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Investigation into the Effect of Mixture Composition on Setting Time of Magnesium Phosphate Cement

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Investigation into the Effect of Mixture Composition on Setting Time of Magnesium Phosphate Cement

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BACKGROUND

The purpose of this paper is to examine the impacts of different mixture compositions on the initial setting time of magnesium phosphate cement (MPC). MPC is a type of cementitious material that is formed through an acid-base chemical reaction between dead burnt magnesia (MgO) and phosphate (PO_4^{3-}) [1]. It exhibits many beneficial properties such as rapid setting time, high early strength, chemical resistance, and ability to cure at low temperatures that give it high potential for use in rapid repairs in the construction industry. Conventionally, it would be formed using ammonium dihydrogen phosphate as the source of phosphate for the reaction, but this leads to the release of some gaseous ammonia. [2] This could lead to potential adverse health effects of workers, as well as corrosion of nearby metals. Potassium dihydrogen phosphate (KDP) is often used as an alternative. The combination of magnesia and KDP within water leads to the formation of k-struvite ($MgKPO_4 \cdot 6H_2O$) crystals, which gives MPC its strength and structure. [1] In addition to its use in the construction industry, MPC is also useful in the medical field. Due to its high strength and nontoxicity, it may be used with certain admixtures as a bone cement. [3]

While the rapid setting time of MPC without any admixtures has advantages in certain applications such as dentistry and orthopedics, it makes its use in large scale construction limited. It simply sets too quickly to be cast at a large scale. Admixtures such as boric acid (H_3BO_3) [4] have been investigated for use as a set retarder in MPC, and sugars like dextrose have been used as a set retarder in conventional portland cement. [6] These help to slow the rapid cure time of the MPC to allow it to be more easily cast.

The reaction that results in the formation of k-struvite from magnesia and KDP is highly exothermic, which is why MPC is able to cure at such low external temperatures. When the reaction is slowed down with the addition of set retarders or alterations to the mixture composition, less heat is produced. When using MPC in large quantities, limiting the heat of hydration is essential.

Additionally, the high cost of MPC compared to portland cement makes it prohibitively expensive in many applications. Fly ash, a fine dust byproduct from the combustion of coal, is a relatively inexpensive admixture that may be used as a filler in the MPC mixture, providing additional volume without greatly impacting the mechanical properties of the MPC. [2] Also, the addition of fly ash, a byproduct of the coal industry, results in a lower carbon footprint than MPC without fly ash.

Another consideration in the development of a mix design for MPC is the ratio of magnesium to phosphate (M/P). In general, a low proportion of magnesium has been linked to suboptimal overall properties. [7] Though the mechanical properties may be worse, the setting time of the MPC is also delayed. In contrast, high M/P ratios are linked with very rapid and imperfect formation of the crystalline structures, which can also have detrimental effects on mechanical properties.

MATERIALS AND METHODS

Materials

Materials used, shown in Figure 1 and Figure 2, include dead burnt magnesia (MgO), potassium dihydrogen phosphate (KDP), Class F fly ash (FFA), boric acid, dextrose, and water. Dead burnt magnesium oxide, in contrast to light burnt and hard burnt magnesium oxide, has been passed through a dead burning kiln at up to 3100 degrees Fahrenheit, producing a high quality and chemically reactive magnesia [8]. The phosphate source chosen, KDP, was selected for its superior qualities over other phosphate sources. Ammonium phosphate, which has previously been used in MPC, has a pungent odor, and corrodes nearby metals [2]. Class F fly ash, characterized by its low calcium content, was used as a filler, providing additional volume to the paste. Fly ash is often used in PC concrete to lower the overall cost without sacrificing mechanical performance. Unlike Class C fly ash, which has a higher calcium content, FFA does not possess cementitious properties of its own [9], but it is counted among the cementitious materials for the purpose of mixture proportioning. These cementitious materials can be seen in Figure 1. Boric acid (H_3BO_3) is a commonly used set retarder in MPC. When used alone in MPC, it has been found to delay setting time and "reduce the intensity of exothermic reactions during the initial setting and hardening stages," [4]. Some sugars such as sucrose, a disaccharide consisting of equal parts of glucose and fructose, have been investigated for use as a set retarder in PC concrete [5]. Dextrose, which is chemically identical to glucose, may also be an effective set retarder. In this study, it is used in conjunction with boric acid to delay the setting time of the MPC. Some additional testing was performed using boric acid or dextrose on their own in the mixtures with water/cement ratios of 0.20 and 0.22 and M/P ratios of 4, but since the results were very inconsistent and limited, additional testing should be performed prior to drawing conclusions about these mixtures. The set retarders used can be seen in Figure 2. The water used was potable tap water. All ingredients were stored inside of climate-controlled areas at the CEREC to keep them at a consistent temperature for the purpose of testing.

Figure 1 Cementitious Materials used in MPC.

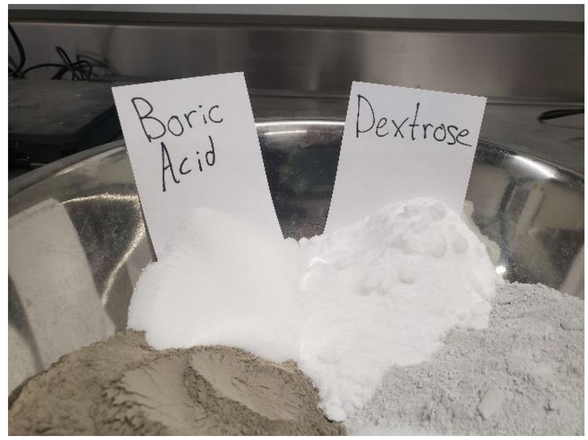

Figure 2 Set Retarders used in MPC.

