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CBU-2002-19
REP-472
October 2002

Accepted for Presentation and Publication at the Seventh CANMET/ACI International Conference on Superplasticizers and Other Chemical Admixtures in Concrete, Berlin, Germany, October 2003

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Influence of Fly Ash and Chemical Admixtures on the Setting Time of Cement Paste and Concrete

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Synopsis: A recurring question about use of fly ash in concrete is dealing with setting and hardening of such mixtures with our with out chemical admixtures. This paper presents literature review on the setting and hardening characteristics of cement paste and concrete as influenced by the inclusion of fly ash and chemical admixtures. The paper also reports the work carried out at the University of Wisconsin-Milwaukee (UWM-CBU) on the effects of Class C fly ashes from various sources on the initial- and final-setting times of non-air-entrained and air-entrained concrete; and the effects of Class C fly ash, gypsum, and various types of chemical admixtures (air-entraining admixture (AEA), water-reducing admixture (WRA), superplasticizer, and retarding admixture) on the initial and final setting times of cement paste. Test results indicated that: (1) both the initial- and final-setting times were relatively unaffected at low-percentage replacement of cement with Class C fly ash, although inclusion of fly ash caused large retardation in the times of setting, up to around 60 percent cement replacement; (2) initial- and final-setting times of cement paste remained essentially the same or were slightly delayed with up to 20 percent cement replacement relative to zero percent fly ash content; beyond this range, the setting times of cement paste were accelerated. Increased rate of setting occurred at cement replacement levels of 40 percent and higher irrespective of type of chemical admixtures used.

Keywords: Air-entraining admixture (AEA), concrete, fly ash, gypsum, high-range water-reducing admixture (HRWRA), paste, retarder, time of setting, water-reducing admixture.

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INTRODUCTION

Immediately upon mixing of cement and water, various chemical reactions occur leading to formation of numerous types of hydration products. The types and amount of hydration products formed depend upon duration of hydration, water-cementitious materials ratio (*W/Cm*), properties of constituent materials, temperature, soluble alkalis, and mineral and chemical admixtures. The formation of hydration products causes increase in stiffness of the cementitious matrix. This stiffening behavior of the matrix is determined by the times of initial and final setting. The initial setting of the matrix refers to the beginning of solidification for a given mixture. It is generally accepted that at this stage concrete can neither be properly re-tamped nor handled or placed. The final setting refers to the stage when the mixture attains sufficient hardness to support stress. The subsequent continuing strength gain is called hardening.

Setting and hardening of cement mortar mixtures are considerably influenced by inclusion of either mineral or chemical admixtures. Generally, the setting and hardening of mortar are delayed when ASTM Class F (low-lime) fly ash is added to it. Mortar incorporating ASTM Class C (high-lime) fly ash, however, has shown either both rapid or delayed setting depending upon the properties and amount of the ash. The setting behavior can be more readily modified when gypsum and chemical admixtures such as water-reducing admixture (WRA), superplasticizer, or retarding and accelerating admixture are used. Even air-entraining admixture is known to slightly modify setting behavior of concrete. A knowledge of setting characteristics of concrete incorporating both mineral and chemical admixtures is needed for efficient scheduling of concrete construction, specifically floor slabs, roadways, pavements, and other flat surfaces. Limited data exist on setting and hardening behavior of paste, mortar, and concrete containing ASTM Class C fly ash and chemical admixtures.

LITERATURE REVIEW

Many investigators have reported on the effects of fly ash on the times of setting of cement paste and concrete.

Dodson[1] investigated the setting characteristics of concretes made with both Class C and Class F fly ashes. He reported that the setting times of concrete are mainly governed by cement content and *W/Cm* when all other parameters are kept equal. He further added that an increase in cement content caused a decrease in the initial- and final-setting times, whereas an increase in *W/Cm* increased setting times. However, in general, addition of fly ash increased the setting times. Ramakrishnan et al.[2] reported on the setting characteristics of concretes made with or without fly ash. They used one high-lime fly ash and two types of cement (ASTM Type I and Type II). They concluded that inclusion of fly ash resulted in higher initial- and final-setting times compared to the concrete without fly ash for both types of cement. Lane and Best[3] reported that fly ash generally slows the setting of concrete, although both initial and final times of setting remain within specified limits. Retardation of setting due to the inclusion fly ash may be affected by the amount, fineness, and chemical composition (particularly, carbon content) of the ash. However, the fineness of cement, the water content of the cementitious paste, and the ambient temperature usually have a much greater effect on times of setting than addition of fly ash. Replacement of 60% of cement with high-carbon fly ash by mass resulted in 200% increase in the time of final setting of control concrete mixture.

Mailvaganam et al.[4] reported the properties of concretes made with a slag and a low-lime fly ash. Concrete mixtures were prepared using various chemical admixtures at two different temperatures (5°C and 20°C). At 20° C, concrete mixtures with 30 % fly ash showed longer initial-setting time than the control mixture, by 1 to 1.75 hours; whereas at 5°C, all mixtures with or without fly ash had the initial-setting time greater than 10 hours. Majko and Pistilli[5] reported the time of setting results for air-entrained concrete mixtures incorporating high volumes of Class C fly ash and WRA. They reported that setting times of concrete mixture increased by 4 to 6 hours with the use of WRA and very high fly ash contents (119 to 178 kg/m^3). Gebler and Klieger[6] studied the times of setting of concretes containing Class F and Class C fly ashes from 10 different sources for high content mixtures. They reported that inclusion of the fly ash increased the initial- and final-setting times of concrete mixtures. Carette and Malhotra[7] reported the setting characteristics of concretes made with fly ashes from different sources. Calcium oxide (CaO) contents of the fly ashes varied between 1 % and 13 %. They concluded that, in general, the fly ashes increased the initial- and final-setting times of concrete.

