

Chemical Reaction Characteristics of Sugarcane-Waste-Fiber-Ash (SWFA), Blended Cement - A Study of the Chemical Shrinkage and Heat of Hydration

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ABSTRACT

Heat of hydration and chemical shrinkage are some of the main causes of cracking in concrete. The heat of hydration and chemical shrinkage characteristics of cement pastes containing Sugarcane Waste Fiber Ash (SWFA) partially replacing cement by weight percentage of between 0 and 20% were investigated in this study using a Semi Adiabatic Method to BS EN 196 – 9:2010 for the heat of hydration, and a volumetric method to ASTM C 1608-07 for the chemical shrinkage. A maximum amount of shrinkage was observed on the paste containing 4% SWFA replacing cement by weight. This was thought to be caused by increased reactions at this SWFA content.

Index Terms: Sugarcane waste fiber ash, blended cement, chemical shrinkage, SWFA

1.0 INTRODUCTION

Past studies have shown that Sugarcane Waste Fiber Ash (SWFA) has good pozzolanic properties [1,2], thus creating a potential for it to be used to partially replace cement in concrete mixes.

Kenya grows sugarcane commercially for the production of sugar with an approximate total installed capacity of 22,150 MT per day. This could give a daily yield of about 890 MT of Sugarcane Waste Fiber Ash (SWFA) [3].

A pozzolanic reaction occurs when a siliceous or aluminous material comes into contact with calcium hydroxide in the presence of moisture to form cementitious compounds [4, 5, 6]. When the two main components of cement (tricalcium silicate and dicalcium silicate) hydrate, calcium hydroxide ($\text{Ca}(\text{OH})_2$ or CH) and calcium silicate hydrate (C-S-H) are released.

Free lime content is the excess uncombined lime in cement. Higher amount of free lime is undesirable as it is the main cause of unsoundness in hydrated cement, thus responsible for cracking of concrete [7], due to orientation of the crystalline growth in one direction [6].

Cement hydration is an exothermic reaction and the rate of evolution of heat is an indication of the rate of hydration [8, 9, 10]. A study of the rate of evolution of heat indicates that there are three peaks in the rate of hydration, in the first three days or so [8]. The higher the concrete temperature, the faster the rate of hydration, and the more rapidly heat is generated in the concrete member [9, 11]. The higher the cement content the greater the potential temperature rise in the concrete [9]. The finer the cement, the more surface area there is for reaction and the more rapid the heat development [8, 9].

When cement paste hydrates under sealed conditions, chemical shrinkage occurs because the hydration product occupies less space than the original reactants [12]. This is caused by the crystallization process which produces volume reduction [13]. This volume change continues to occur as long as cement hydrates [8, 14]. Autogenous shrinkage is a volume change of cement paste, mortar, or concrete caused by chemical shrinkage resulting when there is no moisture transfer to the surrounding environment. It is most prominent in high performance concretes when or where the water to cement ratio is under 0.42 [14, 15]. The hydration rate of a cement paste can be measured by measuring non-evaporable water content, heat of hydration, and chemical shrinkage over time [16].

2.0 EXPERIMENTAL PROCEDURE

2.1 Material and Test Samples

Sugarcane Waste Fiber Ash

SWFA was collected from Mumias Sugar Factory in Western Kenya where it is produced as a waste product of burning sugarcane baggase for electricity generation. Usually baggase with a moisture content of about 50% is fed into boilers where it is burnt at 1000°C to convert water into steam that drives turbines to generate electricity. The ash is then sprayed with water to arrest it so that it

is not blown by wind causing a health hazard. Collection is then done in trucks and transported to the factory’s farm for use as fertilizer. Material collected for this study was ash that was ready for dumping in the factory’s farms. Prior to testing, the ash was dried in an oven at 110°C for 24 hours to remove the little moisture previously added at the factory.

Cement

Bamburi Portland Cement Class 32.5 was used in the preparation of the pastes. Cement was mixed with SWFA in percentage replacements of between 0 and 20% by weight. Figure 1 [2] and Table 1 [2] give the particle size distribution and chemical compositions of these cement-SWFA mixtures.