Methods

Many MPC mixture compositions were developed for the purpose of this study. Each specimen tested was named with a naming convention consistent with the proportions of the constituents. The proportions of the ingredients examined fall generally within the range that has been previously studied in contexts outside of setting time. The ratios examined in this study can be found in Table 1 below.

Table 1 Values for Calculating Proportions of Constituents of MPC Mixtures

Set Retarder Dosages	M/P	Corresponding MgO/KDP mass ratio	FA Dosages	Water/Cement Ratios
4%	4.00	1.18	20%	0.16
6%	6.00	1.78	40%	0.18
	8.00	2.37	60%	0.20
				0.22

Each sample was named with a convention indicating its mix design. First, the M/P ratio is indicated, then the set retarder dosage, then the w/c ratio, then finally the fly ash replacement rate. For example, the sample called M8-BD6-0.20-FFA40 has a M/P ratio of 8, a set retarder dosage of 6%, a water/cement ratio of 0.20, and a fly ash replacement rate of 40%.

The ratios of magnesium to phosphate were initially listed as a molar ratio, and then converted to a mass ratio for the purpose of determining the weights of the magnesia and KDP required. First, an initial mass of KDP required was determined, then it was multiplied by the mass M/P ratio to determine how much magnesia was required. Next, these were added together and multiplied by the percentage of FFA desired.

A 1:3 mass ratio of boric acid to dextrose was examined, so the dosage of set retarder was made of 25% boric acid, and 75% dextrose. To determine the total mass of set retarder required, the sum of magnesia, KDP, and FFA was multiplied by the desired set retarder dosage. Then, to determine the masses of each constituent of the set retarder, the mass of set retarder required was multiplied by 25% and 75% for boric acid and dextrose respectively. Finally, to determine the water quantity required for each mixture, the sum of magnesia, KDP, and FFA was simply multiplied by the water/cement ratio desired. It is worth noting that the w/c ratios examined are significantly lower than what is required for PC concrete. A summary of the process of calculating mixture proportions can be found below in Table 2.

Table 2 Calculations of Masses of MPC Constituents

Formulas for Developing Mixture Composition						
	Dosages of Cementitious Material, Set Retarder, and Water (grams)					
Name of Mix	**MgO**	**KDP**	**FA**	**Boric Acid**	**Dextrose**	**Water**
	= KDP * MgO/KDP mass ratio	KDP - input	= (MgO + KDP) * Fly Ash Dose	= (MgO + KDP + FA) * Retarder Dose * 0.25	= (MgO + KDP + FA) * Retarder Dose * 0.75	= (MgO + KDP + FA) * W/C
Calculation Order	2nd	1st	3rd	4th	5th	6th

Mixing and measuring the components of the MPC occurred in the metallurgy room at the University of Arkansas CEREC. Conditions in this room during the process of measuring and mixing were kept to the standards set in ASTM C511 [10]. Water temperatures were recorded for each mixture. The range of water temperatures spanned from 19.3 degrees Celsius to 23.3 degrees Celsius. The median water temperature recorded was 20 degrees Celsius.

Each component of the mixtures was measured using a scale that was accurate to the nearest 0.2 of a gram. Water and set retarder were added to the mixer and stirred with a plastic spoon to dissolve the set retarder. Mixing was performed in accordance with the procedure for mixing paste in ASTM standard C305 [11]. Cement was added to the water in the mixer shown in Figure 3 and allowed to rest for 30 seconds.

Figure 3 Mixer used for mixing MPC paste.

The first deviation from the standard was that the paste was mixed for 60 seconds at the slow speed of 140 rpms instead of 30 seconds after the initial resting time of the cement in the water. After this initial 60 second mixing time, under-mixed paste from the walls and base of the mixer and the upper side of the mixing paddle was scraped into the center of the mixer bowl. Then, in another deviation from the standard, the second mixing phase of 60 seconds was also done at the slow speed of 140 rpms. This was done to prevent the thin paste from splashing over the walls of the mixing bowl. After the paste was fully mixed, it was transferred into the conical ring to undergo Vicat testing for setting time.

The Vicat testing method of hydraulic cements is used to establish initial and final setting time of the cement paste. The ASTM testing standard C191 describes this procedure [12]. The assembled Vicat apparatus with MPC paste can be seen in Figure 4. Testing of all MPC was done in accordance with test method A of this standard, with some deviations due to the unique nature of the magnesium phosphate cement. Due to the extremely thin consistency of the majority of samples of the paste, the paste was not pressed into a ball prior to being inserted into the conical ring. The paste was poured from the mixing bowl into the ring, which was weighed down on the plate to prevent uplift. Additionally, due to the rapid setting time of some of the samples, some setting specimens were not allowed to rest for a full thirty minutes prior to the first needle penetration. Some of these penetrations were also more frequent than the specified fifteen minutes. Additionally, only a single test was performed for each mix design, not three as the standard specifies. Every one to fifteen minutes, the needle was dropped, and the time and penetration depth were recorded. See Figure 5 below for a close-up of the surface of a test in progress.