Bilodeau and Malhotra[8] reported properties of concrete incorporating high volumes of Class F fly ashes from three different sources. Cementitious materials content was 300, 370 and 430 kg/m^3, and three W/Cm (0.39, 0.31 and 0.27) were used. They concluded that for every W/Cm, the initial- and final-setting times of high-volume fly ash concretes were noticeably increased as compared to those of the control concretes (without fly ash). This could possibly be due to the lower cement content of the high-volume fly ash concretes. Carette et al.[9] reported data on the setting time of high-volume (55 % to 60 %) Class F fly ash concretes. Eight sources of fly ashes and two sources of portland cements were used. The initial- and final-setting times varied from 4:50 to 12:51(hr: min), and 6:28 to 13:24 (hr: min), respectively, except for one mixture whose final-setting time exceeded 13:24 (hr: min). Concrete mixtures showed varying setting times depending upon the source of fly ash. In general, for each fly ash source, concrete made with a low-alkali content cement having 6% C_3A showed longer setting times than concrete made with a high-alkali content cement having 11.9% C_3A. Malhotra and Ramezanianpour[10] have reported that inclusion of Class F fly ash retards the hydration of C_3S at very early stages of hydration and then accelerate at later stages. C_3A contribution from this fly ash increased with increasing its content as a replacement of cement. Thus, fly ash also became a contributor of C_3A and other reactive components at high fly ash contents. Accelerated setting and hardening occurred due to the reactions of C_3A present in the fly ash in addition to contributions of reactions associated with cement hydration in presence of fly ash at cement replacements of about 40% and above. Extremely high rate of setting and hardening occurred at 70% fly ash content and beyond due to the presence of relatively higher amount of C_3A contributed by the fly ash, in addition to that contributed by cement. Hydration of aluminates was very rapid leading to formation of C_3AH_6, C_4AH_{19}, and C_2AH_8 with generation of large amount of heat of hydration[13].

Eren et al.[11] reported the results of setting times of concrete incorporating up to 50 % ground-granulated blast-furnace slag (GGBS) under curing temperatures ranging from 6 to 80^{o}C. They concluded that: (1) increase in temperature decreased the setting times of concrete; (2) setting times of fly ash concretes were longer than those of Type I cement concretes and GGBS concretes; and (3) at temperatures greater than 20^{o}C, the setting times of GGBS concretes were shorter than those of Type I cement concretes. Pinto and Hover[12] studied the effects of inclusion of silica fume and superplasticizer on setting behavior of high-strength concrete mixtures. The influence of temperature was also studied by storing mortar specimens at different temperatures. Use of silica fume caused reduction in the initial time of setting. However, an opposite trend was noted when superplasticizer was used. Statistical analysis revealed significant interaction between the two (silica fume and superplasticizer) when the initial time of setting was taken as a response. The effect of temperature was significant on both initial and final times of setting.

Samadi et al.[13] studied the influence of phosphogypsum (PG) on the times of setting and soundness of cement pastes. In this study, cement paste mixtures were made using ordinary portland cement (OPC) and pozzolanic portland cement (PPC) at a constant water to cement ratio of 0.6 with PG content varying between 0 and 100 percent. In general both initial and final times of setting increased with increasing PG content. The initial time of setting ranged between 100 to 560 minutes and 120 to 710 minutes for pastes containing OPC (ordinary portland cement) and PPC (pozzolana Portland cement), respectively. The corresponding final time of setting ranged between 250 to 1440 minutes and between 270 to 1440 minutes. The paste expansion also increased with increasing PG content. Brooks[14] investigated the effects of silica fume (SF), metakaolin (MK), fly ash (FA), and ground-granulated blast-furnace slag (GGBS) on the setting times of high-strength concrete using the penetration resistance method (ASTM C 403). He also studied the effects of shrinkage-reducing admixture (SRA) on the setting times of normal and high-strength concretes. Based on the test results, he concluded that: (1) the setting times of the high-strength concrete were generally retarded when the mineral admixtures replaced part of the cement. While the SRA was found to have negligible effect on the setting times of normal strength concrete, it exhibited a rather significant retarding effect when used in combination with a superplasticizer; and (2) the inclusion of GGBS at replacement levels of 40% and greater resulted in significant retardation in setting times. In general, as replacement levels of the mineral admixtures were increased, there was greater retardation in setting times. However, for the concrete containing MK, setting time were only observed up to a replacement level of 10%.

Ahmadi[15] studied the initial and final setting times of concrete in hot weather. The effect of field temperature, relative humidity, wind velocity, and admixture on the setting times of concrete were observed. He proposed two equations: (1) the first equation was for determining the initial setting time of concrete with a correlation factor of 0.93 and standard deviation of 5.28%. This equation showed that as the field temperature and field air velocity increased, the initial setting time decreased, and as the field humidity increased, the initial setting time increased; and (2) the second equation for determining the final setting time of concrete with a correlation factor of 0.9 and standard deviation of 5.8% showed similar effects as of initial setting time of concrete.

Targan et al.[16] studied the effects of bentonite, colemanite ore waste (CW), coal fly ash (FA) and coal bottom ash (BA) on the setting time of mortar. Based on the test results, they concluded that (1) setting time of cements was generally accelerated when bentonite replaced a part of cement; (2) in general FA, BA and CW increased the setting time of the cement; and (3) bentonite had a negligible effect on the final setting time of cement containing BA, but it had a significant accelerating effect when used alone or in combination with FA and CW at the higher replacement level. Brooks[17] presented a model for predicting initial setting time of concrete made with and without fly ash. He drew following conclusions from the analysis of setting times of concretes made with Type I cement and fly ash : (1) the proposed model, based on the assumption that initial setting time is given by initial spacing of unhydrated cement particles divided by a rate coefficient, is applicable for predicting the setting times of Type I cement concrete and blended cement concrete containing approximately 60% of the fly ash; (2) the influencing factors quantified in the model are water-to-cementitious materials ratio, fineness, specific gravity, temperature, and the blended oxide ratio: $CaO/(SiO_2 + Fe_2O_3)$; and (3) the model estimated the average setting time to within 16%. Final setting time is related to initial setting time by a factor of 1.35 with an average error of 13%.

