INVESTIGATING THE USE OF THE ADIABATIC CALORIMETRY TO CHARACTERIZE CONCRETE ROAD PAVEMENT MIXES

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ABSTRACT

Determining in-situ concrete properties can be a challenge due to limitations on current test methods where laboratory conditions and in-situ conditions are not the same. The heat released from cementitious materials during the exothermic hydration process can be used to characterize important properties of different mix designs for concrete pavements. In this investigation, an adiabatic calorimeter for concrete mixes was used to measure the real time heat of hydration of concrete mixes for road pavements. The adiabatic calorimeter captures the true temperature of concrete specimens with minimal heat exchange between the concrete and surroundings to determine real-time concrete heat properties. This study investigates three different concrete pavement mix designs that incorporate CEM II 32.5 R, CEM II 42.5 N and CEM I 52.5 N cements respectively. The heat of hydration and the correlation with concrete properties such as 28-day flexural strengths, 7-day, and 28-day compressive strengths, initial and final setting times are discussed in this paper. The results indicate a high correlation ($0.91<R^2<0.99$) between the compressive strength and heat of hydration of the investigated mixes. Low regression values ($0.004<R^2<0.023$) were found for setting time and flexural strength ($R^2=0.0911$). The results also suggest high correlation between the heat flux of the concrete mixes and setting times ($0.95<R^2<1$) and flexural strength ($R^2=0.80$).

1. INTRODUCTION

Empirical tests for determining important concrete properties, such as the compressive strength and setting time remain standardized for the design and construction of concrete pavements. The properties of concrete used for road pavement mixes depend on the cementitious reactions that occur during concrete hydration. One of the challenges with concrete pavements is the temperature changes that can cause thermal cacking during hydration of the concrete (Fultons, 2010). The temperature rise in cement mortars and concrete can be measured through calorimetry testing. The rate of heat of hydration has been shown to provide useful correlations to cement paste properties such as the 28-day compressive strength, initial and final setting times (Mokoena et. al., 2021). Different types of cement with different strengths, additives and compositions are available in South Africa and can be used for pavement concrete designs. The heat of hydration associated with these cements differs and can be measured by means of calorimetry tests. In this study, concrete pavement mix designs were developed using CEM V/A (S-V) 32.5 R (Mix 1), CEM V/A (S-V) 42.5 N (Mix 2), and CEM I 52.5 N (Mix 3) according to ACI 211 test method. CEM I and CEM II along with blends of ground granulated blast furnace slag and fly ash that adhere to SANS 50197-1 and SANS 1491 are suitable to use for the construction of concrete pavements. According to Committee of Transport Officials (COTO), the compressive strength of a concrete pavement should be at least 35 MPa in 28 days, with a flexural strength 4.5 MPa. The cements used in this study are all Portland composite cements from the same supplier but vary in strength and level of extender contents used. This is to evaluate how properties such as the compressive and flexural strengths are related to the heat of hydration as the concrete cures. This study includes a correlation of these properties including the initial and final setting times of each cement type with the heat of hydration.

1.1 Aim of Paper

Typical concrete tests often provide empirical results and can be time-consuming. A method for determining required concrete properties from the heat of hydration during the setting stage of concrete can therefore assist in characterising concrete mixes based on analytical methods that is less labour-intensive.

The objective of this study is to assess and correlate selected concrete properties of different concrete pavement mixes and the heat parameters as a method to characterize concrete mixes. The proposed method is based on the heat of hydration of the concrete mixes during the setting phase of concrete. This method may assist in determining crucial concrete properties at an early stage without the typical 28-days that is usually required to determine important strength parameters for acceptance of the concrete mix.

1.2 Scope of the Paper

Concrete pavement mixes were prepared in the laboratory under controlled conditions, where temperature plays a key role (Singh, 2020). For each concrete mix, the following concrete properties were investigated:

- Heat flux.
- Heat of hydration.
- 7-Day compressive strength.
- 28-Day compressive strength.
- 28-Day flexural strengths.
- Initial setting time.
- Final setting time.

The material used for this investigation includes coarse and fine aggregates properties which also influence the strength and heat of hydration of the concrete mixes.

2. BACKGROUND

2.1 Heat of Hydration

When cement is mixed with water, the mixture releases heat at a fast rate within the first few minutes but promptly decelerates, as shown in Figure 1. This quick rise of heat is seldom considered because it occurs before all ingredients are properly mixed, and it has minimal effect on structures. After the initial reaction, the concrete mix remains in a dormant phase for approximately two hours. The initial set takes place as more heat is generated until the final setting time occurs (Ballim, 2003).

