratio ranged from 0.1 to 0.4 for the mixtures in this study. Information about each of the materials is provided in Appendix A.

Table 3. Summary of raw materials used in MPC mixture proportioning.

Material Name	Source	CMB Serial No.
Magox 93HR 325 Light-Burned MgO	Premier Magnesia, LLC	150132
MagChem 10 Hard-Burned MgO	Martin Marietta	150157
MagChem P98 Dead-Burned MgO	Martin Marietta	150156
Monopotassium Phosphate (MKP) KH ₂ PO ₄	ICL Premium Fertilizers - Peak	150125
Boric Acid	Searles Valley Minerals	150126
Class C Fly Ash	Headwaters Resources, White Bluff Plant, Redfield, AR	140020
Class F Fly Ash	Boral Resources Bowen Plant, Stilesboro, GA	140022
Fine Aggregate	Green Brothers Crystal Springs, MS	120087
3/8-in. Pea Gravel Coarse Aggregate	Green Brothers Crystal Springs, MS	120088
3/8-in. Coarse Aggregate	Osage River Rock Jefferson City, MO	NA
3/4-in. Coarse Aggregate	Osage River Rock Wardsville, MO	NA

3.1 Magnesium oxide (MgO)

The MgO was selected based on the availability, surface area, temperature of calcination, and the rate of reaction. DB MgO was selected due to its lower rate of reaction compared to the other MgO forms. The lower rate was ascribed as proximal to its very high calcination temperature of over 1,500°C, which makes the surface of the particles less reactive. However, for completeness, three classifications (LB, HB, and DB) were tested to gauge their effects on properties such as setting time and workability. Additional information on the MgOs tested is provided in Chapter 4, “Trial Batching,” of this report.

3.1.1 Light-burned (LB)

LB MgO is a reactive grade of magnesium oxide that has been calcined at temperatures ranging from 700–1,000°C. Magox 93HR 325 was the trade name of the LB MgO used in this research. This product is a finely ground,

chemical grade MgO produced from beneficiated Nevada magnesite ores by burning to a moderately high reactivity.

3.1.2 Hard-burned (HB)

HB MgO is a low reactive grade of MgO that has been calcined at temperatures ranging from 1,000–1,500°C. MagChem 10 was the trade name for HB MgO used in this research. This product is a high-purity, low-density, and low-reactivity material that is essentially dust free.

3.1.3 Dead-burned (DB)

DB MgO has been calcined at temperatures ranging from 1,500–2,000°C in a high temperature shaft kiln, yielding a material with very little reactivity. MagChem P98 was the trade name for DB MgO used in this research. This product is a 98% pure MgO product with high density and low reactivity produced from magnesium-rich brine and dolomitic lime.

3.2 Potassium dihydrogen phosphate (KH_2PO_4)

Potassium dihydrogen phosphate (KH_2PO_4), known as “KDP” for short, is a water soluble material with a solubility limit of 188.6 lb per gallon of water. Its molecular weight is 136.09 g, and its density is 2.335 g/cm³. The KDP used in this research was produced by ICL fertilizers under the trade name monopotassium phosphate (MKP), which is synonymous for this phosphate salt.

3.3 Fly ash

ASTM C618 (2017a) defines two classes of fly ash for use in concrete: (1) Class F, usually derived from the burning of anthracite or bituminous coal, and (2) Class C, usually derived from the burning of lignite or subbituminous coal. ASTM C618 also delineates requirements for the physical, chemical, and mechanical properties for these two classes of fly ash. Class F fly ash is pozzolanic with little or no cementing ability on its own. Class C fly ash is principally used as a pozzolan in cement-based composites but also has self-cementing properties. This research used a Class C fly ash sourced from the Headwaters Resources’ White Bluff Plant in Redfield, AR, and a Class F fly ash sourced from the Boral Resources’ Bowen Plant in Stilesboro, GA. The chemical composition of the fly ashes determined by X-ray fluorescence (XRF) elemental analysis are given in Table 4.

Table 4. Chemical oxides of fly ash materials determined by XRF elemental analysis.

Compound	Bowen Class F Fly Ash	Redfield Class C Fly Ash
SiO ₂	56.35	39.84
Al ₂ O ₃	27.66	21.46
Fe ₂ O ₃	6.09	5.73
CaO	1.15	21.23
MgO	0.62	4.13
SO ₃	0.13	1.13
K ₂ O	2.33	0.6
Na ₂ O	0.31	1.5
P ₂ O ₅	0.33	1.34
TiO ₂	0.87	1.6
Mn ₂ O ₃	0.02	0.01
SrO	0.10	0.43
ZnO	0.004	0.013
Cr ₂ O ₃	0.045	0.048
Loss on Ignition (%)	3.46	0.94

3.4 Retarder

MPC is a fast-setting material, and the addition of a retarder was critical to slow the reaction rate and increase the working time. Boric acid (H₃BO₃) was selected as a set retarder due to its ability to retard the MPC and its wide availability. Other retarders, such as sodium tri-poly phosphate (STP), were considered; but due to the significantly higher cost of this material, it was eliminated from the testing plan. The granular boric acid used in this research was manufactured by Searles Valley Minerals in Trona, CA, with a purity of 99.76%.

3.5 Aggregates

Only natural, round gravel aggregates were used due to the low water-to-cement ratios researched in this study. Crushed, angular aggregates were not used because of possible loss in workability due to the tight aggregate-paste bond. Limestone aggregates were eliminated due to reactivity with the binder, causing production of gas and expansion of product.

Three coarse aggregates from two quarries were used in this research. Most mixture proportions used a 3/8-in. NMAS pea gravel from a

4 Trial Batching

Trial batching, or mixture proportioning, is necessary to assess the quality and suitability of the constituent materials to meet performance requirements. This statement is true for OPC concretes and is even more important for alternative binders that are not regulated, more experimental, and/or of rapid-setting nature. MPCs are both rapid setting and unregulated in terms of constituent material guidance for concrete design, sampling, casting, curing, and hardened property testing procedures. Target performance requirements of the MPC concrete for airfield pavements include the following characteristics: (a) minimum 5,000-psi compressive strength at the age of 28 days, (b) minimum 500-psi flexural strength at the age of 28 days, (c) at least 60 min of setting time, (d) good bond strength, (e) adequate workability, (f) good durability, and (g) wide availability of raw materials.

It is important to conduct a thorough constituent materials identification search and mixture proportioning procedure when designing MPCs. Based on the literature review, many typical commercially blended MPC-based products consist of a mixture of high temperature DB (calcined at approximately 1,500°C) MgO, a phosphate salt, a retarder, and various SCMs. The ranges of the raw materials selected are based on mixtures similar to those found in the literature and verified by experimental data. Much of the literature discussed magnesia-to-phosphate (M/P) ratios in terms of molar ratios instead of the mass or volume ratios that are typically used in ready mix concrete applications. These M/P molar ratios typically range from 1.5/1 to 12/1 with no particular ratio being universally accepted as the optimum in the literature. The M/P ratio for any MPC mixture proportion will need to vary depending on the specific constituents being used, W/B ratio, and aggregate-to-binder (A/B) ratio.

The phosphate salt selected to react with the MgO in the current study is potassium dihydrogen phosphate (KH_2PO_4), or KDP. The molar weights of MgO and KH_2PO_4 are 40.3 and 136.09, respectively. This means that the phosphate content is higher than the magnesia content in the cementing system. Table 6 shows the M/P ratio in both a molar ratio and a mass ratio.

Table 6. Magnesia-to-Phosphate (M/P) ratio conversions.

Molar Ratio (M/P)	Mass Ratio (M/P)
2/1	0.592/1
3.4/1	1/1
4/1	1.185/1
6/1	1.777/1
8/1	2.369/1
10/1	2.961/1
12/1	3.554/1
M=MgO P=KH ₂ PO ₄	

A select set of materials and mixture proportioning parameters was designed to go outside of the limits seen in the literature in order to better understand reaction chemistry and its effects on mechanical properties. The W/B ratio for the trial batches ranged from 0.1 to 0.4. The MgO of each calcination classification (LB, HB, and DB) was tested for its effects on setting time and strength. Both a Class F and a Class C fly ash were tested at 50% of binder mass. Although the amount of fly ash was not optimized for each mixture, this amount provided good reactivity and increased set times. In all mixtures, the aggregate contents were held constant. A majority of mixtures contained only a fine aggregate, with a coarse aggregate being added later in the mixture proportioning process after a good cementitious matrix was established. Boric acid was varied between 0 to 5% of total cementitious mass, with most mixtures having a dose of 4%. All test specimens were dry-cured in an ambient lab environment at $73 \pm 2^{\circ}\text{F}$.

4.1 Light-burned MgO, Class C fly ash

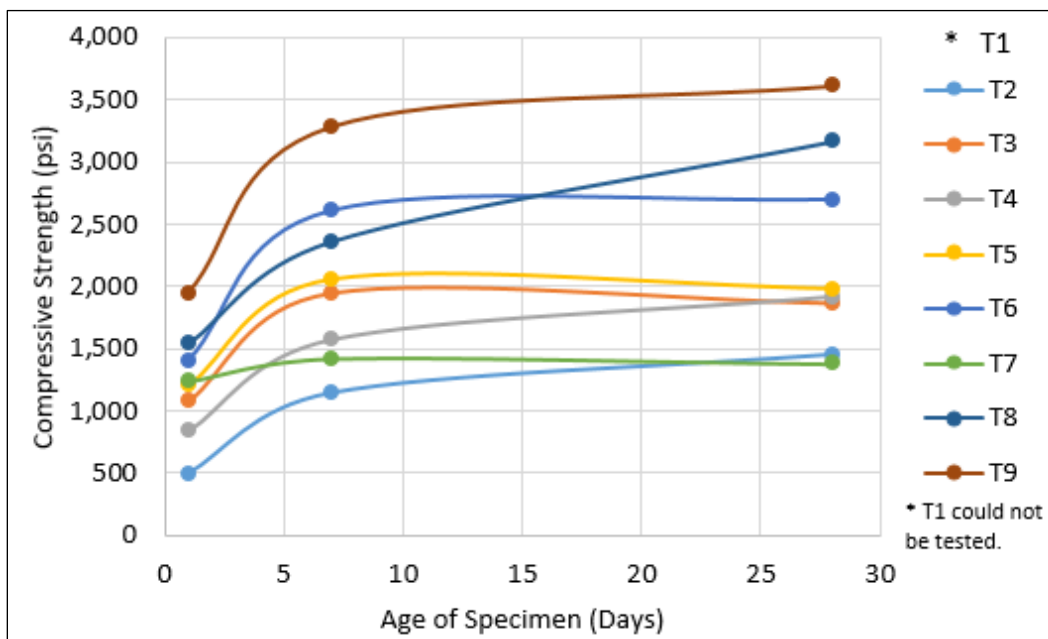
Nine trial batches were made using a LB MgO and a Class C fly ash identified LB-C T1-9. Trial mixture proportions are summarized in Table 7.

Table 7. Trial batch mixing ratios of LB MgO with class C fly ash.

Mixture Description	Mixture ID								
	LB -C T1	LB -C T2	LB -C T3	LB -C T4	LB -C T5	LB -C T6	LB -C T7	LB -C T8	LB -C T9
M/P (MgO to phosphate) molar ratio)	3.4/ 1	3.4/ 1	2/1	4/1	3.4/ 1	2/1	4/1	4/1	3.4/ 1
A/B (aggregate to binder) mass ratio	0	0	0	0	2/1	2/1	2/1	0	2/1
W/B (water to binder) mass ratio	0.4	0.4	0.3	0.3	0.3	0.3	0.3	0.3	0.3
Class C Fly Ash (% mass of binder)	0	50	50	50	50	50	50	50	50
Boric Acid (% total of binder mass)	0	4	4	4	4	4	4	4	4
Consolidation Method T=Tamping V=Vibration	T	T	T	T	T	T	T	V	V

The compressive strength plots determined according to ASTM C109 (ASTM 2016a) using 2-in. cubes are illustrated in Figure 3. Trials 8 and 9 achieved the highest compressive strengths when measured at 28 days. These mixtures were duplicates of T4 and T5 with the exception that T8 and T9 were consolidated by using a vibrating table, whereas all other trials were tamped. This indicates that vibration consolidation produces higher strengths.

Figure 3. Summary of compressive strength of LB MgO trial batch cubes.



Time of setting results provided in Table 8 were determined by ASTM C403 (ASTM 2016f). All batches were cast with 4% boric acid by volume of cementitious material with the exception of T1, which had no boric acid. The maximum hydration temperature determined with an embedded thermometer in the penetrometer sample was measured as the maximum temperature from mixing to 1 hr after final set. The higher M/P ratios exhibited higher hydration temperatures.

Table 8. Summary of time of setting for LB mixtures.

Mixture ID	Penetrometer Method ASTM C403		Maximum Hydration Temperature (°F)
	Initial Set (minutes)	Final Set (minutes)	
LB - T1	<1	<2	180
LB - T2	8	13	130
LB - T3	21	24	120
LB - T4	26	35	170
LB - T5	22	27	160
LB - T6	24	27	110
LB - T7	14	23	140
*LB-T8	N.D.	N.D.	N.D.
*LB-T9	N.D.	N.D.	N.D.
*No Data (N.D.) available for time of set or maximum temperature			

Overall, the LB mixtures had poor workability and exhibited thixotropic behavior by forming a crust on top of the samples before actually setting. This possibly affected the accuracy of the set times recorded. None of the LB materials met the target performance criteria. Based on the testing performed, the LB material is not recommended due to poor workability, extreme reactivity, and low compressive strengths.

4.2 Hard-burned MgO, Class F fly ash

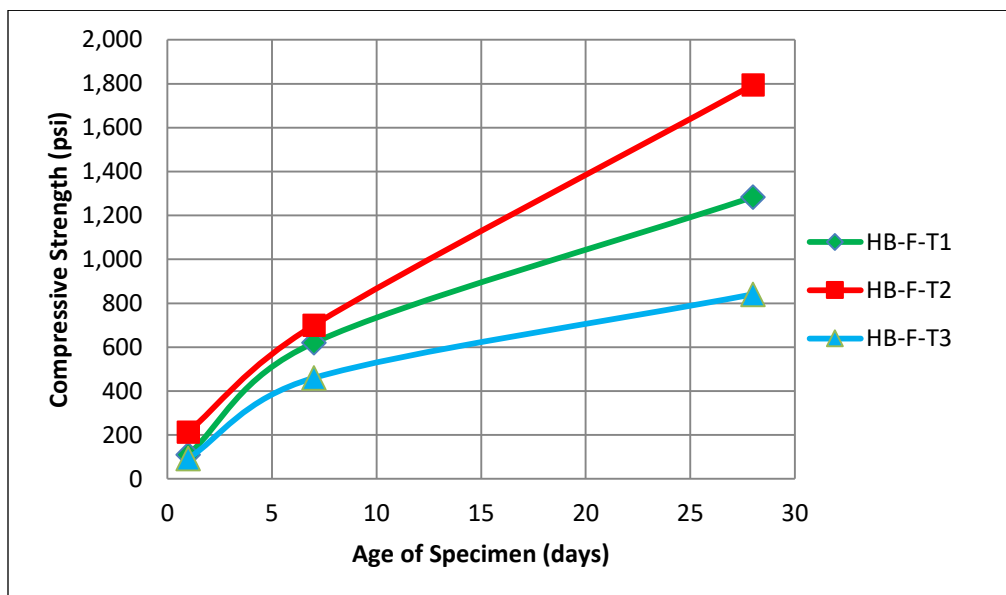
Three trial batches were made using the HB MgO and Class F fly ash identified as mixtures HB-F-T1-3. Trial batch mixture proportions are in Table 9. Initial plans were to make trial batches with a Class F and a Class C fly ash. Initially, the Class F was used due to the fact that an HB MgO would have a high reactivity, and a Class F would help lower reactivity as compared to that of a Class C fly ash.

Table 9. Trial batch mixing ratios of HB MgO with class F fly ash.

Mixture Description	Mixture ID		
	HB-F-T1	HB-F-T2	HB-F-T3
M/P (MgO to phosphate: molar ratio)	3.4/1	2/1	4/1
A/B (aggregate to binder: mass ratio)	2/1	2/1	2/1
W/B (water to binder: mass ratio)	0.30	0.30	0.30
Class F Fly Ash (mass % volume of binder)	50	50	50
Boric Acid (mass % volume of binder)	4	4	4

The compressive strengths for HB-F trial mixtures shown in Figure 4 revealed that the higher the M/P ratio, the lower the compressive strength. HB-F-T3 with a 4/1 MgO-to-phosphate ratio exhibited the lowest strengths, and HB-F-T2 with a 2/1 MgO-to-phosphate ratio exhibited the highest strengths. However, all strengths were below 1,800 psi, which is significantly lower than the target value (5,000 psi).

Figure 4. Compressive strength for HB MgO trial mixtures.



The HB trial mixtures resulted in longer setting times and lower temperatures than expected, as shown in Table 10. This is possibly due to the incorporation of the Class F fly ash and the high dosage of boric acid used in the mixtures. No further testing was investigated on the HB MgO material, based on low compressive strengths and the material's exhibiting thixotropic behavior, resulting in poor workability and consolidation.

Table 10. Summary of time of set and maximum observed temperatures for HB mixtures.

Mixture	Penetrometer Method ASTM C403		Maximum Temperature (°F)
	Initial Set (minutes)	Final (minutes)	
HB-F-T1	140	285	80.9
HB-F-T2	100	170	86.1
HB-F-T3	150	180	98.6

4.3 Dead-burned MgO, Class F fly ash

Six trial batches were made using the DB MgO and Class F fly ash identified as mixtures DB-F-T1-6. The mixture proportions used for the DB material approximated those of the HB mixtures. Trial batch mixture proportions in Table 11 varied the M/P from 2:1, 3.4:1, and 4:1. These M/P ratios were batched at A/B mass ratios of 2:1 and 1:1. Fly ash was held constant at a 50% replacement. The 2:1 A/B ratio mixtures had a 0.3 W/B

ratio while the 1:1 sand to binder S/B mixtures had a 0.25 W/B ratio. Boric acid was held constant at 4% of the mass of the binder.

Table 11. Trial batch mixing ratios of DB cement with Class F fly ash.

Mixture Description	Mixture ID					
	DB-F-T1	DB-F-T2	DB-F-T3	DB-F-T4	DB-F-T5	DB-F-T6
M/P (MgO to phosphate: molar ratio)	3.4/1	2/1	4/1	3.4/1	2/1	4/1
A/B (aggregate to binder: mass ratio)	2/1	2/1	2/1	1/1	1/1	1/1
W/B (water to binder: mass ratio)	0.30	0.30	0.30	0.25	0.25	0.25
Fly Ash (mass % volume of binder)	50	50	50	50	50	50
Boric Acid (mass % volume of binder)	4	4	4	4	4	4

DB-F-T1, 2, and 3 varied the M/P ratio at a 0.30 W/B ratio. These mixtures were almost too fluid; however, the fine aggregate remained in solution, so cubes were cast for testing. The compressive strength results for DB-F trials are given in Figure 5. Of these three mixtures, the 2:1 M/P molar ratio of DB-F-T2 produced the highest 7- and 28-day compressive strengths for a 2-in. cube. The summary of the time of set and maximum temperature observed for DB, Class F fly ash mixtures is provided in Table 12.

Figure 5. Unconfined compressive strength of DB Class F fly ash MPC trials.

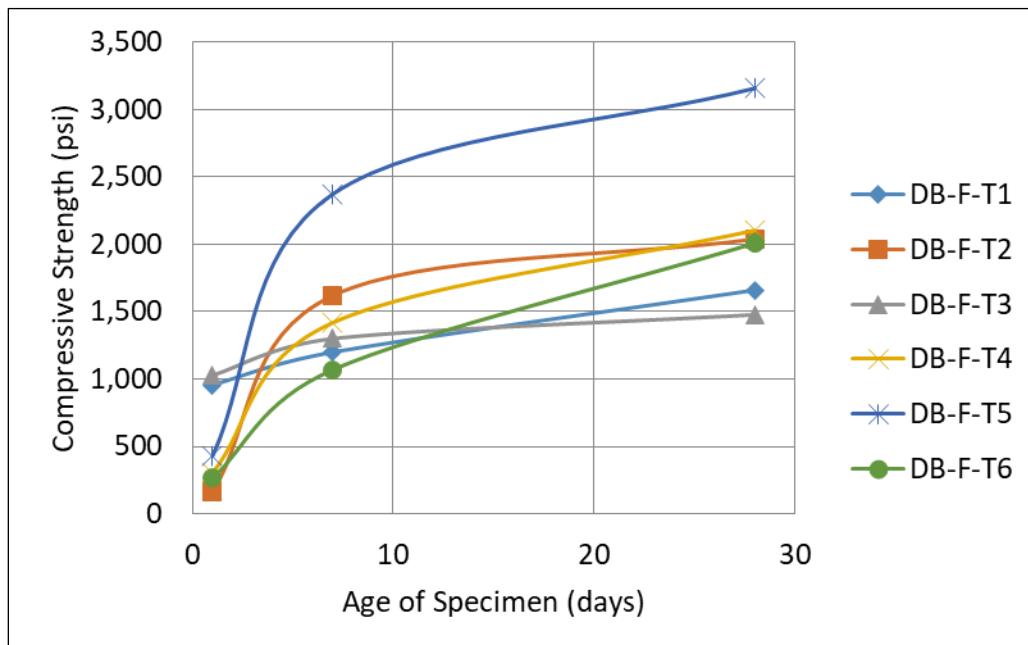


Table 12. Summary of time of set and maximum temperature observed for DB, Class F fly ash mixtures.

Mixture	Penetrometer Method ASTM C403		Maximum Temperature (°F)
	Initial Set (minutes)	Final Set (minutes)	
DB-F-T1	N.T.	N.T.	101.6
DB-F-T2	100	170	86.1
DB-F-T3	150	180	98.6
DB-F-T4	60	150	86.3
DB-F-T5	80	200	87.7
DB-F-T6	60	160	86.2
N.T.= Not Tested			

4.4 Dead-burned MgO, Class C fly ash

Five trial batches were made using the DB MgO and Class C fly ash identified as mixtures DB-C-T4 8. Trial mixture proportions are shown in Table 13. Trials 1 through 3 were replicas of DB-F trials 1, 2, and 3. Segregation of the sand in these mixtures was observed; therefore, the W/B was reduced to 0.20 for trials 4 through 10.

Trials DB-C-T4 6 were 0.25-ft³ batches that varied the M/P molar ratio and were tested for compressive strength using 2-in. cubes. Trials 7 and 8 were cast at 0.75 ft³, and 3- x 6-in. cylinders were cast for compressive strength testing. Trials 7 and 8 added coarse aggregate at a rate of 50% of the mass of the total aggregate. Although the coarse-to-fine aggregate ratio was not optimized, it did produce a more fluid, workable mixture. DB-C-T9 was mixed but not cast, as the mixture was too thick and appeared to be at the coarse aggregate volume limit.

Table 13. Trial batch mixing ratios of DB cement with Class C fly ash.