Takemoto and Uchikawa[18] and Uchiwaka and Uchida[19] described a model for hydration reaction process of cement in the presence of pozzolans. The reactions of C_3A and Class C fly ash resulted in formation of enttringite, monosulphoaluminate hydrate, calcium aluminate hydrates, and calcium silicate hydrate. They reported that presence of pozzolan accelerated hydration of C_3A due to adsorbing Ca^{2+} from the liquid phases and providing precipitation sites for the hydration products.

Tay[20] performed a study to investigate properties of mortar and concrete as influenced by inclusion of pulverized sludge ash. The test data exhibited improved workability and increase in initial and final times of setting with increasing sludge ash content. Sawan and Qasrawi[21] concluded that the use of natural pozzolan cause decrease in workability and increase in the times of setting of mortar under normal condition. However, an opposite trend was obtained in hot weather conditions. Uchikawa et al.[22] evaluated the effects of chemical admixtures on the hydration characteristics of cement. They reported that an admixture having a functional group that produces complex salt with decrease in Ca^{2+} concentration can cause loss in fluidity and delay in the times of setting of cement pastes. Chen and Older[23] investigated the effect of cement with varying in clinker composition with varying amounts and forms of calcium sulfate on the times of setting of mortars.

They indicated that the setting of cement having normal composition was mainly related to hydration of C_3S content. The formation of enttringite occurred at very high C_3A contents.

Matusinovic and Vrbos[24] and Matusinovic and Curlin[25] reported that setting characteristics of high-alumina cement (HAC) were substantially influenced by inclusion of alkali metal salts. The lithium cation had a greater effect on the times of setting than alkali cations did. The results showed that lithium salt or alkali metal salts could be used as a set accelerator for HAC.

Perret et al.[26] investigated the compatibility of six different microfine cements and four different HRWRAs; and the influence of materials and mixture proportions on rheological characteristics and final-setting time of microfine cement-based grouts. Three portland cements and three slag cements, associated with various naphthalene-based and melamine-based HRWRA were investigated. They concluded that: (1) not every microfinecement can be used with every HRWRA; (2) some HRWRAs gave better fluidity, and some gave too long (24 hours) or too short (4 hours) final setting times; and (3) the chemical composition and fineness of cements, as well as the type and chemical characteristics of admixtures lead to different grout properties.

INFLUENCE OF FLY ASH ON SETTING TIMES OF NON-AIR-AIR ENTRAINED CONCRETES (Series 1)

Experimental Details

An experimental program was designed to evaluate the effects of Class C fly ash content and its source on setting times of non air-entrained concrete. Four different Class C fly ashes, obtained from different electric power plants in Wisconsin, were used. The fly ashes corresponding to these power plants are designated as P-4, DPC, Columbia, and Weston. Chemical and physical properties of these fly ashes were determined. Three of the fly ashes (DPC, Columbia, and Weston) exceeded ASTM C 618 requirement for MgO. However, they met all other ASTM C 618 Class C fly ash requirement. Natural sand with 6 mm maximum size was used as a fine aggregate, and a 19 mm maximum size gravel was used as a coarse aggregate throughout this investigation. These aggregates met the ASTM C 33 requirements. Type I cement which met the requirements of ASTM C 150 was used.

Concrete mixture proportions were proportioned with all the four Class C fly ashes. For concrete mixture proportions containing P-4 fly ash, cement replacement varied from 0 to 100 % with an increment of 10%; cement content varied between 360 and 0 kg/m^3; fly ash content between 0 and 488 kg/m^3; *w/cm* between 0.47 and 0.34; sand content between 815 to 833 kg/m^3; and coarse aggregate content between 1070 and 1084 kg/m^3. For concrete mixture proportions containing DPC fly ash, cement replacement varied from 0 to 90 % (0, 10, 30, 50, 70 and 90%); cement content varied between 371 and 36 kg/m^3; fly ash content between 0 and 403 kg/m^3; w/cm between 0.45 and 0.35; sand content between 802 to 840 kg/m^3; and coarse aggregate content between 1070 and 1110 kg/m^3. For concrete mixture proportions containing Columbia fly ash, cement replacement varied from 0 to 90 % (0, 10, 30, 50, 70 and 90%); cement content varied between 360 and 35 kg/m^3; fly ash content between 0 and 394 kg/m^3; w/cm between 0.44 and 0.35; sand content between 816 to 854 kg/m^3; and coarse aggregate content between 1053 and 1085 kg/m^3. For concrete proportions containing Weston fly ash, cement replacement varied from 0 to 100 % with an increment of 20%; cement content varied between 360 and 0 kg/m^3; fly ash content between 0 and 437 kg/m^3; w/cm between 0.44 and 0.34; sand content between 825 to 855 kg/m^3; and coarse aggregate content between 855 and 1080 kg/m^3.

