

QUANTIFICATION OF HYDRATION PRODUCTS IN RICE HUSK ASH (RHA)-BLENDED CEMENT CONCRETE WITH CRUMB WASTE RUBBER TIRES (CWRT) & ITS CORRELATION WITH MECHANICAL PERFORMANCE

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ABSTRACT: Previous studies on the use of rice husk ash (RHA) as a replacement of cement focused on attributing the strength enhancements in mechanical strength in hardened concrete to its pozzolanic reactivity. Investigation of the pozzolanic reaction RHA in terms of cement hydration has yet to be examined. This study intended to assess the pozzolanic activity of RHA by quantification of calcium hydroxide (CH) and calcium silicate hydrate (C-S-H) gel in RHA-blended cement pastes using simultaneous thermogravimetric and differential-thermal analysis (TG-DTA). Moreover, the application of crumb waste rubber tire (CWRT) in concrete as fine aggregate replacement is known to reduce mechanical strength. Thus, the pozzolanic reaction of RHA compensating the strength loss by CWRT was also investigated. Two types of RHA, from open-air burning (RHA-O) and thermal power plant (RHA-T), were used in casting cement pastes and mortars with 0%, 7.5%, 12.5%, and 17.5% replacement of cement (by weight). Finally, concrete with fixed optimal RHA and 5%, 10%, and 15% CWRT (by volume) were tested for compression and splitting tensile strength. Results from TG-DTA revealed reductions in quantified CH contents and increase in C-S-H gel contents in cement pastes with RHA indicating pozzolanic reaction. Decreasing CH content corresponded to increasing compressive strength highlighting the strength enhancement by pozzolanic activity, optimal at 17.5% using RHA-O. For concrete, results showed that incorporating 17.5% RHA-O and 5% CWRT provided comparable compressive strength and higher splitting tensile strength than normal concrete, signifying the compensation of strength loss of CWRT by RHA.

Keywords: Rice husk ash, Pozzolanic effect, Thermogravimetric analysis, Hydration product, Crumb rubber

1. INTRODUCTION

Concrete remains the single most widely used material in the world. Production of cement contributes to about five percent of the global carbon dioxide (CO₂) production due to human activities [1]. One of the waste materials that have been investigated to be an effective supplementary cementitious material is rice husk ash (RHA). Combustion of rice husk to run milling processes will produce a huge amount of RHA, usually dumped on bodies of water that cause pollution and contamination [2]. RHA is described to have high silica (SiO₂) content ranging from 85% to 90% [3] making it very reactive and pozzolanic. Amorphous silica is highly reactive whereas crystalline silica has no pozzolanic activity [4].

Notable increase in mechanical strength has been observed in mortars and concrete with replacements of cement with RHA [3], [5]. The pozzolanic effect of RHA happens when the excess calcium hydroxide (CH) from the hydration of cement reacts with silica (SiO₂) of RHA to form additional calcium silicate hydrate (C-S-H) gel. The C-S-H gel is considered as the main carrier of

strength in a hardened concrete hence increasing its overall mechanical strength [6].

Considering the abundant research on the effect of RHA on the mechanical strength, the mechanism of pozzolanic reaction by RHA in blended mortar and concrete has yet to be studied. Therefore, one of the main objectives of the study is to evaluate the pozzolanic activity of RHA through the quantification of hydration products. Simultaneous thermogravimetric and differential-thermal analysis (TG-DTA) has been considered as the easiest and most widely used methodology for the analysis of hydration reactions in cementitious pastes [7]. In this method, thermogravimetric analysis (TGA) monitors the weight of the sample upon heating to a constant rate while differential-thermal analysis (DTA) detects the temperature at which the reactions in the sample occur manifested by peaks in the curve.

The hydration products have unique decomposition temperatures. This allows the determination of their quantities based on the mass losses associated with the decomposition temperatures. For C-S-H gel, the temperature of decomposition ranges from 110°C to 400°C [7]

whereas for CH, the decomposition ranges from 400°C to 600°C [8]. Using this method, reduction in the quantity of CH can be indicative of reaction with SiO₂ from RHA while increase in C-S-H can be associated with the production of secondary C-S-H gel.

Another potential material replacement in the concrete mix is the crumb waste rubber tire (CWRT) as fine aggregates. Although the addition of CWRT reduces strength [9], the increase in strength provided by the pozzolanic reaction of RHA can compensate for this lack.