Mixture Description	Mixture ID					
	DB-C-T4	DB-C-T5	DB-C-T6	DB-C-T7	DB-C-T8	DB-C-T9
M/P (MgO to phosphate) molar ratio	3.4/1	2/1	4/1	2/1	2/1	2/1
A/B (aggregate to binder) mass ratio	1/1	1/1	1/1	1/1	1/1	1/1
W/B (water to binder) mass ratio	0.20	0.20	0.20	0.20	0.20	0.20
Fly Ash (mass % volume of binder)	50	50	50	50	50	50
Boric Acid (mass % of binder)	4	4	4	4	4	4
Aggregate Replacement	-	-	-	-	50	60
Batch Volume (ft ³)	0.25	0.25	0.25	0.75	0.75	0.75
Compressive Strength Specimen Size (in.)	2x2	2x2	2x2	2x2	3x6	3x6

The compressive strengths for the DB-C trial mixtures are shown in Figure 6. Trial 5 with an M/P ratio of 2:1 provided the best 28-day compressive strength and was, therefore, selected to be scaled up to a larger batch for Trial 7. An increase in strength gain from 5,000 to 7,000 psi was observed at the 28-day age with the larger batch size. This is potentially due to a higher mass of phosphates being available in the larger batch for reaction with the fly ash. DB-C-T8, with the addition of coarse aggregates, was slightly lower in strength but still above the target compressive strength of 5,000 psi at 28 days. Although the strength dropped in T8 compared to T7 with the addition

of coarse aggregate, researchers do not ascribe the loss of strength to that addition. At present, the cause for the loss of strength is unclear.

A summary of the time of set and maximum temperature observed for DB, Class C fly ash mixtures is provided in Table 14.

Figure 6. Compressive strengths of DB MPC mixtures with Class C fly ash.

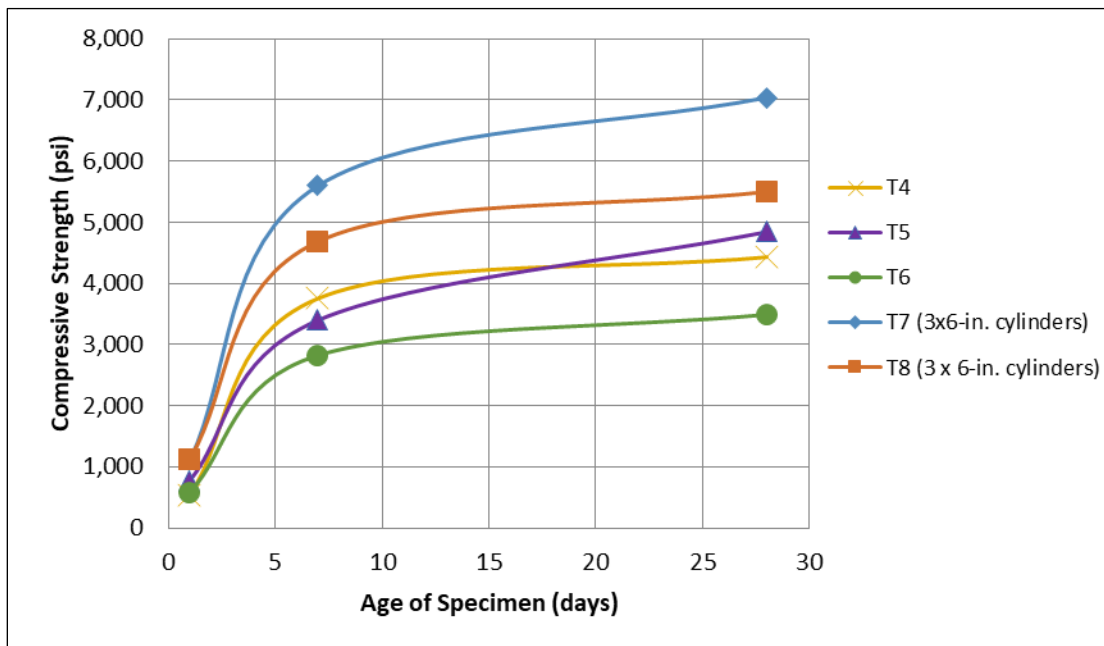


Table 14. Summary of time of set and maximum observable temperature for DB MgO, Class C fly ash mixtures.

Mixture	Penetrometer Method ASTM C403		Maximum Temperature (°F)
	Initial (minutes)	Final (minutes)	
DB-C-T4	35	110	80.9
DB-C-T5	60	150	84.2
DB-C-T6	40	90	82.5
DB-C-T7	35	65	86.0
DB-C-T8	60	90	76.1

4.5 Trial batching discussion

As suspected, the LB trials did not fare well due to the highly reactive nature of LB MgO, coupled with the thixotropic nature of the mixtures, which gave rise to poor workability. The same deficiencies were observed for the HB MgO trials, as the poor workability resulted in unfavorable

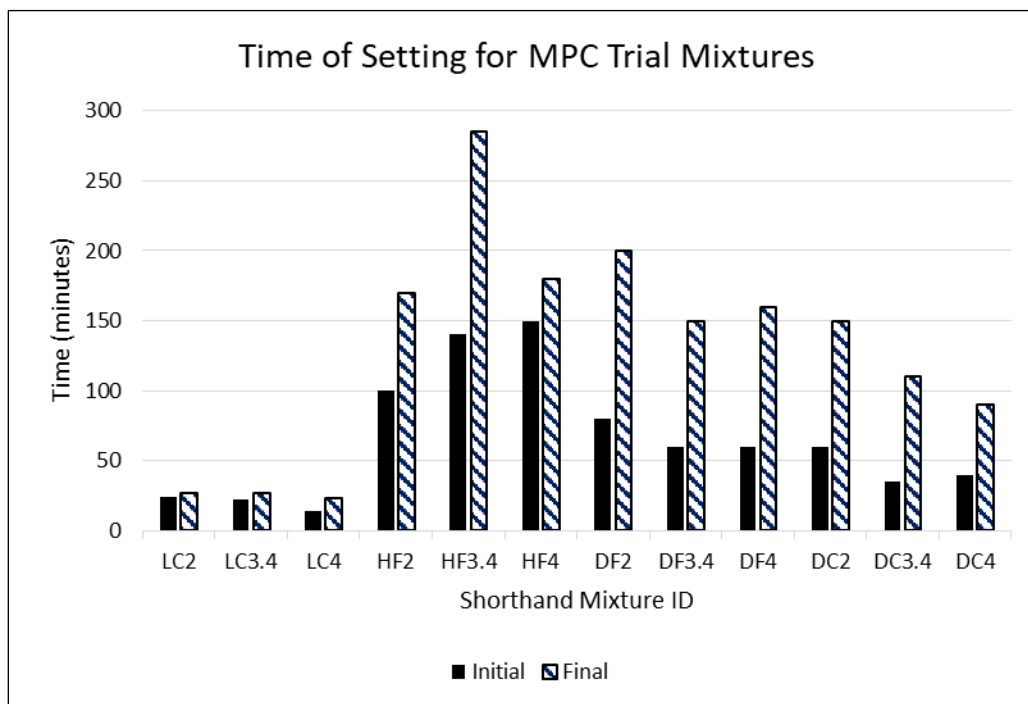
mixture proportions. The HB-F compressive strengths could likely have been improved with a slightly lower W/B ratio and incorporation of a Class C fly ash, but neither of those changes would have improved the workability.

Figure 7 illustrates the time to set for some of the trial mixtures. Each of these trial mixtures contained 50% fly ash, 4% boric acid, and concrete sand at an A/B ratio of 1:1. The x-axis is labeled with shorthand terminology for mixture types. The first letter denotes the MgO burn classification as light (L), hard (H), or dead (D). The second letter denotes the fly ash class as C or F, and the number denotes the M/P molar ratio. The y-axis labels the time to set in minutes determined by ASTM C403 (ASTM 2016f). For all MgO classifications, the set time decreased as the M/P ratio was increased. A surprising result was the extended set times obtained for the HB MgO, and this was accomplished with a 4% boric acid dose by weight of binder with a 50% binder replacement of fly ash. However, the HB and the LB batches illustrated in the figure have W/B ratios of 0.30, while the DB-F mixtures are at 0.25, and the DB-C mixtures have a 0.20 W/B. Typically, higher W/B ratios have longer set times.

The compressive strengths for the DB-F mixtures could be improved upon with a lower W/B. A pavement quality 5,000-psi MPC could possibly be made with a Class C fly ash, but not all Class F fly ashes. These data do corroborate with the theory that the calcium oxide (CaO) in Class C fly ashes are reactive with phosphates and form secondary reaction products. Class F fly ashes tend to have less CaO and act more as a filler in MPCs than their class C counterparts. Therefore, it is recommended that MPCs be made with DB MgO and a Class C fly ash. If a Class C is unavailable, a Class F can potentially be used, and finding a Class F with a relatively high CaO would be beneficial.

A summary table of all MgO cement-based trial batches is provided in Appendix C.

Figure 7. Time of setting for light-, hard-, and dead-burned MPC trial mixtures.



4.6 Suggested design ranges

Below are suggested trial mixture proportion ranges for MPCs.

- W/B ratio: 0.15 – 0.30
- MgO-to-KDP (M/P) molar ratio: 2 – 8
or (0.6 – 2.4 mass ratio)
- Fly ash: 40 to 60% of the mass of binder
- A/B ratio: 0.75 – 1.5
- Coarse-to-Fine aggregate: Proportioning same as OPC concrete
- Boric acid: 2 to 5% total of cementitious mass

These ranges provide a good basis for MPC pavement design. These ranges were sourced from literature and verified during team trial mixture proportioning. The suggested W/B range will vary depending on the selected constituent materials. The fineness of the MgO and the aggregates will dictate the necessary W/B and A/B to provide a workable mixture. The M/P range exceeds the range produced in trial mixtures of 2–4 molar ratio. There are mixtures in literature that provide adequate strength at M/P molar ratios as high as 12. It is important to note that set times decrease as the M/P increases. The M/P ratio will need to be adjusted based on the reactivity and amount of fly ash in the design. A fly ash

content of at least 40% is suggested to increase setting time and drive secondary reactions for the unreacted phosphates in the system. The boric acid content needs to vary depending on application, required strengths, and placement logistics. If borax is used, the range will be slightly higher (i.e., between 3 and 7% total of cementitious mass).

5 Laboratory Testing at Varying Scales

MPCs are rarely cast in volumes larger than 1 ft³. This is due to the highly exothermic, rapid-setting nature, the historically limited application, and the commercially available bag sizes of the proprietary materials on the market. The potential complications that could arise from large-scale MPCs were unknown. Through past experience with calcium sulfoaluminate (CSA) cement, another rapid-setting cement type, larger batch quantities of a rapid-setting cement produce higher exothermic temperatures that reduce setting time. In addition, the retarder dosages need to be adjusted based on the outside ambient temperature. Higher dosages of boric acid, although decreasing the temperature of the mixture and thereby increasing the setting time, have a profound detrimental effect on strength. Therefore, incremental steps of increasing batch size were taken to alleviate some concerns that are associated with large batches of rapid-setting cements. Up to this point, all trial mixtures were batched at quantities less than 0.75 ft³ in a high-shear Hobart planetary mixer. Limited physical and mechanical property tests served as a screening test for the elimination of the materials unsuitable for the fulfillment of the objectives. Mixture DB-C-T8 was selected to be batched at incrementally larger scales for full mechanical and durability properties testing.

5.1 Methodology

In order to evaluate the long-term MPC performance and suitability, additional mechanical, volumetric, and durability property testing was needed. In addition to the physical testing and compressive and flexural strength criteria, the benchmark for additional laboratory testing was based on the pending publication of the technical report (TR) entitled *Evaluation of Rapid-Setting Cementitious Materials and Testing Protocol for Airfield Spall Repair* (Ramsey and Tingle 2018¹). This report is an update to the test protocol established in ETL o8-02, *Testing Protocol for Rigid Spall Repair Materials* (AFCEC 2008) and ERDC/GSL TR-11-13, *Development of Laboratory Testing Criteria for Evaluating Cementitious, Rapid-Setting Pavement Repair Materials* (Priddy 2011). The Ramsey and Tingle TR presents the test methods and results of 26

¹ Ramsey, M. A., and J. S. Tingle. 2018. *Evaluation of rapid-setting cementitious materials and testing protocol for airfield spall repair*. ERDC/GSL TR (Draft). Vicksburg, MS: U.S. Army Engineer Research and Development Center.

cementitious rapid-setting repair products tested at the U.S. Army Engineer Research and Development Center (ERDC) during 2013 to 2017. An evaluation of these test methods and results, along with the historic database of products tested, led to the development of an updated testing protocol for assessing a material's suitability for airfield spall repairs. The selected tests and requirements from the spall material testing protocol applied to the MPC mixture design are shown in Table 15. Additional experimental testing included using the MPC material to prepare a small test slab in the laboratory and coring the concrete for strength determination.

Table 15. Selected tests and requirements from spall material testing protocol applied to MPC mixture design.

Test Property	Test Method	Test Age	Test Criteria
Bond Strength Test Material/Test Material (TM/TM)	ASTM C882 (2013a)	1 day	≥ 1,000 psi
		7 days	≥ 1,500 psi
Bond Strength Portland Cement Mortar/Test Material (PCM/TM)		1 day	≥ 1,000 psi
7 days		≥ 1,250 psi	
Modulus of Elasticity	ASTM C469 (2014d)	28 days	2 ≤ x ≤ 6 Mpsi
Slump Flow	ASTM C1611 (2014c)	Within 5 min of added water	≥ 9 in.
Length Change	ASTM C157 (2014a)	28 days stored in air	-0.04% ≤ x ≤ +0.03%
		28 days stored in water	
Coefficient of Thermal Expansion	ASTM C531 (2012)	-	≤ 7 (in./in./°F × 10 ⁻⁶)
Freeze-Thaw Procedure A	ASTM 666(2015d)	Based on 300 cycles	No established criteria

5.2 Materials and mixing

Two 2.5-ft³ batches of the materials proportioned in Table 16 were cast in a 3-ft³ traditional revolving drum mixer illustrated in Figure 8. Two batches were made in order to cast compressive strength, flexural

strength, bond strength, splitting tensile, modulus of elasticity, time of set, and slump flow. Due to the mixing style and rapid-setting nature, a batching sequence was used that mixed all aggregates, KDP, boric acid, fly ash, and water prior to adding the MgO at the end. The mixture was then mixed for 2 min and 30 sec before discharge and casting. Some minor issues with material sticking to the side of the drum for the first batch were corrected, and the second batch was successfully cast.

Table 16. MPC DB-C T8 mix proportions based on binder and aggregate percentage.

Material	Source	Binder % by Volume	Aggregate % by Volume
MgO Dead-Burned Cement	Martin Marietta - P98 Pulverized Magnesium Oxide	19	-
Monopotassium Phosphate (MKP) (KH_2PO_4)	ICL Premium Fertilizers - Peak	31	-
Class C Fly Ash	Headwaters Resources' White Bluff Plant in Redfield, AR	50	-
Concrete Sand	Green Brothers' Concrete Sand in Redwood, MS	-	50
Pea Gravel	Green Brothers' - 3/8-in. Pea Gravel, Crystal Springs, MS	-	50
Boric Acid	Searles Valley Minerals	4% of total mass of binder	
Water	Tap	0.2 W/B Ratio	

Figure 8. Worthington 3-ft³ steel drum concrete mixer.



The next batch was an 8-ft³ batch made in a 14-ft³ revolving drum mixer to cast a 3-ft x 3-ft x 7.5-in. slab and companion specimens, as shown in Figure 9. It was batched in the same manner as the 2.5-ft³ batches. Ice was added to the batch water at a rate of 40% of the total weight. The slab was cured with a plastic sheet. To reduce chances of dissolving unreacted phosphate, no excess water was added; sheeting was placed to help prevent drying shrinkage. Cores were taken at 7, 14, and 28 days and compared to companion cylinder break strengths.

Figure 9. Displacement of MPC designed concrete into slab form.



5.3 Results and discussion

The results of the MPC mixture DB-C-T8 are in Table 17. Further discussion of each test property follows.

Table 17. Summary of test results of MPC DB-C-T8 mixture.

Property	Standard	Unit	Age	Results by Batch Volume		
				0.75 ft³	2.5 ft³	8 ft³
Temperature	ASTM C1064 (2017d)	°F	-	73	76	66 *iced batch
Unit Weight	ASTM C138 (2017c)	lb/ft³	-	140	140	140
Air Content	ASTM C231 (2017b)	%	-	3.4	3.0	2.8
Flow	ASTM C1611 (2014c)	inch	-	10	10	15
Time of Setting	ASTM C403 (2016f)	minute	Initial Set	60	48	65
			Final Set	90	70	95
Compressive Strength	ASTM C39 (2015a)	psi	1-day	1,130	1,610	N.T.
			7-day	4,680	4,980	4,850
			28-day	5,500	5,530	5,380
Flexural Strength	ASTM C78 (2016c)	psi	1-day	N.T.	270	N.T.
			7-day	410	450	430
			28-day	500	510	495
Bond Strength (TM/TM)	ASTM C882 (2013a)	psi	1-day	N.T.	1,170	N.T.
			7-day	N.T.	1,790	N.T.
Bond Strength (PCM/TM)	ASTM C882 (2013a)	psi	1-day	N.T.	1,040	N.T.
			7-day	N.T.	1,670	N.T.
Modulus of Elasticity	ASTM C469 (2014d)	Mpsi	1-day	N.T.	3,380	N.T.
			7-day	N.T.	4,720	N.T.
			28-day	N.T.	5,200	N.T.
Length Change	ASTM C157 (2014a)	inch	28 days air cured	N.T.	-0.002	N.T.
			28 days water cured	N.T.	0.025	N.T.
			64 weeks air cured	N.T.	-0.045	N.T.
			64 weeks water cured	N.T.	0.054	N.T.
Coefficient of Thermal Expansion	ASTM C531 (2012)	in/in/°F	-	N.T.	7.3	N.T.
Freeze-Thaw Procedure A	ASTM C666 (2015d)	Durability Factor	43 cycles	N.T.	11	N.T.
Drilled Cores	ASTM C42 (2016e)	psi	7 days	N.T.	N.T.	3830
			14 days	N.T.	N.T.	4550
			28 days	N.T.	N.T.	4720
N.T.= Not Tested						

5.3.1 Fresh properties

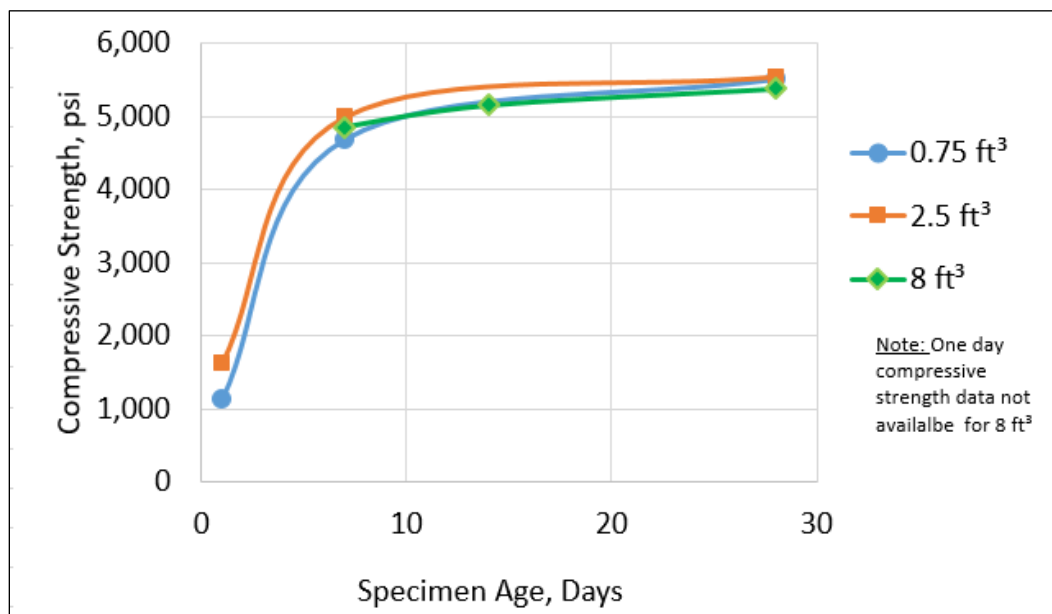
The 0.75-ft³ and 2.5-ft³ concrete batch temperatures measured in accordance with ASTM C1064 (2017d) were 73 and 76°F, respectively. These temperatures indicated a favorable concrete condition with an exothermic heat generation under 80°F. However, it was unknown whether scaling the batch size contributed to the increase in temperature. Caution was therefore taken by icing the mix on the larger 8-ft³ batch volume. The temperature of the fresh concrete reflected a much lower 66°F.

The unit weight measured in accordance to ASTM C138 (2017c) was a consistent 140 lb/ft³ for all concrete batch volumes. The air content measured in accordance to ASTM C231 (2017b) varied between each mixture with a range of 2.8 to 3.4%. The slump flow measured in accordance to ASTM C1611 (2014c) was 10 in. for both the 0.75-ft³ and 2.5-ft³ concrete batches, but increased to 15 in. when measured in the 8-ft³ mix. Although time of setting results varied, all batch volumes resulted in acceptable setting times for practical applications. The 0.75-ft³ batch resulted in an initial set and a final set of 60 and 90 min, respectively. Shorter initial and final set times of 48 min and 70 min, respectively, resulted with the larger 2.5-ft³ batch. The longest initial and final sets were 65 and 95 min, respectively, for the 8-ft³ batch. The variations in the air content, flow, and time of setting could be attributed to icing the batch water for the larger 8-ft³ placement.

5.3.2 Compressive strength

Compressive strength specimens were fabricated in accordance with ASTM C192 (2016d) and tested in accordance with ASTM C39 (2015a) procedures using 4-in. × 8-in. cylinders. The specimens were removed from the molds after 24 hr and air cured under room temperature at 73 ± 3°F until testing ages of 1, 7, and 28 days. Triplicate cylinders were tested at each age at a loading rate of 35 psi/sec until failure. Figure 10 illustrates the similar compressive strength results regardless of the batch volume. The MPC DB-C-T8 mixture achieved the minimum target compressive strength (5,000 psi) at 28 days.

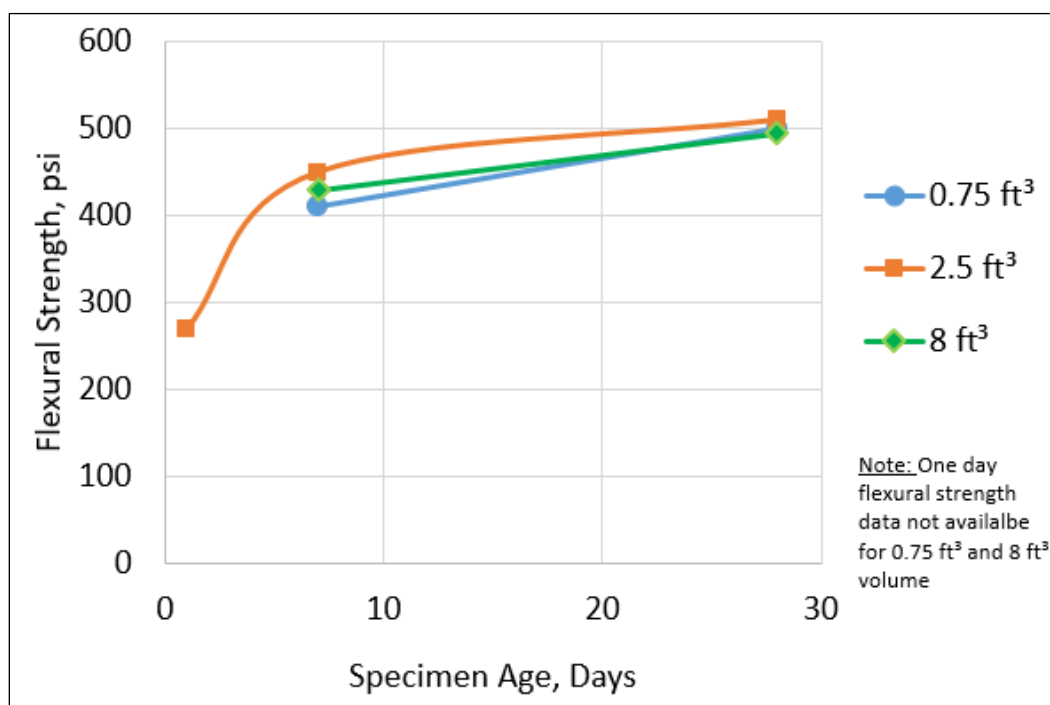
Figure 10. Comparison of compressive strength by volume for the MPC DB-C-T8 mixture.