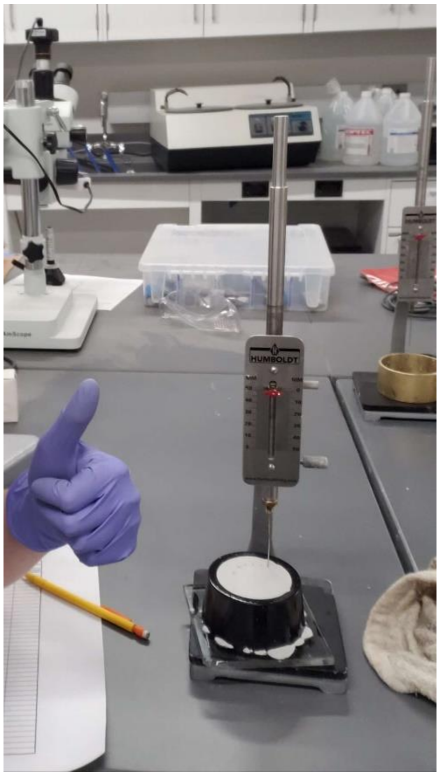

Figure 4 Assembled Vicat Apparatus

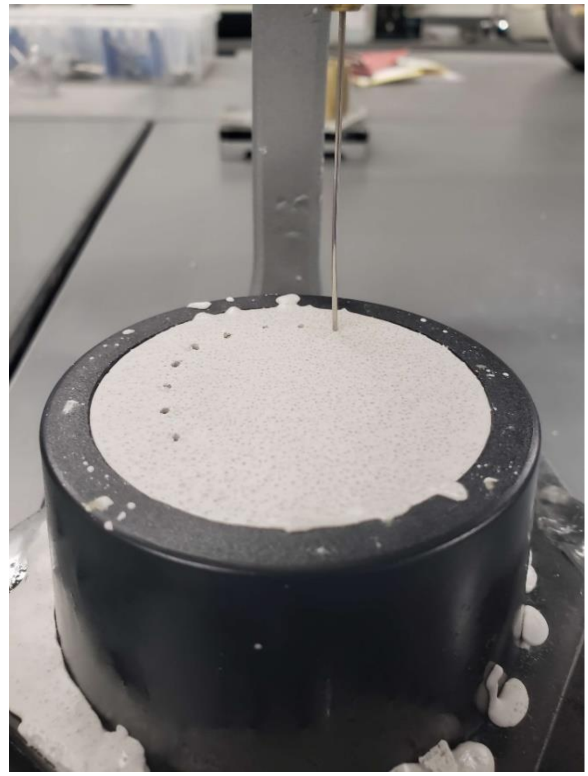

Figure 5 Close-up of Testing Surface

Initial setting time as measured with ASTM C191 is defined as "the elapsed time required to achieve a penetration of 25 mm," [12]. For many tests, interpolation between two points is necessary to determine the initial setting time. Each test was run until the final time of setting was found. Final setting time is "the total time elapsed until the needle does not leave a complete circular impression in the paste surface," according to the ASTM standard [12]. Initial time of setting was the main focus of this study, as it indicates the amount of time available to place and consolidate a mixture before it begins to lose its workability. That said, the setting time of paste typically differs significantly from the setting time of mortar and concrete, so the method is not without its flaws. Additionally, setting time of paste typically varies between different methods used to determine it, so it is difficult to compare Vicat test results with those of other methods, such as penetration resistance [13].

RESULTS AND ANALYSIS

Fly Ash Replacement Rate: The first element of mixture composition considered was fly ash replacement rate. The proportions of MgO and KDP replaced with FFA considered were 20%, 40%, and 60%. Increasing the FFA replacement rate resulted in a delayed initial set. Holding all other mixture proportions constant, increasing the FFA replacement rate from 20% to 40% and from 40% to 60% resulted in a median 14% increase in time of initial setting. Figure 6 compiles the initial setting times of all 72 tests run, organized to show the impact of FFA replacement rate.