Results and Discussion

Initial and final setting times of concrete incorporating various sources of Class C fly ash are shown in Table 1. In general, addition of fly ash up to a certain level (typically up to about 60% replacements) caused delay in the initial and final setting times (doubled) of concrete; and beyond this level of fly ash, a reverse trend was observed. The setting times varied from fly ash to fly ash source. Both the initial and final setting times were relatively unaffected up to 10% cement replacement. Above 10% cement replacement, the effect of fly ash inclusion on the setting behavior of concrete became more pronounced with increasing fly ash replacement levels. The maximum retardation in either the initial or final setting times occurred at 50% for the P-4, DPC, and Columbia fly ash mixtures, and at 60% for the Weston fly ash mixture. However, the final setting time for the Weston fly ash increased with an increase in fly ash content up to 80% cement replacement. The control concrete mixtures without fly ash exhibited less than 5 hours of the initial setting times and less than 6 hours of the final setting times. The maximum initial setting times were 10:25 hr:min at 50% cement replacement for the P-4 ash, 9:40 hr:min at 50% cement replacement for the DPC ash, 12:30 hr:min at 50 and 70% cement replacement for the Columbia ash, and 8:00 hr:min at 80% cement replacement for the Weston ash. The maximum final setting times were 13:30 hr:min at 60% cement replacement for the P-4 fly ash mixture, 12:30 hr:min at 50% cement replacement for the DPC fly ash mixture, 21:40 hr:min at 70% cement replacement for the Columbia fly ash mixture, and 11:50 hr:min at 80% cement replacement for the Weston fly ash mixture. Of all the fly ash mixtures tested, the mixture with Weston fly ash showed the lowest setting times when compared with other three sources of fly ash. When slower than desirable setting times occur, it would be desirable to use set accelerating admixtures.

At high replacements of cement with fly ash (70% or above), the setting of concrete was accelerated. This might be attributed to the fact that at higher cement replacements with fly ash, the concentrations of total C_3A and gypsum present in the mixture becomes low. This resulted in reduced setting times of the mixtures containing low cement and high fly ash contents. As a result, rapid setting of the concrete mixtures occurred. Therefore, under such conditions, it is desirable to use a set retarding admixtures to allow enough time for proper mixing and placing of concrete.

SETTING TIMES OF NON-AIR-ENTRAINED AND AIR-ENTRAINED FLY ASH CONCRETE (Series 2)

Experimental Details

One source (Pleasant Prairie Power Plant, P-4) of Class C fly ash was used. Three nominal compressive strength levels (21, 28, and 35 MPa) of non-air-entrained and air-entrained concrete mixture proportions, by varying the water-to-cementitious materials ratio (0.45, 0.55, and 0.65) were developed. Cement replacement percentage was 35, 45, and 55%. Replacement was on the basis of

1.25 to 1, fly ash replacement of cement. Non-air-entrained concrete mixtures contained cement content between 236 and 115 kg/m^3; fly ash content between 106 and 238 kg/m^3; and *w/cm* between 0.58 and 0.39. Air-entrained concrete mixtures contained cement content between 276 and 132 kg/m^3; fly ash content between 131 and 283 kg/m^3; and *w/cm* between 0.47 and 0.33.

Results and Discussion

Setting time of non-air-entrained concrete mixtures are given in Table 2. For non-air-entrained concrete mixtures, the initial setting time increased by about one hour for the nominal 21 MPa strength concrete as fly ash content was increased from 35% for each 10% increase in fly ash. However, the actual initial setting time of approximately eight hours plus or minus one hour, is practically the same for the 35, 45, and 55% fly ash replacement mixtures. Therefore, these mixtures should perform similarly and they generally would not have any adverse impact on a typical construction project. The final setting time for theses concretes increased by about 90 minutes for each 10% increase in fly ash when compared to the 35% fly ash mixture. However, the actual final setting time, from 8-1/2 hours to 11-1/2 hours, also would not have any significant adverse effect on a typical construction. The change in initial and final setting time for 28 MPa and 35 MPa non-air-entrained fly ash concrete was even less significant when fly ash replacement was increased from 35 to 55%. Therefore, the initial or the final setting time is not significantly different for non-air-entrained fly ash concrete as the fly ash replacement for cement is increased to levels of 55% for the 21, 28 or 35 MPa concretes. In general, however, for the non-air-entrained concretes, as the strength levels increased with increasing cementitious materials ratio, the change in initial or final times of setting was insignificant from 35% to 55% cement replacement with P-4 Class C fly ash.

Setting time data for air-entrained concrete are given in Table 2. It is evident that the initial time of setting for the nominal 21 MPa strength air-entrained fly ash concrete increased by about 1-1/2 hours as the fly ash replacement was increased from 35 to 45%. As the fly ash replacement was further increased from 45 to 55%, there was a reduction of one hour in the initial setting time. The average initial setting time for the three mixtures of 21 MPa air-entrained concrete, containing 35, 45, and 55% fly ash, was 7 hrs: 30 min., with a range of 45 minutes from the average. The final setting time for this concrete increased by about 1-3/4 hrs. as the fly ash replacement was increased from 35 to 45%. When the fly ash replacement was further increased from 45 to 55%, there was reduction of about 45 minutes in the final setting time. The average final setting time for the three mixtures of 21 MPa air-entrained concrete containing 35, 45, and 55% fly ash was 9 hrs: 30 min., with a range of one hour from the average. The initial and the final setting time, therefore, varied within a range of about one hour as fly ash replacement was varied between 35 to 55%. Variation of plus or minus one hour in the initial or the final setting time for concrete will not be significant for a typical construction project. Similar to the non-air-entrained concrete, the change in the initial and final setting time for the 28 MPa and 35 MPa air-entrained fly ash concrete was even less significant as the fly ash replacement was increased from 35 to 55%. In general, air-entrained concrete initial and final setting times were not noticeable affected as the strength levels or fly ash levels were varied.

SETTING TIMES OF CEMENT PASTE AS INFLUENCED BY FLY ASH AND CHEMICAL ADMIXTURES

Four series of tests were performed: (1) to evaluate only the effects of fly ash addition on the setting times of cement paste; (2) to evaluate the effects of fly ash and two levels of air content on the setting times of cement paste; and (3) to evaluate the influence of fly ash and normal dosage of two types of chemical admixtures (WRA and HRWRA) on the setting times of cement paste; (4) to evaluate the combined effects high dosage of fly ash and three dosage rates of two types of chemical admixtures (retarders and gypsum) on the setting times of cement paste.