5.3.3 Flexural strength

Flexural strength specimens were fabricated and tested in accordance with ASTM C78 (2016c). The test specimens were rectangular beams with dimensions of 3 in. x 3 in. x 12 in., with loading applied at third-points of the span. Triplicate beams at each test age were loaded at a specific rate to increase a stress of 150 psi/min in the bottom fiber (maximum tension) of the beams. The flexural strength trends were similar to the compressive strength results. Figure 11 illustrates that the tensile strength measured was invariant of the concrete batch size. The MPC DB-C-T8 mixture achieved the minimum target flexural strength (500 psi) at 28 days.

Figure 11. Comparison of flexural strength by volume for the MPC DB-C-T8 mixture.



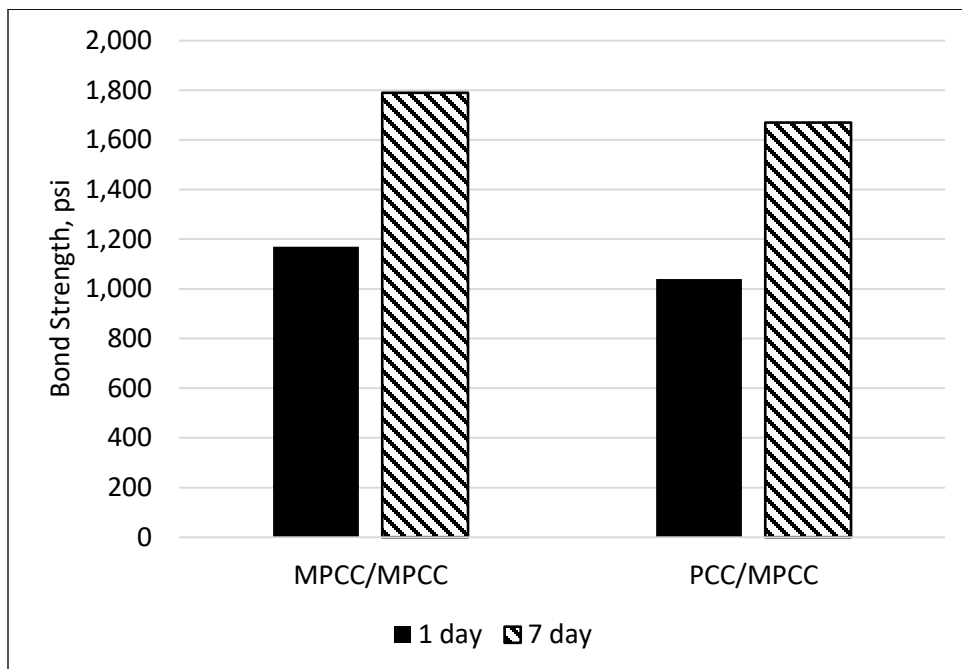
5.3.4 Bond strength

Achieving an adequate bond between repair materials and the existing concrete substructure is a key component for all repair materials. Bond strength specimens were fabricated and tested in accordance with ASTM C882 (2013a) procedures. This slant shear test involved the preparation of specimens in 3-in. x 6-in. cylinder molds. A bond line was produced at approximately 30 degrees from a vertical angle by first casting wedge-shaped dummy sections of either portland cement mortar (PCM) or the test material (TM) itself. One conditioning modification was made to the standard by not soaking the test specimen in water for 24 hr prior to bonding. Dry bonding conditions were used in view of findings by Yang et al. (2000) documenting that moist cure conditions negatively impacted bond strengths.

After curing in ambient, dry air conditions, triplicate composite cylinders were tested in compression at 1 and 7 days. The bond strengths illustrated in Figure 12 were calculated by dividing the load carried by the specimens at failure by the area of the elliptical bonding surface. The bond strengths of TM/TM at 1 day and 7 days were 1,170 psi and 1,790 psi, respectively. The bond strengths of PCM/TM at 1 day and 7 days were 1,040 psi and

1,670 psi, respectively. ASTM C928 (2013b) provides minimum performance requirements for this test of 1,000 psi at 1 day and 1,500 psi at 7 days for cementitious, rapid-setting materials. Based on these criteria, the results of the MPC concrete indicate this material will bond well to the parent substrate and is suitable for repairs and maintenance works.

Figure 12. Bond strength of TM/TM and PCM/TM at 1 and 7 days.



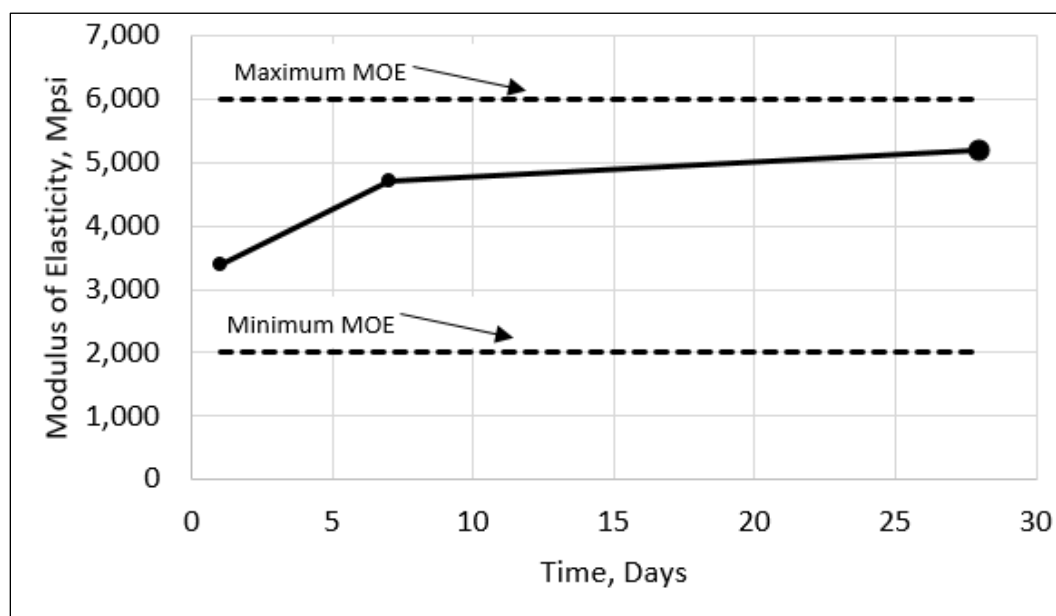
Under standard bond strength test conditions, the parent concrete material is prepared with a wet surface. Similarly, with most field repair applications, the repair surface is dampened during the preparation. A significant advantage of the dry bonding preference of this material is time saved by not having to dampen the surface when preparing the repair section. The reason theorized by Yang et al. (2000) is that the amount of hydration and the W/B ratio are very low; therefore, the shrinkage by moisture change is very low, and there is a very low possibility of causing cracking at the repaired interface. This property makes MPC materials have very strong adaptability in fields.

5.3.5 Modulus of elasticity

Modulus of elasticity is important because a repair material should not have stiffness significantly greater than the parent material. With a higher stiffness, the repair material will assume higher stresses under wheel loading and pavement movement. Modulus of elasticity testing was

accomplished in accordance with ASTM C469 (2014d) procedures. Test specimens were 3-in. × 6-in. cylinders instrumented with an unbounded sensing device attached to the cylinders at midheight for the purpose of measuring vertical deformation. The modulus of elasticity of the DB-C-T8 mixture, illustrated in Figure 13, was calculated as change in stress divided by change in strain, where strain was calculated as vertical deformation divided by gauge length. Clearly all modulus of elasticity results would meet the 2,000–6,000 ksi criteria established in the protocol for cementitious, rapid-setting pavement repair materials (Priddy 2011).

Figure 13. Modulus of elasticity for DB-C-T8 mixture.



5.3.6 Length change

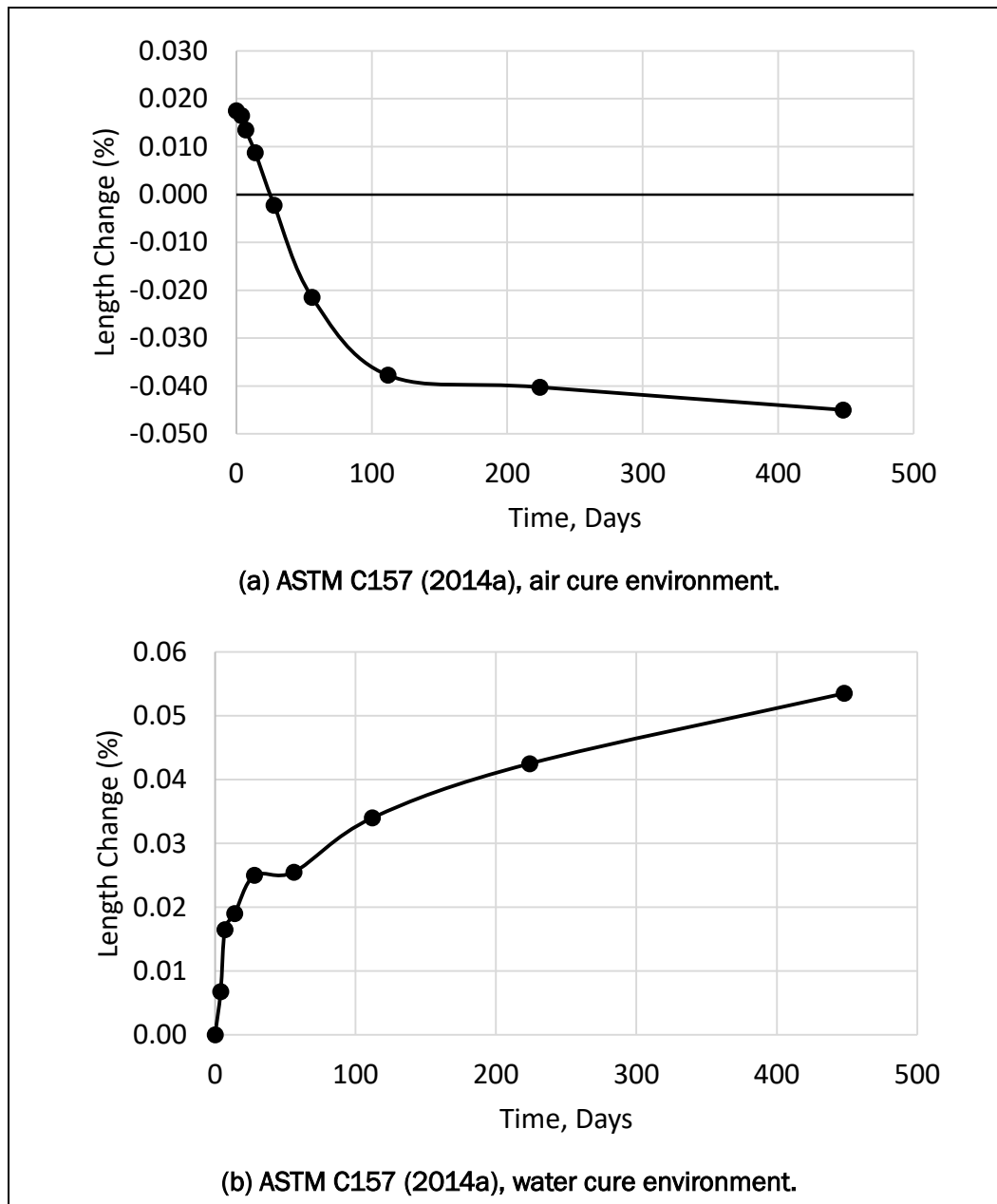
Length change is important because excessive expansion or contraction of a spall repair will result in a loss of bond to the parent material. Additionally, if the spall repair is large, excessive expansion can result in the deterioration of the surrounding pavements. Length change testing was accomplished in accordance with ASTM C157 (2014a) procedures with both air- and water-storage curing. Test prismatic specimens with dimensions of 3 in. × 3 in. × 11.25 in. with embedded gauge studs were fabricated from the MPC DB-C-T8 material with readings taken at 4, 7, 14, and 28 days and after 8, 16, 32, and 64 weeks by using a length comparator. The length change was calculated using the following equation:

$$\Delta L_{age} = \frac{L_{age} - L_0}{L_0}$$

Here, ΔL_{age} is the length change of the specimen at any age, L_{age} is the length of the specimen testing at any age, L_0 is the initial length of the specimen at 24 hr after casting of the specimen.

The length change results of the MPC concrete are illustrated in Figure 14 (a) air-cure environment and (b) water-cure environment. All of the results are an averaged value of three specimens. Similar values were found between the drying shrinkage and expansion results. The 28-day length change was 0.025% when cured in water and -0.020% when cured in air. According to the laboratory testing criteria for evaluating cementitious, rapid-setting pavement repair materials (Priddy 2011), the maximum acceptable length change is $\pm 0.03\%$ at 28 days. Based on this criterion, the MPC concrete material is dimensionally compatible. At 64 weeks, the shrinkage and expansion were -0.045% and 0.054%, respectively. No specific criteria at this age were identified in the benchmark criteria.

Figure 14. Length change over time in (a) air-cure environment and (b) water-cure environment.



5.3.7 Coefficient of thermal expansion

The coefficient of thermal expansion (CTE) is important for reasons similar to those for the modulus of elasticity. A repair material with a CTE that is significantly greater than the parent material will experience greater volume changes with changes in temperature (volumetric expansion due to externally applied forces). The difference in movements for the repair versus the parent material tends to deteriorate their bond.

CTE testing was accomplished in accordance with ASTM C531 (2012) procedures. Test bar specimens with dimensions of 1-in. x 1-in. x 11.25-in. were measured at 24 hr then daily for two weeks to determine linear shrinkage. Next, the test specimens cycled between environmental conditions of 210°F for 3 days, then 73°F for 24 hr until a constant CTE expansion was determined.

The final CTE of the DB-C-T8 mixture was 7.3 in./in./°F x 10⁻⁶. A similar CTE value of 7.8 in./in./°F x 10⁻⁶ was reported by Seehra et al. (1993). Although the results are slightly higher than the spall repair material testing protocol value of ≤ 7 in./in./°F x 10⁻⁶, the CTE is similar to typical values found in normal weight concrete (between 4.1 in./in./°F x 10⁻⁶ and 7.3 in./in./°F x 10⁻⁶). This indicates thermal compatibility between the MPC concrete and the parent concrete material.

5.3.8 Freezing and thawing durability

Freezing and thawing durability testing was accomplished in accordance to procedure A of ASTM C666 (2015d). Test prisms with dimensions of 3 in. x 4 in. x 16 in. were moist-cured for 14 days before being subjected to freeze-thaw cycles until failure (60% loss in dynamic modulus) or at a maximum of 300 cycles. The specimens were cycled between 4 and -18°C in 2 hr, so that 12 freezing and thawing cycles were executed in 24 hr. Fundamental transverse frequency was measured approximately every 36 cycles. Results are reported as the durability factor, which is a function of the number of cycles survived by the specimens and the relative dynamic modulus of elasticity at the time the test is terminated.

The relative dynamic modulus of elasticity, P_c , is defined by the equation

$$P_c = \frac{(n_1)^2}{(n)^2} \times 100$$

where:

- P_c = relative dynamic modulus of elasticity after c cycles
- n = fundamental transverse frequency at 0 cycles
- n_1 = fundamental transverse frequency after c cycles.

The durability factor, DF, is defined by the following equation:

$$DF = \frac{P \times N}{M}$$

where:

DF = durability factor of the test specimen

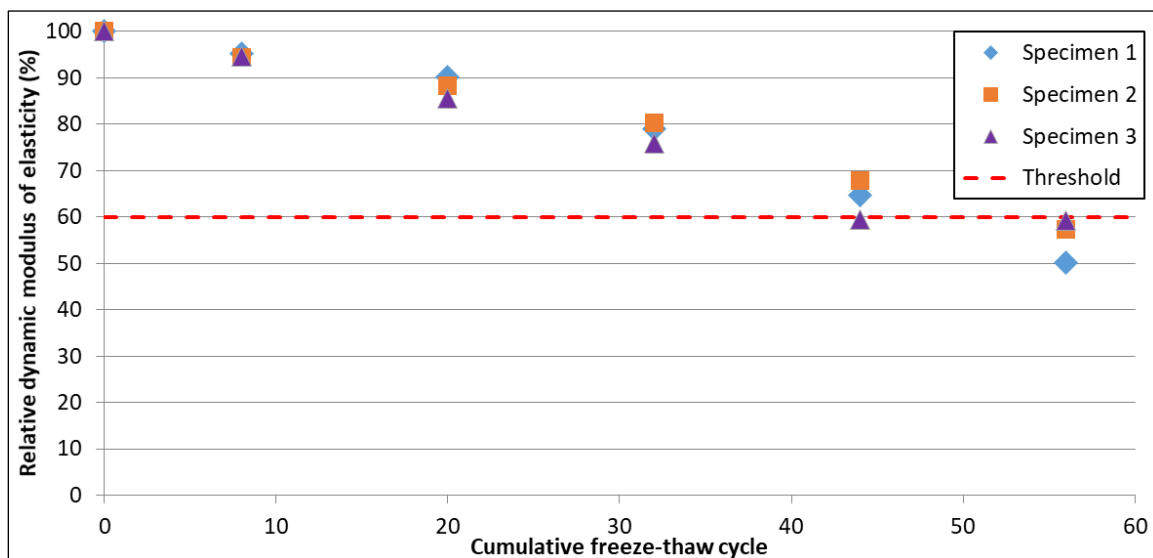
P = relative dynamic modulus of elasticity at N cycles, %

N = number of cycles at which P reaches the specified minimum value for discontinuing the test or the specified number of cycles at which the exposure is to be terminated, whichever is less

M = specified number of cycles at which the exposure is to be terminated (300 cycles in accordance to procedure A).

The graph in Figure 15 presents the relative dynamic elastic modulus with respect to the cumulative number of freeze-thaw cycles. For the tests conducted on the specimens for the DB-C-T8 mixture, the DF was 11 at 50 cycles. However, no surface scaling or spalling was observed on the specimens. The early freeze-thaw failure could be attributed to the low air content in the mixture.

Figure 15. Relative dynamic elastic modulus over freeze-thaw cycles.

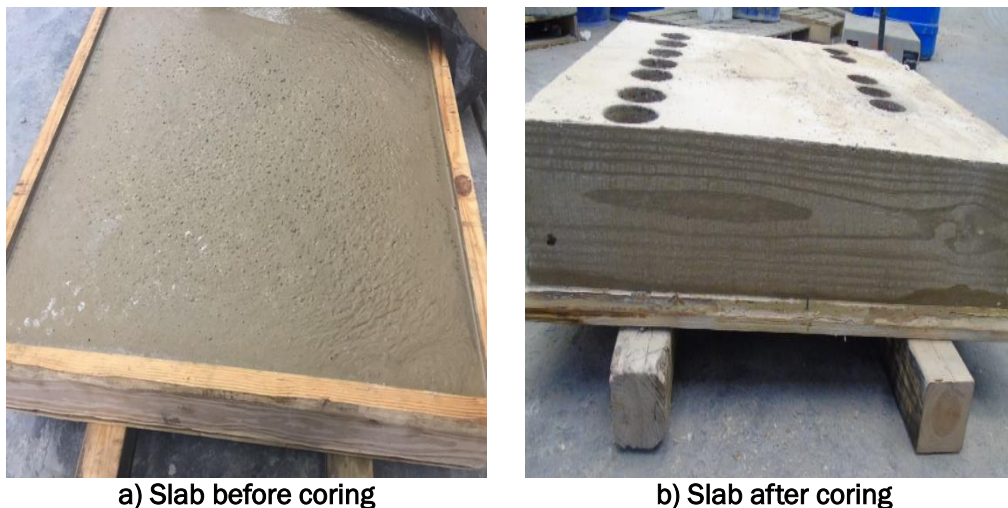


5.3.9 Drilled cores of concrete

Cores were drilled from a 3-ft x 3-ft x 7.5-in. slab of hardened MPC DB-C-T8 concrete in accordance with ASTM C42 (2016e). Samples were not taken until 7 days of curing to ensure the concrete was strong enough to permit sample removal without disturbing the bond between the mortar

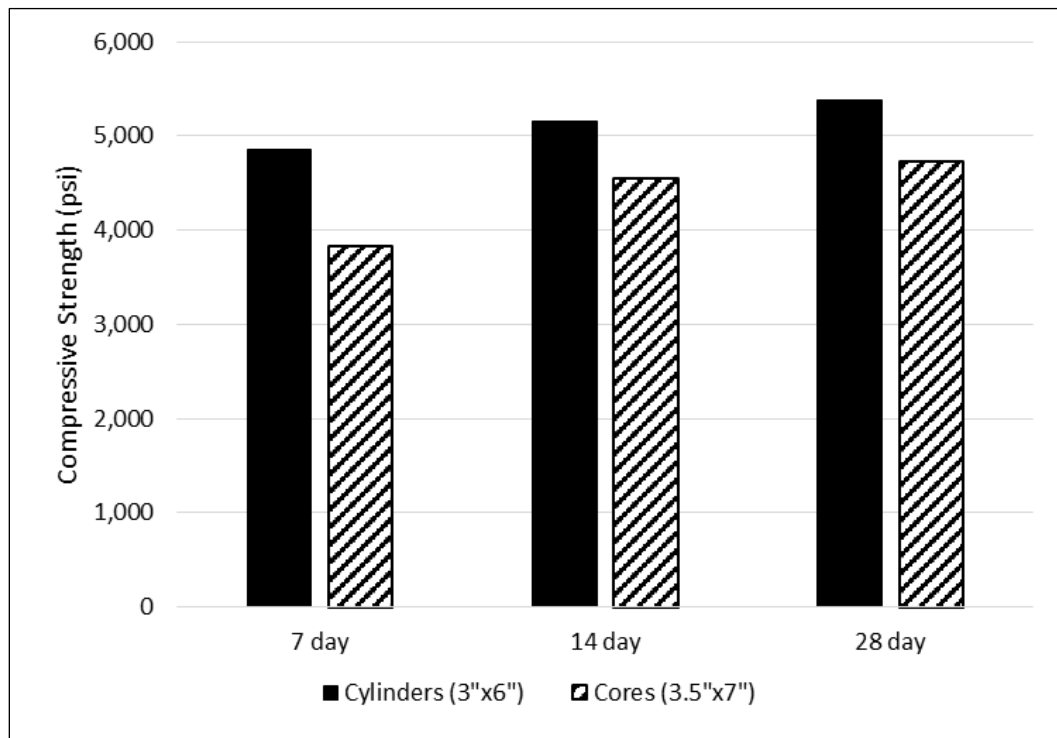
and the coarse aggregate. A core drill with diamond impregnated bits attached to a core barrel was used to take specimens of 3.5-in. x 7-in. dimensions with the purpose to determine the rate of strength gain. Dry coring was attempted due to concerns that excessive water could potentially reduce the strength of the cores by reacting with phosphates in the material. However, this forced excessive stress and friction on the core drill and barrel, so dry coring was not possible. Triplicate cores were extracted using the standard equipment water flow rate for each of three test ages (7, 14, and 28 days). Figure 16 illustrates the slab before and after final coring.

Figure 16. MPC concrete slab cast from DB-C-T8 mixture.