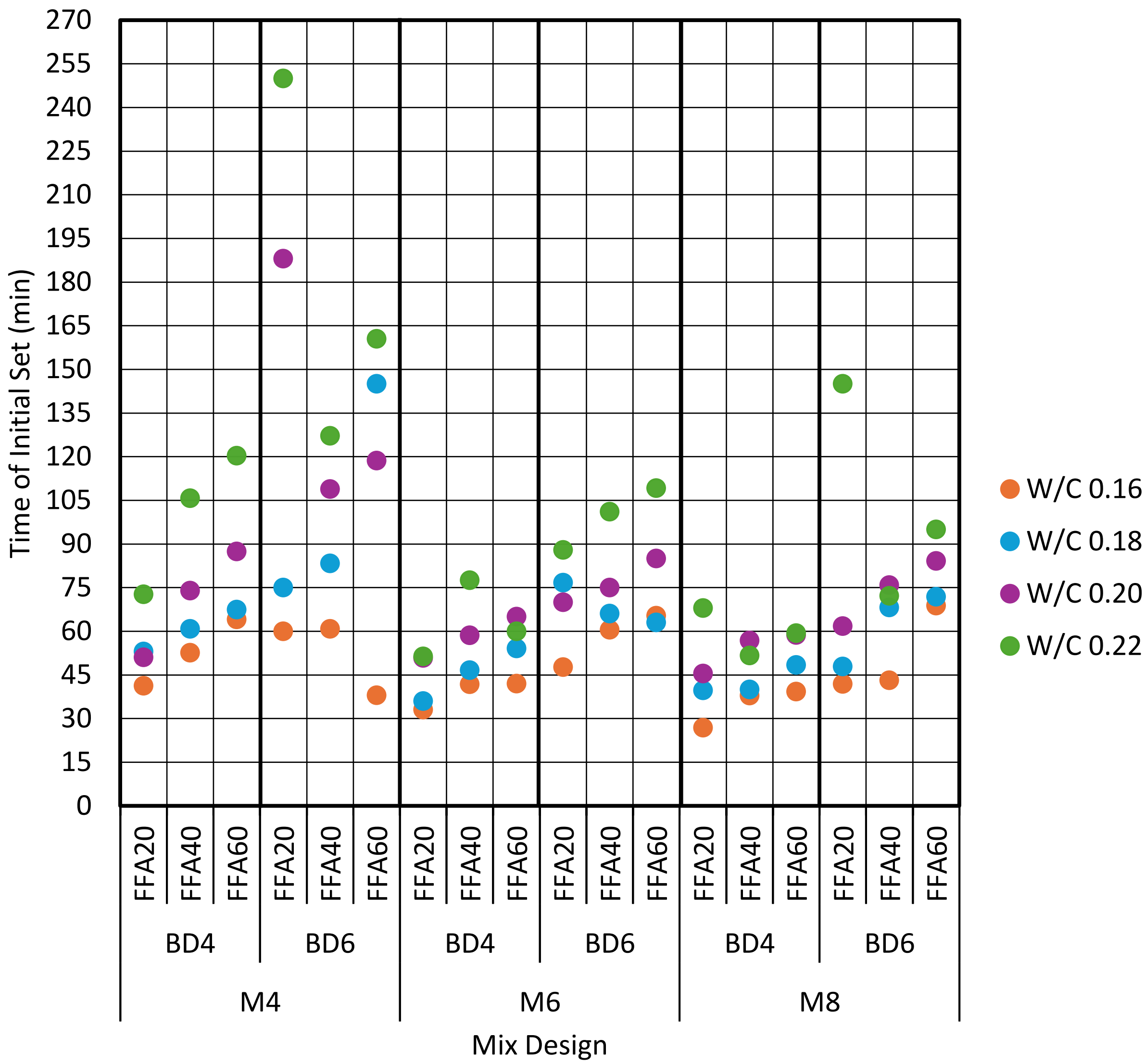

Figure 6 Time of initial setting organized to show impacts of fly ash replacement rate.

Fly ash appears to have a relatively consistent impact on setting time across the different water/cement ratios studied: the median percent increase in setting time ranges from 12% to 15%. The standard deviation for the full data set is 26%, indicating a large amount of variation in the data.

Increasing the concentration of FFA meant that the magnesia and KDP were somewhat diluted by fly ash particles. The fly ash likely increased the distance between the reactive components in the mixtures, slowing the reaction. Temperature data was not recorded, but it was observed that the mixtures that had higher fly ash replacement rates and set more slowly did not produce as much heat while curing.

Mixtures with more fly ash were notably more viscous than the mixtures with less fly ash. This is likely due to the fineness of the fly ash, as well as its lower solubility in water than the other mixture components. This may not have impacted the time of initial setting of the MPC paste, but it is likely that the thicker paste would affect the workability of MPC concrete. Despite this, even mixtures with a 60% FFA replacement rate liquified when agitated.

Set Retarder Dosage: The addition of boric acid alters the setting time of MPC by forming a coating on the surface of the magnesia particles. This slows their dissolution [4]. The mechanism of action by which dextrose delays the setting of portland cement concrete is by adsorption onto the surface of the particles, delaying their interaction with the surrounding water [5]. It can be inferred that a similar process occurs in the MPC paste. The usage of both boric acid and dextrose further delays the initial setting time in MPC paste.

Two variations in total amount of set retarder were also considered. The concentrations of 4% and 6% were examined. A median increase of 42% in the time of initial setting was observed when increasing the set retarder dosage from 4% to 6%. Altering the amount of set retarder had the greatest impact of all of the factors considered on adjusting the initial setting time of the MPC paste. The adjustment of the set retarder dosage had the greatest impact on mixtures with a water/cement ratio of 0.22: a median 60% increase in time of initial set. The adjustment of the set retarder dosage had the least impact on mixtures with a water/cement ratio of 0.2: only a median 36% increase in time of initial set. Figure 7 compiles the initial setting times of all 72 tests run, organized to show the impact of set retarder dosage.