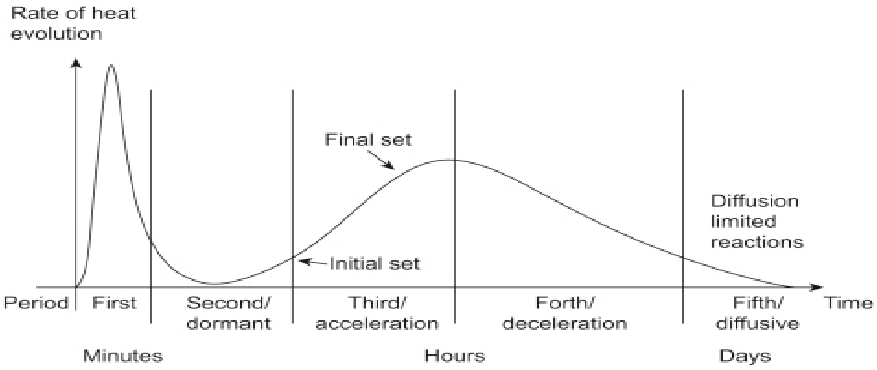


Figure 1: Heat of hydration curve (Vazquez & Pique, 2016)

2.2 Calorimetry Testing

The temperature rise in cement mortars and concrete can be investigated using different calorimetry tests available namely (i) isothermal, (ii) semi-adiabatic, and (iii) adiabatic calorimeters, depending on the temperature conditions and heat exchange applied during testing. Isothermal calorimeters enable the measurement of heat produced by samples maintained at a constant temperature, while the semi-adiabatic calorimeter measures heat of hydration with limited heat exchange with the external environment but is not completely prevented. Adiabatic calorimeters measure the temperature rise in a sample with minimal heat exchange with the external environment (Ballim & Graham, 2009).

Adiabatic calorimetry describes a method whereby the temperature conditions of where the specimen is kept are controlled either by a heated water jacket, heated air around the specimen or heated containers. Semi-adiabatic uses insulation alone to slowdown the rate of heat loss from concrete specimen; no additional heat source is utilized to increase the device’s efficiency. The insulation can either be provided by thermos flask or those placed in polystyrene cube moulds (Springenschmid, 2004). Both calorimeters are used to determine the adiabatic temperatures of concrete specimen although semi-adiabatic is easier to use and the adiabatic calorimeter provides more accurate heat of hydration curves (RILEM, 1997). The loss of heat should be minimized to achieve adiabatic conditions. Since the cement hydration rate is accelerated by temperature to the extent of twice for every 10°C, any significant departure from adiabatic conditions will result in the cement being exposed to a lower temperature than it should and prevent the proper rate of reaction from occurring. Adiabatic test conditions for concrete mixes are achieved when the sample's temperature loss does not exceed 0.02 K/h (Springenschmid, 2004). In comparison to the isothermal and semi-adiabatic calorimeter, the adiabatic calorimeter offers realistic simulation of the heat of hydration in concrete structures where the heat generated is not lost to the environment but contributes to the concrete’s temperature thus affecting the heat evolution (Ballim & Graham, 2009).

An adiabatic calorimeter is, in theory, one in which heat is contained inside the calorimeter, typically by enclosing it in an adiabatic shield that is kept at the calorimeter's temperature. In actuality, during the experiment, temperature gradients in the calorimeter and shield result in a net heat exchange. Measurements taken at different intervals throughout an experiment are typically used to account for heat exchange. For instance, in the intermittent heating method, drift rates, heat transfer coefficients, and the empty calorimeter's heat capacity, determined at a later stage, all play a role in the thermal corrections made to the energy input during the experiment (West, 1963). Currently in South Africa, there is currently the Wits calorimeter by Ballim and Gibbon (1996) whereby one litre sample of concrete is placed in a water bath so that the sample and the water which are separated by a stationary pocket of air. The calorimeter is attached to a computer which uses an analogue-to-digital conversion card to monitor the signal from a temperature probe inserted into the concrete sample. The computer can automatically turn the heater on or off to keep the temperature the same (Ballim & Graham, 2009).

Figure 2 shows the new age adiabatic calorimeter which provides an instantaneous measurement of heat of hydration and heat generation of concrete during hydration. The calorimeter cell aids the equipment with minimized heat loss from the concrete for true adiabatic conditions and measures the heat of hydration considering the temperature reached by the concrete. It measures the heat evolution of concrete within 3 to 7 days. The adiabatic calorimeter has two probes which measure the temperature of the sample and the chamber. The equipment measures concrete temperatures, cell temperature, intrinsic temperature rise, and cumulative heat development at specified time intervals.