After coring, all water was wiped from the surface, and the cores with unbonded caps were allowed to dry for 1 hr prior to the testing in accordance with ASTM C39 (2015a) procedures. Figure 17 illustrates the compressive strength of the core specimens compared to companion cylinders cast from the same DB-C-T8 material. According to ACI 318 (2014), the concrete represented by the cores is considered structurally adequate if the average strength of three cores is at least 85% of the specified strength. The tested core strengths were 21% less than the companion cylinders at 7 days and 12% less at 14 and 28 days. Results indicate the 14- and 28-day core results are acceptable, but the concrete was possibly prematurely cored at 7 days and not strong enough to withstand damage during removal.

Figure 17. Comparison of cylinder and core specimens from MPC DB-C-T8 mixture.



6 Field Testing

Following the full-scale laboratory testing of DB-C-T8, four variations of concretes named MPC Mixes 1-4 were developed to blend commercially in bulk 2,800-lb super sacks. Prior to field testing the super sack MPC materials, a partial sample of the commercially blended materials was tested in the laboratory to characterize the products and compare mechanical and physical properties to earlier mixes performed in-house.

Field placement of concretes were conducted in two intervals at the Vicksburg, MS, test site to simulate craters in an airfield. Placement of MPC Mix 1 occurred in April 2018, and placements of MPC Mixes 2-4 occurred in May 2018. A volumetric mixer was used to place the commercially blended MPC material in slabs constructed 8.5 ft wide x 8.5 ft long x 8 in. deep with a companion test specimen for each of the four mixture designs.

6.1 Materials

Batch weights of MPC Mixes 1-4 are provided in Table 18. The raw materials were provided to CTS Cement Manufacturing Corp. to commercially blend the bulk proportions for uniformity. MPC Mix 1 was scaled up using all original materials in the mixture design DB-C-T8 detailed in earlier chapters. All aggregates were oven dried in-house and stored in barrels before shipment for dry blending of all materials. This proportion assumes a zero-% moisture. Mix 2 is a replicate of the original mixture design with the exception of using the CTS Cement Manufacturing Corp.'s locally sourced coarse aggregate. Mix 3 increases the NMA to 3/4 in. Mix 4 increases the A/B ratio from 50/50 to 52/48. Mixes 3 and 4 also used the CTS Cement Manufacturing Corp.'s locally sourced coarse aggregate.

Table 18. Batch weights of MPC mixture designs for super sack proportions.

Material	Source	Batch Weight (lb)			
		MPC Mix 1 CMB #180021	MPC Mix 2 CMB #180022	MPC Mix 3 CMB #180022	MPC Mix 4 CMB #180023
MgO Dead Burned	Martin Marietta - P98 Pulverized Magnesium Oxide	260	285	285	275
Monopotassium Phosphate (MKP) (KH ₂ PO ₄)	ICL Premium Fertilizers	419	458	458	442
Class C Fly Ash	Headwaters Resources, White Bluff Plant Redfield, AR	679	743	743	716
Concrete Sand	Green Brothers Crystal Springs, MS	677	666	666	788
3/8-in. Coarse Aggregate	Green Brothers Crystal Springs, MS	712	-	-	-
3/8-in. Coarse Aggregate	Osage River Rock Jefferson City, MO	-	778	467	829
3/4-in. Coarse Aggregate	Osage River Rock Wardsville, MO	-	-	391	-
Boric Acid	Three Elephants	54	59	59	57

6.2 Characterization and variability testing

6.2.1 X-ray Fluorescence

The bulk chemistry composition for randomly sampled super sacks of the MPC commercial blend materials in Mixes 1-4 was characterized by using X-ray fluorescence (XRF). The results in Table 19 show the similarity in the cementitious chemical compounds.

Table 19. Chemical composition of MPC commercial blend mixes.

Compound	MPC Mix 1 CMB #180021	MPC Mix 2 CMB #180022	MPC Mix 3 CMB #180023	MPC Mix 4 CMB #180023
SiO ₂ (%)	23.15	23.47	24.32	24.32
Al ₂ O ₃ (%)	12.3	12.36	12.29	12.3
Fe ₂ O ₃ (%)	3.42	3.37	3.58	3.57
CaO (%)	22.78	22.53	20.76	20.86
MgO (%)	28.01	27.99	29.13	29.16
SO ₃ (%)	2.26	2.27	1.49	1.54
K ₂ O (%)	1.43	1.37	1.48	1.48
Na ₂ O (%)	1.48	1.42	1.5	1.51
P ₂ O ₅ (%)	2.29	2.24	2.46	2.45
TiO ₂ (%)	1.01	0.99	1.02	1.03
Mn ₂ O ₃ (%)	0.01	0.01	0.01	0.01
SrO (%)	0.26	0.26	0.27	0.27
ZnO (%)	0.002	0.002	0.002	0.001
Cr ₂ O ₃ (%)	0.042	0.042	0.042	0.042
Loss on Ignition (%)	1.65	1.66	1.64	1.45

6.2.2 Cement content

The cement content of the randomly sampled super sacks of the MPC commercial blend materials in Mixes 1-4 was analyzed by sieve analysis in accordance to the ASTM C136 (2014b). The average percentage of material finer than the No. 200 size sieve was taken and reported in Table 20. The cement content ranged between 24.7 and 31.05 % with a coefficient of variation (COV) between 0.12 and 4.01 %.

Table 20. Cement content of random samples of the MPC super sack material.

Sample ID	Cement Content (%)	Std. Dev.	COV (%)
MPC Mix 1 CMB #180021	29.74	0.04	0.12
MPC Mix 2 CMB #180022	31.05	0.86	2.78
MPC Mix 3 CMB #180023	24.7	0.99	4.01
MPC Mix 4 CMB #180023	25.23	0.81	3.20

6.3 Laboratory experimental methods

Approximately 300 lb was sampled from the super sacks of material and dry blended in order to obtain a homogeneous material before mixing. A volume of 2.5 ft³ was used when batching and mixing the MPC concretes with a 0.2 W/B ratio, using the same mixing procedures detailed earlier. This batch volume was selected for the best comparison to the original laboratory mixtures discussed earlier.

Mixing was performed for 2½ min in a drum-type mechanical mixer apparatus per ASTM C192 (2016d) requirements. After mixing, the concrete was tested for physical and fresh properties of temperature, time of setting, air content, and unit weight. The fresh concrete was placed in forms to produce compressive and flexural test specimens. The forms were covered with plastic to prevent moisture loss from specimens for 24 hr before being demolded. All samples were kept in a temperature controlled room (73 ± 2°F) at 50% relative humidity until the age of testing. Unlike traditional concrete, which is moist-cured, these specimens were dry-cured.

6.4 Laboratory results of sampled MPC Mixes 1-4 super sacks

The results of the overall laboratory tests of fresh and hardened properties of the sampled super sacks of MPC Mixes 1-4 are summarized in Table 21.

Table 21. Results of fresh and hardened properties of sampled MPC super sacks.

Test Property	Specifics	MPC Mix 1 CMB #180021	MPC Mix 2 CMB #180022	MPC Mix 3 CMB #180023	MPC Mix 4 CMB #180023
Unit Weight ASTM C138	lb/ft ³	140	141	141	142
Air Content ASTM C231	%	3.3	2.0	2.6	1.2
Flow ASTM C1611	inch	13	10.5	9	13
Temperature ASTM C1064	°F	74	75	76	77
Time of Set (minutes) ASTM C403	Initial	35	38	23	45
	Final	45	46	36	65
Compressive Strength (psi) ASTM C39	1-day	1,110	1,130	1,240	950
	7-day	5,340	5,200	5,400	3,900
	28-day	5,420	5,170	5,620	4,650
Flexural Strength (psi) ASTM C78	7-day	445	400	440	350
	28-day	505	500	530	465

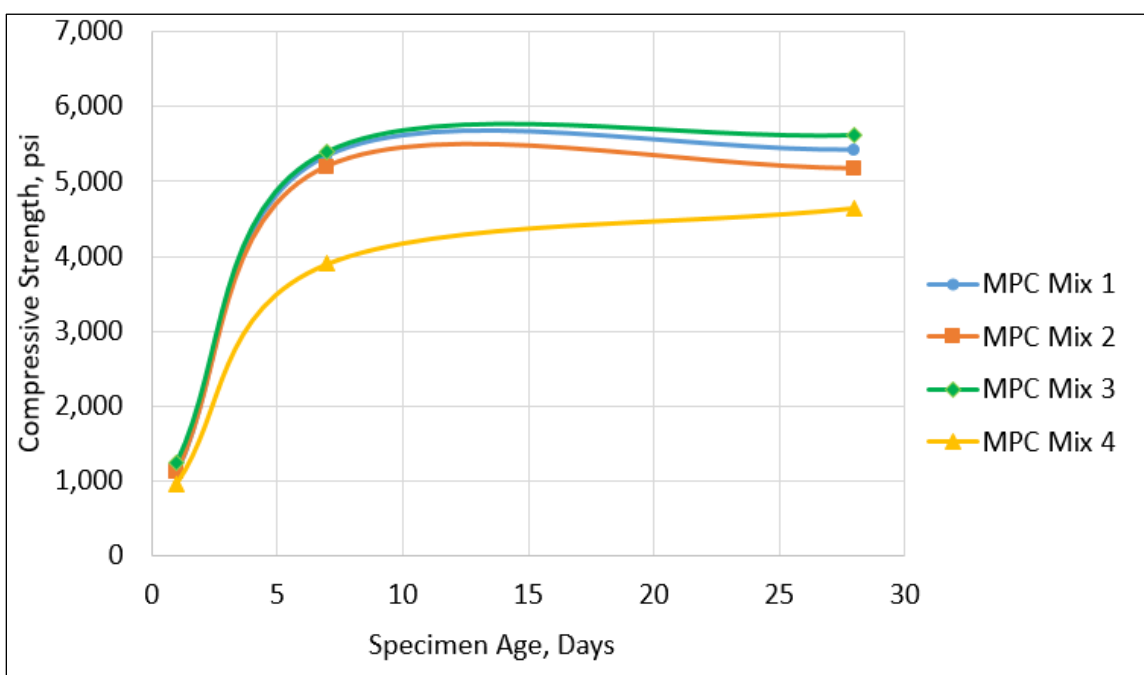
6.4.1 Fresh properties

For each batch, fresh concrete properties measured included unit weight (ASTM 2017c), air content (ASTM 2017b), flow (ASTM 2014c), temperature (ASTM 2017d), and time of setting (ASTM 2016f). The unit weight averaged 141 lb/ft³, which is consistent with the original laboratory mix result of 140 lb/ft³. The air content varied between each mixture with a range of 1.2 to 3.3%. The flow ranged from 9 to 13 in., so workability was maintained in each mixture. The flow was also consistent with the previously tested flow result of 10 in. The sampled concrete temperature ranged from 74 to 77°F when measured with a liquid-in-glass thermometer. The time of setting resulted in the most surprising of the fresh properties. All mixtures failed to meet the initial set requirement of at least 60 min. The original laboratory mix resulted in an initial set of 60 min and final set of 90 min. MPC Mix 3 produced the quickest initial and final setting times of 23 min and 36 min, respectively. MPC Mix 4 resulted in the longest initial and final setting times of 45 min and 65 min, respectively.

6.4.2 Compressive strength

Compressive strengths for the sampled super sacks of MPC Mixes 1-4 materials at 1, 7, and 28 days are shown in Figure 18. It can be seen from Figure 18 that Mix 3 exhibits marginally higher strengths over Mix 1 and 2, and Mix 4 resulted in the lowest of the four MPC mixture proportions. As expected, Mixes 1 and 2 produced similar strength trends. Only MPC Mixes 1, 2, and 3 achieved the minimum 28-day compressive strength requirement of 5,000 psi.

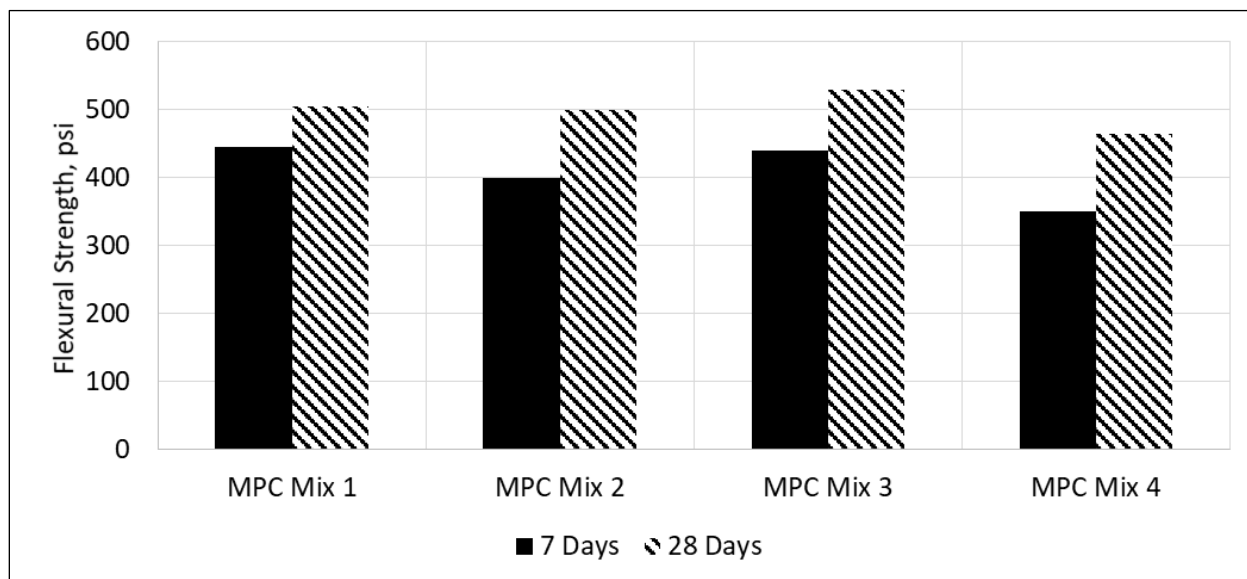
Figure 18. Compressive strength of sampled super sack of MPC Mixes 1-4 materials.



6.4.3 Flexural strength

Flexural strengths for the sampled super sacks of MPC Mixes 1-4 materials at 7 and 28 days are shown in Figure 19. Similar to trends in the compressive strength, Mix 3 exhibits marginally higher strengths over Mixes 1 and 2, and Mix 4 results in the lowest strengths of the four MPC mixture proportions. Only MPC Mixes 1, 2, and 3 achieved minimum 28-day flexural strength requirement of 500 psi.

Figure 19. Flexural strength of sampled super sack of MPC Mixes 1-4 materials.



6.5 Discussion of sampled super sacks of MPC Mixes 1-4

Overall, the sampled super sacks of MPC Mixes 1-4 were consistent with the full-scale testing results of the original MPC mixture design DB-C-T8. All fresh properties were similar to the original laboratory mixture results, with the exception of the time of setting, which was much shorter. The reduced time of setting could be due to the small sample portion tested from the large super sack of material. Variability in the materials, including boric acid that slows the time of setting, could have existed. The target 28-day compressive and flexural strengths were achieved (5,000 psi and 500 psi) on all but Mix 4.

6.6 Field experimental methods

For each slab, 2 yd³ (equivalent of two super sacks) of concrete was mixed by using ERDC's portable volumetric mixer, as illustrated in Figure 20, and placed in slabs constructed 8.5 ft wide x 8.5 ft long x 8 in. deep for each of the four mixture designs.

Figure 20. Loading the volumetric mixer for concrete placement of MPC materials.



For each batch, fresh concrete properties measured included unit weight (ASTM 2017c), air content (ASTM 2017b), temperature (ASTM 2017d), and time of setting (ASTM 2016f). Testing of the fresh properties was started within 5 min of obtaining the final portion of the composite sample. Figure 21 illustrates the sampling of the freshly mixed concrete.

Figure 21. Sampling freshly mixed concrete.



Twelve 4- x 8-in. concrete cylinder specimens were prepared for compressive strength testing at 1, 7, 14, and 28 days (3 cylinders at each age). Three 3- x 3- x 11.25-in. beam specimens were also prepared for flexural strength at 28 days. Figure 22 illustrates the field specimen preparation for mechanical property testing. All specimens were cured under plastic at the test site until the testing age.

Figure 22. Field specimen preparation for compressive and flexural strength.



Each batch of concrete was delivered to the crater from the chute of the volumetric mixer after approximately 5 min of mixing. Concrete rakes and shovels were used to homogeneously spread the material in the pit. Once filled, the cap was struck level and finished with a bull float. Figure 23 and Figure 24 illustrate the mixing, placing, and finishing process for the MPC Mixes 1-4.

Figure 23. Mixing and placing MPC Mixes 1-4 concrete.



(a) MPC Mix 1



(b) MPC Mix 2



(c) MPC Mix 3



(d) MPC Mix 4

Figure 24. Finishing MPC Mixes 1-4 concrete.



(a) MPC Mix 1



(b) MPC Mix 2



(c) MPC Mix 3



(d) MPC Mix 4



(e) Surface temperature monitoring



(f) Covering test section with plastic

6.7 Results and discussion of field testing

The fresh properties (unit weight, air content, temperature, and time of setting) and mechanical properties (compressive and flexural strengths) from the field tested MPC Mixes 1-4 are in Table 22.

Table 22. Results of fresh and hardened properties of field tested MPC super sacks.

Test Property	Specifics	MPC Mix 1 CMB #180021	MPC Mix 2 CMB #180022	MPC Mix 3 CMB #180023	MPC Mix 4 CMB #180023
Unit Weight ASTM C138	lb/ft ³	130	136	135	136
Air Content ASTM C231	%	2.1	2	2	2
Temperature ASTM C1064	°F	73	71.4	69.6	72.8
Time of Set (minutes) ASTM C403	Initial	70	55	50	60
	Final	90	65	60	80
Compressive Strength (psi) ASTM C39	1-day	180	260	320	240
	7-day	1,215	1,255	1,340	1,150
	14-day	2,380	2,820	3,200	1,780
	28-day	3,550	3,700	4,190	2,800
Flexural Strength (psi) ASTM C78	7-day	125	130	290	110
	28-day	370	370	440	305

6.7.1 Fresh properties

For each batch, measured fresh concrete properties included unit weight (ASTM 2017c), air content (ASTM 2017b), temperature (ASTM 2017d), and time of setting (ASTM 2016f). The unit weight of the field-mixed material ranged from 130-136 lb/ft³ compared to an average 140 lb/ft³ unit weight in the laboratory mixture. The air content averaged 2%. This could have been caused by placing the material with a more fluid consistency than was used in the laboratory mixtures. The temperature ranged from 69.6 to 73°F, which was lower than the laboratory measurement of 76°F and sampled super sack average ranging from 74 to 77°F. This was possibly due to the differences in temperature recording. An infrared temperature gun was used to measure the surface temperature

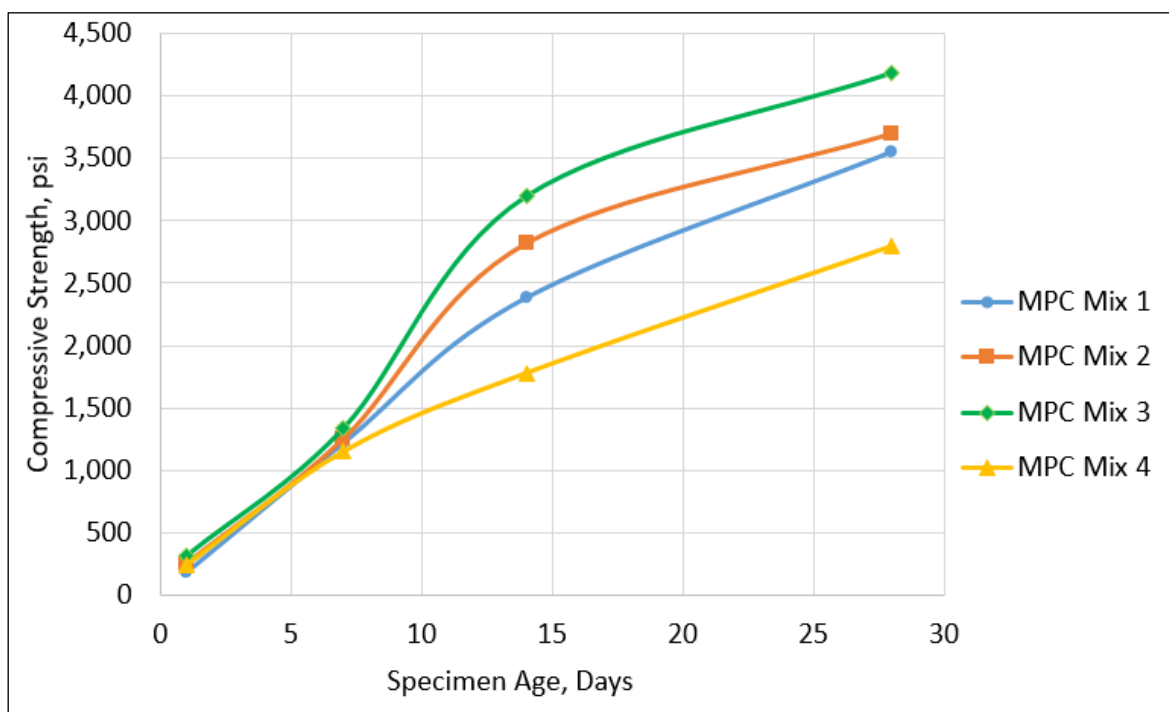
of the concrete slab compared to internal immersion of a liquid-in-glass thermometer for laboratory mixtures.

All mixture surface temperatures were monitored by an infrared thermometer and stayed below 80°F. The unit weight averaged 141 lb/ft³. MPC Mix 3 produced the quickest initial and final setting times of 50 and 60 min, respectively. MPC Mix 1 resulted in the longest initial and final setting times of 70 and 90 min, respectively.

6.7.2 Compressive strength

The comparison of MPC Mixes 1-4 at 1, 7, 14, and 28 days is illustrated in Figure 25. All mixture proportions had low 1- and 7-day strengths. Mix 3 reached the highest 28-day strength of 4,190 psi.

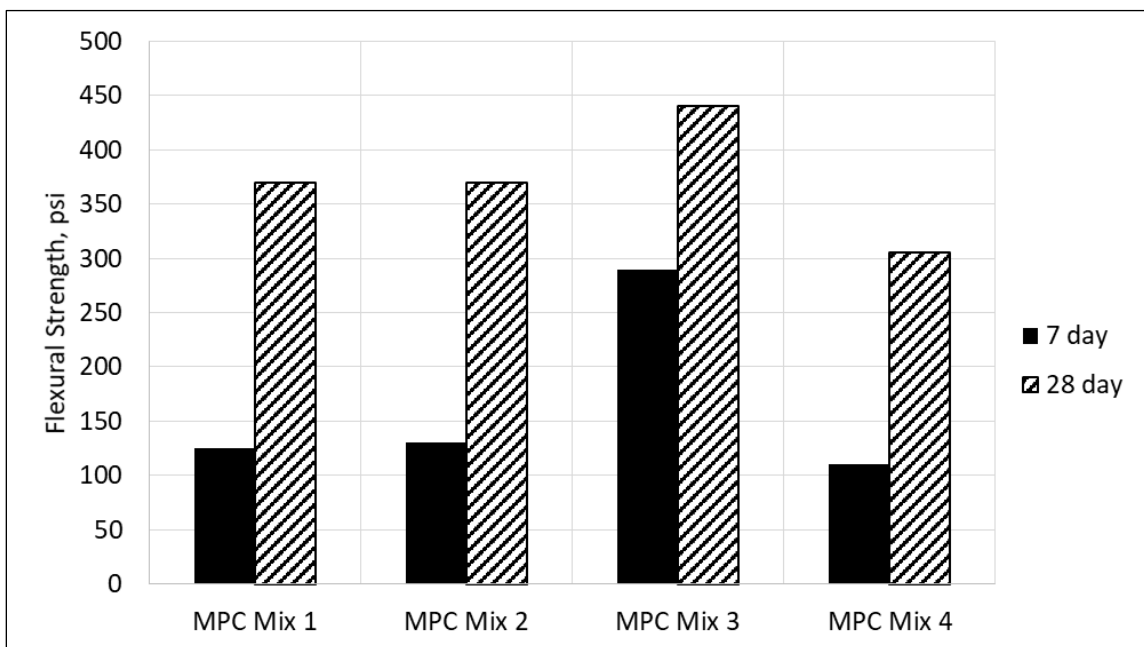
Figure 25. Compressive strength of MPC Mixes 1-4 field tested companion specimens.