Experimental Details

A portland cement conforming to the requirements of ASTM C 150 was used. An ASTM Class C fly ash, obtained from one source, Pleasant Prairie (P-4), was used. The fly ash met all ASTM C 618 requirements for Class C fly ash. Five chemical admixtures: an air entraining admixture (ASTM C 260), a water-reducer (ASTM C 494, Type A), a retarder (ASTM C 494, Type B), and a HRWRA (ASTM C 494, Type F) were obtained from a local ready-mixed concrete company, the Tews Company, Milwaukee, WI. A total of 82 cement paste mixtures were prepared for evaluating their setting and hardening characteristics. Each mixture was composed of cement, fly ash, and water. Fly ash was used as a replacement of cement ranging from 0 to 100 percent by mass. A ratio of fly ash addition to cement replaced was kept at 1.25. All ingredients were mixed in a laboratory mixer in accordance with ASTM C 305. Normal consistency of pastes containing cement/fly ash was determined in accordance with ASTM C 187. Air content of each paste mixture was determined according to ASTM C 185. Test specimens for each mixture were prepared for measuring the initial and final times of setting using the Vicat apparatus (ASTM C 191).

Results and Discussion

Effect of fly ash on setting times of pastes without admixtures

The initial and final times of setting were essentially the same due to the inclusion of fly ash at 10% compared to the 0% fly ash mixture (Table 3). Beyond 10% fly ash content, accelerated setting occurred and the rate of setting increased with fly ash content. Very rapid rate of setting and hardening occurred at 40% fly ash content and above. The entrapped air content was essentially the same for all mixtures at about 6.5 ± 0.5 percent.

Effect of air entrainment and content on setting times of paste

Effects of air entrainment and content at two dosage levels on setting times of fly ash mixtures are given in Table 4. At the low air content level, the time of settings were essentially the same up to 20% fly ash content, beyond which the times of setting were accelerated. Very high rate of setting occurred at 40% fly ash content and beyond. Thus at high fly ash contents, setting characteristics of air-entrained mixtures followed a similar general trend as indicated by the fly ash mixtures without AEA (Table 3). At the high air content level also, the general setting characteristics of fly ash mixtures remained the same as that obtained at the low air content level. However, in general, as the air content increased, from the entrapped air only (Table 3) to entrained air with increasing amounts of air (Table 4), the setting times were less affected by the amount of fly ash and, therefore, setting times were equal to the 0% fly ash content paste as the amount of fly ash increased.

Effect of fly ash with normal dosages of chemical admixtures on setting times of paste

In this series of tests, fly ash content varied from 0 to 100% with normal dosages of individual chemical admixtures (five different types).

Fly ash with a normal dosage of water-reducer

Effects of normal dosage of water-reducer on setting characteristics of fly ash mixtures are given in Table 5. Both initial and final times of setting were reduced above the fly ash content of 20%. Beyond 20% fly ash content, the setting was accelerated. High rates of setting occurred at 40% fly ash content and above. The hydration reaction was accelerated due to the reduction in water to cementitious materials ratio, as well as more cement particles hydrating early due to dispersion of the cementitious particles when the water-reducing admixture (WRA) was used.

Fly ash with a normal dosage of superplasticizer

Effects of normal dosage of superplasticizer on setting characteristics of fly ash mixtures are given in Table 5. In general, water demand for superplasticized mixtures decreased with increasing fly ash content. The combined effect of fly ash and superplasticizer was a noticeable reduction in the times of setting beyond 20% fly ash content, and there was sharp reduction after 30% fly ash content. In general paste containing WRA or HRWRA had similar affects on times of setting of pastes containing 0% to 50% fly ash content.

Fly ash with a normal dosage of retarder

Effects of normal dosage of retatder on setting characteristics of fly ash mixtures are given in Table 6. The water demand for mixtures with retarder decreased with increasing fly ash content. The time of setting for mixtures containing the retarder was essentially the same for fly ash content up to 20%. Beyond the 20% fly ash level, the corresponding setting times were not helped by the use of the retarder; even with double and triple amount of retarder dosages the practical needs of sufficient retardation was not possible to achieve. The rate of setting became very high at 50% fly ash content and above. At high fly ash contents, general trend of setting and hardening of paste mixtures remained similar as that obtained for the fly ash mixtures without retarder (Table 3).

Fly ash with a normal dosage of gypsum

Effects of normal dosage of gypsum on setting characteristics of fly ash mixtures are given in Table 7. The water demand for gypsum mixtures decreased with increasing fly ash content. The results showed no significant difference in the initial and final setting times of the mixtures up to

10% fly ash content when gypsum was used. Beyond 10% fly ash content, the times of setting decreased for gypsum mixtures with increasing fly ash content. The general setting behavior of mixtures followed essentially similar trend as indicated by fly ash mixtures without gypsum (Table 3). Inclusion of gypsum in the fly ash mixtures caused a noticeable reduction of the initial and final setting times compared to the paste mixtures without gypsum (Table 3).

Effect of High Fly Ash Contents with High Dosages of Chemical Admixtures on Setting Times of Paste

At high fly ash content (above 40%), very rapid rate of setting of mixtures occurred. Use of normal dosage of retarder and gypsum did not cause enough delay to compensate for the rapid rate of setting resulting from the presence of the high-levels of fly ash. Therefore, high dosages of these admixtures were used at fly ash contents of 70, 85, and 100%. The retarder and gypsum were used at their respective double and triple dosages.