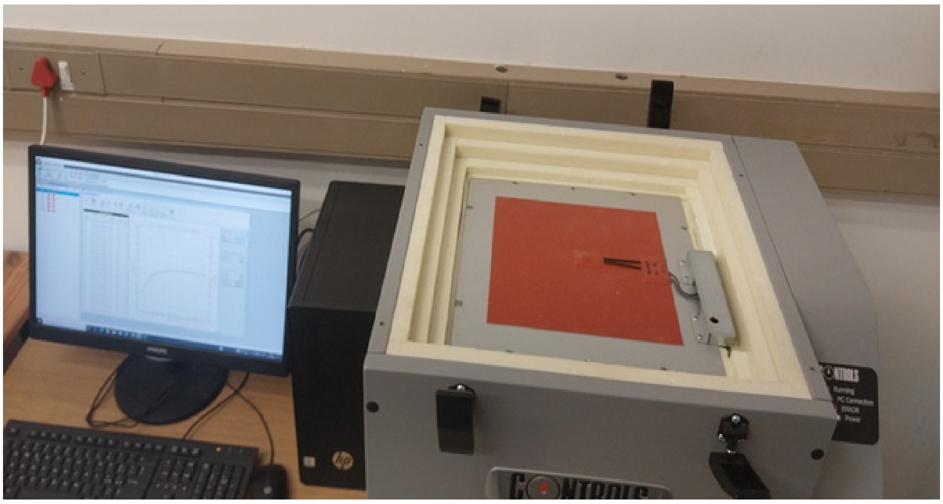


Figure 2: Adiabatic calorimeter at CSIR

Calorimetry tests can offer more accurate results for characterization by measuring the heat of hydration of cement and concrete compared to empirical tests. Semi-adiabatic calorimeters have been shown to be more effective compared to isothermal and full adiabatic calorimeters in terms of thermal testing and are often more economical (Weakley, 2010). Some older models of adiabatic calorimeters are considered difficult because of the unaccounted temperature loss that occurs during testing (Christensen, 2006). The adiabatic calorimeter used in this study accounts for heat losses through an external insulating enclosure and calorimeter cell conditioning system controlled by a Proportional Integral Derivative (PID) closed loop system. Studies have shown how the temperature of concrete during hydration has a strong correlation with the strength development and these have been successfully proven using different methods of measuring heat in different curing conditions (Wang et al, 2008).

The National Concrete Pavement Technology Centre developed a framework for monitoring the performance of concrete pavements through calorimetry equipment in three phases. The goal of their study was to find, create, and assess a low-cost, dependable calorimetry instrument and test procedure for monitoring the evolution of pavement concrete's heat.

The first phase conducted in 2006 identified the user needs for the calorimetry tests and application of calorimeter test results. It also investigated the current test protocols for determining the heat of hydration of concrete using calorimetry and other techniques. It was determined that the heat evolution test results can be used for predicting concrete maturity and strength, setting times, mix design proportions and showing changes in cementitious materials. In 2008, the second phase was done to develop a standard test procedure and establish test methods for interpreting calorimeter test results. It was determined that the isothermal testing device was easier to apply and repeatable. The conclusions made were that calorimeter tests can be used to distinguish between the heat evolution of mortars composed of various materials and under various curing conditions. It can detect cementitious changes and material compatibility and predict setting times and strength gain.

The third phase focussed on confirmation of the tests on larger applications and to develop specifications for calorimeter testing of *in-situ* concrete (Wang et al, 2008).

2.3 Effects of Aggregates

Aggregates make up 70% to 80% of the concrete volume and their effect has a major influence on the properties of concrete. The same concrete mix design with same cement quality and different coarse aggregates can therefore result in different properties. The difference in concrete performance due to different aggregates is influenced by their mineralogical composition and the proportions used. The physico-mechanical properties of aggregates play a significant role and are therefore classified for different concrete applications and properties. Aggregates can be classified into three groups. Group I is ultramafic rocks which represent lowest concrete strengths due to their high alteration degree and poor mechanical parameters. Group II is the mafic rocks which represent a wide range of concrete performance based on the petrographic properties. Group III is the albite rocks which represent highest concrete strengths due to their low degree of alteration and superior mechanical properties (Petrounias et al, 2018).