6.7.3 Flexural strength

The flexural strengths of MPC Mixes 1-4 at 7 and 28 days are compared in Figure 26. Mix 1 and Mix 2 resulted in almost identical results at both ages. Mix 4 achieved the lowest strength of 110 psi at 7 days and 305 psi at 28 days. Mix 3 reached the highest strength of 290 at 7 days and 440 psi at 28 days.

Figure 26. Flexural strength of MPC Mixes 1-4 field tested companion specimens.



6.7.4 Discussion of field testing

Some challenges were encountered with the MPC Mix 1 upon opening the sealed super sacks of material. Although the product did not appear hydrated, the materials were densely compacted solid and had to be broken up into powder form before mixing. This was not difficult with the laboratory mix since it was only a partial 300-lb sample of the super sack material and was easily dry blended in a drum concrete mixer. However, this was a major problem for the field testing in preparing the volumetric mixer for approximate 2-yd³ yields. Large portions of the product had to be physically broken down with hammers, as shown in Figure 27. The volumetric mixer still experienced malfunctions during mixing due to the compaction of the MPC materials.

Figure 27. Large portions of compacted MPC material being broken down.



Although the super sacks of MPC Mix 1 materials were tightly sealed and lined with foil to minimize exposure to moisture, additional measures were needed to reduce potential hydration of the materials. MPC Mixes 2-4 were designed using local source aggregates, oven dried and blended commercially by CTS Cement Manufacturing Corp. This was to ensure no moisture was encountered in the shipment of aggregates. Mix 3 increased the NMA to $\frac{3}{4}$ -in., and Mix 4 increased the A/B ratio in attempts to reduce the highly compacted material in the super sacks. Additional QA measures were taken by the blending company to measure the free moisture at multiple intervals during packaging and to “pack set” the bulk super sack products. Despite these measures, the material for Mixes 2-4 still exhibited the same issues as Mix 1, indicating that the size and makeup of the dry materials is such that excessive packing can occur easily, even when each mix ingredient is sufficiently dry.

Another challenge encountered during field testing was determining the proper mixer settings to produce the target W/B ratio, as shown in Figure 28. The volumetric mixer uses a gate to allow more or less dry material to enter the mix auger, resulting in a lower or higher W/B ratio, respectively. The gate was adjusted at first to achieve a reasonable workable mixture, but additional fine adjustments were not conducted so that the required sampling could take place. The proper gate setting could easily be determined with additional testing.

Figure 28. Excessive water initially delivered from volumetric mixer.



During the placement of MPC Mix 4, segregation of the material was observed, and the material appeared more fluid. Strength results were also the lowest for Mix 4. This was possibly caused by the lower binder and increased aggregate content in the mixture design. Due to the low compressive strength results, no field section was trafficked under simulated aircraft loading.

7 Conclusions and Recommendations

The purpose of this study was to develop optimized MPC concrete mixture proportions from raw materials to meet characterization criteria for airfield pavements sponsored by the AFCEC. Raw materials used in this research included three types of MgO (light-burned [LB], hard-burned [HB], and dead-burned [DB]), one phosphate salt (potassium dihydrogen phosphate, or KDP), two types of fly ash (Class F and Class C), a retarder (boric acid), two gradations of coarse aggregates (3/8 in. and 3/4 in.), and one natural river sand.

A total of 24 mixture proportions were trial batched and tested for compressive strength and time of setting. Trial batching with the LB and HB MgO resulted in high exothermic reactivity, poor thixotropic workability, short setting times, and low compressive strengths. Therefore, these materials are not recommended. The trial batch named DB-C-T8 using DB MgO, KDP, Class C fly ash, boric acid, and locally available aggregates was selected for full-scale laboratory testing-based target accomplishments for the workability, time of setting, and compressive strength properties.

The Class C fly ash performed better than the Class F fly ash for MPC during trial batching. This is possibly due to the higher levels of calcium reacting with some of the remaining phosphates in the system, which leads to a denser, stronger, and more water-resistant material. The Class C fly ash slightly increased the set times and improved the workability of the MPCs.

Laboratory test results indicated the commercially blended MPC design is a good material for rapid repair of concrete and can be conventionally used. Measured engineering properties of compressive, flexural, splitting tensile, and bond strengths; modulus of elasticity; time of setting; length change; coefficient of thermal expansion; and freeze-thaw durability were comparable to the protocol used to evaluate rapid-setting cementitious materials for airfield spall repair.

Following the full-scale laboratory testing of DB-C-T8, four variations of the concrete proportions named MPC Mixes 1-4 were developed and commercially blended in bulk super sacks. The materials were densely compacted; and large, solid portions of the product had to be broken down

during the batching of the concrete. Although multiple QA measures were taken to reduce the MPC material's exposure to moisture, further investigation is needed for possible improvements in the bulk packaging of the MPC material to reduce compaction of the materials.

Large-scale (2 yd³) placements were achieved using the four specialized designs of MPC Mixes 1-4. This is noteworthy since no literature reference was discovered for an MPC concrete placement at this large volume. The setting time was about 60 min with the temperature under 80°C. Target 28-day compressive and flexural strengths of 5,000 psi and 500 psi, respectively, were accomplished with laboratory mixture proportioning but not with field testing. Due to failure to meet the target strengths, trafficking using simulated aircraft loads was not administered to the field test sections.

The undesirable mechanical property test results are attributed to the excessive water delivered to the material during the batching of the concrete. This excess can be overcome in future testing by placing tighter controls in operating procedures of the volumetric mixer and adjusting the gate settings to reduce the water when batching the MPC material.

In addition, the lower strengths may be a result of differences in curing conditions. The field specimens were not demolded in 24 hr then aged in an ambient air environment protected from moisture. Rather, specimens aged in their plastic molds on site until the test age. The confined test environment possibly sealed in the bleed water and caused any unhydrated phosphates to react, leading to lower strengths. The actual test sections possibly acquired sufficient strength, but coring was not performed, so a definitive statement cannot be made. It is recommended to obtain core samples from future slab sections for compressive strength testing to compare with comparison cylinder specimens at designated test ages.

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Appendix A: Material Data Reports

A.1 Light-burned MgO: Magox 93HR 325

MATERIAL SAFETY DATA SHEET				
PREMIER MAGNESIA			MSDS No.: 2621	
Phone: PREMIER MAGNESIA: 1-800-227-4287			Date Prepared: 10/10	
CHEMTREC, 24-Hr Emergency Assistance: 1-800-424-9300			This Revision:	
SECTION 1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION				
Material / Product Name(s): MAGOX® 93 HR 325 CAS Number: 1309-48-4 Chemical Family: Mineral Oxide General Use: A chemical grade magnesium oxide powder. Manufacturer / Supplier: PREMIER MAGNESIA, LLC 300 Barr Harbor Suite 250 West Conshohocken, PA 19428-2998				
SECTION 2. INGREDIENTS / COMPOSITION				
Ingredient name:	CAS Number:	Percent:	IARC/NTP/OSHA:	Exposure Limits:
Calcined Magnesite (Magnesium Oxide)	1309-48-4	100	No	Nuisance Particulate OSHA PEL: TWA 15mg/m ³ ; respirable: 5mg/m ³ . ACGIH TLV: TWA Total dust: 10mg/m ³ ; respirable dust: 5mg/m ³ .
Quartz* (SiO ₂)	14808-60-7	0-1	Yes	ACGIH TLV: TWA respirable quartz 0.05mg/m ³ .
<u>Typical Chemical Analysis, Wt % (Loss Free Basis)</u>				
LOI (1000°C)	3.72			
Acid Insol	2.00			
R ₂ O ₃	0.92			
CaO	3.37			
MgO	93.72			
The oxides shown in the typical chemical analysis do not exist in the magnesium oxide as free, uncombined oxides, but are combined mineralogically as calcium-magnesium silicates, aluminates and ferrites.				
*Quartz. Product may contain a trace of quartz, a polymorph of crystalline silica, which is classified by IARC as a "Known Human Carcinogen - Group 1". NTP lists respirable crystalline silica amongst substances which may "reasonably be anticipated to be carcinogens".				
SECTION 3. HAZARDS IDENTIFICATION				
HMIS				
HEALTH HAZARD		1 - SLIGHT		
FLAMMABILITY HAZARD		0 - MINIMAL		
REACTIVITY HAZARD		1 - SLIGHT		
PERSONAL PROTECTION		B - Glasses, Gloves		
EMERGENCY OVERVIEW:				
A brownish, free flowing, fine granular material. Not a fire or spill hazard. Contact with water may cause product to swell, generate some heat, and burst its container. Low toxicity. Dust is classified as a "nuisance particulate not otherwise regulated".				
Target Organs: Chronic overexposure may cause lung damage.				
Primary route(s) of entry: Inhalation				
Acute effects: Particulate may cause eye and upper respiratory irritation.				
Page 1 ---		HAZARD IDENTIFICATION continues on page 2		--- Page 1

MATERIAL SAFETY DATA SHEET	
PREMIER MAGNESIA Phone: PREMIER MAGNESIA: 1-800-227-4287 CHEMTRAC, 24-Hr Emergency Assistance: 1-800-424-9300	MSDS No.: 2621 Date Prepared: 10/10 This Revision:
HAZARD IDENTIFICATION continued from page 1 Chronic effects: Product dust is classified as a "nuisance particulate, not otherwise regulated" as specified by ACGIH and OSHA. The excessive, long-term inhalation of mineral dusts may contribute to the development of industrial bronchitis, reduced breathing capacity, and may lead to the increased susceptibility to lung disease. Signs & symptoms of overexposure: Eye contact: Particulate is a physical eye irritant. Skin contact: Low toxicity by skin contact. Inhalation: Chronic overexposure by inhalation of airborne particulate may irritate upper respiratory system as well as the throat. Ingestion: An unlikely route of exposure. If ingested in sufficient quantity, may cause gastrointestinal disturbances. Symptoms may include irritation, nausea, vomiting and diarrhea.	
SECTION 4. FIRST AID MEASURES	
Eye contact: Flush eyes, including under the eyelids, with large amounts of water. If irritation persists, seek medical attention. Skin contact: Wash affected areas with mild soap and water. Inhalation: Remove victim to fresh air. If not breathing, give artificial respiration. Get immediate medical attention. Ingestion: Ingestion is an unlikely route of exposure. If ingested in sufficient quantity and victim is conscious, give 1-2 glasses of water or milk. Never give anything by mouth to an unconscious person. Leave decision to induce vomiting to qualified medical personnel, since particles may be aspirated into the lungs. Seek immediate medical attention.	
SECTION 5. FIRE FIGHTING MEASURES	
NFPA code: Flammability: <u>0</u> , Health: <u>1</u> , Reactivity: <u>1</u> , Special: <u>0</u> . Flash point: Not Combustible Unusual Fire Hazard / Extinguishing Media: Water reacts with magnesium oxide producing magnesium hydroxide and heat. Do not allow water to get inside containers; reaction with water will cause product to swell, generate heat, and burst its container. If contact is unavoidable, use sufficient water to safely absorb the heat that may be generated. Wetted product is not a health or environmental hazard. Hazardous Decomposition Products: None Firefighting Instructions: Firefighters should wear NIOSH-approved, positive pressure, self-contained breathing apparatus and full protective clothing when appropriate.	
SECTION 6. ACCIDENTAL RELEASE MEASURES	
Spill procedures: Carefully, clean up and place material into a suitable container, being careful to avoid creating excessive dust from dried product. If conditions warrant, clean up personnel should wear approved respiratory protection, gloves, and goggles to prevent irritation from contact and/or inhalation.	
SECTION 7. HANDLING AND STORAGE	
Storage: Store in dry, protected storage. Product is stable under normal conditions of dry storage. Do not allow water to get inside containers; reaction with water will cause product to swell, generate heat, and burst its container. Exposed, unprotected magnesium oxide will absorb moisture and carbon dioxide from the air. Minimize dust generation during material handling and transfer.	
SECTION 8. EXPOSURE CONTROLS AND PERSONAL PROTECTION	
Engineering controls: Provide sufficient ventilation, in both volume and air flow patterns to control mist/dust concentrations below allowable exposure limits.	
Page 2 --- EXPOSURE CONTROLS AND PERSONAL PROTECTION continues on page 3 --- Page 2	

MATERIAL SAFETY DATA SHEET

PREMIER MAGNESIA

MSDS No.: 2621

Phone: PREMIER MAGNESIA: 1-800-227-4287

Date Prepared: 10/10

CHEMTRAC, 24-Hr Emergency Assistance: 1-800-424-9300

This Revision:

EXPOSURE CONTROLS AND PERSONAL PROTECTION continued from page 2**Personal protective equipment:** The use of eye protection, gloves and long sleeve clothing is recommended.**Respiration protection:** Provide workers with NIOSH approved respirators in accordance with requirements of 29 CFR 1910.134 for level of exposure incurred.**Hygienic Practices:** Avoid contact with skin, eyes and clothing. After handling this product, wash hands before eating or drinking.**SECTION 9. PHYSICAL AND CHEMICAL PROPERTIES****Appearance:** The product is brownish, fine granular, and free flowing; odorless.**Boiling Point:** Not Applicable**Specific Gravity (g/cc):** 3.56**Melting Point:** >3800°F (>2100°C)**Bulk Density (lbs./cu.ft.):** 60 - 70**Water Solubility:** Slight <1%**% Volatile by volume:** 0**pH (10% aqueous slurry):** 10-11**Evaporation rate:** Not Applicable**SECTION 10. STABILITY AND REACTIVITY****Hazardous Polymerization:** Will not occur**Chemical Incompatibilities:** Magnesium oxide is soluble in aqueous acids generating heat and steam; violent reaction or ignition with interhalogens (e.g., bromine pentafluoride; chlorine trifluoride). Incandescent reaction with phosphorus pentachloride. Water will react with magnesium oxide producing magnesium hydroxide and heat.**Hazardous Decomposition Products:** Heat and Steam**SECTION 11. TOXICOLOGICAL INFORMATION****Magnesium Oxide** CAS #1309-48-4 Toxic and Hazard Review: low toxicity - a nutrient and/or dietary supplement food additive. THERAP CAT: antacid. (Sax) an experimental tumorigen. Inhalation of fume (not MgO dust particular) produced upon decomposition of magnesium compounds can produce a febrile reaction and leukocytosis in humans.**TOXICITY DATA:** ihl-hmn TCLo:400mg/m³, itr-ham TDLo:480 mg/kg/30w-I:ETA.**Quartz** CAS #14808-60-7. Toxic and Hazard Review (Sax): Experimental poison by intratracheal and intravenous routes. An experimental carcinogen, tumorigen, and neoplastigen. Human systemic effects by inhalation: cough, dyspnea, liver effects. Listed by IARC as a "Known Human Carcinogen" Group 1. Listed by NTP.**TOXICITY DATA:** No LD₅₀ in RTECS. ihl-hmn: TCLo 16 mppcf / 8 hrs / 17.9Y-I: PUL; ihh-hmn LCLo: 300 µg / m³ / 10 Y-I;LVR; Other species toxicity data (NIOSH RTECS): inv-rat LDLo: 90mg/kg, itr-rat LDLo: 20mg/kg; ivn-mus LDLo: 40mg/kg; inv-mus: 20mg/kg.**SECTION 12. ECOLOGICAL INFORMATION****Ecotoxicological / Chemical Fate Information:** No data available on any adverse effects of this material on the environment.**SECTION 13. DISPOSAL INFORMATION****Waste Management/Disposal:** This product does not exhibit any characteristics of a hazardous waste. The product is suitable for landfill disposal. Follow all applicable federal, state and local regulations for safe disposal.**SECTION 14. TRANSPORT INFORMATION****US Department of Transportation:** Not regulated by DOT as a hazardous material. No hazard class, no label or placard required, no UN or NA number assigned.**Canadian TDG Hazard Class & PIN:** Not regulated.

MATERIAL SAFETY DATA SHEET

PREMIER MAGNESIA

MSDS No.: 2621

Phone: PREMIER MAGNESIA: 1-800-227-4287

Date Prepared: 10/10

CHEMTRAC, 24-Hr Emergency Assistance: 1-800-424-9300

This Revision:

SECTION 15. REGULATORY INFORMATION

SARA TITLE III: This product does not contain any substances reportable under Sections 302, 304 or 313. Sections 311 and 312 do apply. (Routine Reporting and Chemical Inventories)

TSCA: All substances in this product are listed in the Chemical Substance Inventory of the Toxic Substances Control Act.

CERCLA Hazardous Substance List, RQ: No

California Proposition 65: This product contains chemicals known to the State of California to cause cancer, birth defects or other reproductive toxins.

SECTION 16. OTHER INFORMATION

ACRONYMS AND REFERENCES USED IN PREPARATION OF MSDS':

ACGIH:	American Conference of Governmental Industrial Hygienists
CAS#:	CAS Registration Number is an assigned number to identify a material. CAS stands for Chemical Abstracts Service.
CERCLA:	Comprehensive Environmental Response, Compensation & Liability Act
EPCRA:	Emergency Planning and Community Right-to-Know Act of 1986
HMISTM:	Hazardous Materials Identification System (National Paint & Coatings Association)
IARC:	International Agency for Research on Cancer
MSHA:	Mine Safety and Health Administration
mg/m ³ :	Milligrams per cubic meter
NIOSH:	National Institute for Occupational Safety and Health
NFPA:	National Fire Protection Association
NTP:	National Toxicology Program
OSHA:	Occupational Safety and Health Administration
PEL:	Permissible Exposure Limit (OSHA)
REL:	Recommended Exposure Limit (OSHA)
SARA:	Superfund Amendments and Reauthorization Act
TITLE III:	Emergency Planning and Community Right-to-Know Act
Section 302:	Extremely Hazardous Substances
Section 304:	Emergency Release
Section 311:	Community Right-to-Know, MSDSs or List of Chemicals
Section 312:	Community Right-to-Know, Inventory and Location, (Tier I/II)
Section 313:	Toxic Chemicals, Toxic Chemical Release Reporting, Form R
TLV:	Threshold Limit Values (ACGIH)
TWA:	Time Weighted Average
29CFR1910.134:	OSHA Respiratory Protection Standard

REFERENCES:

Sax, N. Irving: Dangerous Properties of Industrial Materials, Ninth Edition, Van Nostrand Reinhold Co., Inc., 1996.
 Kirk, R. and Othmer, D., Encyclopedia of Chemical Technology, Third Edition, Wiley-Interscience, New York, NY 1982.
 Clansky, K.B., Suspect Chemicals Sourcebook, 1992-2nd Edition, Roytech Publications, Bethesda, Maryland.
 Sax, N. Irving and Lewis, R.J. Hawley's Condensed Chemical Dictionary, Eleventh Ed., Van Nostrand Reinhold Co., Inc., NY
 Manufacturers / Suppliers, Material Safety Data Sheets on Raw Materials Used
 American National Standard for Hazardous Industrial Chemicals - Material Safety Data Sheets - Preparation, American National Standards Institute, Inc., 11 West 42nd St, New York, NY 10036.

Prepared/revised: Mark A. Shand October 27, 2010

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A.2 Hard-burned MgO: MagChem 10



MagChem® 10 Hard Burned Magnesium Oxide

DESCRIPTION	MagChem 10 grades are high purity, hard burned magnesium oxides with relatively high density and low reactivity and produced from magnesium-rich brine and dolomitic lime. MagChem 10 granular grades are essentially dust free.						
USES	Milled MagChem 10 grades have a combination of low reactivity, high purity and fine particle size which makes them suitable for the production of magnesium salts, particularly in reactions with strong acids. Milled MagChem 10 grades also find applications in fiberglass, aluminum metal processing, and fuel additives. Screened MagChem 10 products are widely used as a raw material in manufacturing refractories and ceramic products.						
COMPOSITION							

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A.3 Dead-burned MgO: MagChem P98



MagChem® P98 Dead Burned Milled Magnesium Oxide

DESCRIPTION	MagChem P98 products are high purity magnesium oxides produced from magnesium-rich brine and dolomitic lime. The products are fired in a shaft kiln to produce dead burned magnesium oxide with high density and low reactivity.			
USES	MagChem P98 products are well suited for refractory and ceramic applications and other applications where a slow, controlled chemical reaction rate is required.			
COMPOSITION		Typical	Specification	
	Magnesium Oxide (MgO), %	98.0	97.7 min.	
	Silicon Oxide (SiO ₂), %	0.7	0.8 max.	
	Calcium Oxide (CaO), %	0.95	1.1 max.	
	Iron Oxide (Fe ₂ O ₃), %	0.15	0.3 max.	
	Aluminum Oxide (Al ₂ O ₃), %	0.19	0.3 max.	
	Loss on Ignition, %	---	0.3 max	
MagChem P98 grades are available in a variety of milled sizes from minus 1/8" to powder.				
Typical Screen Sizes				
	% Passing	1/8"	-30 Mesh	Pulverized
	4 Mesh	100		
	6 Mesh	95		
	8 Mesh	64	100	
	16 Mesh	16	99.0	100
	30 Mesh	2	88	99.8
	50 Mesh			98
	100 Mesh			91
	200 Mesh		16	75
	325 Mesh			60

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A.4 Potassium dihydrogen phosphate (KH_2PO_4)



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1 Identification of the substance/mixture and of the company/undertaking

1.1 Product identifier

Trade name: **MKP**

Synonyms

Phosphoric acid, monopotassium salt; Mono potassium phosphate; Potassium dihydrogen orthophosphate, multi-MKP, PeaK, Krista MKP, Krista

Article number: 9744080200, 9744080100

CAS Number:

7778-77-0

EC number:

231-913-4

Index number: None

Registration number 01-2119490224-41-0015

1.2 Relevant identified uses of the substance or mixture and uses advised against

Relevant identified uses:

Fertilizer

Detergents

Water treatment

Food additives

Fire retarding agent

Processing aid/ Additive

No uses advised against.

1.3 Details of the supplier of the safety data sheet

Manufacturer/Supplier:

Rotem Amfert Negev Ltd.

ICL Specialty Fertilizers

Mishor Rotem, Mobile Post Arava 86800

ISRAEL

Phone: +972-8-6598877

Fax: +972-8-6598987

E-mail: novapeak@iclfertilizers.com

Only Representative/Supplier:

P.M. Chemicals S.r.l.

Via Monteverdi 11, 20131, Milano,

Italy

Phone: +39-02-20487221

Fax: +39-02-2049449

E-mail: info.pmchemicals@pmchemicals.it

1.4 Emergency telephone number:

In Europe call: +31-205-815100 (24 hours a day, 365 days a year)

In Israel call: +972-8-6504777 (24 hours a day, 365 days a year)

+972-8-6504915

2 Hazards identification

2.1 Classification of the substance or mixture

Classification according to Regulation (EC) No 1272/2008

The substance is not classified according to the CLP regulation.