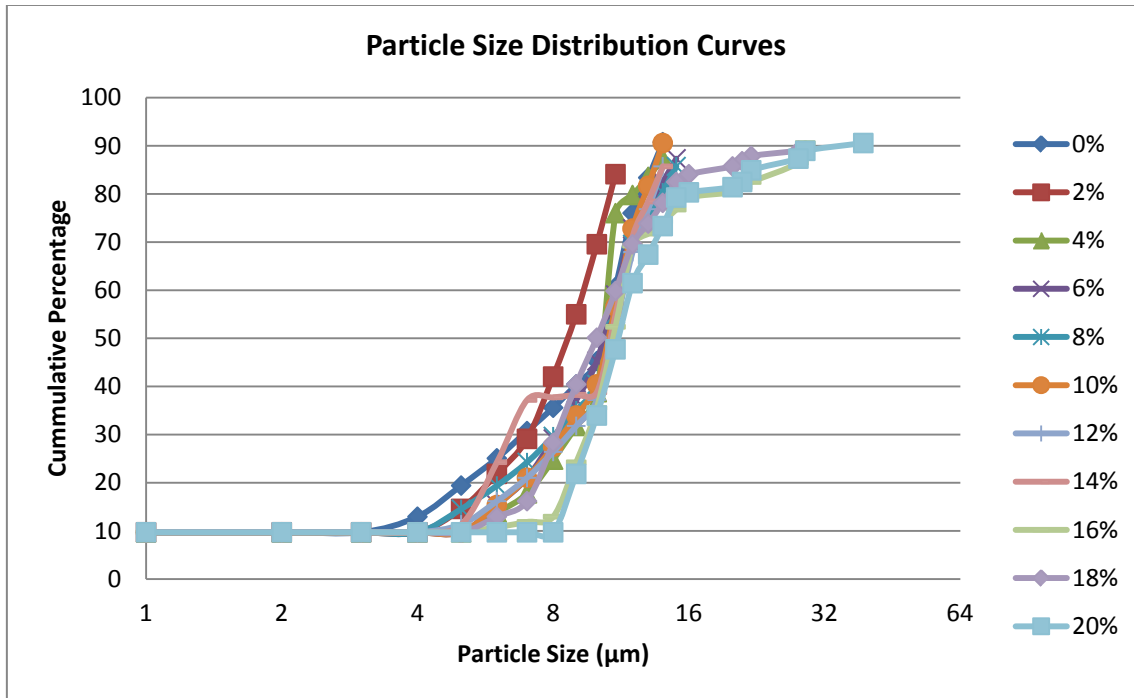


Figure 1: Particle Size Distribution of cement-ash mixtures [2]

Table 1: Chemical Composition of Cement – Ash Mixtures [2]

Sample Ref (% Replacement)	SiO ₂	Al ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	TiO ₂	MnO	Fe ₂ O ₃	LOI	TOTAL
0	41.32	6.74	35.2	0.71	1.41	2.17	0.94	0.12	4.5	4.3	97.41
2	39.19	7.05	34	0.72	1.42	2.19	0.84	0.12	4.53	4.2	94.26
4	42.27	7.1	33.6	0.72	1.37	2.18	0.94	0.12	4.44	4.08	96.82
6	41.4	6.95	32.8	0.73	1.34	2.17	0.97	0.12	4.36	4.71	95.55
8	42.08	6.98	33.6	0.72	1.33	2.11	0.97	0.13	4.49	4.52	96.93
10	41.08	6.92	33.2	0.74	1.32	2.16	0.88	0.13	4.51	4.66	95.60
12	39.68	7.14	31.8	0.73	1.26	1.98	0.9	0.15	4.66	4.93	93.23
14	44.02	6.92	29.8	0.72	1.23	2.13	0.93	0.14	4.43	4.98	95.30

16	42.82	6.95	29.6	0.74	1.26	2.16	0.86	0.13	4.45	4.89	93.86
18	40.22	6.91	29.8	0.75	1.22	2.03	0.96	0.15	4.5	5.78	92.32

Sand

Sand conforming to EN 190-1, complying to particle size distribution given in Table 2 was used.

Table 2: EN 196-1 Standard Sand Particle Size Distribution

Square Mesh Size	Cummulative (%) Retained
0.08	99 ± 1
0.16	87 ± 5
0.50	67 ± 5
1.00	33 ± 5
1.60	7 ± 5
2.00	0

2.2 Testing

Chemical Shrinkage

SWFA was blended with Portland cement in weight percentages of 0, 4, 8, 12, 16 and 20%. Two samples weighing 50g of the SWFA - cement mix was mixed thoroughly with 20g water to achieve a w/c ratio of 0.4. The time of first contact of the SWFA – cement powder with water was recorded.

These mixes were then put into 50 ml glass vials to a depth of between 5 and 10mm. The weight of the glass vials had been previously determined to the nearest 0.01g. The weight of the vials plus cement paste was then taken, again to the nearest 0.01g.

Clean de-aerated water was then carefully added into the glass vials to the top before covering with a stopper tightly fitted with a 1 ml capillary tube with graduations of 0.01 ml. Some more clean de-aerated water was then added into the capillary tube to bring the water to some level near the top of the capillary tube. A paraffin drop was then added on top of the water to control evaporation.

The prepared specimens were then placed into a constant temperature water bath at 23 °C such that the top of the glass vials were just above the water level in the bath. The temperature in the water bath was maintained at 23 +/- 2 °C. The time and initial levels of the water in the capillary tubes were recorded. Figure 2 illustrates the chemical shrinkage test set up.