The heat of hydration of concrete is influenced by the tricalcium silicate and tricalcium aluminate of cement. Dolomite aggregates, specifically fine aggregate improved the compressive strength and create a dense matrix through its pozzolanic cementitious properties. Dolomite powder, when used as cementitious additive tends to lower the heat of hydration due to the smaller particle size that increases the surface area available to contribute to the cementitious reactions. (Szybilski & Nocuń-Wczelik, 2015). The influence of dolomite aggregates is understood to be mostly positive due to their sound petrographic characteristics, medium degree of alteration and physico-mechanical properties. However, the angular shape and rough surfaces of dolomite aggregates tend to have a negative effect on the fresh properties of concrete such as consistency, which results in an increase in water demand to achieve the desired consistency (Petrounias et al, 2018).

3. METHODOLOGY

3.1 Concrete Mix Design

The American Concrete Institute’s (ACI) Standard Practice for selecting proportions for normal, heavyweight, and mass concrete (ACI 211.1-91) was used to develop the concrete pavement mix proportions. The selected method used for this study was based on the structural requirements for concrete pavements. According to COTO, the minimum 28-day compressive strength for concrete pavements is 35 MPa. The ACI method follows nine steps of proportioning cementitious materials, water, fine and coarse aggregates. A target slump of between 25 mm and 75 mm mm was specified for concrete pavement applications. The nominal aggregate size was selected based on the consolidation and compaction factors that may cause honeycombing or large voids. The maximum nominal size used was 20 mm. Subsequently, the mixing water and air content was estimated based on the maximum nominal size, particle shape, grading analysis, concrete temperature, amount of entrained air and the use of admixtures. A water-cement ratio of 0.48 was then selected to comply with COTO standard specification for concrete pavements. The aggregates were oven-dried at 110ºC overnight until a constant aggregate mass was achieved before mixing. Mix design trials were conducted in the laboratory to determine the final concrete mix designs.

Three concrete mixes for pavements were investigated, with the same mix design proportions and varying cement types, all sourced from the same supplier. This was to eliminate all other variables except cement type. CEM II 32.5 R, CEM II 42.5 N and CEM I 52.5 N were used as the only cementitious material as per Table 1. No extenders or admixtures were incorporated into the mixes. Crusher dust and natural coarse (dolomite) aggregates which abides to the COTO specifications and SANS 1083 were used for this study.

Table 1 shows the three concrete mix quantities used in this study. The cement classes include CEM II 32.5 R which is a Portland-composite cement extended with siliceous fly ash, blast furnace slag and limestone. The second concrete mix is made of the Portland-limestone cement CEM II/A-L 42,5 N and lastly Portland Cement CEM I 52,5 N.

Table 1: Concrete mixes quantities

Concrete mix	Cement type	Quantities per (kg/m^3)			
		Cement	Coarse aggregates	Fine aggregates	Water
Mix 1	CEM II 32.5 R	360	849	1035	178
Mix 2	CEM II 42.5 N				
Mix 3	CEM I 52.5 N				

Dolomite aggregates which fall under Group II were used for the experimental work. Hydrochloric acid was used to confirm the geology of the aggregates received from the quarry. Cold hydrochloric acid reacts mildly with dolomite, forming a drop of acid on its surface. However, heated acid results in a visible fizz due to higher temperature reactions. The latter reaction occurs because acid and rock react more vigorously at higher temperatures (Ivanishin & Nasr-El-Din, 2021).

3.2 Concrete Preparation and Standard Testing

Eighteen 100 x 100 mm cubes were prepared and cured as per SANS 5861-1, SANS 5861-2 and SANS 5861-3. The 7- and 28- day compressive strengths were determined as per SANS 5863. Three 100 x 100 x 500 mm beams per mix were also prepared and tested for the 28-day flexural strengths according to SANS 5864 using two-point method. The test method used for determining the initial and final setting times was SANS 50196-3 which is based on the penetration resistance of the three different cement pastes.

3.3 Adiabatic Calorimeter Testing

The adiabatic calorimeter test method follows the EN 12390-15 European standard. The concrete ingredients were stored in the same room as the calorimeter set at a constant temperature of 22°C for at least 24 hours before mixing. A concrete mixer was then used to mix the cement, fine, coarse aggregates and water. The concrete was then cast into a polystyrene mould according to SANS 5861-3 and compacted using a vibration table to eliminate air-entrained according to the test method as illustrated in Figure 3. The masses of the individual ingredients, fresh concrete, room temperature, the time water was added into the dry ingredients, concrete temperature after mixing were all recorded into the adiabatic calorimeter software before starting the test. Results, including the cement's heat of hydration were then calculated within the software based on the concrete mix input parameters and heat parameters of the calorimeter.

