



EVALUATION OF THE EFFECT OF FLY ASH ON THE CEMENT HEAT OF HYDRATION ACCORDING TO ASTM C1702

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Abstract: In the construction of mass concrete structures, special attention should be given to the cement heat of hydration (HH). The amount of HH released can be substantial that it might compromise the integrity of the structure. Uncontrolled HH can lead to the formation delayed ettringite formation and thermal stresses that may lead to the failure of the structure. One method to control the HH is through the integration of pozzolanic supplementary cementitious materials (PCSM) in the concrete mix that are known to lower the HH. Previous research shows that Fly Ash (FA) can be an effective replacement of cement especially in mass concreting as it not only enhances the compressive strength but also it lowers the HH. However, one study shows that heat released using FA class-C can exceed the HH of plain cement. As such, there is a need to investigate this discrepancy and verify the results. This study presents a re-evaluation of the effect of FA on the HH of a cement HH using a high precision isothermal calorimeter. Eighteen paste mixtures were made for two different types of cements replaced at different percentages using FA classes C and F. The experiments were conducted in accordance with ASTM C1702.

1 INTRODUCTION

The hydration of cement is an exothermic reaction that can sometimes release a substantial amount of heat that can be problematic for the structure. In mass concreting, this amount of heat is considerable that it increases the temperature of the concrete. The problem occurs when a temperature gradient occurs due to the difference in the rate of cooling between the inner core and the outer surface of concrete. As the surface of concrete cools and shrinks faster than its core thermal cracking start to occur. Another problem that is caused by the rise in the temperature of the core of concrete is the formation of the delayed ettringite, which causes an expansion in the volume and cracking of concrete (Botang 2013, Patil 2015, Gajda and Vangeem 2002, Gajda 2016). As such the specifications usually limits the temperature difference between the outer and the inner of concrete to 20°C (36°F) with a maximum temperature of 70°C (158°F) (PCA 1997).

The methods used to treat the HH vary in nature. They can be classified into methods of constructions used and supplementary cementitious materials (SCM) incorporated in the concrete mix. The method of construction includes, precooling of concrete aggregates, post cooling of concrete, insulation of the concrete member, and pouring of concrete in thin lifts (PCA 1997, Botang 2013, Patil 2015, Gajda 2016). Some specifications require the incorporation of SCMS that are known to lower the HH in mass concreting (Alhozaimy et al. 2015). Previous studies addressed the effect of a variety of SCMs on the cement HH; the studies included fly ash (FA), silica fume, metakaolin, granulated slag, and others (Rojas 1996, Rojas and Frias 1995, Cheng-Yi et al. 1985, Frias 2000, Snelson et al. 2008, Persson 1997, Schindler 2003, Langan

2002, Ballim et al. 2009). Fly ash and granulated slag have been proven to be the most effective in lowering the HH of the concrete mix compared to other (Cengiz Duran Atis 2002, Snelson et al. 2008, Sokkary et al. 2004).

There are several factors that influence the amount of HH released, which are: the amounts of tricalcium silicate (C3S) tricalcium aluminate(C3A) in cement, water-cement(w/c) ratio, curing temperature, and cement fineness (Kirby et al. 2012, Orosz et al. 2017). The quantification of HH has been previously done using isothermal calorimetry because of its sensitivity and high precision (Pane et al. 2005, Mostafa et al. 2005, Xu et al. 2006). The sample used for the HH quantifications varied between cement pastes, mortar, and concrete. However, the quality and precision of the obtained raw data depends on the type of isothermal calorimeter used. Hence, it is expected to have variations in results not only because of the differing chemical composition of the material used but also because of the degree of precision of the instrument used. Another source of variation is the standard used for testing the material to quantify the HH. There are different standard tests that can be used to determine the HH. The Standard Test Method for Heat of Hydration of Hydraulic Cement (ASTM C186-05) is a more common test and has been used extensively throughout the literature. This test allows the determination of HH of hydraulic cement at any age, the temperature rise in mass concrete, and if the cement under test meets the HH requirement of the applicable hydraulic cement specification. Another high-precision standard that is specific to isothermal calorimetry is the Standard Test Method for Measurement of Heat of Hydration of Hydraulic Cementitious Materials Using Isothermal Conduction Calorimetry (ASTM C1702-17). However, it has not been very common in determining the HH of cementitious materials according to the literature reviewed.