Classification according to Directive 67/548/EEC or Directive 1999/45/EC Not applicable.

Information concerning particular hazards for human and environment:

No hazards to be particularly mentioned.

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- 2.2 Label elements
- Labelling according to Regulation (EC) No 1272/2008 Void
- Hazard pictograms Void
- Signal word Void
- Hazard statements Void
- 2.3 Other hazards
- Results of PBT and vPvB assessment
- PBT: Not applicable.
- vPvB: Not applicable.

* 3 Composition/information on ingredients

- 3.1 Chemical characterization: Substances
- CAS No. Description
- 7778-77-0 potassium dihydrogenorthophosphate
- EC number: 231-913-4
- SVHC None

* 4 First aid measures

- 4.1 Description of first aid measures
- General information: No special measures required.
- After inhalation: Supply fresh air; consult doctor in case of complaints.
- After skin contact:
Generally the product does not irritate the skin.
Rinse with warm water.
If skin irritation continues, consult a doctor.
- After eye contact:
Rinse opened eye for several minutes under running water. If symptoms persist, consult a doctor.
- After swallowing:
Rinse out mouth and then drink plenty of water.
If symptoms persist consult doctor.
- NOTE: Never give an unconscious person anything to drink.
- 4.2 Most important symptoms and effects, both acute and delayed No further relevant information available.
- 4.3 Indication of any immediate medical attention and special treatment needed
No further relevant information available.

* 5 Firefighting measures

- 5.1 Extinguishing media
- Suitable extinguishing agents:
The product is not flammable.
Use fire extinguishing methods suitable to surrounding conditions.
- For safety reasons unsuitable extinguishing agents: None
- 5.2 Special hazards arising from the substance or mixture
In case of fire, the following can be released:
Phosphorus oxides (e.g. P₂O₅)
- 5.3 Advice for firefighters
- Protective equipment:
Wear fully protective suit.

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Mouth respiratory protective device.

Additional information

Collect contaminated fire fighting water separately. It must not enter the sewage system.

6 Accidental release measures

6.1 Personal precautions, protective equipment and emergency procedures

Avoid formation of dust.

Use respiratory protective device against the effects of fumes/dust/aerosol.

Wear protective clothing.

6.2 Environmental precautions: Do not allow to enter sewers/surface or ground water.

6.3 Methods and material for containment and cleaning up: Pick up mechanically.

6.4 Reference to other sections See Section 13 for disposal information.

7 Handling and storage

7.1 Precautions for safe handling

Ensure good ventilation/exhaustion at the workplace.

Prevent formation of dust.

Information about fire - and explosion protection:

The product is not flammable.

No special measures required.

7.2 Conditions for safe storage, including any incompatibilities

Requirements to be met by storerooms and receptacles:

Store in dry conditions.

Protect from heat and direct sunlight.

Information about storage in one common storage facility:

Store away from oxidizing agents.

Do not store together with alkalis (caustic solutions).

Further information about storage conditions:

Protect from humidity and water.

Store in a cool place.

7.3 Specific end use(s) No further relevant information available.

8 Exposure controls/personal protection

8.1 Control parameters

Ingredients with limit values that require monitoring at the workplace: Not required.

DNELs

For workers:

Long-term-systemic effects (inhalation) DNEL: 4.07 mg/m³

For general population:

Long-term-systemic effects (inhalation) DNEL: 3.04 mg/m³

PNECs

PNEC aqua (freshwater): 0.05 mg/L

PNEC aqua (marine water): 0.005 mg/L

PNEC aqua (intermittent releases): 0.5 mg/L

PNEC STP: 50 mg/L

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Additional information:

Ventilation must be sufficient to maintain TLV-TWA below 3 mg/m³, respirable particles, and 10 mg/m³, inhalable particles [ACGIH recommendation for Particles (Insoluble or poorly soluble). Not Otherwise Specified (PNOS)]

8.2 Exposure controls

Personal protective equipment:

General protective and hygienic measures:

The usual precautionary measures are to be adhered to when handling chemicals.

Keep away from foodstuffs, beverages and feed.

Immediately remove all soiled and contaminated clothing

Wash hands before breaks and at the end of work.

Do not eat or drink while working.

Respiratory protection:

Use suitable respiratory protective device in case of insufficient ventilation.

Filter P2

Protection of hands:



Protective gloves

Material of gloves

The selection of the suitable gloves does not only depend on the material, but also on further marks of quality and varies from manufacturer to manufacturer.

Penetration time of glove material

The exact break through time has to be found out by the manufacturer of the protective gloves and has to be observed.

Eye protection: Safety glasses

Body protection: Protective work clothing

Limitation and supervision of exposure into the environment

Based on all data available this product is not considered to pose a risk to the environment.

Risk management measures

Do not allow undiluted product or large quantities of it to reach ground water, water course or sewage system.

9 Physical and chemical properties

9.1 Information on basic physical and chemical properties

General Information

Appearance:

Form: Crystalline

Colour: White

Odour: Odourless

pH-value (208 g/l) at 20°C: 4,2-4,5

Change in condition

Melting point/Melting range: 252,6°C

Boiling point/Boiling range: > 450°C

Flash point:

Not applicable.

This product is inorganic substance.

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· Flammability (solid, gaseous):	Product is not flammable. (based on molecular structure)
· Ignition temperature:	Not applicable
· Decomposition temperature:	>450°C Thermal decomposition on losing water.
· Self-igniting:	Product is not selfigniting. (based on molecular structure)
· Danger of explosion:	Product does not present an explosion hazard. (based on molecular structure)
· Explosion limits:	None
· Oxidizing properties	None The substance does not contain any groups associated with oxidising properties.
· Vapour pressure at 25°C:	4,5x10 ⁻¹⁵ Pa
· Density at 20°C:	2,34 g/cm ³
· Bulk density at 20°C:	1150-1200 kg/m ³
· Solubility in / Miscibility with water at 20°C:	208 g/l
· Segregation coefficient (n-octanol/water):	Not applicable This substance is inorganic chemical.
· Viscosity:	Not applicable This product is solid. Viscosity is only relevant to liquids.
· 9.2 Other information	No further relevant information available.

10 Stability and reactivity

- **10.1 Reactivity** Reacts with alkali (lyes).
- **10.2 Chemical stability** No decomposition if used and stored according to specifications.
- **10.3 Possibility of hazardous reactions** Reacts with oxidizing agents.
- **10.4 Conditions to avoid**
Water
To avoid thermal decomposition do not overheat.
- **10.5 Incompatible materials:**
Alkalies
Oxidizing agents
- **10.6 Hazardous decomposition products:**
Formation of toxic gases is possible during heating or in case of fire.
Phosphorus oxides (e.g. P₂O₅)
- **Additional information:** This product is hygroscopic.

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11 Toxicological information

11.1 Information on toxicological effects

Acute toxicity:

LD/LC50 values relevant for classification:
no classification is necessary

14887-42-4 Potassium pentahydrogen bis(phosphate)

Dermal	LD50	>2000 mg/kg (rabbit) (OECD 402)
--------	------	---------------------------------

7558-80-7 sodium dihydrogenorthophosphate

Inhalative	LC50/4 h	>0,83 mg/l (rat) (OECD 403) the maximum attainable concentration
------------	----------	---

7778-77-0 potassium dihydrogenorthophosphate

Oral	LD50	>2000 mg/kg (rat)
------	------	-------------------

Primary irritant effect:

Effect	Species	Method
7558-80-7 sodium dihydrogenorthophosphate		
Sensitisation	OECD 429, EC B.42	none (mouse)
7778-77-0 potassium dihydrogenorthophosphate		
Irritation of skin	OECD 404	not irritating (rabbit)
Irritation of eyes	OECD 405, EC B.5	not irritating (rabbit)

7558-80-7 sodium dihydrogenorthophosphate

Sensitisation	OECD 429, EC B.42	none (mouse)
---------------	-------------------	--------------

7778-77-0 potassium dihydrogenorthophosphate

Irritation of skin	OECD 404	not irritating (rabbit)
--------------------	----------	-------------------------

Irritation of eyes	OECD 405, EC B.5	not irritating (rabbit)
--------------------	------------------	-------------------------

Additional toxicological information:

When used and handled according to specifications, the product does not have any harmful effects to our experience and the information provided to us.

Toxicokinetics, metabolism and distribution

This product dissociates into potassium and phosphate ions, which are normal body and nutritional components.

This substance is not considered to have bioaccumulative potential as it is highly soluble in water and phosphate levels in the body are regulated via homeostasis.

Repeated dose toxicity

no classification is necessary

No reliable study with this product is present.

This study is conducted on an analogous substance. (read-across)

7785-88-8 Sodium aluminum phosphate

Oral	NOAEL	>323 mg/kg bw/day (dog) (90 days, subchronic)
------	-------	---

CMR effects (carcinogenicity, mutagenicity and toxicity for reproduction)

Mutagenicity:

None

Sodium and potassium phosphates are routinely used in the nutrient broths that support bacterial colonies in the laboratory and as such bacteria are constantly exposed to these inorganic phosphates. The constant exposure of bacteria to these materials suggests that they pose no inherent risk of genotoxicity.

Carcinogenicity:

no data available

(no carcinogenicity study needs to be performed as this substance is not genotoxic)

Toxicity for reproduction:

no classification is necessary

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7758-11-4 dipotassium hydrogenorthophosphate, OECD 422 :
developmental toxicity: NOAEL> 1000 mg/kg bw/day; rat, oral
7778-77-0 potassium dihydrogenorthophosphate:
developmental / maternal toxicity: NOAEL> 282 mg/kg bw/day; rat, oral
developmental / maternal toxicity: NOAEL> 320 mg/kg bw/day; mouse, oral

12 Ecological information**12.1 Toxicity****Aquatic toxicity:**

Inorganic phosphates are not considered to be toxic to aquatic species.
No reliable study with this product is present.

This study is conducted on an analogous substance. (read-across)

6922-99-4 Tripotassium trihydrogen diphosphate dihydrate

EC50/48 h (static) >100 mg/L (Daphnia magna) (OECD 202, freshwater)

EC50/72 h (static) >100 mg/L (algae) (OECD 201, freshwater)

LC50/96 h >100 mg/L (fish Oncorhynchus mykiss) (OECD 203, freshwater, semi-static)

12.2 Persistence and degradability

The substance is inorganic; therefore no biodegradation tests are applicable.

This product dissociates into potassium and phosphate ions, which cannot be further degraded.

12.3 Bioaccumulative potential

Does not accumulate in organisms

This substance is highly water soluble and dissociating.

This product dissociates into potassium and phosphate ions, which are ubiquitous in the environment.

12.4 Mobility in soil

This substance is highly water soluble and dissociating.

Low potential for adsorption (based on substance properties).

Other information:

Product should not get in higher quantities into waste water because it may act as a plant nutrient and cause eutrophication.

Behaviour in sewage processing plants:

Type of test	Effective concentration	Method	Assessment
7758-11-4 dipotassium hydrogenorthophosphate			
EC50/3 h	>1000 mg/L	(activated sludge)	(OECD 209)
	NOEC (3 h): 1000 mg/L		



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Trade name: MKP

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13 Disposal considerations· **13.1 Waste treatment methods**· **Recommendation**

This product is used as fertiliser. However, large spills can kill vegetation. Prevent large quantities from entering waterways. If uncontaminated, sweep up or collect, and reuse as product. If contaminated with other materials, collect in suitable containers.

Can be reused without reprocessing.

Disposal must be made in accordance with Local Authority requirements.

· **Uncleaned packaging:**· **Recommendation:**

Packaging may be reused or recycled after cleaning.

Disposal must be made in accordance with Local Authority requirements.

· **Recommended cleansing agents:** Water, if necessary together with cleansing agents.

14 Transport information· **14.1 UN-Number**

None

· **14.2 UN proper shipping name**

None

· **14.3 Transport hazard class(es)**· **DOT, ADR, IMDG, IATA**· **Class**

None

· **14.4 Packing group**

None

· **14.5 Environmental hazards:**· **Marine pollutant:**

No

· **14.6 Special precautions for user**

Not applicable.

· **14.7 Transport in bulk according to Annex II of**

MARPOL73/78 and the IBC Code

None

· **Transport/Additional information:**

Not dangerous according to the above specifications.

· **UN "Model Regulation":**

None

15 Regulatory information· **15.1 Safety, health and environmental regulations/legislation specific for the substance or mixture**

Directive 2000/60/EC (phosphates)

· **Labelling according to Regulation (EC) No 1272/2008** Void

· **Hazard pictograms** Void

· **Signal word** Void

· **Hazard statements** Void

· **National regulations:**

· **Additional classification according to Decree on Hazardous Materials, Annex II:** None

· **Other regulations, limitations and prohibitive regulations**

· **Substances of very high concern (SVHC) according to REACH, Article 57** None

· **Registration status (Chemical Inventories listing) :**

United States (TSCA) : listed

Australia (AICS) : listed

Japan (ENCS) : listed

Korea (KECI) : listed

Philippines (PICCS) : listed



ROTEM AMFERT NEGEV LTD.

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Safety data sheet
according to 1907/2006/EC and 453/2010/EC

Printing date 08.09.2011

2-S00-26-010-EU version 7

Revision: 08.09.2011

Trade name: MKP

(Contd. of page 8)

China (IECSC) : listed
NTP (National Toxicology Program) : Substance is not listed
IARC (International Agency for Research on Cancer) : Substance is not listed
15.2 Chemical safety assessment: A Chemical Safety Assessment has been carried out.

16 Other information

· Department issuing MSDS: EHS UNIT in ISRAEL

· Contact:

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E-mail : lati@dsw.co.il

· Abbreviations and acronyms:

ADR: Accord européen sur le transport des marchandises dangereuses par Route (European Agreement concerning the International Carriage of Dangerous Goods by Road)

RID: Règlement international concernant le transport des marchandises dangereuses par chemin de fer (Regulations Concerning the International Transport of Dangerous Goods by Rail)

IMDG: International Maritime Code for Dangerous Goods

IATA: International Air Transport Association

ICAO: International Civil Aviation Organization

GHS: Globally Harmonized System of Classification and Labelling of Chemicals

EINECS: European Inventory of Existing Commercial Chemical Substances

CAS: Chemical Abstracts Service (division of the American Chemical Society)

DNEL: Derived No-Effect Level (REACH)

PNEC: Predicted No-Effect Concentration (REACH)

LC50: Lethal concentration, 50 percent

LD50: Lethal dose, 50 percent

NOAEL: No Observable Adverse Effect Level

NOEC: No-Observed Effect Concentration

OECD: Organisation for Economic Co-operation and Development

· Sources

REACH dossier, 2010

REACH CSR, 2010

· * Data compared to the previous version altered

Reason for revision: Compliance with Reg. 453/2010 EC, amending Reg. 1907/2006 EC.

The sections where alterations took place are marked with an asterisk in the left border

· Disclaimer

Although the information and recommendations set forth herein (hereinafter "information") are presented in good faith and believed to be correct as of the date hereof, Rotem Amfert Negev Ltd. makes no representations as to the completeness or accuracy thereof. Information is supplied upon the condition that the persons receiving same will make their own determination as to its safety and suitability for their purposes prior to use. In no event will Rotem Amfert Negev Ltd. be responsible for damages of any nature whatsoever resulting from the use of or reliance upon information.

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EU

A.5 Class F fly ash



REPORT OF FLY ASH ANALYSIS

Project Name: Bowen
Sample ID #: 130830040
Sample Date: August 2013
Silos 2,3,4 & 5

Tested By: QC
Report Date: 21-Oct-13

CHEMICAL TESTS	RESULTS	ASTM C 618 CLASS F/C	AASHTO M 295 CLASS F/C
Silicon Dioxide (SiO ₂), %	56.35		
Aluminum Oxide (Al ₂ O ₃), %	27.66		
Iron Oxide (Fe ₂ O ₃), %	6.09		
Sum of SiO ₂ , Al ₂ O ₃ , Fe ₂ O ₃ , %	90.10	70.0/50.0 min.	70.0/50.0 min.
Calcium Oxide (CaO), %	1.15		
Magnesium Oxide (MgO), %	0.62		
Sulfur Trioxide (SO ₃), %	0.13	5.0 max.	5.0 max.
Sodium Oxide (Na ₂ O), %	0.31		
Potassium Oxide (K ₂ O), %	2.33		
Total Alkalies (as Na ₂ O), %	1.84		
Available Alkalies (as Na ₂ O), %			

PHYSICAL TESTS	RESULTS	ASTM C 618 CLASS F/C	AASHTO M 295 CLASS F/C
Moisture Content, %	0.15	3.0 max.	3.0 max.
Loss on Ignition, %	3.46	6.0 max.	5.0 max.
Amount Retained on No. 325 Sieve, %	21.72	34 max.	34 max.
Specific Gravity	2.25		
Autoclave Soundness, %	-0.02	0.8 max.	0.8 max.
Strength Activity Index with Portland Cement at 7 days, % of Control	77.3	75 min.*	75 min.*
Strength Activity Index with Portland Cement at 28 days, % of Control	83.2	75 min.*	75 min.*
Water Required, % of Control	97.5	105 max.	105 max.
Loose Bulk Density, lbs/ft ³	52.3		

Meets ASTM C 618 and AASHTO M 295, FDOT Section 929, TxDOT DMS 4610, SCDOT and MDOT for class F Fly Ash

*Meeting the 7 day or 28 day strength activity index will indicate specification compliance.

Melissa Garcia

Melissa Garcia
Quality Assurance Manager

A.6 Class C fly ash



Adding Value to Energy™

Headwaters Resources certifies that pursuant to ASTM C-618-15 protocol for testing, the test data listed herein was generated by applicable ASTM methods and meets requirements of ASTM C-618 and AASHTO M-295 for Class C fly ash.

Report of Class "C" Fly Ash White Bluff Plant, Redfield, Arkansas Unit #1

DATE: May 8, 2016 LABORATORY NUMBER: UNIT #1 MARCH

<u>CHEMICAL ANALYSIS</u>		COMPOSITE DATE MARCH 2016		ASTM C-618-15 SPECIFICATIONS		AASHTO M-295-11 SPECIFICATIONS	
				CLASS C	CLASS F	CLASS C	CLASS F
Silicon Dioxide (SiO ₂)	36.95 %						
Aluminum Oxide (Al ₂ O ₃)	21.02 %						
Iron Oxide (Fe ₂ O ₃)	5.64 %						
Sum of SiO ₂ , Al ₂ O ₃ , & Fe ₂ O ₃	63.60 %			50 Min.	70 Min.	50 Min	70 Min.
Magnesium Oxide (MgO)	4.82 %						
Sulfur Trioxide (SO ₃)	1.80 %			5.0 Max.	5.0 Max.	5.0 Max.	5.0 Max.
Moisture Content	0.14 %			3.0 Max.	3.0 Max.	3.0 Max.	3.0 Max.
Loss On Ignition	0.73 %			6.0 Max.	6.0 Max.	5.0 Max.	5.0 Max.
Sodium Oxide (Na ₂ O)	1.57 %						
Potassium Oxide (K ₂ O)	0.51 %						
Total Alkali as Na ₂ O equivalent	1.90 %						
Calcium Oxide (CaO)	23.86 %						
<u>PHYSICAL ANALYSIS</u>							
Fineness: Amount retained on 325 sieve %	6.70 %			34% Max.	34% Max.	34% Max.	34% Max.
Water Requirement, % Control	94 %			105%Max	105%Max	105%Max	105%Max
Specific Gravity	2.56						
Autoclave Expansion, %	0.03 %			0.8% Max	0.8% Max	0.8% Max	0.8% Max
Strength Activity Index With Portland Cement, 7 Day	98 %			75% Min.	75% Min.	75% Min.	75% Min.

AUTHORIZED SIGNATURE:

P.O. Box 38 Thompsons, Texas 77481-0038
P: 281.343.0079 F: 281.343.0872

A.7 Boric acid



THREE ELEPHANT BORIC ACID GRANULAR TECHNICAL

Material Safety Data Sheet



PESTICIDE: EPA Registration No. **64745-3** [Not for Food or Drug
Use - - ONLY For Manufacturing Use]

Manufactured by:
Searles Valley Minerals
13200 Main Street
P.O. Box 367, Trona, CA 93592-0367

Section I - CHEMICAL PRODUCT & COMPANY IDENTIFICATION

PRODUCT NAME: Three Elephant Boric Acid Granular Technical --- Pesticide: EPA Reg # 64745-3

MANUFACTURER:
Searles Valley Minerals
P.O. Box 367
Trona, CA 93592-0367

EMERGENCY PHONE NUMBER:
24 Hour Information Service: 760-372-2291
CHEMTREC: 800-424-9300

PREPARATION/REVISION DATE: May 2, 2006
Supersedes: Apr, 12, 2004, February 20, 2002 & October 22, 1999

Section II - COMPOSITION/INFORMATION ON INGREDIENTS

NOTE: See Section 15 for Exposure Limits.

PRODUCT NAME: Boric Acid Granular Technical

FORMULA: H_2BO_3

CHEMICAL NAME: Boric Acid

SYNONYMS: OrthoBoric Acid, Boracic Acid

COMPONENTS:

Material: Boric Acid

CAS Number: 10043-35-3

Percent: 99.755% (Label Claim = 100%)

Boric Acid is hazardous under the OSHA Hazard Communication Standard based on animal chronic toxicity studies of similar organic Borates; see Section 11 for details on Toxicological Data.

Section III - HAZARDS IDENTIFICATION

EMERGENCY OVERVIEW: Boric Acid is a white odorless, powdered substance that is not flammable, combustible, or explosive, and it presents no unusual hazard if involved in a fire. Boric Acid presents little or no hazard (to humans) and has low acute oral and dermal toxicities. Care should be taken to minimize the amount of Boric Acid released to the environment to avoid ecological effects.

ROUTES OF EXPOSURE: In the occupational setting, inhalation is the most important route of exposure. Dermal absorption is usually not important because Boric Acid is not absorbed through the intact skin.

INHALATION: Mild irritation to nose and throat may occur when the PEL or TLV are exceeded (see Section 15).

EYE CONTACT: Exposure to Boric Acid dust does not cause eye irritation in normal industrial use.

DERMAL CONTACT: Boric Acid is non-irritating to the intact skin. Can be readily absorbed through broken or abraded skin.

INGESTION: Boric Acid products are not intended for ingestion. Amounts greater than one teaspoonful, when ingested, may cause gastrointestinal problems.

CANCER: Boric Acid is not considered a carcinogen.

REPRODUCTIVE: A human study of occupationally exposed Borate worker population showed no adverse reproductive effects. Animal studies of similar organic Borates demonstrated reproductive effects in males.

TARGET ORGANS: No target organs have been determined in humans. High dose animal ingestion studies indicate that the testes is the target organ.

SIGNS AND SYMPTOMS OF EXPOSURE: Symptoms of accidental over-exposure to Boric Acid have been associated with ingestion or by absorption through large areas of damaged skin. These may include nausea, vomiting, and diarrhea, with delayed effects of skin redness and peeling.

See Section 4 also. See Section 11 for details on Toxicological Data.

Section IV - EMERGENCY & FIRST AID PROCEDURES

HAZARDS TO HUMANS AND DOMESTIC ANIMALS: CAUTION Harmful if swallowed or inhaled. Causes moderate eye irritation. Avoid contact with eyes or clothing. Avoid breathing dust. Wash thoroughly with soap and water after handling. Remove contaminated clothing and wash clothing before reuse.