In this paper, two types of RHA will be examined: RHA from open-air burning (RHA-O) and RHA from a thermal plant (RHA-T). Using TG-DTA, C-S-H and CH contents from the hydration of RHA-blended cement pastes will be quantified and correlated to the compressive strength of mortar with and without RHA to evaluate strength improvement in relation to pozzolanic effect. The final output of this study is a blended concrete with optimal amount of RHA and CWRT.

2. RESEARCH SIGNIFICANCE

There are very limited studies on the pozzolanic effect of RHA through hydration of RHA-cement paste, as well as the combined effect of RHA and CWRT in concrete. This research provides an understanding on the mechanism for strength development of these waste materials in hardened concrete. Likewise, RHA-CWRT concrete is a viable for commercial use in the future such as for structural applications or an alternative to the conventional concrete pavements for use in roads and bridge decks. Aside from the environmental benefits, the application of rubber for pavement can give a concrete alternative with unique mechanical and dynamic properties.

3. THEORETICAL BACKGROUND

3.1 Hydration of Portland Cement With RHA

The hydration of C₃S and C₂S in Portland cement produces CH and C-S-H gel in the form C_{1.7}SH₄ where: C = CaO, S = SiO₂ and H = H₂O [10]. The reaction of amorphous silica (SiO₂) in RHA with the excess CH from the hydration of Portland cement forms another type of C-S-H given by the formula [11]:



3.2 Quantification of Hydration Products By TG-DTA

Various authors have observed the following decomposition reactions that occur in cement pastes

upon heating during TG-DTA: a) 30 °C to 105 °C: dehydration of evaporable water [12], b) 110 °C to 400 °C: dehydration or loss of chemically bound water from C-S-H molecules [7], c) 400 °C to 600 °C: dehydroxylation of CH [8] following the reaction:



and d) 600 °C to 1100 °C: decarbonation (loss of carbon dioxide) in CaCO₃ [10] based from the reaction:



The amount of CH can be estimated using [10]:

$$CH (\%) = CH_{\text{loss}} \cdot \frac{MW (CH)}{MW (H)} \quad (4)$$

where CH (%) is the percentage amount of CH, CH_{loss} is the mass loss in percentage due to dehydroxylation of CH, and MW (CH) and MW (H) are the molecular weights of CH and water, respectively. Calcium silicate hydrate (C-S-H) gel content can be computed using the equation [7]:

$$C-S-H (\%) = C-S-H_{\text{loss}} \cdot \frac{MW (C-S-H)}{\text{Moles of water} \cdot MW (H)} \quad (5)$$

where C-S-H (%) is the percentage amount of C-S-H, C-S-H_{loss} is the mass loss in percentage due to dehydration of C-S-H gel between 110°C - 400°C and MW (C-S-H) and MW (H) are the molecular weights of C-S-H gel and water, respectively.

4. METHODOLOGY

4.1 Preparation of RHA

The burning of rice husks was performed in an open area using a cylindrical hollow steel plate. The fire was maintained for one hour without controlling the combustion temperature and cooled for 24 hours. Two types of RHA were utilized in this study: (a) RHA from open-air burning (RHA-O) and (b) RHA from a thermal plant (RHA-T). Both RHA were further ground using Los Angeles Abrasion machine to attain the required fineness of ASTM C618.

4.2 Characterization of OPC and RHA

Characterization of OPC and RHA were conducted as follows: a) Blaine Air-Permeability Test (ASTM C204) and No. 325 mesh fineness test (ASTM C430) for fineness, b) Scanning Electron Microscope (SEM) Imaging using JEOL JSM-5310 for microstructure, c) X-ray fluorescence spectrometry (XRF) for chemical compositions and

d) X-ray Diffraction (XRD) analysis for crystallinity. The degree of crystallinity (DOC) of silica in each RHA was estimated using Eq. (6) based on areas of crystalline peaks and residual area in the diffraction curve attributed to the area of amorphous peaks [13]:

$$\text{DOC}(\%) = 100 \times \frac{\text{Crystalline area}}{\text{Crystalline area} + \text{Amorphous area}} \quad (6)$$

The weight fraction of amorphous silica is given by:

$$W_{\text{amorphous}} (\%) = 100 \times (1 - \text{DOC}) \quad (7)$$

4.3 Thermogravimetric and Differential-Thermal Analysis (TG-DTA) of RHA Cement Pastes

Two sets of cement pastes were prepared with RHA-O and RHA-T, at levels of replacement of pure cement equal to 0%, 7.5%, 12.5% and 17.5% (Table 1). All pastes were mixed following ASTM C305, casted into plastic jars (Ø30mm) with 10 mm height and stored in a water bath 24 hours after casting.