2 PROBLEM STATEMENT

Although the literature contains several studies that focused on the evaluation of effect of FA on the HH of cement, the results are quite inconclusive. Most studies conclude that the incorporation of FA in the concrete mix can lower the HH significantly. However, one study shows the addition of FA class-C to the mix can increase the HH over time compared to plain Portland Cement (PC) (Schindler and Folliard 2003). This study is dated to 2003. The ability to measure and record the HH has drastically has changed since then as more accurate instruments and knowledge have become available.

3 OBJECTIVE

The purpose of this study is to re-analyze and re-evaluate the effects of different classes of FA on the HH of two different types of cements, namely type I, and III using a high precision isothermal calorimeter Calmetrix I-Cal2000. The study aims at drawing a conclusion on the performance of FA class-C since the results regarding its effect on the cement HH is quite contradictory. The study also serves as an alternative evaluation of the effect of FA class-F using ASTM C1702-17 standard, which is specific for isothermal calorimetry.

4 LITERATURE REVIEW

The effect of the FA on the cement HH has been studied throughout the years. The earliest study goes back to Meland (1983). This study investigated the effect of using FA on the HH when used as 10% and 20% cement replacements. The study investigated the HH of a plain cement pasted, a mixture that incorporated FA as a cement replacement in 10% and 20%, and a mixture that included FA and plasticizer. The results showed that both mixtures that contained FA released lower HH compared to the control mix (Meland, 1983). Tokyay (1988) assessed the effect of three different types of Turkish fly ash on the cement HH. The FAs were used as 10%, 20%, and 40% cement replacements. The study concluded low-calcium FA is more effective at reducing the HH than the high-calcium types. In 1993, De Rojas et al. conducted a study to evaluate the effect of FA on the HH when used as a partial replacement of cement. The results show that increasing the percentage of FA reduces the temperature of the mixture. De Rojas and Frias (1995) conducted another study to compare the effect of FA versus Silica Fume (SF) on reducing the HH. The results showed that FA is more effective than SF. Several other studies concluded the same results

about the effectiveness of FA in decreasing the peak temperature and the total HH released (Frias et al. 2002, Atis 2002, Maia et al. 2011, Alhozaimy et al. 2015).

Some other studies focused on the effect of the water/binder (w/b) ratio on the HH of mixtures that incorporated FA. As study by Langan et al. (2002) showed that increasing the w/b ratio delay the peak temperature and decrease the heat evolution for mixtures that contained FA class-C. On the contrary, the results obtained from Pane and Hansen (2005) show that decreasing the w/b ratio lowers the HH. The effect of FA combined with other SCMs, such as slag and metakaolin was tested. The results show that the FA when combined with slag becomes very effective in reducing the HH (Lee et al. 2014). However, the HH tends to increase in the presence of metakaolin (Snelson et al. 2008).

A study was conducted to compare the effect of different classes of Fly ash, namely class F and C, on the HH of a cement mixture (Schindler and Folliard 2003). The study sheds the light that the performance of FA varies based on the amount of the calcium compounds contents it includes. The study concluded that FA class-F is more effective in reducing the HH compared to class-C. In fact, FA class-C tends to increase the amount of HH released compared to a plain cement mixture due to the high contents of calcium (Schindler and Folliard 2003).

Class-C and class-F FA were both tested and compared to each other in mixtures that contained replacements of 15%, 25%, and 35% of PC (Schindler and Folliard 2003). Both class-C and class-F fly ashes with the highest replacement amount (35%) had the lowest adiabatic temperature rise when compared to the control sample (100% cement). However, after 40-50 hours, the heat release of the samples made with class-C FA showed an increase exceeding the HH released by the control sample. It was concluded that class-F FA is a better option for reducing the HH than FA class-C.