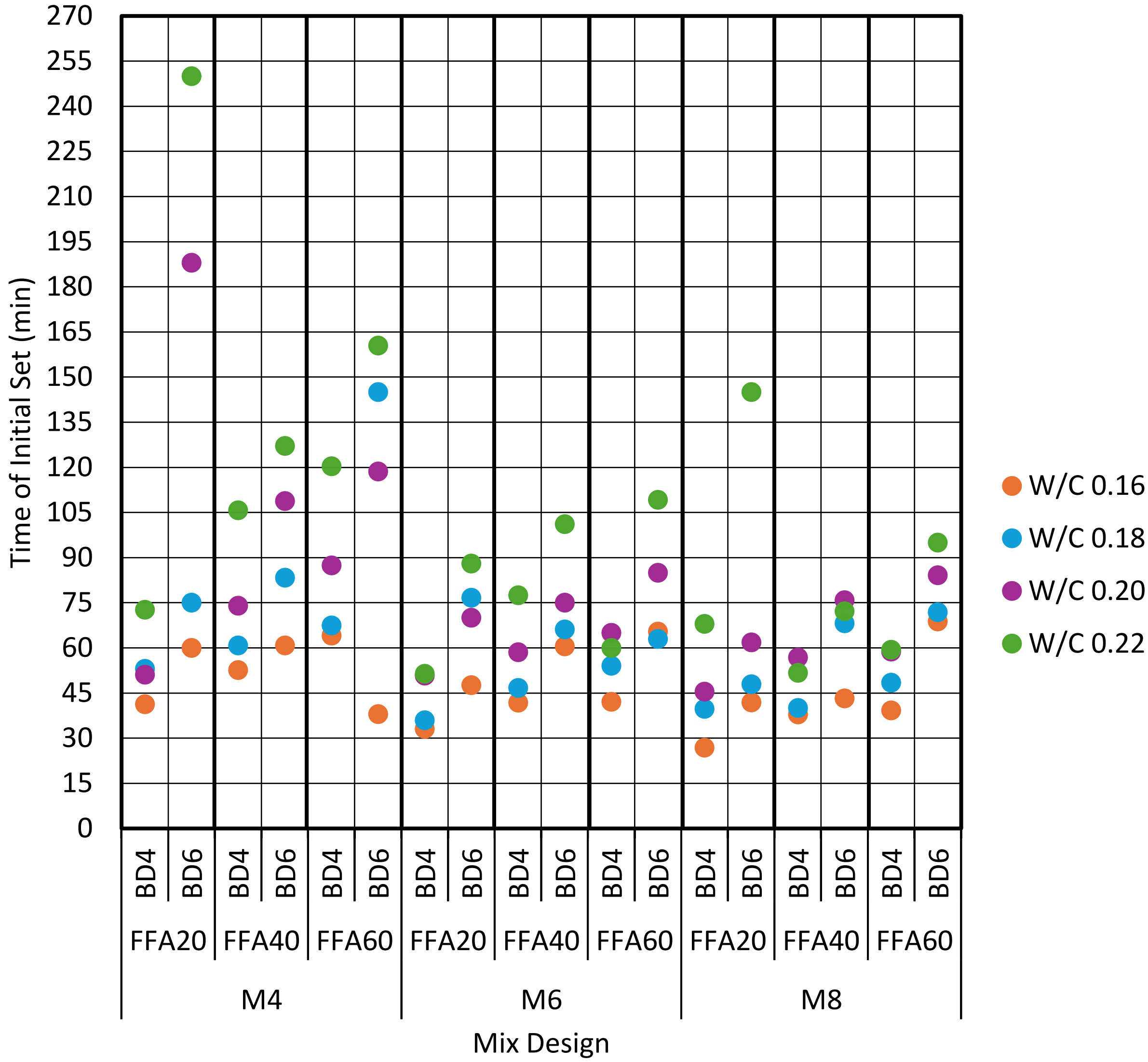

Figure 7 Time of initial setting organized to show impacts of set retarder dosage.

Set retarder dosage appears to have a somewhat inconsistent impact on time of initial setting: the median percent increase in time of initial setting ranges from 36% to 60%. The standard deviation of the full set of median percentage changes was 57%, also indicating a high level of variation.

Due to the small mass of set retarder added, there was little noticeable difference in consistency between similar mixtures with 4% set retarder and mixtures with 6% set retarder immediately after mixing. The mixture M8-BD4-0.16-FFA20 had the quickest time of initial setting: only 27 minutes. Increasing the set retarder dosage to 6% resulted in a time of initial setting of 42 minutes, a 56% increase. The mixture M4-BD4-0.20-FFA20 had an initial setting time of 51 minutes. The time of initial setting for this mixture was increased to 188 minutes upon increasing the set retarder dosage to 6%. This was a 268% increase, the largest percentage increase caused by the adjustment of set retarder dosage alone.

Magnesium to Phosphate Ratio: Higher magnesium to phosphate ratios were correlated with a quicker time of initial setting. Increasing the M/P ratio from 4 to 6 and from 6 to 8 resulted in a median percentage change in the initial setting time of -19%. Since increasing the M/P ratio caused the MPC paste to set more quickly, it can be inferred that a reduction in M/P ratio would cause the MPC paste to set more slowly.

The median percentage change when increasing the M/P ratio from 4 to 6 was -23%, and the median percentage change when increasing the M/P ratio from 6 to 8 was -9%. This difference indicates that the relationship between M/P ratio and setting time is not linear. The standard deviation of the full dataset is 26%, indicating high variation in the impact of M/P ratio on setting time. The initial setting times of all 72 tests can be found below in Figure 8, organized to show the effect of changing the M/P ratio.