Figure 3: Casting concrete into the polystyrene mould (Controls Group, p. 53)

The duration for mixing, casting and placing the mould into the equipment was, on average, 13 minutes. Two PT 100 temperature sensors were positioned, with one inside the concrete sample for measuring the temperature of concrete. The second sensor was positioned under the lid of the calorimeter cell to measure the temperature of the equipment. Once the test commenced, the temperature sensors measured the temperature variations of the calorimeter and concrete, and the heat generated by the concrete sample.

4. RESULTS

4.1 Concrete Properties

A summary of the mechanical and physical properties of concrete determined in this study are provided in Table 2, namely compressive strengths, flexural strength and setting times. The compressive strengths for each mix are shown in Figure 4 where an increase in early- and late- day compressive strengths is observed with an increase in cement strength, given the same mix design was used for all three mixes. The initial and final setting times are presented in Figure 5, where CEM I 32.5 R exhibited the fastest setting times for both initial and final set, followed by CEM I 52.5 N. Mix 1 (CEM I 32.5R) and Mix 3 (CEM I 52.5 N) showed similar flexural strengths as shown in Figure 6. All three concrete mixes exceed the minimum compressive (35 MPa) and flexural strength requirements (4.5 MPa) for concrete pavements as specified by COTO (2020).

Table 2: Concrete specimen test results

Concrete Mix	7-Day Compressive Strength (MPa)	28-Day Compressive Strength (MPa)	28-Day Flexural Strength (MPa)	Initial setting times (Min)	Final Setting Times (Min)
CEM I 32.5 R	20	39	7	150	210
CEM II 42.5 N	24	40	5	230	300
CEM I 52.5 N	34	50	7	170	250