5 METHODOLOGY

A high-precision isothermal calorimeter was used to ensure the accuracy and consistency of the results, and the repeatability of the experiments in a controlled test environment. The isothermal calorimeter contains two separate channels that can accommodate two samples for testing at the same time. The tests were performed according to the ASTM C1702, which provides guidelines for measuring the heat of hydration of hydraulic cementitious materials specific to using an isothermal calorimeter. To conduct an ASTM C1702 test, the sample water is preconditioned for 2 hours at 23° C. After the water calibration is completed, the sample mix is made, and the samples are placed inside the isothermal calorimeter channels. With the software provided by the isothermal calorimeter manufacturer, the experiments are left running for 24 hours. As a result, raw data of the heat flow produced by the cement hydration reaction is generated and stored. The generated raw data can then be used to create thermal power and cumulative energy graphs for a more in-depth heat of hydration analysis.

According to ASTM C1702, a mixture sample is comprised of 50 grams of the cementitious binder mixed with water at a w/b ration of 0.5. In this research two different types of Portland Cement were considered, which are: Type I and III. FA class-F and class-C was used in different percentages as mass replacements of the different cement types. The replacement percentages used were 10%, 20%, 30% and 40% based on the percentages used in the literature. As such, eighteen mixtures representing the different combinations of cement type, FA class, and percentage replacements are needed to accomplish the goals of this study.

6 EXPERIMENTATION AND RESULTS

The heat flow of the different mixtures was measured using isothermal conduction calorimetry for 24 hours at constant temperature of 23° C According to ASTM C1702. Thirty-two different curves were generated reflecting the thermal power over time and the total heat released by the eighteen mixtures.

6.1 Cement Heat Curves

In order to evaluate the effect of different classes of FA on the HH, it is essential to generate the basic heat curves of the different types of cements that are used in the study as a medium for assessment for comparison purposes. The heat flow and heat of hydration curves for cement type I and III are given in Figures 1a and 1b, respectively. In agreement with the literature, the thermal power measured revealed that cement type III generates a rapid heat evolution in contrast with cement type I. According to the ASTM C 186 standards, cement type III reaches a sulfate depletion point at the time of 7 hours after hydration. The sulfate depletion point consists of a seizure formed by the action of aluminate activity due to the lack of a SCM in the mixture (Caldarone 2008).

6.2 Fly Ash Class-C

6.2.1 Cement Type I

The effect of FA class-C on the HH of PC Type I is shown in Figure 2. The results show that increasing the amount of FA class-C in the mix decreases the heat released and delays the peak temperature. The heat flow curves -see Figure 2 a- show that the increasing the replacement percentages extend the dormant period. This is due the prolonged reaction of Calcium Sulfate (CaSO_4) with Tricalcium aluminate (C_3A) that occurs during the dormant period. Also, the higher the percentage of FA class-C incorporated in the mix is, the longer the delay in the initial and final setting time will be.

To further analyze the effect of fly ash C on the HH of cement Type I, a cumulative energy (HH) graph was generated, as shown in Figure 2b. The results of this experiment confirm the results obtained from previous studies that assessed the impact of FA in cement mixtures on the HH, as in (Meland 1983, Tokyay 1988, De Rojas et al. 1993, De Rojas and Frias 1995, Frias et al. 2002, Atis 2002, Maia et al. 2011, Alhozaimy et al. 2015). However, it still contradicts the results reported by Schindler and Folliard in (2003), that indicated that using FA class C in cement mixtures increases the HH.