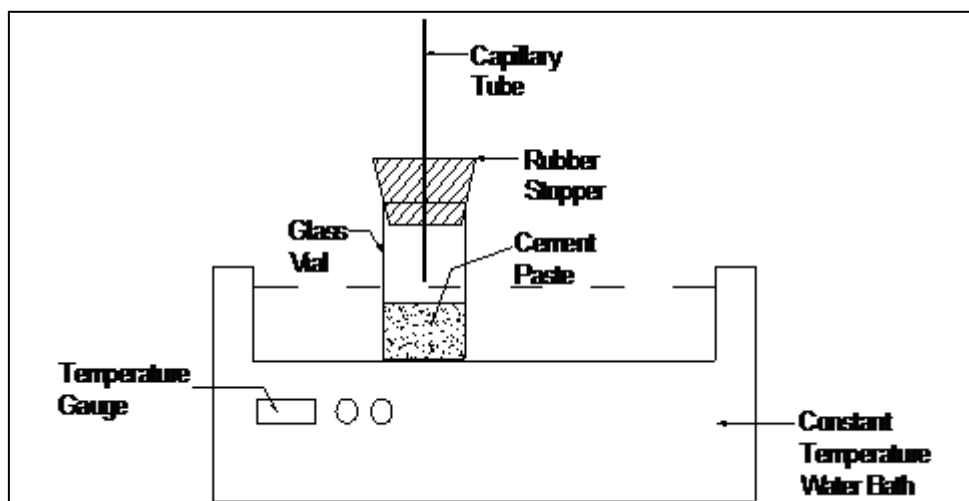


Figure 2: Chemical shrinkage test set up

Periodically (every 30 minutes) the water level in the capillary tube and its corresponding time to the nearest minute were recorded. This was carried out in the first 8 hours after which the readings were spaced at bigger time intervals. The first reading was taken after

1 hour for all samples and this was used as the zero point in all calculations (this is to allow time for the specimen to achieve temperature equilibrium within the water bath).

2.3 Analysis

Using the mass of cement powder of 50g, the chemical shrinkage per unit mass of cement at time t was computed using equation 1 [17].

$$CS_{(t)} = \frac{[h_{(t)} - h_{(60\text{min})}]}{m_c} \quad (1)$$

Where:

- $CS_{(t)}$ = Chemical shrinkage at time t (ml/g cement)
- $h_{(t)}$ = Water level in capillary tube at time t (ml)
- $h_{(60\text{ min})}$ = Water level in capillary tube 60 minutes after setting the test
- m_c = Mass of cement in the vial (g)

Heat of Hydration

Cement-Ash pastes described above were tested for heat of hydration for the first 24 hours of hydration. Heat of hydration measurements were carried out to BS EN 196 – 9:2010 [18], using the semi adiabatic method.

The principle of the semi adiabatic method consists of introducing a sample of freshly made mortar into a calorimeter in order to determine the quantity of heat generated in accordance with the development of the temperature [18]. At a given point in time, the heat of hydration of the cement contained in the sample is equal to the sum of the heat accumulated in the calorimeter and the heat lost into the ambient atmosphere throughout the period of the test. The temperature rise of the mortar is compared with the temperature of an inert sample in a reference calorimeter. Three calorimeters similar to the one in Figure 3 were built and calibrated prior to use in these tests.