Fly ash with retarder

Effects of high dosage of retatder on setting characteristics of fly ash mixtures are given in Table 6. In general, when the amount of the retarder increased beyond normal dosage, the initial and final times of setting were considerably delayed, especially for 85% and 100% fly ash levels. These results show that a delay in the times of setting can be achieved even at 100% fly ash level with high dosage of the retarder. Under this condition, the final time of setting was delayed by over 300 % compared to the normal dosage of the retarder. However, the rate of setting was still very high even at the triple dosage of the retarder; and, at these high setting rates, practical benefits of necessary retardation was not realized.

Fly ash with gypsum

Effects of high dosage of gypsum on setting characteristics of fly ash mixtures are given in Table 7. Due to very rapid setting, initial time of setting could not be recorded at the fly ash content of 70% and beyond at the normal dosage of gypsum, probably due to early enttringite formation. However, when dosage of gypsum was doubled or tripled, initial times of setting, though not slow enough, could only be recorded for the fly ash levels of 70% and 85%. Thus, the use of high dosage of gypsum did not help significant in modifying the setting characteristic of mixtures incorporating fly ash.

CONCLUSIONS

Following are the general conclusions from this study:

1. Both the initial- and final-setting times of the concretes were significantly influenced by both the source and amount of fly ash. Both the initial- and final-setting times were relatively unaffected at 10% cement replacement. Although inclusion of fly ash caused large retardation in the setting times, for up to around 60% cement replacement, the rate of strength development were appropriate for most construction applications. Therefore, setting time should not be taken as a sole parameter for selecting a fly ash for a particular

application. However, in order to improve construction productivity and efficient construction planning, fly ash content should be reduced and/or chemical admixtures should be added to control the setting times.

2. For non-air-entrained and air-entrained fly ash concretes having compressive strengths of 21, 28, and 35 MPa, the initial- and final-setting time were not significantly different when fly ash replacement for cement was increased up to 55 percent.

3. The water demand of cement paste mixtures decreased with increasing fly ash content. Further decrease in the water demand occurred when were used. Inclusion of fly ash in cement paste mixtures caused small delay in the initial- and final-setting times up to 20% fly ash content depending upon type of chemical admixtures used. Beyond this limit, the setting was accelerated. Fast setting occurred at fly ash content of about 40% and beyond depending upon type of chemical admixtures used.

4. Use of gypsum and water-reducer of normal dosages caused acceleration in the setting of cement paste. However, no appreciable effect of normal dosage of superplasticizer on the setting was observed.

ACKNOWLEDGEMENT

The authors express deep sense of gratitude to the We Energies, Milwaukee, WI for financial support for this investigation. The Center was established in 1988 with a generous grant from the Dairyland Power Cooperative, La Crosse, WI; Madison Gas and Electric Company, Madison, WI; National Minerals Corporation, St. Paul, MN; Northern States Power Company, Eau Claire, WI; Wisconsin Electric Power Company, Milwaukee, WI; Alliant Energy Corporation, Madison, WI; and Wisconsin Public Service Corporation, Green Bay, WI. Their financial support, and support from Manitowoc Public Utilities, Manitowoc, WI, is gratefully acknowledged.

REFERENCES

1. Dodson, V. H., The effects of Fly Ash on the Setting of Concrete - Chemical or Physical. Proceedings of the Symposium on Fly Ash Incorporation in Hydrated Cement Systems, S. Diamond, Ed., Material Research Society, Boston, pp. 166-171, 1981.

2. Ramakrishnan, V., Coyle, W. V., Brown, J., Tiustus, A., and Venkataramanujam, P. C., Performance Characteristics of Concretes Containing Fly Ash. Proceedings of the Symposium on Fly Ash Incorporation in Hydrated Cement Systems, Materials Research Society, Boston, Ed. S. Diamond, pp. 166-171, 1981.

3. Lane, R. O., and Best, J. F., Properties and Use of Fly Ash in Portland Cement Concrete. Concrete International, Vol. 4, No. 7, pp. 81-92, July 1982.

4. Mailvaganam, N. P., Bhagrath, R. S., and Shaw, K. L., Effects of Admixture on Portland Cement Concretes Incorporating Blast Furnace Slag and Fly Ash. Proceedings of the First

International Conference on the Use of Fly Ash, Silica Fume, Slag, and Other Mineral By-Products in Concrete, Montebello, Canada, ACI Special Publication SP-76, Ed. V.M. Malhotra, pp. 519-537, 1983.

5. Majko, R. M., and Pistilli, M. F., Optimizing the Amount of Class C Fly Ash in Concrete Mixtures. ASTM Cement, Concrete and Aggregates, Vol. 6, No. 2, pp. 105-119, 1984.
6. Gebler, S. H., and Krieger, P., Effect of Fly Ash on Physical Properties of Concrete. Proceedings Second International Conference on Fly Ash, Silica Fume, Slag, and Natural Pozzolans in Concrete (SP-91), Madrid, Spain, pp. 1-50, 1986.
7. Carette, G. G., and Malhotra, V. M., Characterization of Canadian Fly Ashes and Their Relative Performance in Concrete. Canadian Journal of Civil Engineering, Vol. 14, No. 5, pp. 667-682, 1987.
8. Bilodeau, A. and Malhotra, V. M., Concrete Incorporating High Volumes of ASTM Class F Fly Ashes: Mechanical Properties and Resistance to Deicing Salt Scaling and to Chloride-Ion Penetration. Proceedings Fourth International Conference on Fly Ash, Silica Fume, Slag, and Natural Pozzolans in Concrete (SP-132), Istanbul, Turkey, pp. 319-350, 1992.
9. Carette, G. G., Bilodeau. A., Chevrier, R. L., and Malhotra, V. M., Mechanical Properties of Concrete Incorporating High Volumes of Fly Ash from Sources in the USA. ACI Materials Journal, Vol. 90, No. 6, pp. 535-544, Nov. – Dec.1993.
10. Malhotra, V. M., and Ramezanianpour, A. A., Fly Ash in Concrete. Second Edition, CANMET, Ottawa, Canada, 307 pages, 1994.
11. Eren, O., Brooks, J. J., and Celik, T., Setting Times of Fly Ash and Slag-Cement Concretes as Affected by Curing Temperature. Cement, Concrete, and Aggregates, Vol. 17, No. 1, pp. 11-17, June 1995.
12. Pinto, R. C. A., and Hover, K. C., Effect of Silica Fume and Superplasticizer Addition on Setting Behavior of High-Strength Mixtures. Transportation Research Board, No. 1574, pp. 56-62, 1996.
13. Samadi, M. M., Hadad, R. H., and Akour, A. M., Potential Use of Phosphogypsum in Concrete. Cement and Concrete Research, Vol. 29, No. 9, pp. 1419-1425, 1999.
14. Brooks, J. J., Effect of Admixtures on the Setting Times of High-Strength Concrete. Cement and Concrete Composites, Vol. 22, No. 4, pp. 293-301, August 2000.
15. Ahmadi, B. H., Initial and Final Setting Time of Concrete in Hot Weather. Materials and Structures, Vol. 33, No. 232, pp. 511-514, October 2000.
16. Targan, S., Olgun, A., Erdogan, Y, and Sevinc, V., Effects of Supplementary Cementing Materials on the Properties of Cement and Concrete. Cement and Concrete Research, Vol. 32,