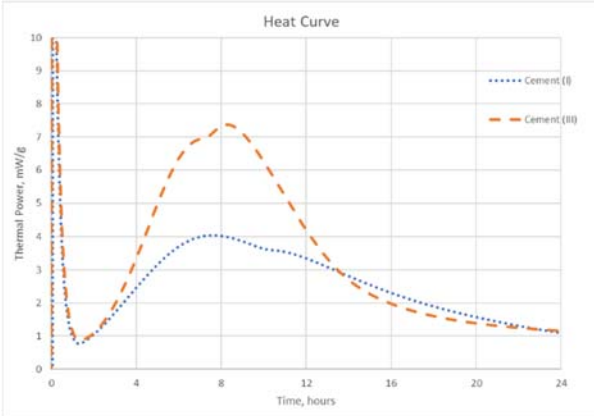


Figure 1a. Heat flow of cement type I and III

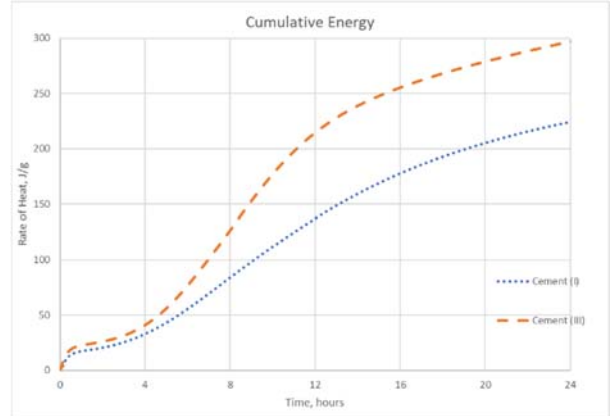


Figure 1b. Heat of hydration of cement type I and III

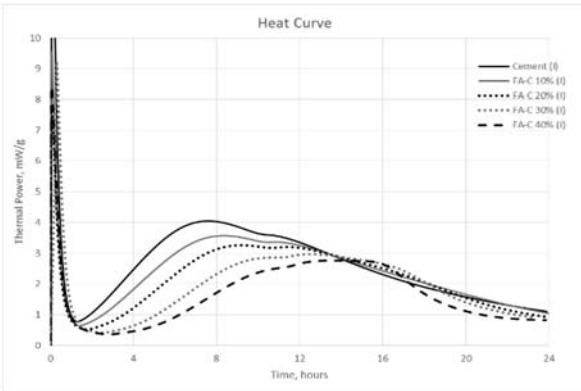


Figure 2a. Heat flow of cement Type I mixed with FA-class C

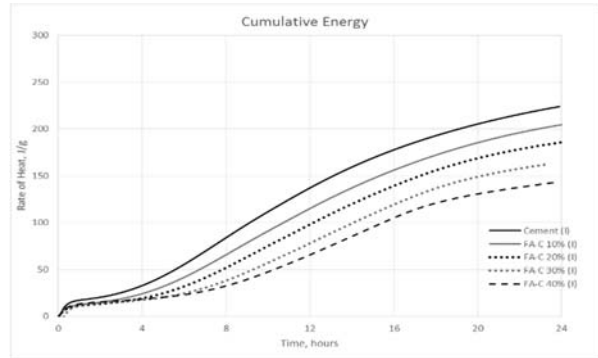


Figure 2b. Heat of hydration of cement Type I mixed with FA-class C

6.2.2 Cement Type III

The heat curves in Figure 3a show the contrast of heat release between different FA class-C replacement percentages of PC Type III. It is very clear that the heat evolution for PC Type III is higher than in Type I. Consistent effects are noticed on the HH of PC Type III with increasing the replacement levels of FA class-C. The highest heat index occurred at 10% FA class-C replacement level, which produced a thermal power of 6.7 mW/g. The increase in percentage of FA class-C replacement decreases the heat evolution; at 40 % FA class-C replacement, the heat curve lowers drastically. Additionally, the sulfate depletion and acceleration of aluminat activity are extended, which extend the dormant period. Similar to the effect of PC Type I, the setting time is increased with increasing the replacement level of FA class-C.

The cumulative energy (HH curve) collected from the isothermal calorimetry test is shown in Figure 3b. It can be observed that the OPC type III yield higher rate of HH, thus greater early compressive strength compared to PC Type I. Also, it should be noted that the FA class-C replacement maintained lower level of HH compared to OPC (Type I). In general, it can be deduced that the higher the percentage of FA class-C replacing PC Type III, the lower the HH will be.