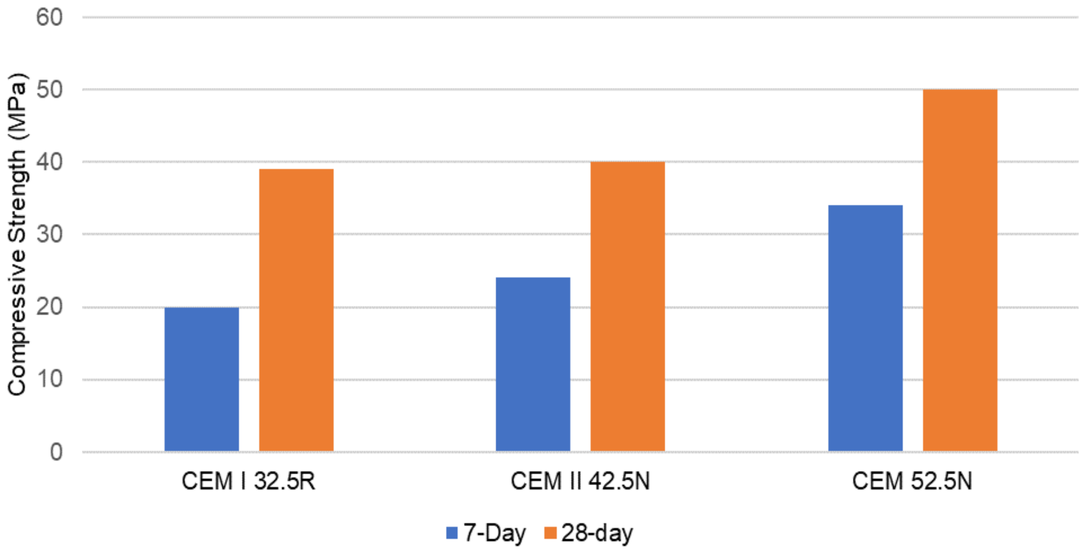


Figure 4: 7- and 28-Day compressive strengths of all mixes

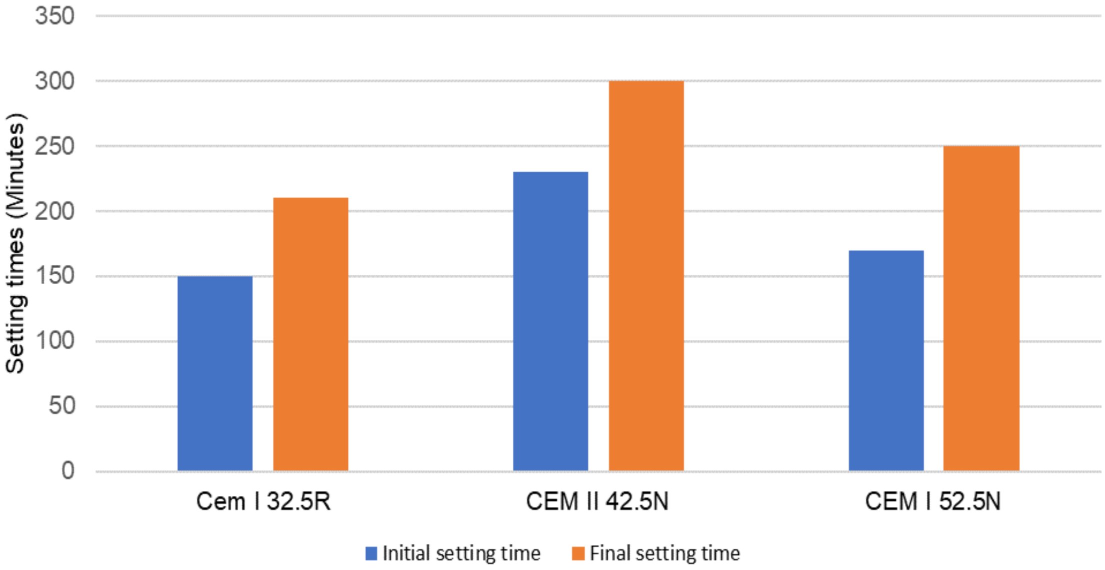


Figure 5: Setting times of the cement pastes.

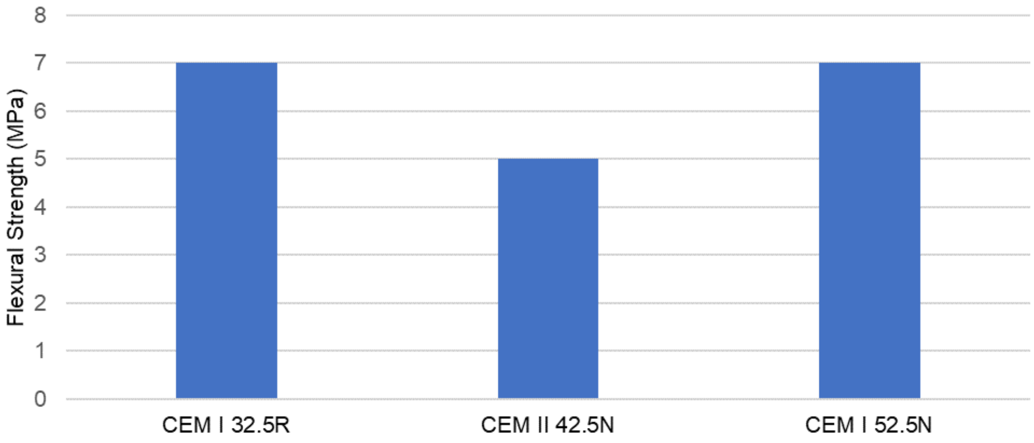


Figure 6: 28-Day flexural strength of concrete mixes

4.2 Heat of Hydration

Table 3 shows the highest temperatures of the concrete mixes recorded by the 7th day. CEM I 32.5 R and CEM II 42.5 N exhibited similar heat of hydration on the 7th day. The heat of hydration of CEM I 52.5 N mix is much lower than the other two mixes.

Table 3: Heat of hydration parameters

Concrete Mix	Highest temperature recorded (°C)	Heat of hydration for cement (kJ/kg)	Heat of hydration for concrete (kJ/kg)
CEM I 32.5 R	51.40	268	44.4
CEM II 42.5 N	54.00	303	44.0
CEM I 52.5 N	64.65	148	21.4

Figure 7 shows the heat of hydration of the concrete mixes investigated. The adiabatic calorimeter recorded the heat of hydration of the concrete mixes for seven days. Mix 1 (CEM I 32.5 R) and Mix 2 (CEM II 42.5 N) show a similar initial steady rise in the heat generated that stabilises after approximately 4 days. Mix 3 (CEM I 52.5 N) was retested and showed that the equipment had likely reached its temperature threshold hence a reduction in the heat of hydration at 12000 minutes showing an unexpected heat release from this point. The heat of hydration plot for Mix 3 (CEM I 52,5 N) does not support true adiabatic conditions after this point and may result from the apparatus limitations requiring further testing and investigation.