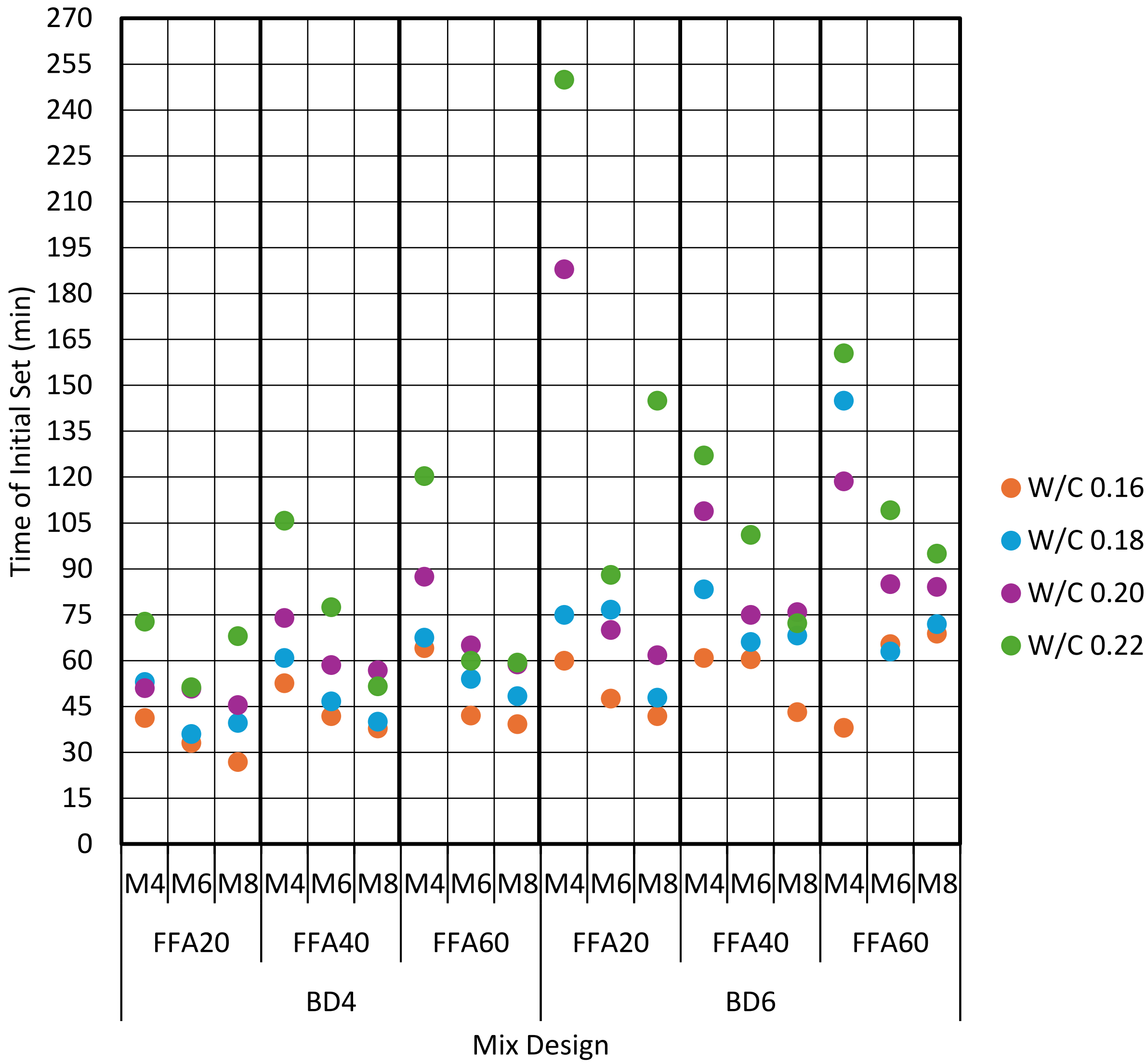

Figure 8 Time of initial setting organized to show impacts of magnesium to phosphate ratio.

Similar to the effect of increasing the fly ash concentration, the mixtures with higher M/P ratios were notably thicker than those with lower M/P ratios. This is likely due to the fineness of the magnesia, and potentially also because of the more rapid reactions that occurred within the mixtures with high M/P ratios. Though temperature data was not recorded, mixtures with a high M/P ratio got significantly warmer to the touch than similar mixtures with a lower M/P ratio. Due to the impacts on performance of MPC concrete, it is unlikely that adjusting M/P ratio is the most efficient and effective way of adjusting the initial setting time.

Water/Cement Ratio: Increasing water cement ratio is known to impact setting time, and therefore workability of concrete, at the cost of the mechanical properties of the cured concrete. Increasing the water cement ratio from 0.16 to 0.18, from 0.18 to 0.20, and from 0.20 to 0.22 resulted in an overall median percentage increase in the time of initial setting of 24%. Figure 9 shows the time of initial setting for all 72 samples organized to show the impact of adjusting the w/c ratio.