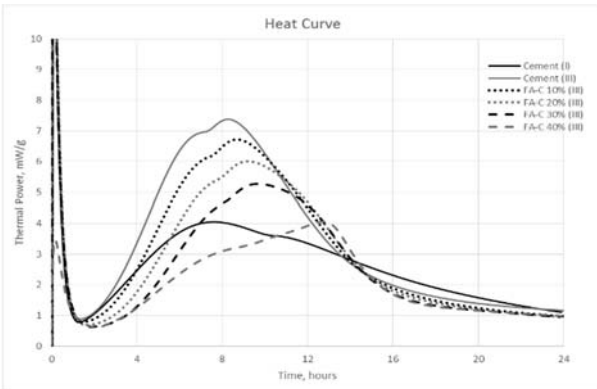


Figure 3a. Heat flow of cement Type III mixed with FA-class C

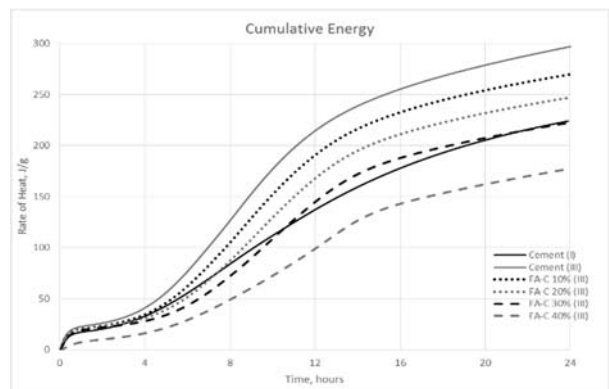


Figure 3b. Heat of hydration of cement Type III mixed with FA-class C

6.3 Fly Ash Class-F

6.3.1 Cement Type I

The results obtained from the isothermal tests for the replacement of Portland cement type I with FA class-F are shown in Figure 4. The effect of FA class-F on the HH of PC Type I is consistent with the results from the literature. The higher the amount of FA class-F used for replacing PC Type I, the lower the HH will be. The highest and the lowest heat peak recorded was at 3.6 mW/g and 2.8 mW/g using 10% and 40% FA class-F replacements, respectively. Also, the setting time is affected as well, the increase in replacement level slightly delays the final setting time. The main hydration peak span decreases in length drastically from 10% replacement level to 20%. This means that the dormant period lasts longer higher replacement level of FA class-F.

The cumulative HH data obtained from the isothermal test on the replacement of PC Type I with FA class-F, is shown in Figure 2b. The cumulative energy graph is formulated by integrating the thermal power graph. As shown, there is a significant drop of energy from 198 J/g at 10% replacement level to 158 J/g at 20% replacement.

6.3.2 Cement Type III

Portland cement type III has higher heat release than other types of PC. Nonetheless, FA class-F has similar effects on PC Type III compared to other type of cements. The HH of mixtures containing FA class-F as a replacement of PC Type III in the amounts 10%, 20%, 30%, and 40% were obtained using isothermal conduction calorimetry, as shown in Figure 5. The thermal power graph (Figure 5a) shows that increasing the amount of FA class-F decreases the amount of heat released. Also, the dormant period is prolonged by increasing the replacement percentages delaying the peak temperature. The delayed heat peaks (slightly shifted to the right) indicates that there is a small delay in the final setting time. of the cementitious materials when increasing the replacement of FA class-F. It should be noted that increasing the replacement percentage of FA class-F from 30% to 40% does not decrease the heat peak, but rather delay it.