No. 10, pp. 1551-1558, October 2002.

17. Brooks, J. J., Prediction of Setting Time of Fly Ash Concrete. ACI Materials Journal, Vol. 99, No. 6, pp. 591-597, November-December 2002.

18. Takemoto, K., and Uchikawa, H., Hydration of Pozzolanic Cement. Proceedings of the 7th International Congress on the Chemistry of Cement, Sub-Theme IV-2, Paris, France, pp. 1-28, 1980.

19. Uchikawa, H., and Uchida, S., Influence of Pozzolana on the Hydration of C_3A. Proceedings of the 7th International Congress on the Chemistry of Cement, Sub-Theme IV, Paris, France, pp 24 –29, 1980.

20. Tay, J. .H., Properties of Pulverized Sludge Ash Blended Cement. ACI Materials Journals, Vol. 84, No. 5, pp. 358-364, 1987.

21. Sawan, J. S., and Qasrawi, H. Y., Early Age Properties of Pozzolanic Mortar Under Hot Weather. ASCE Journal of Materials in Civil Engineering, Vol. 3, No. 3, pp. 165-178, 1991.

22. Uchikawa, H., Hanehara, S., Shiraska, T., and Sawaki, D., Effect of Admixture on Hydration of Cement, Adsorptive Behavior of Admixture and Fluidity and Setting of Fresh Concrete Paste. Cement and Concrete Research, Vol. 22, No. 6, pp. 115-1129, 1992.

23. Chen, Y., and Odler, L., On the Origin of Portland Cement Setting. Cement and Concrete Research, Vol. 22, No. 6, pp. 1130-1140, 1992.

24. Matusinovic, T., and Vrbos, N., Alkali Metal Salts as Set Accelerator for High Alumina Cement. Cement and Concrete Research, Vol. 23, No.1, pp. 177-186, 1993.

25. Matusinovic, T., and Curlin, D., Lithium Salt as a Set Accelerator for High Alumina Cement. Cement and Concrete Research, Vol. 23, No. 4, pp. 885-895, 1993.

26. Perret, S., Palardy, D., and Ballivy, G., Rheological Behaviour and Setting Time of Microfine Cement-Based Grouts. ACI Materials Journal, Vol. 97, No. 4, pp. 472-478, Jul.-Aug. 2000.

27. Naik, T. R. and Singh, S. S., Influence of Fly Ash on Setting and Hardening Characteristics of Concrete Systems. ACI Materials Journal, Vol. 94, No. 5, pp. 355-360, 1997.

28. Naik, T. R. and Ramme, B. W., Setting and Hardening of High Fly Ash Content Concrete. Presented at the American Coal Ash Association's 8th International Coal Ash Utilization Symposium, Washington D.C., Oct. 28-31, 1987.

29. Naik, T. R., Singh, S. S., and Ramme, B. W., Time of Setting Influenced by Fly Ash and Chemical Admixtures. Proceedings, Seventh CANMET/ACI International Conference on Fly Ash, Silica Fume, Slag, and Natural Pozzolan in Concrete, Madras, India, July 2001.

Table 1 - Setting Times at Various Cement Replacements with Four Different Class C Fly Ash Sources (Series 1)[27]

Setting Times for Concrete Mixtures Containing (hr:min)								
Cement Replacement, Percent	P-4		DPC		Columbia		Weston	
	Initial Set	Final Set	Initial Set	Final Set	Initial Set	Final Set	Initial Set	Final Set
0	4:35	5:55	4:35	6:00	4:20	5:45	4:20	5:45
10	5:05	6:30	5:00	6:50	6:35	8:20	-	-
20	7:20	9:05	6:15	8:10	-	-	5:25	7:20
30	8:30	10:15	7:30	9:30	10:00	12:35	-	-
40	10:40	12:25	9:20	11:40	-	-	7:05	9:55
50	10:25	13:25	9:40	12:30	12:30	15:40	-	-
60	10:10	13:30	4:45	12:10	-	-	8:00	10:45
70	6:30	9:55	2:30	3:35	12:30	21:40	-	-
80	2:40	3:55	1:30	1:45	-	-	5:50	11:50
90	1:30	1:50	*	*	4:30	7:25	-	-
100	0:40	0:50	*	*	-	-	*	*

* Tests could not be performed - concrete set too quickly.