All hardened cement pastes cured for 120 days were dried in an oven at 105 °C for at least 24 hours, pulverized using mortar and pestle, and then sieved using No. 200 sieve. Simultaneous thermogravimetric and differential-thermal analysis (TG-DTA) was performed using Perkin Elmer Simultaneous Thermal Analyzer (STA) 6000 available in Advanced Device and Materials Testing Laboratory (DOST-ADMATEL). Approximately 20 mg of each powdered sample was weighed and filled in a platinum crucible. The heating program was set in the following manner: (a) heat from 30 to 995 °C at 10 °C per min and (b) hold for 5 min at 995 °C. All samples were heated in Nitrogen gas atmosphere.

Quantification of CH and C-S-H contents in pure and blended cement paste samples were based on the proposed methods from previous works [7], [8] calculated using Eq. (4) and (5), respectively.

Table 1 Mix proportion of pure Portland cement and RHA-cement pastes

Mix Design	Material			Water, mL
	Cement, g	RHA, g		
		RHA-O	RHA-T	
CP	100	0	0	48.5
7.5PO	92.5	7.5	-	48.5
12.5PO	87.5	12.5	-	48.5
17.5PO	82.5	17.5	-	48.5
7.5PT	92.5	-	7.5	48.5
12.5PT	87.5	-	12.5	48.5
17.5PT	82.5	-	17.5	48.5

4.4 Mortar Mix Proportioning and Testing

Mortar cubes having 50 mm edge with 7.5%, 12.5% and 17.5% cement replacement (by weight) by each RHA type (RHA-O and RHA-T) were made using ASTM C109 and mixing per ASTM C305 as shown in Table 2. Mortar fragments were subjected to SEM imaging after testing.

Table 2 Mixture proportion of 6-cube batch RHA mortars

Mix Design	Material				
	Cement, g	RHA, g		Sand, g	Water, mL
		RHA-O	RHA-T		
CM	500	0	-	1375	242
7.5MO	462.5	37.5	-	1375	242
12.5MO	437.5	62.5	-	1375	242
17.5MO	412.5	87.5	-	1375	242
7.5MT	462.5	-	37.5	1375	242
12.5MT	437.5	-	62.5	1375	242
17.5MT	412.5	-	87.5	1375	242

4.5 Concrete Mix Proportioning and Testing

From the compression test on RHA mortars, the most optimal RHA type is RHA-O at 17.5% cement replacement. Concrete cylinders were prepared at fixed 17.5% RHA-O with CWRT at replacement levels 5%, 10% and 15% by volume. Mix proportioning per 1m³ of each batch in Table 3 was formulated according to ACI 211.1 at w/c equal to 0.5. Three replicates of Ø100mm×200mm cylinders were tested for compression and splitting tensile strength after 28 days as per ASTM C39 and ASTM C496, respectively.

5. RESULTS AND DISCUSSION

5.1 Properties of RHA and OPC

5.1.1 Chemical composition

Both RHA-O and RHA-T manifested high silica (SiO₂) contents equal to 93.38% and 82.73%, respectively, as shown in Table 4. Jamil et al. [11] demonstrated that amorphous silica from RHA reacts with CH from cement hydration process to form secondary C-S-H gel.

5.1.2 Crystallinity of silica in RHA

The silica phase of RHA-T is partly crystalline and partly amorphous due to narrow peaks at around 21.8° at the top of a broad peak between 10° and 28° and another at 29.4° as shown in Fig. 1. These sharp peaks are indicative of crystalline silica while the broad peak is attributed to amorphous silica. The broad smooth hump between 10° and 30° of RHA-O indicated amorphous silica content. From the DOC method, RHA-O and RHA-T contained

Table 3 Mixture proportion of concrete cylinders with RHA and CWRT in kg for 1 m³ concrete production.