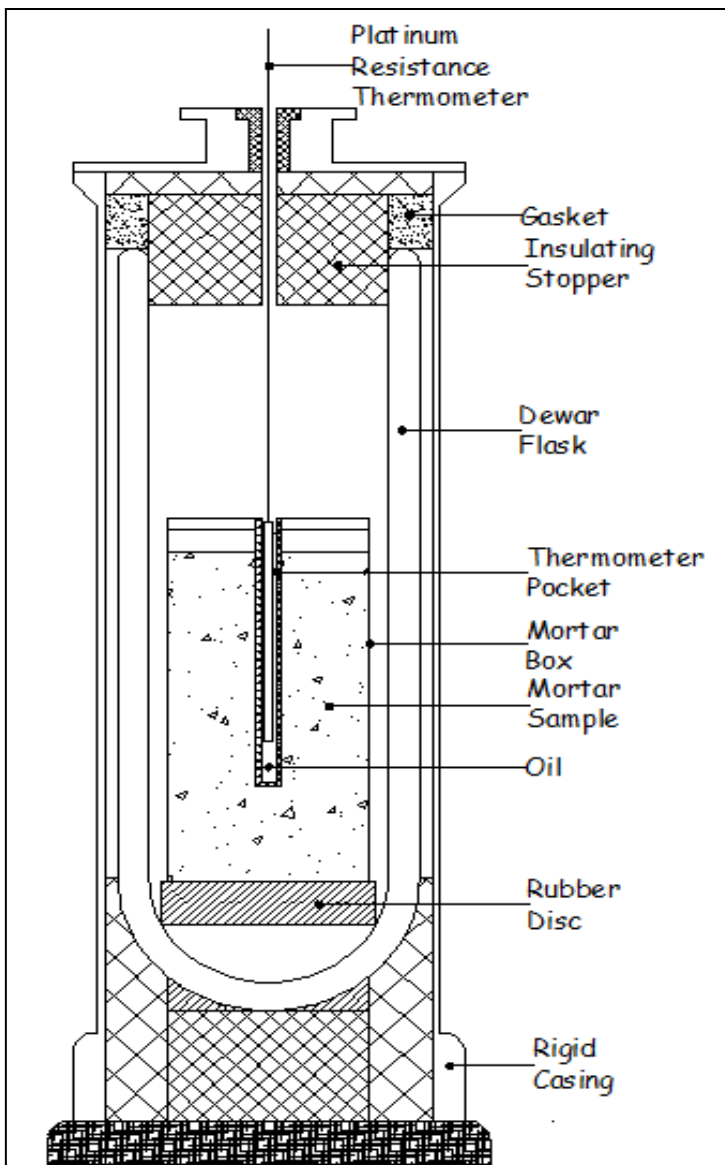


Figure 3: Schematic of a typical calorimeter

The calorimeters in this study consisted of steel casing dewar flasks contained in wooden boxes padded with 1 inch insulating material. Samples were contained in steel mortar boxes as per the code specification [18]. Temperature was taken via a digital thermometer with a probe capable of being enclosed within the calorimeter with its display outside the casing. Accuracy of measured temperature by this thermometer was $\pm 0.1^\circ\text{C}$, to satisfy the $\pm 0.3^\circ\text{C}$ specified in the code [18].

Calibration of the calorimeters was carried out using an aluminium calibration cylinder as described in the code [18].

Measurement of heat of hydration

The determination of heat of hydration consisted of taking temperature readings of a freshly mixed mortar at specific moments in time and comparing that to the temperature of an inert sample in a calorimeter under similar conditions as the sample. The heat of hydration, Q, in Joules per gram of cement, at elapsed time, t, was calculated using Equation 2 [18].

$$Q = \frac{c}{m_c} \theta_t + \frac{1}{m_c} \int_0^t \alpha x \theta_t x dt \tag{2}$$

Where:

- m_c – mass of cement contained in the test sample in grams
- t – hydration time, in hours
- c – total thermal capacity of the calorimeter in Joules per Kelvin
- α – coefficient of heat loss of the calorimeter, in Joules per hour per Kelvin

θ_t – difference in the temperature of the test calorimeter compared with that of reference calorimeter at time t, in Kelvins.

The coefficient of heat loss was calculated using Equation 3 [18]

$$\alpha = a + b \times \theta_c \tag{3}$$

The coefficients a and b are characteristic of a particular calorimeter.

The total capacity of the calorimeter c, including the mortar box and mortar sample under test was calculated using Equation 4 [18].

$$c = 0.8(m_c+m_s) + 3.8m_w + 0.50m_b + \mu \tag{4}$$

Where;

- 0.8 is the thermal capacity per unit of mass of cement plus sand, in Joules per Kelvin per gram.
- 3.8 is the average thermal capacity per unit mass of water, in Joules per Kelvin per gram.
- 0.50 is the thermal capacity per unit mass of the mortar box, in Joules per Kelvin per gram.
- μ is the thermal capacity of the empty calorimeter in Joules per Kelvin.
- m_c is the mass of cement, in grams (360.0±0.5) g
- m_s is the mass of sand, in grams (1080±1) g
- m_w is the mass of water, in grams (180.0±0.5) g
- m_b is the mass of empty mortar box plus lid, in grams

3.0 RESULTS AND DISCUSSION

3.1 Chemical Shrinkage

Table 3 shows average chemical shrinkage results for 0, 4, 8, 12, 16 and 20% SWFA cement blends for a period of 24hours from preparation of cement paste. Figures 4 and 5 show plots of the average chemical shrinkage values for periods of up to 24 hours, and up to 5.5 hours from time of preparation of the cement pastes, respectively.

Table 3 Average chemical shrinkage for SWFA cement blends

Time in Hours	0% SWFA	4% SWFA	8% SWFA	12% SWFA	16% SWFA	20% SWFA
0.0	-2.50	-1.90	2.20	1.50	1.45	3.00
0.5	1.90	-0.20	0.40	0.50	0.65	0.70
1.0	0.00	0.00	0.00	0.00	0.00	0.00
1.5	-0.80	-0.10	-1.20	-0.30	-0.05	-0.50
2.0	-0.70	-0.35	-2.10	-0.35	-0.05	-0.70
2.5	-1.05	-0.60	-2.44	-0.35	-0.05	-0.95
3.0	-1.40	-0.70	-2.77	-0.30	-0.10	-1.00
3.5	-3.36	-0.80	-4.28	-0.20	-0.15	-1.00
4.0	-5.32	-0.90	-5.78	-0.40	-0.40	-0.95
4.5	-7.28	-1.00	-7.29	-0.60	-0.65	-1.25
5.5	-9.24	-1.50	-8.79	-0.60	-3.05	-1.80
24.0	-12.30	-15.00	-10.30	-7.70	-9.15	-10.60