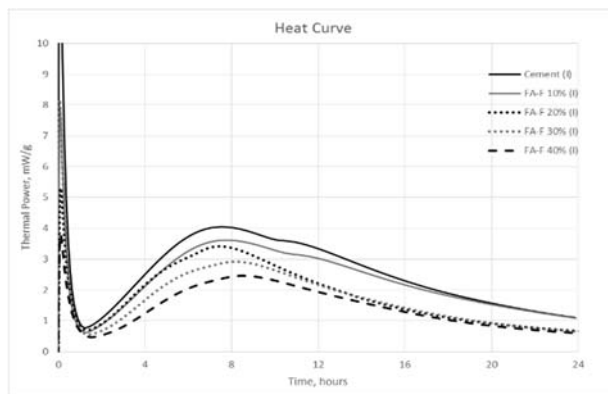


Figure 4a. Heat flow of cement Type I mixed with FA-class F

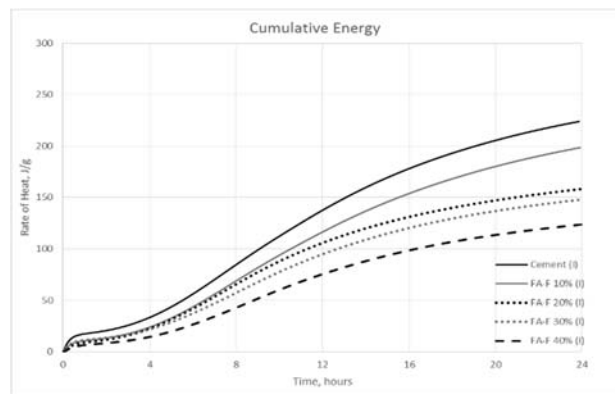


Figure 2b. Heat of hydration of cement Type I mixed with FA-class F

The cumulative HH curves for PC Type III mixtures made with FA class-F is given in Figure 5b. The energy released in these experiments is higher than with PC Type I. A pattern can be observed; when the replacement level of fly ash F increases, the HH decreases.

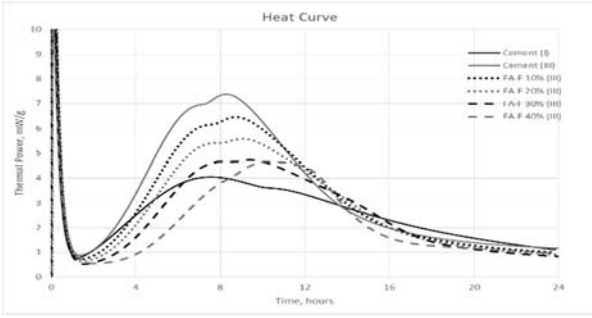


Figure 5a. Heat flow of cement Type III mixed with FA-class F

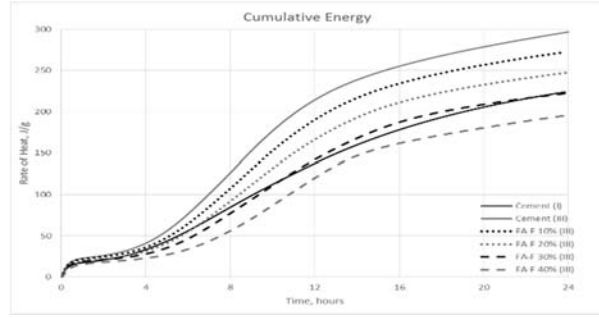


Figure 2b. Heat of hydration of cement Type III mixed with FA-class F

7 ANALYSIS AND FINDINGS

From the results it can be determined that PC Type I and Type III have similar patterns. However, when comparing the maximum heat flow from both cements, PC Type III recorded 7.4 mW/g and PC Type I reached to 4 mW/g, as shown in Figure 6. There is a substantial difference in heat peaks between these two cements. Nonetheless, both cements peaked at an indistinguishable amount of time. PC Type I peaked after 8.6 hours, where PC Type III peak occurred at 8.2 hours of testing. Among other similarities, the dormant periods for both cement types lasted for about 6 hours. The total HH differs when comparing PC Type I and Type III. PC Type I had a total HH of 224 J/g, in contrast, Type III recorded 311 J/g, see Figure 7.