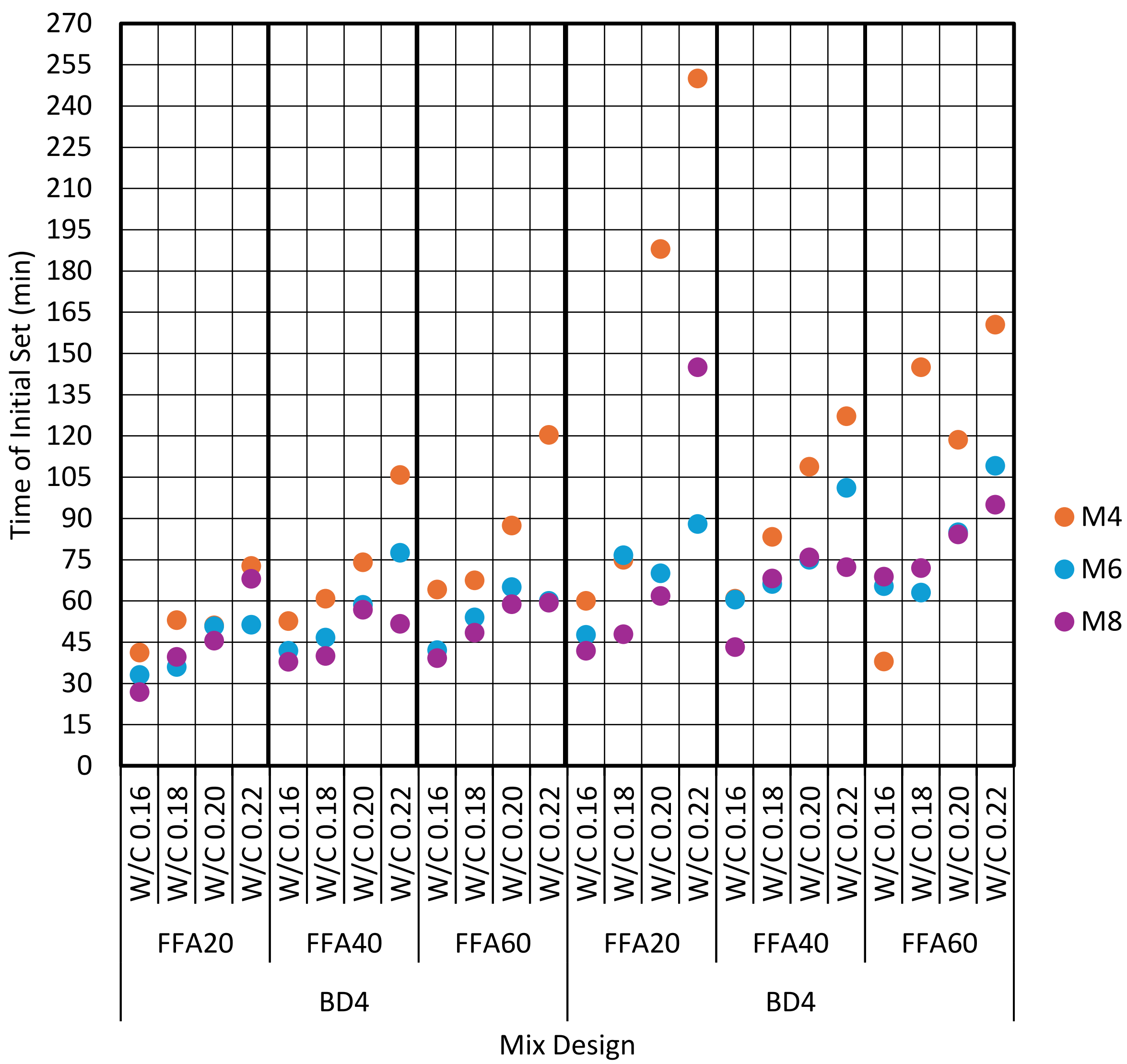

Figure 9 Time of initial setting organized to show impacts of water to cement ratio.

In the mixtures with a M/P ratio of 4, the median percentage change was 30%. In the mixtures with a M/P ratio of 6, the median percentage change was 23%. In the mixtures with a M/P ratio of 8, the median percentage change was only 16%. This indicates that increasing w/c ratio has a lesser impact on the setting time of MPC paste with a higher M/P ratio.

Increasing the amount of water in the mixtures of MPC paste had a large impact on the consistency of the mixtures. The mixtures with a w/c ratio of 0.22 were incredibly thin and runny, and the mixtures with a w/c ratio of 0.16 were very thick and viscous. When agitated, even the thicker mixtures seemed to thin out, indicating that MPC paste has some degree of thixotropic properties.

General: Each test was run until the time of final setting on the Vicat apparatus, and all data was recorded. Some level of interpretation is required when determining final set, so the data is highly variable, especially with the samples that took a long time to set. Tables containing the time of initial setting and the time of final setting can be found in the appendix.

Since only one test was performed on each mixture composition of MPC paste, additional testing would significantly improve the level of certainty in these results. Additionally, testing was done over the span of several months in a room that had some amount of foot traffic, potentially introducing confounding variables that could be reduced by testing in a shorter time period and in a more controlled environment.

CONCLUSIONS

Several conclusions can be drawn based on the findings of this study:

1. Increasing fly ash replacement rate in MPC paste delays the time of initial setting. It also lowers the overall price of MPC concrete. Higher FFA replacement rates also produce thicker MPC paste samples.
2. Increasing the dosage of 25% boric acid, 75% dextrose set retarder in MPC paste delays the time of initial setting. Adjusting dosage of set retarder has very little impact on the consistency of MPC paste mixtures.
3. Increasing the ratio of magnesium to phosphate in MPC paste hastens the time of initial setting. That said, M/P ratio should not be relied on alone to adjust the setting behavior of the MPC concrete due to its impact on mechanical properties.
4. A higher water to cement ratio in MPC paste delays the time of initial setting. Lowering the concentrations of the constituents of the MPC slows the rate of reaction. Since adjusting w/c ratio has impacts on mechanical properties of concrete, this should not be considered as a primary way to adjust setting time.
5. Time of final setting of MPC paste is highly variable, regardless of mixture proportions.
6. Additional research should be undertaken to further understand the influence of mixture composition of MPC on setting time. More proportions of components should be considered, and additional tests should be run to corroborate the results.
7. Additional testing should be performed on samples of MPC paste, mortar, and concrete to further understand the impacts of mixture composition on properties beyond setting time. Investigation into the thixotropic nature of MPC paste in particular should be done.

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APPENDIX

Table 3 Initial and final setting time of MPC paste mixtures with w/c ratio of 0.16.