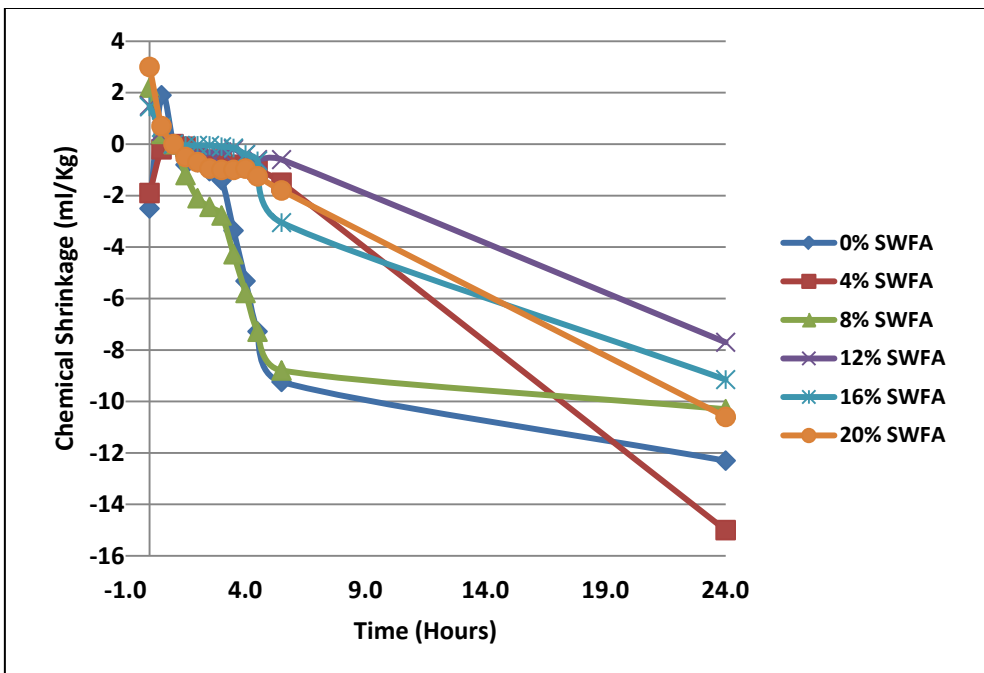


Figure 4 Average chemical shrinkage of 0, 4, 8, 12, 16, & 20% SWFA replacing cement (0 - 24 hours)

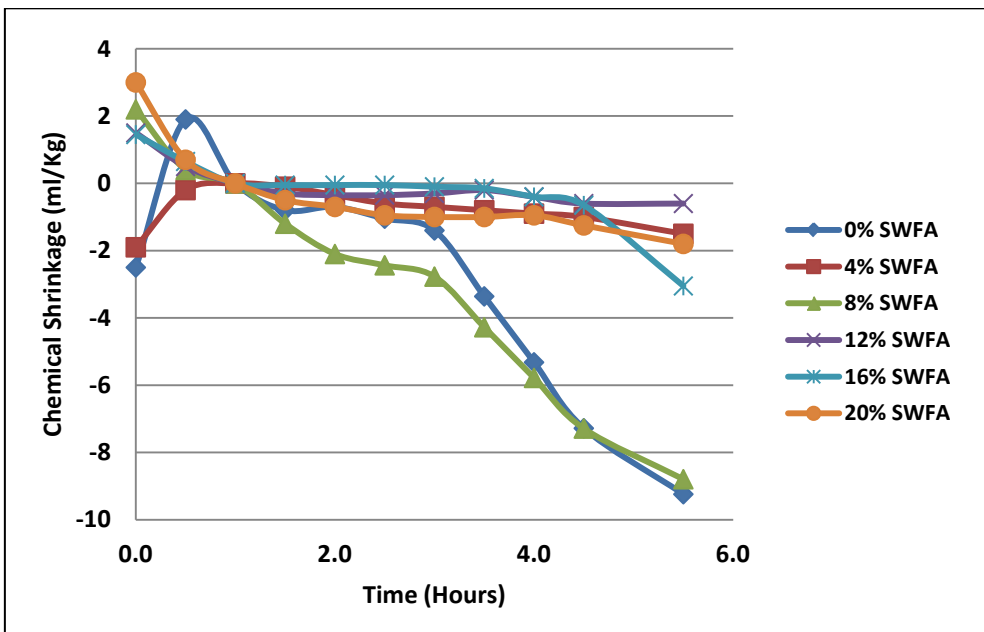


Figure 5 Average chemical shrinkage of 0, 4, 8, 12, 16, & 20% SWFA replacing cement (0 - 5.5 hours)

Figure 4 suggests that the mix with 4% replacement of cement with SWFA gave the largest value of chemical shrinkage at 24 hour age of the pastes. This implies that the 4% mix had the largest amount of reactions occurring after 24 hours of hydration [17]. Figure 4 also seems to suggest an acceleration of the reaction in the 4% mix after the 5.5 hour mark.