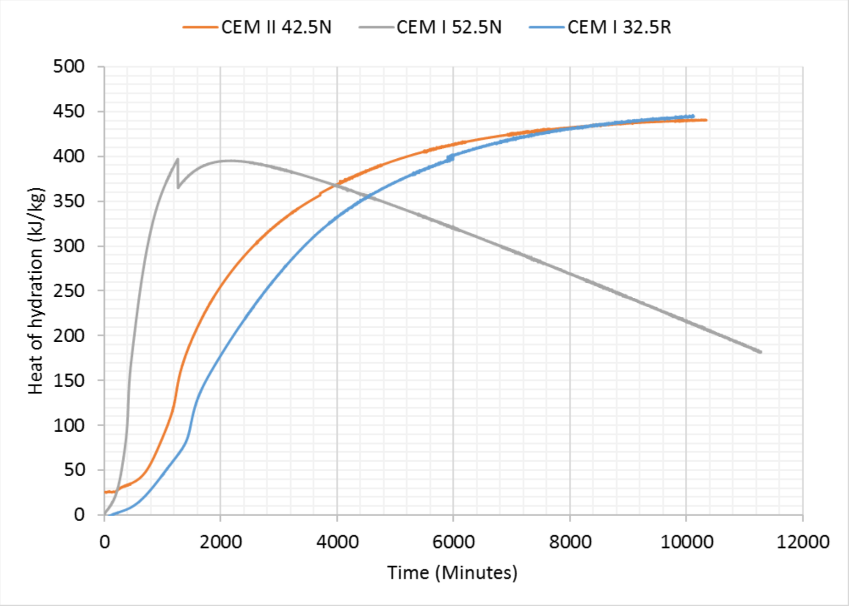


Figure 7: Heat of hydration of concrete mixes

4.3 Rate of Heat Evolution

The maximum heat rate within the acceleration phase of each concrete mix and the duration to reach the observed maximum is shown in Table 4. The CEM I 32.5 R concrete mix showed the highest value given its early strength gain denoted by the ‘R’ classification.

Table 4: Maximum heat rate and corresponding duration

Concrete mix	Maximum heat rate (W/kg)	Time (Minutes)
CEM I 32.5R	54.72	2672
CEM II 42.5N	13.13	1714
CEM I 52.5N	31.74	1037

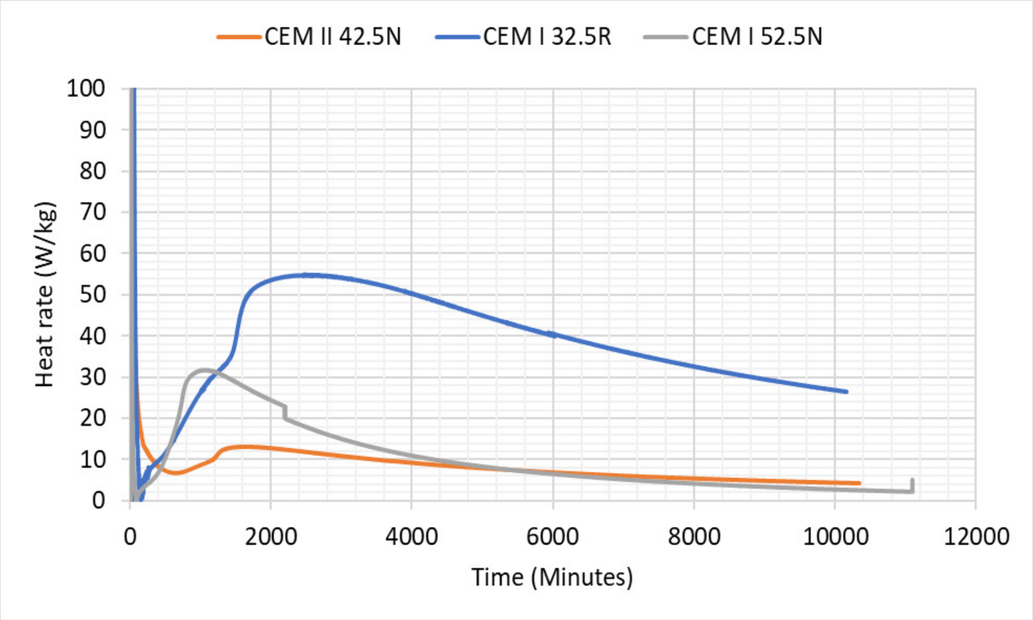
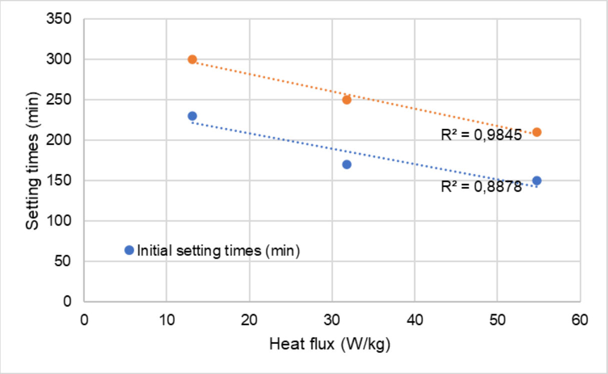