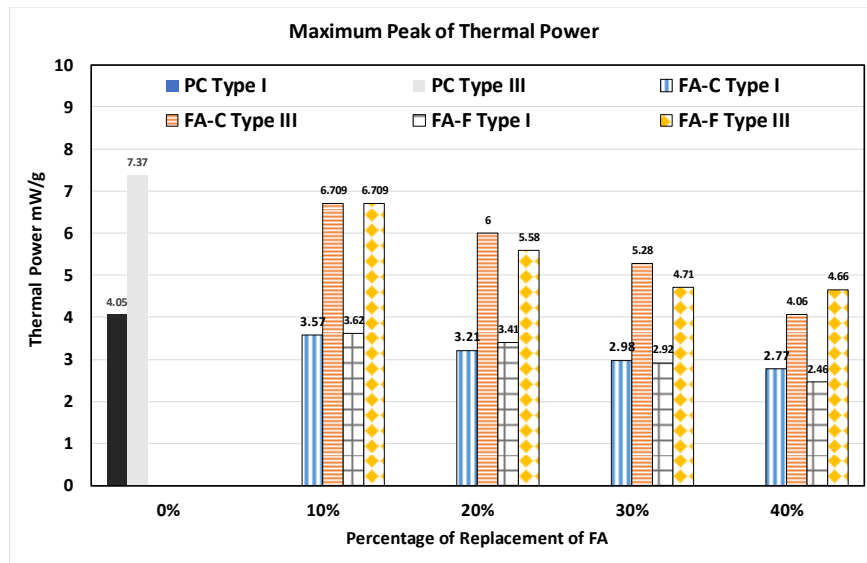


Figure 6. Comparison of maximum peaks of thermal power of PC Type I and III and samples containing different replacement levels of FA-C and FA-F

PC Type I with FA class-C recorded a maximum value of heat flow of 3.57 mW/g at 10% replacement level. The heat peak occurred at 10.8 hours of isothermal calorimetry testing. Also, the maximum total HH reached 229 J/g at 10% replacement percentage. In addition, the duration of the dormant period begins to extend continuously with the increment of FA class-C replacement level. The durations of the dormant periods range from 6.4-10.5 hours. In contrast, PC Type I with FA class-F recorded a maximum thermal power of 3.62 mW/g at 10% replacement level. The maximum heat index was recorded after 8.7 hours of testing. Furthermore, the maximum total HH recorded for PC Type I with FA class-F was 198 J/g at 10%

replacement level. The dormant period slightly extends when the replacement level increases. The dormant period durations span from 6.3-7 hours, see Table 1.

Table 1: FA class C and F samples run with PC Type I

Parameters	PC Type I									
	FA class-C					FA class-F				
	0%	10%	20%	30%	40%	0%	10%	20%	30%	40%
Peak of Heat Flow (mW/g)	4.05	3.57	3.21	2.98	2.77	4.05	3.62	3.41	2.92	2.46
Time of Max Peak of Heat Flow (hours)	8.6	10.8	12.45	15.47	14.4	8.6	8.65	7.47	8.2	8.56
Max Total HH (J/g)	223.94	228.66	185.95	162.47	143.26	223.94	198.48	158.47	147.9	123.87
Dormant Period (hours)	6.35	7.17	9.65	10	10.46	6.25	6.35	6.16	6.82	7

PC Type III with FA class -C recorded a maximum value of heat flow at 6.709 mW/g at 10% replacement level. The maximum heat peak occurred at 8.2 hours of isothermal calorimetry testing, and it had the maximum total HH with 180.84 J/g. The dormant period, which is the stage before the hydration acceleration, range from 1.5-3.5 hours. On the other hand, PC Type III with FA class -F recorded a maximum thermal power of 6.46 mw/g at 10% replacement level, and it was recorded at 7.35 hours of testing. Furthermore, the maximum total HH was gotten from the 10% FA class-F replacement level with 272.52 J/g. The dormant period for most of the tested samples had a duration span ranging from 1 and 3.3 hours, with the exception of the 40% replacement level that had an extended dormant period between 1.5 and 4.2 hours, see Table 2.