STATEMENT OF PRACTICAL TREATMENT: **If swallowed:** Call a physician or poison control center. Drink 1 or 2 glasses of water and induce vomiting by touching back of throat with finger [or if available, by administering syrup of ipecac]. If person is unconscious, do not give anything by mouth and do not induce vomiting. **If Inhaled:** Remove victim to fresh air. If not breathing, give artificial respiration, preferably by mouth-to-mouth. Get medical attention. **If In Eyes:** Flush eyes with plenty of water. Call a physician if irritation persists.

Section V - FIRE FIGHTING MEASURES

GENERAL HAZARD: Boric Acid is not flammable, combustible, or explosive. Boric Acid presents no unusual hazards when involved in a fire. This product is an inherent fire retardant.

UEL/LEL: Not Applicable

FLASH POINT: Not Applicable

AUTOIGNITION TEMPERATURE: Not Applicable

FLAMMABILITY CLASSIFICATION: Flammability Classification (29 CFR 1910.1200), Non-flammable solid.

EXTINGUISHING MEDIA: Any fire extinguishing media may be used on nearby fires.

Section VI - ACCIDENTAL RELEASE MEASURES

ENVIRONMENTAL HAZARD: Do not discharge effluent containing this product into lakes, streams, ponds, estuaries, oceans or other waters unless in accordance with the requirements of a National Pollutant Discharge Elimination System (NPDES) permit and the permitting authority has been notified in writing prior to discharge. Do not discharge effluent containing this product to sewer systems without previously notifying the local sewage treatment plant authority. For guidance, contact your State Water Board or Regional Office of the EPA.

Section VII - HANDLING & STORAGE

It is a violation of Federal Law to use this product in a manner inconsistent with its labeling]

CAUTION: Keep out of Reach of Children

HYGIENIC PRACTICES: Wash hands thoroughly with soap and water after handling, and before eating, drinking, or smoking.

STORAGE AND DISPOSAL: Do not contaminate water, food or feed by storage or disposal. **STORAGE:** Store in a cool, dry area away from heat. **PESTICIDE DISPOSAL:** Wastes resulting from the use of this product may be disposed of on site or at an approved waste disposal facility. **CONTAINER DISPOSAL:** Completely empty bags into application equipment. Then dispose of empty bag in a sanitary landfill or by incineration, or, if allowed by state and local authorities, by burning. If burned, stay out of smoke. Observe all Federal, state and local regulations concerning disposal of waste pesticide and containers. **FORMULATORS AND REPACKAGERS USING THIS PRODUCT ARE RESPONSIBLE FOR OBTAINING ENVIRONMENTAL PROTECTION AGENCY (EPA) REGISTRATION FOR THEIR PRODUCTS.** [Refer to PR Notice 95-1 for the applicability of the *Environmental Hazards* statement to your product.] This product is a soluble inorganic powder which may be used for the formulation of products for the following **registered end-use patterns:** 1) Algacides for water treatment in swimming pools; 2) Bacteriostats for use in impregnating or otherwise applying to absorbent material(s) to inhibit the growth of odor-causing bacteria when applied at a rate of 0.015 to 0.37% w/w (approximately) equivalent boron; 3) Insecticides for mop, spot and crack and crevice treatment in homes, residential, industrial, institutional and commercial buildings and in transportation equipment; 4) Insecticide/fungicide for wood treatment.

Section VIII - EXPOSURE CONTROLS/PERSONAL PROTECTION

ENGINEERING CONTROLS: Use local exhaust ventilation to keep airborne levels below exposure limits (see Section 15).

EYE PROTECTION: Use goggles or vented safety glasses in excessively dusty conditions.

SKIN PROTECTION: (Not required under normal conditions.) Use protection if excessively dusty or if skin is damaged.

RESPIRATORY PROTECTION: Use appropriate NIOSH/MSHA certified respirators when levels are expected to exceed exposure limits (see Section 15).

Section IX - PHYSICAL & CHEMICAL PROPERTIES

SOLUBILITY IN WATER: 4.7% at 20°C; 27.5% at 100°C

APPEARANCE: White granular or powder solid, odorless.

MOLECULAR WEIGHT: 61.83

BOILING POINT: Not Applicable

MELTING POINT: 169°C

pH VALUE: At 20°C: 7.26 (100 ppm solution)

FLASH POINT: None

SPECIFIC GRAVITY (H₂O = 1 at 4°C): 1.44

VAPOR PRESSURE: Not Applicable

BULK DENSITY: 57.0 Lbs./ CuFt.

Section X - STABILITY & REACTIVITY DATA

STABILITY: Stable under normal conditions; forms partial hydrate in moist air. When heated, water is lost forming Metaboric Acid (HBO₂). On further heating, the material is converted to boric oxide (B₂O₃).

INCOMPATIBILITY: Boric Acid reacts as a weak acid that may cause corrosion of base metals. Reaction with strong reducing agents such as metal hydrides or alkali metals will generate hydrogen gas that could create an explosive hazard.

HAZARDOUS DECOMPOSITION PRODUCTS: None known.

HAZARDOUS POLYMERIZATION: Will not occur.

Section XI - TOXICOLOGICAL EFFECTS

EYES: Boric Acid, when applied to the eyes of albino rabbits (Draize test), produced effects of mild erythema, and mild to moderate discharge in 5 of 6 rabbits. All signs subsided by the fourth day after application. Fifty years of occupational exposure history indicates no human eye injury from exposure to Boric Acid.

SKIN: Boric Acid was applied to the skin of albino rabbits. Slight to no irritation persisted 72 hours after application. No evidence of tissue damage was found. Low acute dermal toxicity; LD₅₀ for rabbits is expected to be greater than 2,000 mg/kg of body weight (test conducted per 16 CFR 1500.41). Boric Acid is not absorbed through intact skin.

INHALATION: Human epidemiological studies show no increase in pulmonary disease in occupational populations with chronic exposure to Boric Acid and Sodium Borate dust (See Section 4 also).

INGESTION: Low acute oral toxicity; LD₅₀ for Sprague-Dawley rats is 3,500 to 4,100 mg/kg of body weight. (See Section 4 also).

CARCINOGENICITY: Boric Acid is not listed as a carcinogen by the Environmental Protection Agency (EPA), the State of California, or the International Agency for Research on Cancer (IARC). A report issued by the National Toxicology Program showed "no evidence of carcinogenicity" from a full two-year bioassay on Boric Acid on mice at feed doses of 2,500 to 5,000 ppm in the diet. No mutagenic activity was observed for Boric Acid in a recent battery of four short-term mutagenicity assays.

REPRODUCTIVE: A human study of occupationally exposed Borate worker population showed no adverse reproductive effects. Animal studies indicate that Boric Acid reduces or inhibits sperm production, causes testicular atrophy, and, when given to pregnant animals during gestation, may cause developmental changes. These feed studies were conducted under chronic exposure conditions leading to doses many times in excess of those that could occur through inhalation of dust in the occupational setting.

Dietary levels of Boric Acid of 6,700 ppm in chronic feeding studies in rats and dogs produced testicular changes [Weir, Fisher, 1972]. In chronic feeding studies of mice on diets containing 5,000 ppm Boric Acid, testicular atrophy was present, while mice fed 2,500 ppm Boric Acid showed no significant increase in testicular atrophy. In another chronic Boric Acid study, degeneration of seminiferous tubules was present together with a reduction of germ cells in mice fed 4,500 ppm Boric Acid. In a reproduction study on rats, 2,000 ppm of dietary Boric Acid had no adverse effect on lactation, litter size, weight and appearance [Weir, Fisher, 1972]. In a continuous breeding study in mice, there was a reduction in fertility rates in males receiving 4,500 ppm Boric Acid, but not for females receiving 4,500 ppm Boric Acid [Fail et al., 1992].

Boric Acid at dietary levels of 1,000 ppm administered to pregnant female rats throughout gestation caused a slight reduction in fetal weight, but was considered close to NOAEL. Doses of 2,000 ppm and above caused fetal malformations and maternal toxicity. In mice, the no effect level for fetal weight reduction and maternal toxicity was 1,000 ppm Boric Acid. Fetal weight loss was noted at dietary levels of 2,000 ppm and above. Malformations (agenesis or shortening of the thirteenth rib) were seen at 4,000 ppm [Heindal et al., 1992].

Section XII - ECOLOGICAL DATA

NOTE: Boron is the element in Boric Acid that is used to characterize Borate product ecological effects. To convert Boric Acid to boron multiply by 0.1748.

FISH TOXICITY: Boron naturally occurs in seawater at an average concentration of 5 mg B/liter. In laboratory studies the acute toxicity (96-hr LC₅₀) for under-yearling Coho salmon (*Oncorhynchus kisutch*) in seawater was determined as 40 mg B/L (added as Sodium Metaborate). The Minimum Lethal Dose for minnows exposed to Boric Acid at 20°C for 6 hours is 18,000 to 19,000 mg/l in distilled water, 19,000 to 19,500 in hard water.

Rainbow trout (*S. gairdneri*)

24-day LC₅₀ = 150.0 mg/B/L

36-day NOEC-LOEC = 0.75-1 mg/B/L

Goldfish (*Carassius auratus*)

7-day NOEC-LOEC = 26.50 mg/B/L

3-day LC₅₀ = 178 mg/B/L

BIRD TOXICITY: Dietary levels of 100 mg/kg resulted in reduced growth of female mallards. As little as 30 mg/kg fed to mallard adults adversely affected the growth rate of offspring.

INVERTEBRATE TOXICITY:

Daphnids

48-hour LC₅₀ = 133 mg/B/L

21-day NOEC-LOEC = 6-13 mg/B/L

PHYTOTOXICITY: Although boron is an essential micro-nutrient for healthy growth of plants, it can be harmful to boron-sensitive plants in higher quantities. Plants and trees can easily be exposed by root absorption to toxic levels of boron in the form of water-soluble Borate leached into nearby waters or soil. Care should be taken to minimize the amount of boron released to the environment.

ENVIRONMENTAL FATE DATA:

Persistence/Degradation: Boron is naturally occurring and is commonly found in the environment. Boric Acid decomposes in the environment to natural Borate.

Soil Mobility: The product is soluble in water and is leachable through normal soil.

Section XIII - DISPOSAL CONSIDERATIONS

DISPOSAL GUIDANCE: See Section 7.

Section XIV - TRANSPORT REGULATIONS

US DEPARTMENT of TRANSPORTATION (DOT) IDENTIFICATION NUMBER: Boric Acid is not a DOT Hazardous Material or Hazardous Substance.

INTERNATIONAL TRANSPORTATION: Boric Acid has no U.N. number, and is not regulated under international rail, highway, water, or air transport regulations.

Section XV - REGULATORY INFORMATION

TSCA NUMBER: 10043-35-3

RCRA (40 CFR 261): Not listed under any section.

CERCLA (SUPERFUND): Not listed under any section.

CLEAN WATER ACT (CWA): Boric Acid is not regulated by any water quality criteria under Section 304, is not listed as priority pollutant under Section 307, and is not listed as a hazardous substance under Section 311.

SAFE DRINKING WATER ACT (SDWA): Not regulated under SDWA, 42 USC 300g-1, 40 CFR 141 et seq. Consult state and local regulations for possible water quality advisories involving boron.

OCCUPATIONAL EXPOSURE LIMITS: Boric Acid is listed/regulated by OSHA, CAL OSHA, or ACGIH as "Particulate Not Otherwise Classified" or "Nuisance Dust".

OSHA: Permissible Exposure Limit: 150mg/m³, total dust

5 mg/m³, respirable dust

ACGIH: Threshold Limit Value: 2 mg/m³

CALIFORNIA OSHA: Permissible Exposure Limit: 5 mg/m³

INTERNATIONAL AGENCY for RESEARCH on CANCER: Not listed as a carcinogen.

NTP ANNUAL REPORT ON CARCINOGENS: Not listed as a carcinogen.

OSHA CARCINOGEN: Not listed as an OSHA carcinogen.

CONEG MODEL LEGISLATION: Meets all CONEG requirements relating to heavy metal limitations on components of packaging materials.

CALIFORNIA PROPOSITION 65: Not listed as carcinogen or reproductive toxin.

FEDERAL DRUG AGENCY (FDA): Pursuant to 21 CFR 175.105, 176.180, and 181.30, Boric Acid (non-pesticide) is approved by the FDA for use in adhesive components of packaging materials, as a component of paper coatings on such materials, or for use in the manufacture thereof, which materials are expected to come in contact with dry food products.

WORKPLACE HAZARDOUS MATERIALS INFORMATION SYSTEMS (WHMIS): Boric Acid is regulated as a Controlled Product and is classified as D2A because of reproductive toxicity.

FIFRA: This product is a PESTICIDE.

Section XVI - OTHER INFORMATION

OTHER INFORMATION:

Product Label Text Hazard Information (see appropriate sections as relates to pesticide use):

- May be harmful if swallowed.
- May cause reproductive harm or birth defects based on animal data.
- Avoid contamination of food or feed.
- Not for food or drug use
- Practice good housekeeping.
- Refer to all sections of this MSDS.
- KEEP OUT OF THE REACH OF CHILDREN.

National Fire Protection Association (NFPA) Classification:

4 = Severe, 3 = Serious, 2 = Moderate, 1 = Slight, 0 = Minimal

Health	0
Flammability	0
Reactivity	0

Hazardous Materials Information Systems (HMIS):

4 = Extreme, 3 = High, 2 = Moderate, 1 = Slight, 0 = Insignificant

Blue: (Acute Health)	1*
Red: (Flammability)	0
Yellow: (Reactivity)	0

* Chronic Effects (for explanation see Section 11)

NOTICE

Judgements as to the suitability of information herein for purchaser's purposes are necessarily purchaser's responsibility. Therefore, although reasonable care has been taken in the preparation of such information, Searles Valley Minerals extends no warranties, makes no representations, and assumes no responsibility as to the accuracy or suitability of such information for application to purchaser's intended purposes or for consequences of its use.

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A.8 Concrete sand

Serial No.:	120087	TPP:		Date:	Sep-12	Tested By:	CEERD-GM-C
WIC:		District:	CMB	Contract No.:	Lab Stock		
Producer:	Green Bros			Date Recd:	Oct-12		
Sampled By:	B. Green			Matl Type:	Natural Sand		
ASTM C 136 Sieve Analysis:							
	Run 1		Cumulative Percent				
Sieve Size	Mass Ret, g	% Ret.	Ret.	Pass			Avg
3/8 in.	0.00	0.00%	0.00%	100.00%			100%
No. 4	3.80	0.84%	0.84%	99.16%			99%
No. 8	33.06	7.30%	8.14%	91.86%			92%
No. 16	38.05	8.40%	16.54%	83.46%			83%
No. 30	90.75	20.03%	36.57%	63.43%			63%
No. 50	243.74	53.81%	90.38%	9.62%			10%
No. 100	41.92	9.25%	99.63%	0.37%			0%
No. 200	1.36	0.30%	99.93%	0.07%			0%
Pan	0.31	0.07%	100.00%				
Total	452.99	100.00%					
Fineness Modulus:			2.521				2.52
ASTM C 128 Bulk Specific Gravity & Absorption:							
Flask No.			3				Avg
SSD Mass, g			500.50				
Mass Flask+Water, g			679.40				
Mass Flask+Water+Material, g			989.70				
Mass Displaced Water, g			190.20				
Water Temp C			22.5				
Relative Density (Bulk Specific Gravity)(SSD)			2.631				2.63
SSD Mass, g			500.80				
Oven Dry Mass, g			499.00				
Moisture Loss, g			1.80				
Absorption			0.36%				0.4%

A.9 Three-eighths-inch (3/8-in.) pea gravel

Serial No.:	120088	TPP:		Date:	Sep-12	Tested By:	CEERD-GM-C
WIC:		District:	CMB	Contract No.:	Lab Stock		
Producer:				Date Recd:	Oct-12		
Sampled By:	B. Green			Matl Type:	Pea Gravel		
ASTM C 136: Sieve Analysis:							
			Cumulative Percent				
Sieve Size	Mass Ret, g	% Ret.	Ret.	Pass			Avg
3 in	0.0	0.00%	0.00%	100.00%			100%
1-in	0.0	0.00%	0.00%	100.00%			100%
3/4-in	0.0	0.00%	0.00%	100.00%			100%
1/2-in	30.9	0.61%	0.61%	99.39%			99%
3/8-in	1208.4	23.76%	24.37%	75.63%			76%
No. 4	3649.5	71.77%	96.14%	3.86%			4%
No. 8	165.1	3.25%	99.39%	0.61%			1%
No. 16	3.2	0.06%	99.45%	0.55%			1%
No. 30	0.9	0.02%	99.47%	0.53%			1%
No. 50	4.0	0.08%	99.55%	0.45%			0%
Pan	23.0	0.45%	100.00%				
Total	5085.0	100.00%					
Fineness Modulus:			6.184				6.18
ASTM C 127: Bulk Specific Gravity & Absorption:							
SSD Mass in Air, g					3933.8		Avg
SSD Mass in Water, g					2366.1		
Displaced Water, g					1567.7		
Water Temp C					22.8		
Relative Density (Bulk Specific Gravity)(SSD)					2.509		2.51
Oven Dry Mass, g					3810.3		
Moisture Loss, g					123.5		
Absorption					3.24%		3.2%

A.10 Three-fourths-inch (3/4-in.) river aggregate

Capital Sand Company, Inc.													
Jefferson City, MO													
Osage 1in River Rock							Plant Location: Wardsville, Mo.						
Source of Material: Osage River													
Sieve Analysis ASTM C 136 (Percent Passing)													
													WASHED
		(25mm)	(19mm)	(12.5mm)	(9.5mm)	(4.75mm)	(2.36mm)	(1.18mm)	(0.60mm)	(0.30mm)	(0.15mm)	(.075mm)	ASTM
Date	Tester	1"	3/4"	1/2"	3/8"	#4	#8	#16	#30	#50	#100	#200	C 117
9/18/2017	KR	100	98	70	35	5	1	0.5	0.3	0.1	0.06	0	N
9/25/2017	KR	100	98	61	19	0.8	0.3	0.2	0.2	0.2	0.08	0.08	N
11/13/2017	KR	100	100	72	36	3	0.8	0.4	0.07	0	0	0	N

Appendix B: Trial Mixture Proportions at SSD for 1 Cu Yd

B.1 HB-F T1

REPORT OF CONCRETE MIXTURE PROPORTIONS									
Project: Magnesium Phosphate Concrete									
Mixture ID: 252-15 Hard F-T1									
Proportioned: 09-Sep-15									
1. MIXTURE PROPORTIONS									
Material	Aggregate, %		Solid Volume		Mass, S.S.D.		Bulk Sp. Gr.	Absorption,	
	by vol.	by wt.	ft ³	m ³	lb/yd ³	kg/m ³	S.S.D.	%	
Hard Burn			1.872	0.0693	418	248	3.58		
KDP			5.165	0.1913	419	249	1.3		
Class F Fly Ash			5.875	0.2176	836	496	2.28		
Concrete Sand	100.0	100.0	5.019	0.1859	821	487	2.62	0.50	
0	0.0	0.0		0.0000	0	0	2.76	0.65	
Batch water			8.043	0.2979	502	297.9	1.0		
Boric Acid					66	10			
Air			1.026	0.0380					
Totals:	100	100	27	1	2996	1778			
2. MIXTURE CHARACTERISTICS									
W/(C+M), by weight:		0.300		Cementitious Factor, kg/m ³ (lb/yd ³):		993		1673	
3. HARDENED PROPERTIES									
1 Day (psi)		7 Day (psi)		28 Day (psi)		Initial Set (min)		Final Set (min)	
110		620		1284		140		285	
Initial Temp (F)		Final Temp (F)		Initial Temp (F)		Final Temp (F)			
75.9		80.9		75.9		80.9			
4. MATERIALS:									
Magnesium Oxide: Marten Marietta MagChem 10 Hard Burned									
Potassium Phosphate: Peak MonoPotassium Phosphate									
Fly Ash: A.W. Cook Class F from Georgia									
FINE AGGREGATE: Concrete Sand from Green Bros. Redwood, MS									
ADMIXTURES: 3 Elephants Boric Acid									

B.2 HB-F T2

REPORT OF CONCRETE MIXTURE PROPORTIONS										
Project: Magnesium Phosphate Concrete										
Mixture ID: 252-15 Hard F-T2										
Proportioned: 09-Sep-15										
1. MIXTURE PROPORTIONS										
			Aggregate, %		Solid Volume		Mass, S.S.D.		Bulk Sp. Gr.	Absorption,
	Material		by vol.	by wt.	ft ³	m ³	lb/yd ³	kg/m ³	S.S.D.	%
	Hard Burned				1.395	0.0517	312	185	3.58	
	KDP				6.179	0.2288	501	298	1.3	
	Class F Fly Ash				5.714	0.2116	813	483	2.28	
	Concrete Sand	100.0	100.0		4.870	0.1804	796	473	2.62	0.50
	0	0.0	0.0		0.0000	0	0	0	2.76	0.65
	Batch water				7.817	0.2895	488	289.5	1.0	
	Borax						65	9		
	Air				1.026	0.0380				
	Totals:	100	100		27	1	2910	1727		
2. MIXTURE CHARACTERISTICS										
W/(C+M), by weight:			0.300		Cementitious Factor, kg/m ³ (lb/yd ³):				965	1626
3. HARDENED PROPERTIES										
		1 Day (psi)	7 Day (psi)	28 Day (psi)	initial Set (min)	Final Set (min)	Initial Temp (F)	Final Temp (F)		
		214	700	1795	255	275	74.9	88.3		
4. MATERIALS:										
Magnesium Oxide: Marten Marrietta MagChem 10 Hard Burned										
Potassium Phosphate: Peak MonoPotassium Phosphate										
Fly Ash: A.W. Cook Class F from Georgia										
FINE AGGREGATE: Concrete Sand from Green Bros. Redwood, MS										
ADMIXTURES: 3 Elephants Boric Acid										

B.3 HB-F T3

REPORT OF CONCRETE MIXTURE PROPORTIONS									
Project: Magnesium Phosphate Concrete									
Mixture ID: 252-15 Hard F-T3									
Proportioned: 09-Sep-15									
1. MIXTURE PROPORTIONS									
Material	Aggregate, %		Solid Volume		Mass, S.S.D.		Bulk Sp. Gr.	Absorption,	
	by vol.	by wt.	ft ³	m ³	lb/yd ³	kg/m ³	S.S.D.	%	
Hard Burn			2.044	0.0757	457	271	3.58		
KDP			4.791	0.1774	389	231	1.3		
Class F Fly Ash			5.941	0.2200	845	502	2.28		
Concrete Sand	100.0	100.0	5.072	0.1878	829	492	2.62	0.50	
0	0.0	0.0		0.0000	0	0	2.76	0.65	
Batch water			8.127	0.3010	507	301	1.0		
Borax					67	10			
Air			1.026	0.0380					
Totals:	100	100	27	1	3027	1796			
2. MIXTURE CHARACTERISTICS									
W/(C+M), by weight:			0.300		Cementitious Factor, kg/m ³ (lb/yd ³):			1003	1691
3. HARDENED PROPERTIES									
	1 Day (psi)	7 Day (psi)	28 Day (psi)	Initial Set (min)	Final Set (min)	Initial Temp (F)	Final Temp (F)		
	93	460	841	105	200	76.8	80.4		
4. MATERIALS:									
Magnesium Oxide: Marten Marietta MagChem 10 Hard Burned									
Potassium Phosphate: Peak MonoPotassium Phosphate									
Fly Ash: A.W. Cook Class F from Georgia									
FINE AGGREGATE: Concrete Sand from Green Bros. Redwood, MS									
ADMIXTURES: 3 Elephants Boric Acid									