M4-BD4-0.16-FFA20	
Initial Set	41
Final Set	75

M4-BD4-0.16-FFA40	
Initial Set	53
Final Set	95

M4-BD4-0.16-FFA60	
Initial Set	64
Final Set	115

M6-BD4-0.16-FFA20	
Initial Set	33
Final Set	60

M6-BD4-0.16-FFA40	
Initial Set	42
Final Set	70

M6-BD4-0.16-FFA60	
Initial Set	42
Final Set	95

M8-BD4-0.16-FFA20	
Initial Set	27
Final Set	70

M8-BD4-0.16-FFA40	
Initial Set	38
Final Set	70

M8-BD4-0.16-FFA60	
Initial Set	39
Final Set	90

M4-BD6-0.16-FFA20	
Initial Set	60
Final Set	170

M4-BD6-0.16-FFA40	
Initial Set	61
Final Set	150

M4-BD6-0.16-FFA60*	
Initial Set	38
Final Set	150

M6-BD6-0.16-FFA20	
Initial Set	48
Final Set	100

M6-BD6-0.16-FFA40	
Initial Set	61
Final Set	130

M6-BD6-0.16-FFA60	
Initial Set	65
Final Set	125

M8-BD6-0.16-FFA20	
Initial Set	42
Final Set	90

M8-BD6-0.16-FFA40	
Initial Set	43
Final Set	85

M8-BD6-0.16-FFA60	
Initial Set	69
Final Set	160

Table 4 Initial and final setting time of MPC paste mixtures with w/c ratio of 0.18.

M4-BD4-0.18-FFA20	
Initial Set	53
Final Set	90

M4-BD4-0.18-FFA40	
Initial Set	61
Final Set	125

M4-BD4-0.18-FFA60	
Initial Set	68
Final Set	150

M6-BD4-0.18-FFA20	
Initial Set	36
Final Set	80

M6-BD4-0.18-FFA40	
Initial Set	47
Final Set	90

M6-BD4-0.18-FFA60	
Initial Set	54
Final Set	140

M8-BD4-0.18-FFA20	
Initial Set	40
Final Set	90

M8-BD4-0.18-FFA40	
Initial Set	40
Final Set	105

M8-BD4-0.18-FFA60	
Initial Set	48
Final Set	120

M4-BD6-0.18-FFA20	
Initial Set	75
Final Set	300

M4-BD6-0.18-FFA40	
Initial Set	83
Final Set	240

M4-BD6-0.18-FFA60*	
Initial Set	145
Final Set	330

M6-BD6-0.18-FFA20	
Initial Set	77
Final Set	235

M6-BD6-0.18-FFA40	
Initial Set	66
Final Set	165

M6-BD6-0.18-FFA60	
Initial Set	63
Final Set	140

M8-BD6-0.18-FFA20	
Initial Set	48
Final Set	130

M8-BD6-0.18-FFA40	
Initial Set	68
Final Set	175

M8-BD6-0.18-FFA60	
Initial Set	72
Final Set	170

Table 5 Initial and final setting time of MPC paste mixtures with w/c ratio of 0.20.

M4-BD4-0.20-FFA20	
Initial Set	51
Final Set	120

M4-BD4-0.20-FFA40	
Initial Set	74
Final Set	140

M4-BD4-0.20-FFA60	
Initial Set	87
Final Set	155

M6-BD4-0.20-FFA20	
Initial Set	51
Final Set	85

M6-BD4-0.20-FFA40	
Initial Set	59
Final Set	115

M6-BD4-0.20-FFA60	
Initial Set	65
Final Set	135

M8-BD4-0.20-FFA20	
Initial Set	46
Final Set	85

M8-BD4-0.20-FFA40	
Initial Set	57
Final Set	130

M8-BD4-0.20-FFA60	
Initial Set	59
Final Set	145

M4-BD6-0.20-FFA20*	
Initial Set	188
Final Set	390

M4-BD6-0.20-FFA40	
Initial Set	109
Final Set	330

M4-BD6-0.20-FFA60	
Initial Set	119
Final Set	345

M6-BD6-0.20-FFA20	
Initial Set	70
Final Set	270

M6-BD6-0.20-FFA40	
Initial Set	75
Final Set	255

M6-BD6-0.20-FFA60	
Initial Set	85
Final Set	240

M8-BD6-0.20-FFA20	
Initial Set	62
Final Set	215

M8-BD6-0.20-FFA40	
Initial Set	76
Final Set	225

M8-BD6-0.20-FFA60	
Initial Set	84
Final Set	240

Table 6 Initial and final setting time of MPC paste mixtures with w/c ratio of 0.22.

M4-BD4-0.22-FFA20	
Initial Set	73
Final Set	145

M4-BD4-0.22-FFA40	
Initial Set	106
Final Set	225

M4-BD4-0.22-FFA60	
Initial Set	120
Final Set	235

M6-BD4-0.22-FFA20	
Initial Set	51
Final Set	90

M6-BD4-0.22-FFA40	
Initial Set	78
Final Set	125

M6-BD4-0.22-FFA60	
Initial Set	60
Final Set	150

M8-BD4-0.22-FFA20*	
Initial Set	68
Final Set	175

M8-BD4-0.22-FFA40	
Initial Set	52
Final Set	110

M8-BD4-0.22-FFA60	
Initial Set	59
Final Set	135

M4-BD6-0.22-FFA20*	
Initial Set	250
Final Set	525

M4-BD6-0.22-FFA40	
Initial Set	127
Final Set	245

M4-BD6-0.22-FFA60	
Initial Set	161
Final Set	285

M6-BD6-0.22-FFA20	
Initial Set	88
Final Set	160

M6-BD6-0.22-FFA40	
Initial Set	101
Final Set	195

M6-BD6-0.22-FFA60	
Initial Set	109
Final Set	215

M8-BD6-0.22-FFA20*	
Initial Set	145
Final Set	305

M8-BD6-0.22-FFA40	
Initial Set	72
Final Set	150

M8-BD6-0.22-FFA60	
Initial Set	95
Final Set	190