Table 2– Initial and Final Setting Times of Concrete Mixtures (Series 2)[28]

Mixture Number	28-day Compressive Strength (MPa)	Class C Fly Ash (%)	Time of Setting (hr:min)	
			Initial	Final
Non-Air-Entrained Concrete				
P-1	21	35	6:55	8:30
P-2	21	45	7:45	9:55
P-3	21	55	8:45	11:20
P-4	28	35	7:35	9:25
P-5	28	45	7:30	9:50
P-6	28	55	7:55	10:25
P-7	35	35	6:30	8:15
P-8	35	45	7:15	9:25
P-9	35	55	7:00	9:15
Air-Entrained Concrete				
P-10	21	35	6:40	8:40
P-11	21	45	8:15	10:25
P-12	21	55	7:15	9:45
P-13	28	35	7:30	9:45
P-14	28	45	6:40	9:10
P-15	28	55	6:55	9:30
P-16	35	35	6:45	8:20
P-17	35	45	7:30	9:40
P-18	35	55	5:40	7:10

Table 3 – Setting Times of Cement Paste Incorporating Class C Fly Ash without any Admixtures[29]

Mixture Number	Class C Fly Ash (%)	Times of Setting		Water Content (mL)	Air Content (%)
		Initial (hr:min.)	Final (hr:min.)		
1	0	3:50	4:15	244	6.7
2	10	3:45	4:02	234	6.7
3	20	1:49	2:05	227	6.9
4	30	1:00	2:10	220	7.1
5	40	0:17	0:48	213	7.1
6	50	0:15	0:45	206	7.4

Table 4 – Setting Times of Cement Paste Incorporating Class C Fly Ash and Air-Entraining Admixture[29]

Mixture Number	Class C Fly Ash (%)	AEA (mL)	Times of Setting		Water Content (mL)	Air Content (%)
			Initial (hr:min.)	Final (hr:min.)		
Air-Entraining Admixture (Low-Air Level)						
1	0	1.0	4:10	4:55	196	19.8
2	10	1.0	3:50	4:25	186	18.1
3	20	1.2	4:15	5:00	181	20.0
4	30	1.6	2:00	2:40	173	18.9
5	40	1.6	0:50	2:00	166	18.1
6	50	1.6	0:41	1:48	159	16.8
Air-Entraining Admixture (High-Air Level)						
1	0	1.8	3:00	4:30	198	25.1
2	10	2.2	4:30	5:15	184	24.2
3	20	2.6	4:40	5:25	170	22.4
4	30	3.0	1:50	4:05	164	25.3
5	40	3.6	1:20	2:40	151	23.3
6	50	4.4	0:45	1:35	144	23.0

Table 5 – Setting Times of Cement Paste Incorporating Class C Fly Ash, Water-Reducer and Superplasticizer[29]

Mixture Number	Class C Fly Ash (%)	Times of Setting		Water Content (mL)	Water Content (%)
		Initial (hr:min.)	Final (hr:min.)		
Mixtures Incorporating Fly Ash and Water-Reducer					
1	0	1:47	2:30	164	25
2	10	2:15	3:08	158	24
3	20	2:30	3:55	156	23
4	30	1:06	1:50	155	22
5	40	0:30	1:15	153	21
6	50	0:30	0:50	150	21
Mixtures Incorporating Fly Ash and Superpalsticizer					
1	0	3:15	3:30	164	25
2	10	3:45	4:10	163	24
3	20	2:50	3:25	165	24
4	30	1:10	2:10	166	24
5	40	0:30	0:55	150	21
6	50	0:20	0:25	142	19

Table 6 – Setting Times of Cement Paste Incorporating Class C Fly Ash and Retarder[29]

Mixture Number	Class C Fly Ash (%)	Times of Setting		Water (mL)	Water Content (%)
		Initial (hr:min.)	Final (hr:min.)		
1	0	3:05	3:45	174	27
2	10	3:42	4:22	168	25
3	20	3:00	3:55	166	24
4	30	1:15	2:12	155	22
5	40	0:47	1:47	151	21
6	50	0:18	0:52	146	20
7	60	0:12	0:27	140	19
8	70	0:20	0:47	140	18
9	85	0:10	0:37	139	18
10	100	*	0:14	135	17
[b]11	70	0:28	0:54	137	18
[b]12	85	0:30	0:42	131	17
[b]13	100	0:26	0:35	128	16
[c]14	70	0:17	0:37	131	17
[c]15	85	0:23	0:43	130	17
[c]16	100	0:44	0:54	129	16

Mixtures 1 to 10 - Normal dosage of 150 ml/cwt (3 liq. 0z./cwt)
[b] Mixture Number 11, 12, and 13: Double dose of retarder used
[c] Mixture Number 14, 15, and 16: Triple dose of retarder used
* Set too quickly to conduct test

Table 7 – Setting Times of Cement Paste Incorporating Class C Fly Ash and Gypsum[29]

Mixture Number	Fly Ash (%)	Times of Setting		Water Content (mL)	Water Content (%)
		Initial (hr:min.)	Final (hr:min.)		
1	0	2:50	3:15	186	29
2	10	2:40	3:10	180	27
3	20	1:45	2:30	176	26
4	30	0:50	1:50	170	24
5	40	0:35	1:00	166	23
6	50	0:10	0:30	160	22
7	60	0:15	0:35	157	21
8	70	-	0:15	156	20
9	85	-	0:15	154	20
10	100	-	0:10	153	19
[b]11	70	0:23	0:40	164	22
[b]12	85	0:15	0:27	162	21
[b]13	100	-	0:13	160	20
[c]14	70	0:27	0:35	176	23
[c]15	85	0:08	0:30	174	22
[c]16	100	-	0:13	172	21

Mixtures 1 to 10 - Normal dosage of 3 kg/cwt (4 lbs/cwt), $CaSO_4.1/2\ H_2O$

[b] Mix No. 11, 12, and 13: Double dose of gypsum used

[c] Mix No. 14, 15, and 16: Triple dose of gypsum used.