From Figure 5, 0% and 8% Cement replacement are seen to have the fastest reaction rates in the period 0-5.5 hours. Figure 4 suggests that this fast rate of reaction slows down in the later hours of the first 24 hours. From Figure 4, 12%, 16% and 20% SWFA cement blends are observed to have the lowest reaction rates and therefore the lowest rates of the set.

These results are in agreement with mechanical strength results achieved for concrete cube samples prepared using cement containing between 0% and 20% SWFA [19]. In the tests for cube compressive strength, results suggested an increase in compressive strength of the concrete cubes at about 6% SWFA content followed by a steady decrease in strength of the cubes with increasing SWFA content. SWFA contains a high SiO₂ content (Table 1). SiO₂ in a hydrating cement paste reacts with Ca(OH)₂, a product of the hydration of

C₃S and C₂S to form CSH which is the primary bonding compound in a cement paste. This process boosts the total amount of reactions taking place within the paste causing an increase in both the heat of hydration and the chemical shrinkage, both of which can be used as a direct measure of the amount of reactions occurring in the hydrating cement paste. Addition of SWFA to cement up to 6% SWFA content provides the SiO₂ necessary for the reaction to occur, this however also reduces the amount of C₃S and C₂S in the paste, and consequently the amount of Ca(OH)₂ produced. 6% SWFA content was found to be the optimum content for the mix. Beyond this point, addition of more SWFA meant less and less C₃S and C₂S available for reaction and therefore lesser Ca(OH)₂ produced causing the drop in strength.

Finer particles of RHA have been found to cause higher drying shrinkage in cement RHA blend cements [20]. This is due to increased pore refinement of the RHA concrete matrix occasioned by the higher packing efficiency of the smaller particles. This is in contrast to observations on drying shrinkage of fly ash in which fly ash fineness was observed to have very minimal effect on drying shrinkage of fly ash-cement blends [21]. The lower fineness (i.e. bigger particles of SWFA) [2], could have contributed to the reduction in shrinkage with increase in ash content due to two reasons: (i) a reduced packing efficiency, and (ii) reduced hydration reactions.

Figure 6 shows the values of chemical shrinkage for SWFA – Cement paste at the age of 24 hours for 0, 4, 8, 12, 16 & 20% SWFA content. Maximum shrinkage value was achieved with the SWFA - Cement paste containing 4% SWFA. Increased chemical reaction (due to pozzolanic activity occasioned by presence of increased SiO₂ in SWFA) is thought to have caused the increase in shrinkage indicating an increase in hydration reaction in this mix. As the content of SWFA increased, the paste became coarser, resulting in reduced amount of reaction [20, 21], consequently returning lower amount of shrinkage. These shrinkage results are qualitatively in agreement with SWFA – cement concrete mechanical strength results [19].

Reduced shrinkage presents an advantage where concrete's early age cracking is a problem. Similar observations have also been made with coarser cements [12] where coarser cements were found to have a reduced early age cracking.

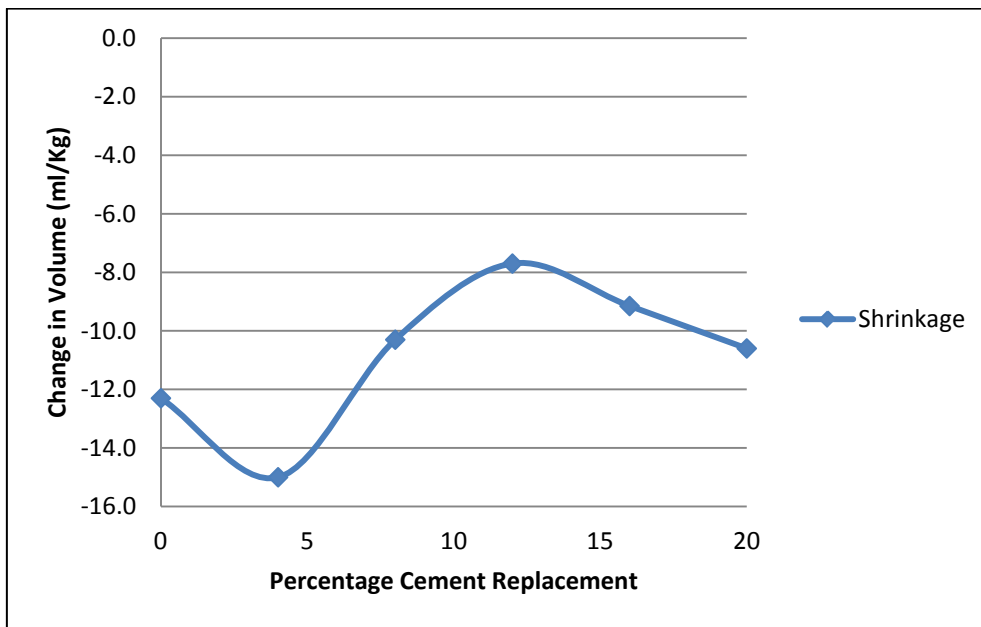


Figure 6 Variation of 24 hour chemical shrinkage with SWFA content

Within the first 24 hours, addition of approximately 4% SWFA to cement causes a significant increase in shrinkage. This would result in the creation of more bonds from the formation of CHS gel resulting in improved mechanical strength [21], reduced pore sizes [22], and better durability [23] in concretes containing SWFA in these quantities.