Mix	RHA (%)	CWRT (%)	Cement	RHA	FA ^a	CWRT	CA ^b	Water
ROC0	0	0	418	0	779	0	1151	241
ROC10	0	10	418	0	701	38	1151	241
R17.5C0	17.5	0	345	73	779	0	1151	241
R17.5C5	17.5	5	345	73	740	19	1151	241
R17.5C10	17.5	10	345	73	701	38	1151	241
R17.5C15	17.5	15	345	73	662	57	1151	241

Note: ^a fine aggregate, ^b coarse aggregate

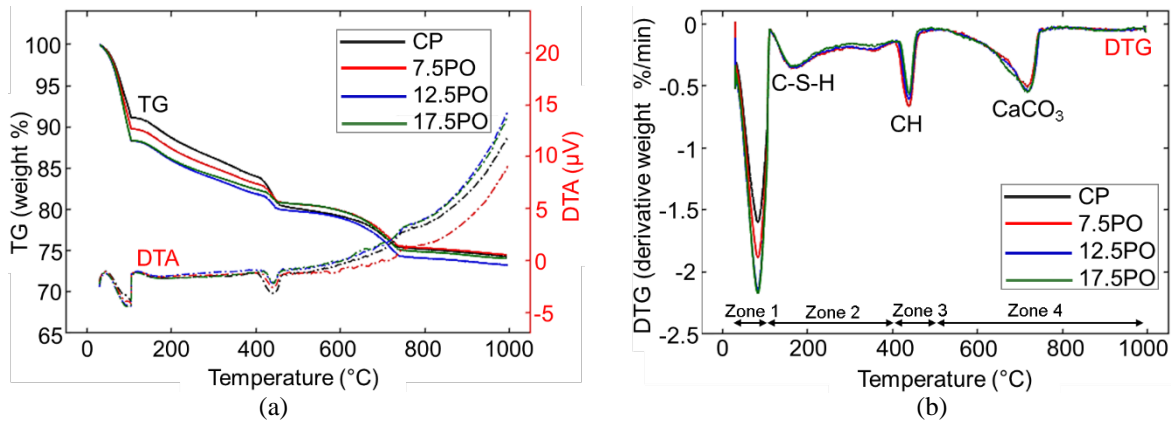


Fig. 2 TG-DTA results for RHA-O cement pastes: (a) TG-DTA curve and (b) DTG curve

90.91% and 76.18% amorphous silica, respectively. Hence, it can be concluded that RHA-O will be more reactive than RHA-T since it has more amorphous silica content.

Table 4 Chemical composition of RHA-O, RHA-T and OPC

Chemical analysis (%)	RHA-O	RHA-T	OPC
Silicon Dioxide (SiO ₂)	93.38	82.73	16
Aluminum Oxide (Al ₂ O ₃)	-	-	3.69
Ferric oxide (Fe ₂ O ₃)	1.35	1.1	2.93
Calcium oxide (CaO)	1.65	11.22	67.6
Moisture content (%)	0.36	0.29	-

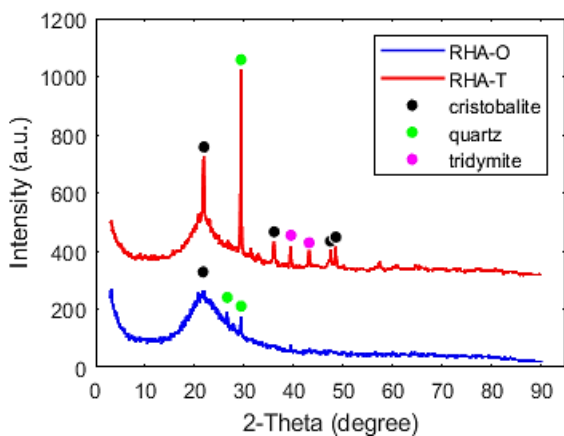


Fig. 1 XRD pattern of RHA-O and RHA-T

5.2 Thermogravimetric Analysis of Cement Pastes

Figures 2a show the profiles of thermogravimetric (TG) curves of 120-day cement pastes with RHA-O typical to cement pastes with RHA-T where gradual weight loss with increasing heating temperature can be observed. Differential Thermal (DTA) curves are also presented in the figure showing the temperatures at which reactions in the samples occur.