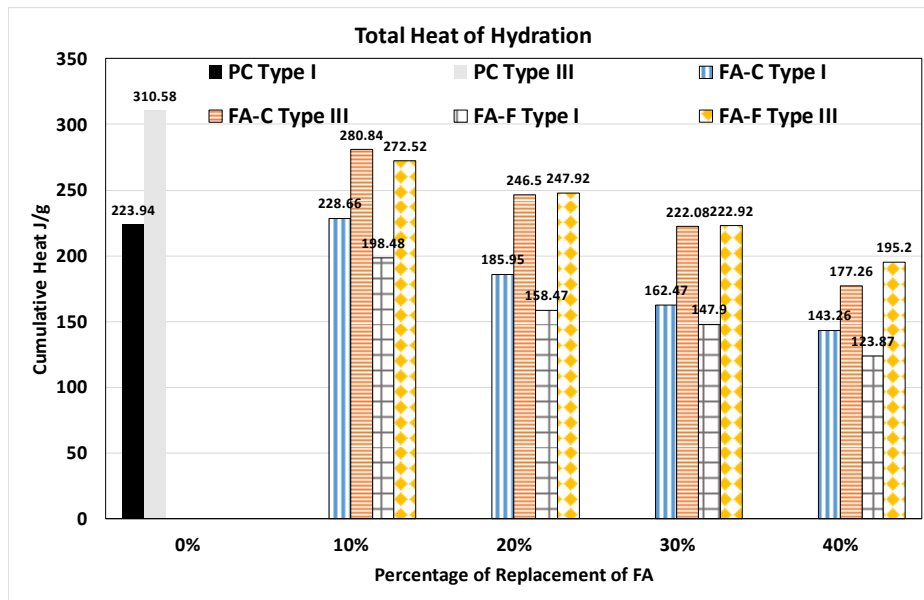


Figure 7. Comparison of the total HH comparison of PC Type I and III and samples containing different replacement levels of FA-C and FA-F

In comparison with PC Type I results, both follow the same pattern, as the percent replacement increases, the lower the maximum value of heat flow. Nevertheless, values for PC Type III are higher, because of its higher Tricalcium Aluminate (C3A) content, which is characterized for providing rapid strength at an early age by releasing large amounts of heat during the first days.

Table 2: FA class C and F samples run with PC Type III

Parameters	PC Type III									
	FA class-C					FA class-F				
	0%	10%	20%	30%	40%	0%	10%	20%	30%	40%
Peak of Heat Flow (mW/g)	7.37	6.709	6	5.28	4.06	7.37	6.46	5.58	4.71	4.66
Time of Max Peak of Heat Flow (hours)	8.2	8.65	9.32	9.73	12.85	8.2	8.75	9.1	9.5	10.4
Max Total HH (J/g)	310.58	280.84	246.5	222.08	177.26	310.58	272.52	247.92	222.98	195.2
Dormant Period (hours)	6.83	7.2	7.65	7.88	10.55	6.83	7.35	7.63	7.95	8.36

8 CONCLUSIONS

This study focusses on the evaluation of the effects of fly ash on the cement HH with the purpose of updating previous results using a high precision isothermal calorimeter. Based on the results and findings of this study, the following conclusions can be drawn:

1. The thermal power of PC constantly decreases when it is replaced with FA. The larger amount of PC replaced with FA, the lower the peak temperature rises.
2. PC with FA class-C has higher heat evolution than PC with FA class-F. However, there is a difference in maximum heat index between PC Type I and Type III. PC Type III has significantly higher heat evolution in comparison with Type I. Therefore, from the tests performed, PC Type III replaced with FA class-C have the higher heat outputs.
3. The maximum heat peak occurrence delays in time the higher the replacement level of FA on PC. Nevertheless, in PC with FA class-C the maximum heat peaks delays much more than in PC with FA class-F.
4. The total HH is decreased with the increase of replacement of PC with FA. The higher the replacement level, the lower the maximum HH.
5. The dormant period extends with the increment of replacement of FA in PC. FA class-C dormant period extends in larger intervals of time, where in FA class-F, the dormant period extends, but not
6. Significantly.

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