The accelerated rate of reaction for 4% SWFA content (Figure 4) is not evident in the first few hours (approximately 0 – 6 hours) but is seen to pick up later towards 24 hours from time of preparation of the cement paste. Concretes in which the binder is replaced with SWFA at 4% can be expected to gain strength faster than concrete without SWFA. This poses the obvious advantage of a possible early removal of formwork in concrete construction. However, increased reaction rate implies increased heat of hydration output rate and such would have to be addressed properly prior to making use of this advantage. Control of excess heat can be achieved by use of cold water for mixing, covering of curing concrete elements with wet material like sand and limiting the element sizes.

3.2 Heat of Hydration

Heat of hydration of hydrating cement can be used to characterize the setting and hardening behavior of cement, and predicting the temperature rise which is very important when large volumes of plain concrete are placed [24]. Traditionally, cement reactions are recorded by calorimetric methods. This is a direct method similar to volume change. Direct methods offer the advantage that the reaction does not have to be stopped to take a reading due to their simplicity [25].

Heat of hydration tests were carried as outlined in Section 2.2. The first step in the calibration process involved the determination of the coefficient of heat loss, α . Table 4 summarizes the values of a , b as given in Equation 3 and coefficient of linear correlation, R^2 , and thermal capacity of empty calorimeter, μ .

Table 4 Calibration values a and b , coefficient of linear correlation R^2 , and thermal capacity of empty calorimeter

Calorimeter	a	b	R^2	μ
A	84.78	1.861	0.997	347.52
B	101.6	1.058	0.987	515.03
C	102.9	0.896	0.998	380.23

Table 5 summarizes the average heat of hydration values for SWFA-Cement mortars containing 0, 5, 10, 15 and 20% SWFA.

Table 5 Heat of hydration for SWFA-cement mortars

Age (Hours)	Heat of Hydration (J/g)				
	0% SWFA	5% SWFA	10% SWFA	15% SWFA	20% SWFA
0.1	0.62	2.82	18.52	5.96	10.17
0.5	5.67	6.32	26.82	11.26	18.67
1.5	7.78	8.24	27.78	12.69	19.26
2.5	8.78	9.42	27.47	13.36	19.55
3.5	9.11	10.40	28.12	14.33	20.17
4.5	11.04	11.38	28.79	15.32	20.75
8.5	14.02	24.69	28.49	16.94	21.94
10.5	15.90	23.13	29.77	18.90	23.15
11.5	16.60	25.43	30.44	20.54	25.25
12.5	16.63	27.41	31.08	22.49	26.80
15.5	20.68	28.41	48.53	24.14	28.01
16.5	19.89	29.72	43.46	25.77	29.09
17.5	20.86	31.68	45.57	32.38	36.58
18.5	21.20	34.30	47.95	33.66	31.39
19.5	23.77	35.32	48.35	32.97	36.31
20.5	29.59	38.87	48.04	35.57	45.06
21.5	34.84	41.19	48.03	38.20	44.99
22.5	37.52	43.47	47.37	41.15	44.37
23.5	38.23	45.11	47.35	44.71	43.75
24.5	38.88	45.79	47.04	44.48	43.16
25.5	38.58	45.80	46.39	44.79	42.82

Figure 7 plots the values in Table 5, for the variation of the average heat of hydration of mortars containing 0 – 20% SWFA by weight of the cementing material with time in the first 24 hours of hydration. Figure 8 plots the values of heat of hydration of SWFA – Cement mortars at the age of 24 hours.