B.4 DB-C T1

REPORT OF CONCRETE MIXTURE PROPORTIONS									
Project: Magnesium Phosphate Concrete									
Mixture ID: 257-15 Dead C-T1									
Proportioned: 14-Sep-15									
1. MIXTURE PROPORTIONS									
Material	Aggregate, %		Solid Volume		Mass, S.S.D.		Bulk Sp. Gr.	Absorption,	
	by vol.	by wt.	ft ³	m ³	lb/yd ³	kg/m ³	S.S.D.	%	
Dead Burn			1.872	0.0693	418	248	3.58		
KDP			5.165	0.1913	419	249	1.3		
Class C Fly Ash			5.875	0.2176	836	496	2.28		
Concrete Sand	100.0	100.0	5.019	0.1859	821	487	2.62	0.50	
0	0.0	0.0		0.0000	0	0	2.76	0.65	
Batch water			8.043	0.2979	502	297.9	1.0		
Boric Acid					66	10			
Air			1.026	0.0380					
Totals:	100	100	27	1	2996	1778			
2. MIXTURE CHARACTERISTICS									
W/(C+M), by weight:		0.300	Cementitious Factor, kg/m ³ (lb/yd ³):				993	1673	
3. HARDENED PROPERTIES									
1 Day (psi)		7 Day (psi)		28 Day (psi)		Initial Set (min)		Final Set (min)	
Initial Temp (F)		Final Temp (F)							
Hardened Properties not measured due to aggregate fallout.									
4. MATERIALS:									
Magnesium Oxide: Marten Marietta P98 Pulverized									
Potassium Phosphate: Peak MonoPotassium Phosphate									
Fly Ash: Headwaters Class C from Redfield, AR									
FINE AGGREGATE: Concrete Sand from Green Bros. Redwood, MS									
ADMIXTURES: 3 Elephants Boric Acid									

B.5 DB-C T2

REPORT OF CONCRETE MIXTURE PROPORTIONS									
Project: Magnesium Phosphate Concrete									
Mixture ID: 257-15 Dead C-T2									
Proportioned: 14-Sep-15									
1. MIXTURE PROPORTIONS									
Material	Aggregate, %		Solid Volume		Mass, S.S.D.		Bulk Sp. Gr.	Absorption,	
	by vol.	by wt.	ft ³	m ³	lb/yd ³	kg/m ³	S.S.D.	%	
Dead Burned			1.395	0.0517	312	185	3.58		
KDP			6.179	0.2288	501	298	1.3		
Class C Fly Ash			5.714	0.2116	813	483	2.28		
Concrete Sand	100.0	100.0	4.870	0.1804	796	473	2.62	0.50	
0	0.0	0.0		0.0000	0	0	2.76	0.65	
Batch water			7.817	0.2895	488	289.5	1.0		
Borax					65	9			
Air			1.026	0.0380					
Totals:	100	100	27	1	2910	1727			
2. MIXTURE CHARACTERISTICS									
W/(C+M), by weight:		0.300	Cementitious Factor, kg/m ³ (lb/yd ³):			965	1626		
3. HARDENED PROPERTIES									
1 Day (psi)	7 Day (psi)	28 Day (psi)	Initial Set (min)	Final Set (min)	Initial Temp (F)	Final Temp (F)			
Hardened Properties not measured due to aggregate fallout, w/c too high									
4. MATERIALS:									
Magnesium Oxide: Marten Marietta P98 Pulverized									
Potassium Phosphate: Peak MonoPotassium Phosphate									
Fly Ash: Headwaters Class C from Redfield, AR									
FINE AGGREGATE: Concrete Sand from Green Bros. Redwood, MS									
ADMIXTURES: 3 Elephants Boric Acid									

B.6 DB-C T3

REPORT OF CONCRETE MIXTURE PROPORTIONS									
Project: Magnesium Phosphate Concrete									
Mixture ID: 257-15 Dead C-T3									
Proportioned: 14-Sep-15									
1. MIXTURE PROPORTIONS									
Material	Aggregate, %		Solid Volume		Mass, S.S.D.		Bulk Sp. Gr.	Absorption,	
	by vol.	by wt.	ft ³	m ³	lb/yd ³	kg/m ³	S.S.D.	%	
Dead Brun			2.044	0.0757	457	271	3.58		
KDP			4.791	0.1774	389	231	1.3		
Class C Fly Ash			5.941	0.2200	845	502	2.28		
Concrete Sand	100.0	100.0	5.072	0.1878	829	492	2.62	0.50	
0	0.0	0.0		0.0000	0	0	2.76	0.65	
Batch water			8.127	0.3010	507	301	1.0		
Borax					67	10			
Air			1.026	0.0380					
Totals:	100	100	27	1	3027	1796			
2. MIXTURE CHARACTERISTICS									
W/(C+M), by weight:			0.300		Cementitious Factor, kg/m ³ (lb/yd ³):			1003	1691
3. HARDENED PROPERTIES									
1 Day (psi)		7 Day (psi)	28 Day (psi)	Initial Set (min)	Final Set (min)	Initial Temp (F)	Final Temp (F)		
Hardened Properties not measured due to aggregate fallout. w/c too high									
4. MATERIALS:									
Magnesium Oxide: Marten Marietta P98 Pulverized									
Potassium Phosphate: Peak MonoPotassium Phosphate									
Fly Ash: Headwaters Class C from Redfield, AR									
FINE AGGREGATE: Concrete Sand from Green Bros. Redwood, MS									
ADMIXTURES: 3 Elephants Boric Acid									

B.7 DB-C T4

REPORT OF CONCRETE MIXTURE PROPORTIONS								
Project: Magnesium Phosphate Concrete								
Mixture ID: 287-15 Dead C-T4								
Proportioned: 14-Oct-15								
1. MIXTURE PROPORTIONS								
Material	Aggregate, %		Solid Volume		Mass, S.S.D.		Bulk Sp. Gr.	Absorption,
	by vol.	by wt.	ft ³	m ³	lb/yd ³	kg/m ³	S.S.D.	%
Dead Burn			1.706	0.0632	381	226	3.58	
KDP			4.707	0.1743	382	227	1.3	
Class F Fly Ash			5.354	0.1983	762	452	2.28	
Concrete Sand	100.0	100.0	9.320	0.3452	1524	904	2.62	0.50
0	0.0	0.0		0.0000	0	0	2.76	0.65
Batch water			4.887	0.1810	305	181	1.0	
Boric Acid					61	9		
Air			1.026	0.0380				
Totals:	100	100	27	1	3354	1990		
2. MIXTURE CHARACTERISTICS								
W/(C+M), by weight:		0.200		Cementitious Factor, kg/m ³ (lb/yd ³):			905	1525
3. HARDENED PROPERTIES								
	1 Day (psi)	7 Day (psi)	28 Day (psi)	Initial Set (min)	Final Set (min)	Initial Temp (F)	Final Temp (F)	
	535	3753	4430	35	110	70.8	80.9	
4. MATERIALS:								
Magnesium Oxide: Marten Marietta P98 Pulverized								
Potassium Phosphate: Peak MonoPotassium Phosphate								
Fly Ash: Headwaters Class C from Redfield, AR								
FINE AGGREGATE: Concrete Sand from Green Bros. Redwood, MS								
ADMIXTURES: 3 Elephants Boric Acid								

B.8 DB-C T5

REPORT OF CONCRETE MIXTURE PROPORTIONS									
Project: Magnesium Phosphate Concrete									
Mixture ID: 287-15 Dead C-T5									
Proportioned: 14-Oct-15									
1. MIXTURE PROPORTIONS									
Material	Aggregate, %		Solid Volume		Mass, S.S.D.		Bulk Sp. Gr.	Absorption,	
	by vol.	by wt.	ft ³	m ³	lb/yd ³	kg/m ³	S.S.D.	%	
Dead Burn			1.274	0.0472	285	169	3.58		
KDP			5.640	0.2089	458	272	1.3		
Class F Fly Ash			5.216	0.1932	743	441	2.28		
Concrete Sand	100.0	100.0	9.086	0.3365	1486	882	2.62	0.50	
0	0.0	0.0		0.0000	0	0	2.76	0.65	
Batch water			4.887	0.1810	297	176	1.0		
Boric Acid					61	11.0			
Air			1.026	0.0380					
Totals:	100	100	27	1.0048	3269	1940			
2. MIXTURE CHARACTERISTICS									
W/(C+M), by weight:		0.200		Cementitious Factor, kg/m ³ (lb/yd ³):		882		1486	
3. HARDENED PROPERTIES									
1 Day (psi)		7 Day (psi)		28 Day (psi)		Initial Set (min)		Final Set (min)	
775		3397		4840		60		150	
Initial Temp (F)		Final Temp (F)		Initial Temp (F)		Final Temp (F)			
75		84.2		75		84.2			
4. MATERIALS:									
Magnesium Oxide: Marten Marietta P98 Pulverized									
Potassium Phosphate: Peak MonoPotassium Phosphate									
Fly Ash: Headwaters Class C from Redfield, AR									
FINE AGGREGATE: Concrete Sand from Green Bros. Redwood, MS									
ADMIXTURES: 3 Elephants Boric Acid									

B.9 DB-C T6

REPORT OF CONCRETE MIXTURE PROPORTIONS									
Project: Magnesium Phosphate Concrete									
Mixture ID: 287-15 Dead C-T6									
Proportioned: 14-Oct-15									
1. MIXTURE PROPORTIONS									
Material	Aggregate, %		Solid Volume		Mass, S.S.D.		Bulk Sp. Gr.	Absorption,	
	by vol.	by wt.	ft ³	m ³	lb/yd ³	kg/m ³	S.S.D.	%	
Dead Brun			1.862	0.0690	416	247	3.58		
KDP			4.364	0.1616	354	210	1.3		
Class C Fly Ash			5.412	0.2004	770	457	2.28		
Concrete Sand	100.0	100.0	9.401	0.3482	1537	912	2.62	0.50	
0	0.0	0.0		0.0000	0	0	2.76	0.65	
Batch water			4.936	0.1828	308	182.8	1.0		
Borax					61	9			
Air			1.026	0.0380					
Totals:	100	100	27	1	3385	2009			
2. MIXTURE CHARACTERISTICS									
W/(C+M), by weight:			0.200		Cementitious Factor, kg/m ³ (lb/yd ³):			914	1540
3. HARDENED PROPERTIES									
1 Day (psi)		7 Day (psi)	28 Day (psi)	Initial Set (min)	Final Set (min)	Initial Temp (F)	Final Temp (F)		
585		2822	4486	40	90	75	82.5		
4. MATERIALS:									
Magnesium Oxide: Marten Marietta P98 Pulverized									
Potassium Phosphate: Peak MonoPotassium Phosphate									
Fly Ash: Headwaters Class C from Redfield, AR									
FINE AGGREGATE: Concrete Sand from Green Bros. Redwood, MS									
ADMIXTURES: 3 Elephants Boric Acid									

B.10 DB-C T7

REPORT OF CONCRETE MIXTURE PROPORTIONS									
Project: Magnesium Phosphate Concrete									
Mixture ID: 021-16 Dead C-T7									
Proportioned: 21-Jan-16									
1. MIXTURE PROPORTIONS									
Material	Aggregate, %		Solid Volume		Mass, S.S.D.		Bulk Sp. Gr.	Absorption,	
	by vol.	by wt.	ft ³	m ³	lb/yd ³	kg/m ³	S.S.D.	%	
Dead Burned KDP			1.274	0.0472	285	169	3.58		
Class C Fly Ash			5.641	0.2089	458	272	1.3		
Concrete Sand	100.0	100.0	9.085	0.3365	1486	882	2.62	0.50	
0	0.0	0.0		0.0000	0	0	2.76	0.65	
Batch water			4.757	0.1762	297	176.2	1.0		
Borax					59	8			
Air			1.026	0.0380					
Totals:	100	100	27	1	3267	1939			
2. MIXTURE CHARACTERISTICS									
W/(C+M), by weight:		0.200		Cementitious Factor, kg/m ³ (lb/yd ³):			881	1484	
3. HARDENED PROPERTIES									
1 Day (psi)	7 Day (psi)	28 Day (psi)	Initial Set (min)	Final Set (min)	Initial Temp (F)	Final Temp (F)			
1100	5596	7037	TOS was not tested		68.7	Not Measured			
4. MATERIALS:									
Magnesium Oxide: Marten Marrietta P98 Pulverized									
Potassium Phosphate: Peak MonoPotassium Phosphate									
Fly Ash: Headwaters Class C from Redfield, AR									
FINE AGGREGATE: Concrete Sand from Green Bros. Redwood, MS									
ADMIXTURES: 3 Elephants Boric Acid									

B.11 DB-C T8

REPORT OF CONCRETE MIXTURE PROPORTIONS									
Project: Magnesium Phosphate Concrete									
Mixture ID: 021-16 Dead C-T8									
Proportioned: 21-Jan-16									
1. MIXTURE PROPORTIONS									
Material	Aggregate, %		Solid Volume		Mass, S.S.D.		Bulk Sp. Gr.	Absorption,	
	by vol.	by wt.	ft ³	m ³	lb/yd ³	kg/m ³	S.S.D.	%	
Dead Burned			1.274	0.0472	285	169	3.58		
KDP			5.641	0.2089	458	272	1.3		
Class C Fly Ash			5.216	0.1932	742	441	2.28		
Concrete Sand	50.0	48.7	4.543	0.1682	743	441	2.62	0.50	
Pea Gravel	50.0	51.3		0.1682	782	464	2.76	0.65	
Batch water			4.757	0.1762	297	176.2	1.0		
Borax					59	8			
Air			1.026	0.0380					
Totals:	50	49	22	1	3307	1962			
2. MIXTURE CHARACTERISTICS									
W/(C+M), by weight:		0.200		Cementitious Factor, kg/m ³ (lb/yd ³):			881	1484	
3. HARDENED PROPERTIES									
	1 Day (psi)	7 Day (psi)	28 Day (psi)	Initial Set (min)	Final Set (min)	Initial Temp (F)	Final Temp (F)		
	1127	4683	5498						
4. MATERIALS:									
Magnesium Oxide: Marten Marietta P98 Pulverized									
Potassium Phosphate: Peak MonoPotassium Phosphate									
Fly Ash: Headwaters Class C from Redfield, AR									
FINE AGGREGATE: Concrete Sand from Green Bros. Redwood, MS									
ADMIXTURES: 3 Elephants Boric Acid									

B.12 DB-C T9

REPORT OF CONCRETE MIXTURE PROPORTIONS									
Project: Magnesium Phosphate Concrete									
Mixture ID: 021-16 Dead C-T9									
Proportioned: 21-Jan-16									
1. MIXTURE PROPORTIONS									
Material	Aggregate, %		Solid Volume		Mass, S.S.D.		Bulk Sp. Gr.	Absorption,	
	by vol.	by wt.	ft ³	m ³	lb/yd ³	kg/m ³	S.S.D.	%	
Dead Burned KDP			1.157	0.0428	258	153	3.58		
Class C Fly Ash			4.737	0.1754	674	400	2.28		
Concrete Sand	50.0	48.7	5.319	0.1970	870	516	2.62	0.50	
Pea Gravel	50.0	51.3		0.1970	916	544	2.76	0.65	
Batch water			4.320	0.1600	270	160	1.0		
Borax					53	8			
Air			1.026	0.0380					
Totals:	50	49	22	1	3403	2020			
2. MIXTURE CHARACTERISTICS									
W/(C+M), by weight:		0.200	Cementitious Factor, kg/m ³ (lb/yd ³):			800	1348		
3. HARDENED PROPERTIES									
1 Day (psi)	7 Day (psi)	28 Day (psi)	Initial Set (min)	Final Set (min)	Initial Temp (F)	Final Temp (F)			
Mixture was never batched.									
4. MATERIALS:									
Magnesium Oxide: Marten Marrietta P98 Pulverized									
Potassium Phosphate: Peak MonoPotassium Phosphate									
Fly Ash: Headwaters Class C from Redfield, AR									
FINE AGGREGATE: Concrete Sand from Green Bros. Redwood, MS									
ADMIXTURES: 3 Elephants Boric Acid									

REPORT OF CONCRETE MIXTURE PROPORTIONS									
Project: Magnesium Phosphate Concrete									
Mixture ID: 021-16 Dead C-T10									
Proportioned: 21-Jan-16									
1. MIXTURE PROPORTIONS									
Material	Aggregate, %		Solid Volume		Mass, S.S.D.		Bulk Sp. Gr	Absorption,	
	by vol.	by wt.	ft ³	m ³	lb/yd ³	kg/m ³	S.S.D.	%	
Dead Burned KDP			1.497	0.0555	335	199	3.58		
Class C Fly Ash			4.266	0.1580	607	360	2.28		
Concrete Sand	50.0	48.7	5.440	0.2015	890	528	2.62	0.50	
Pea Gravel	50.0	51.3		0.2015	937	556	2.76	0.65	
Batch water			4.320	0.1600	270	160	1.0		
Borax					53	8			
Air			1.026	0.0380					
Totals:	50	49	22	1	3444	2044			
2. MIXTURE CHARACTERISTICS									
W/(C+M), by weight:		0.200		Cementitious Factor, kg/m ³ (lb/yd ³):			800	1348	
3. HARDENED PROPERTIES									
1 Day (psi)	7 Day (psi)	28 Day (psi)	Initial Set (min)	Final Set (min)	Initial Temp (F)	Final Temp (F)			
Mixture was never batched.									
4. MATERIALS:									
Magnesium Oxide: Marten Marrietta P98 Pulverized									
Potassium Phosphate: Peak MonoPotassium Phosphate									
Fly Ash: Headwaters Class C from Redfield, AR									
FINE AGGREGATE: Concrete Sand from Green Bros. Redwood, MS									
ADMIXTURES: 3 Elephants Boric Acid									

Appendix C: Summary of Trial Batch Results

Mix ID	Batch Volume ft ³	Consolidation Method	M/P MgO to Phosphate molar ratio	W/B Water to Binder mass ratio	A/B Aggregate to Binder mass ratio	FA/CA Fine to Coarse Aggregate mass ratio	Fly Ash % binder mass	Boric Acid % binder mass	Time of Setting (min)		Max Temp °F	Compressive Strength (psi)			Specimen Size (inch)
									Initial	Final		1 day	7 day	28 day	
LB-C T1	0.14	Tamping	3.4/1	0.36	0	1/0	0	0	<1	<2	180	-	-	-	2 x 2
LB-C T2	0.14	Tamping	3.4/1	0.36	0	1/0	50	4	8	13	130	500	1145	1450	2 x 2
LB-C T3	0.25	Tamping	2/1	0.30	0	1/0	50	4	21	24	120	1075	1940	1865	2 x 2
LB-C T4	0.25	Tamping	4/1	0.30	0	1/0	50	4	26	35	170	840	1570	1910	2 x 2
LB-C T5	0.25	Tamping	3.4/1	0.30	2/1	1/0	50	4	22	27	160	1210	2055	1980	2 x 2
LB-C T6	0.25	Tamping	2/1	0.30	2/1	1/0	50	4	24	27	110	1400	2610	2700	2 x 2
LB-C T7	0.25	Tamping	4/1	0.30	2/1	1/0	50	4	14	23	140	1235	1420	1380	2 x 2
LB-C T8	0.25	Vibration	4/1	0.30	0	1/0	50	4	N.T.	N.T.	N.T.	1540	2360	3160	2 x 2
LB-C T9	0.25	Vibration	3.4/1	0.30	2/1	1/0	50	4	N.T.	N.T.	N.T.	1950	3290	3615	2 x 2
HB-F T1	0.25	Vibration	3.4/1	0.30	2/1	1/0	50	4	140	285	80.9	110	620	1284	2 x 2
HB-F T2	0.25	Vibration	2/1	0.30	2/1	1/0	50	4	100	170	86.1	215	700	1795	2 x 2
HB-F T3	0.25	Vibration	4/1	0.30	2/1	1/0	50	4	150	180	98.6	95	460	840	2 x 2
DB-F T1	0.25	Vibration	3.4/1	0.30	2/1	1/0	50	4	N.T.	N.T.	101.6	960	1200	1660	2 x 2
DB-F T2	0.25	Vibration	2/1	0.30	2/1	1/0	50	4	100	170	86.1	160	1620	2040	2 x 2
DB-F T3	0.25	Vibration	4/1	0.30	2/1	1/0	50	4	150	180	98.6	1030	1300	1480	2 x 2
DB-F T4	0.25	Vibration	3.4/1	0.25	1/1	1/0	50	4	60	150	86.3	300	1420	2100	2 x 2
DB-F T5	0.25	Vibration	2/1	0.25	1/1	1/0	50	4	80	200	87.7	430	2375	3160	2 x 2
DB-F T6	0.25	Vibration	4/1	0.25	1/1	1/0	50	4	60	160	86.2	265	1070	2010	2 x 2
DB-C T4	0.25	Vibration	3.4/1	0.2	1/1	1/0	50	4	35	110	80.9	535	3755	4430	2 x 2
DB-C T5	0.25	Vibration	2/1	0.2	1/1	1/0	50	4	60	150	84.2	775	3400	4840	2 x 2
DB-C T6	0.25	Vibration	4/1	0.2	1/1	1/0	50	4	40	90	82.5	585	2820	3490	2 x 2
DB-C T7	0.75	Vibration	2/1	0.2	1/1	1/0	50	4	35	65	86	1100	5600	7040	3 x 6
DB-C T8	0.75	Vibration	2/1	0.2	*1/1	1/1	50	4	60	90	76.1	1130	4685	5500	3 x 6

*50% CA

Unit Conversion Factors

Multiply	By	To Obtain
cubic feet	0.02831685	cubic meters
cubic inches	1.6387064 E-05	cubic meters
cubic yards	0.7645549	cubic meters
degrees Fahrenheit	(F-32)/1.8	degrees Celsius
feet	0.3048	meters
gallons (U.S. liquid)	3.785412 E-03	cubic meters
inches	0.0254	meters
ounces (mass)	0.02834952	kilograms
ounces (U.S. fluid)	2.957353 E-05	cubic meters
pounds (force)	4.448222	newtons
pounds (force) per square foot	47.88026	pascals
pounds (force) per square inch	6.894757	kilopascals
pounds (mass)	0.45359237	kilograms
pounds (mass) per cubic foot	16.01846	kilograms per cubic meter
pounds (mass) per cubic inch	2.757990 E+04	kilograms per cubic meter
tons (2,000 pounds, mass)	907.1847	kilograms
tons (2,000 pounds, mass) per square foot	9,764.856	kilograms per square meter
yards	0.9144	meters

REPORT DOCUMENTATION PAGE

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14. ABSTRACT Magnesium phosphate cements (MPCs) have been used in proprietary products for pavement repairs for over 30 years. However, these products generally are intended for small repair sections less than 0.5 ft ³ due to high heat generations and short working times. The objective of this research was to develop optimal mixture proportions of MPCs for use in pavement applications. Materials used in this research included three types of magnesium oxide (MgO), one phosphate salt, two types of fly ash, a retarder, two course aggregate gradations, and one sand. From these materials, 24 mixture proportions were batched with variations including water content, retarder, and fly ash to determine the effects on physical and mechanical properties. Laboratory testing at various scales was conducted on a mixture consisting of a dead-burned MgO, potassium dihydrogen phosphate, Class C fly ash, boric acid, and local aggregates. Four variations were developed and commercially blended in bulk super sacks. These concrete mixtures met the workability, setting time, and strength requirements. A volumetric mixer was used to batch 2 yd ³ field placements in test sections 8.5 ft wide x 8.5 ft long x 8 in. deep. This was noteworthy because no literature was discovered for MPC concrete placements at this large volume.					
15. SUBJECT TERMS Magnesium phosphate cement Mixture proportioning Runways (Aeronautics)—Maintenance and repair		Strength properties Setting time Repair of concrete Military bases		Concrete—Mixing Magnesium phosphate Cement—Mixing-	
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