Figure 8: Heat flux

The rate of heat evolution for all three mixes is shown in Figure 8. This illustrates the rate of heat from the initial reaction, dormant phase, initial set, final set and finally deceleration. The CEM I 32.5 R concrete mix showed the highest heat rate (54.72 W/kg) within 2672 minutes, while the CEM I 52.5 N mix had reached a maximum heat rate of approximately 32 W/kg at 1037 minutes. The concrete mix with CEM II 42.5 N had the lowest values but a similar heat of hydration compared to the CEM 1 32,5 R concrete mix after 7 days.

5. DISCUSSIONS

Figures 9 to 11 show the resulting trends between the tested parameters. Given, the non-adiabatic conditions observed after 1200 minutes for Mix 3 (CEM 52,5 N), the relationships between the total heat generated and the physical concrete properties are not discussed. However, since the maximum heat rate was achieved before the heat release, the resulting trends are therefore discussed in relation the maximum heat rate and not the total heat of hydration. Figure 9 shows a co-efficient of determination (R^2) of 0.888 and 0.985 between the maximum heat rate and the initial and final setting times, respectively by linear regression. Figure 10 shows that the 28-day flexural strength and maximum heat rate has a lower R^2 value of 0.696 by linear regression. Figure 11 shows a low R^2 value between the 7- and 28-day compressive strengths and the maximum heat rate.



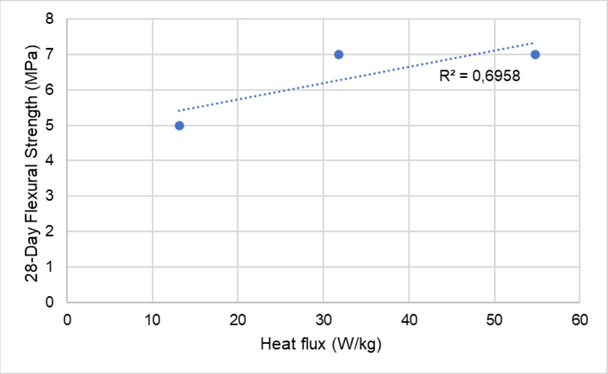


Figure 9: Heat flux vs setting time and heat of hydration vs setting times

Figure 10: Heat flux vs flexural strength and heat of hydration vs flexural strength

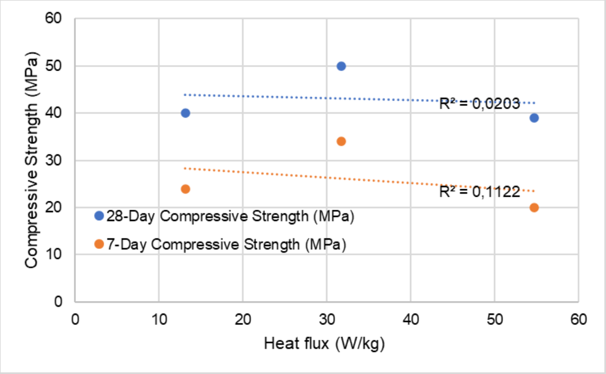


Figure 11: Heat flux vs compressive strength and heat of hydration vs compressive

6. CONCLUSIONS

The aim of this study was to use an adiabatic calorimeter to assess the correlation between the rate of heat evolution from early-age hydration reactions of concrete and the corresponding physical properties of three different concrete mixes using three different cements. The properties assessed include the 7- and 28- day compressive strength, flexural strength, initial and final setting times. The results revealed a strong correlation between the setting times and maximum heat rate achieved by each concrete mix, but lower R^2 values for all other parameters. The study shows that the setting times of concrete mixes can be deduced from heat parameters obtained through adiabatic calorimeter testing of concrete. Due to the current limitations on the adiabatic calorimeter used in this study, further investigations are required to establish the trend between the heat of hydration and the assessed physical properties of concrete mixes. Additional studies are also recommended to include a more diverse range of cements from different cement suppliers to improve on the results obtained in this paper.

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