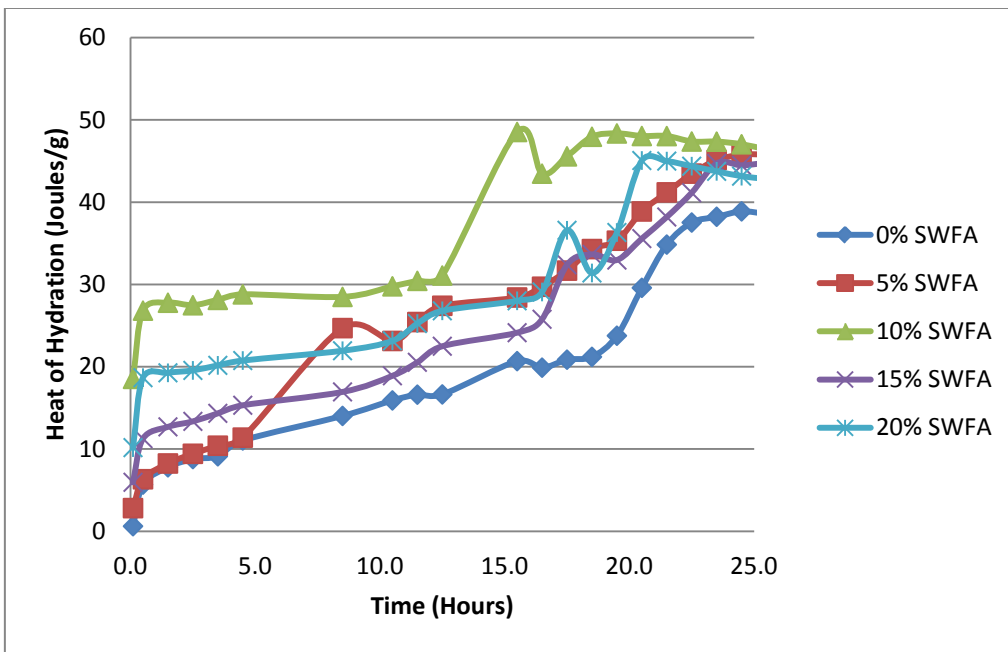


Figure 7 Average heat of hydration for SWFA - cement mortars

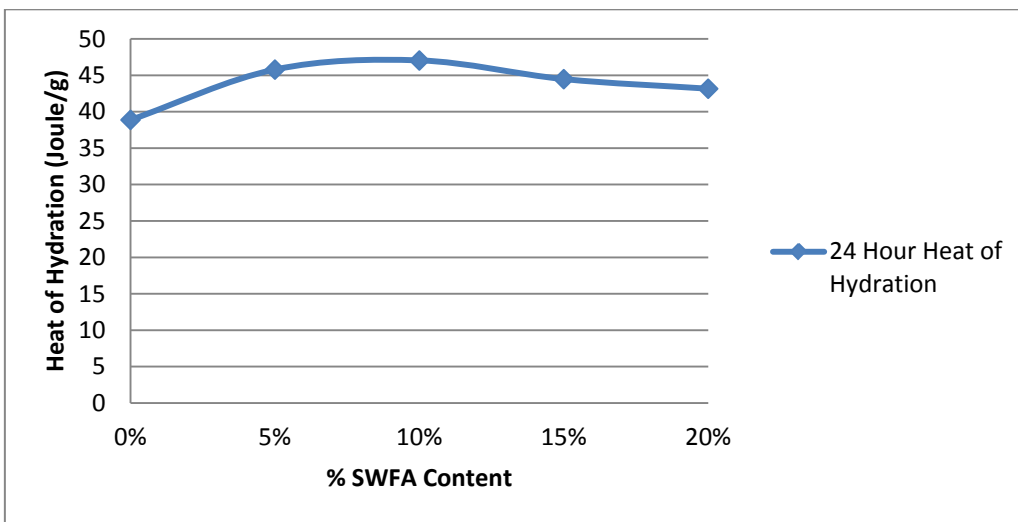


Figure 8 24 hour heat of hydration vs SWFA content

Figures 7 and 8 suggest that the lowest liberated heat of hydration was from pure cement (0% SWFA) and highest heat emitted by the sample with 10% SWFA content. The amount of heat liberated during the hydration process is a direct indication of the amount of hydration reactions occurring at the particular time [18], the more the heat of hydration, the higher the amount of hydration reactions occurring. The figures suggest that highest volumes of hydration reactions occurred in the samples between 5% and 10% SWFA. This result agrees with the chemical shrinkage results (Figures 4 and 5) which also gives a direct indication of the amount of hydration reactions occurring. The pozzolanic activity occasioned by the SWFA is thought to have caused the increased reactions in the samples containing 5 and 10% SWFA.

The increase in hydration reactions with increase in SWFA content was caused by an increase of the main cementitious compounds (C_3S and C_2S) caused by the fresh supply of silica from SWFA in the mix (Table 1) [2]. This increase continued up to the point where the available calcium hydroxide was depleted (between 5% and 10% SWFA content) after which the amount of main cementitious compounds could no longer increase with increase in SWFA content.

The coarseness of the cementing material occasioned by SWFA content (Figure 1) [2] caused a decrease in the available area for reaction resulting in the reduced reaction amount and heat of hydration seen in samples containing SWFA quantities beyond 10%.

The observed reduction in the heat of hydration might be a delayed heat production as opposed to a reduced heat production as has been observed in other studies [24].

The reduction in the heat of hydration with increasing cement coarseness observed elsewhere [24] poses a big advantage especially in the use of mass concrete where heat of hydration can result in cracking of the concrete.

5. CONCLUSIONS

Results obtained from this study led to the following conclusions:

- 1 Within the first 24 hours, addition of approximately 4% SWFA to cement causes a significant increase in the chemical shrinkage in cement pastes.
- 2 The increased chemical shrinkage for 4% SWFA content paste is not evident in the first few hours (approximately 0 – 6 hours) but picks up later towards 24 hours from time of preparation of the cement paste.
- 3 Heat of hydration is increased in cement pastes containing between 5% and 10% SWFA by weight

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