5.2.1 Thermogravimetric curves

From the DTG curves in Fig. 2b, four zones were defined where the sharp peaks occurred for all samples. Zone 1 is the first mass loss step corresponding to dehydration or loss of evaporable water [12]. Zone 2 characterizes the dehydration or loss of chemically bound water from C-S-H molecules [7]. Zone 3 indicates the dehydroxylation of calcium hydroxide (CH) [7]. Lastly, Zone 4 is associated with the decarbonation of calcium carbonate or calcite (CaCO₃) [8]. The temperature of peaks and decompositions for all cement pastes did not vary significantly thus no major change in phase chemistry of the products.

5.2.2 Calcium silicate hydrate (C-S-H) content

The formation of C-S-H gel with chemical composition 1.7CaO·SiO₂·4H₂O for cement pastes with pure Portland cement [10] and 2Ca_{1.5}SiO_{3.5}

$\cdot 2H_2O$ for RHA-blended cement pastes [11] were considered for its quantification by TG-DTA. Figure 3 presents the computed C-S-H gel from 120 days of hydration with mass loss from 110 °C to 400 °C. The formation of C-S-H gel increased from 22.37% in CP (control) to 33.09%, 32.06% and 29.54% with 7.5PO, 12.5PO, and 17.5PO, respectively. Similarly, the C-S-H content in 7.5PT, 12.5PT, and 17.5PT increased by 31.82%, 29.89% and 30.14%, respectively. However, C-S-H gel does not vary significantly with the replacement level of RHA.

It can be inferred that the addition of both RHA types as cement replacement can cause a significant increase in the C-S-H content. The mechanism of C-S-H increase can be explained by the pozzolanic activity of RHA where secondary C-S-H is produced from the consumption of CH from Portland cement hydration [11]. For cement pastes with RHA-O, the amount of C-S-H is higher than pastes with RHA-T except at 17.5% replacement. This is due to higher amorphous silica content and higher fineness of RHA-O as compared to RHA-T.

5.2.3 Calcium hydroxide (CH) content

The quantity of CH in cement pastes was calculated from the mass loss from 400°C to 500°C as shown in Fig. 3. The amount of CH decreased from 16.22% in CP to 10.32%, 8.73%, and 7.35% for 7.5PO, 12.5PO and 17.5PO, respectively. The same trend was observed for cement pastes with RHA-T where CH content diminished to 10.43%, 8.30% and 7.89% for 7.5PT, 12.5PT and 17.5PT, respectively. Further, CH in blended cement decreases with increasing RHA content. Between RHA-O and RHA-T at same level of replacement, CH content does not vary significantly.

The reduction in the CH content confirmed the pozzolanic activity of RHA-O and RHA-T. This is consistent with the results of the quantification of C-S-H gel where cement pastes with RHA exhibited more C-S-H gel content than with plain Portland cement. Boualleg et al. [14] reported that as the hydration of pure cement progresses, portlandite ions (Ca^{++} and OH^-) are released and captured by the pozzolan to form a new type of C-S-H gel. Based on the results, the reduction of CH is greatest in cement paste with 17.5% RHA-O (17.5PO) indicating the greatest pozzolanic reaction.

5.3 Effect of RHA in Mortar

5.3.1 Compressive strength

After 3 days, the compressive strengths of all mortars with RHA are lower than the control mortar (CM) except to the mortar with 7.5% RHA-O

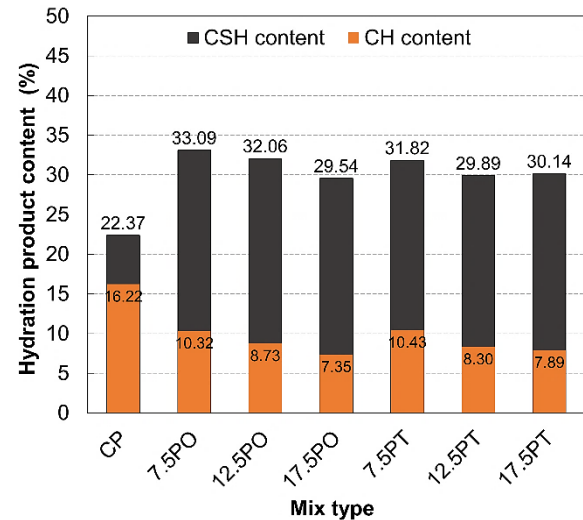


Fig. 3 Hydration product contents of cement pastes after 120 days

(7.5MO) as shown in Fig. 4. For 28-day samples, 12.5MO and 17.5MO exhibited comparable strength increase equal to 29.1% and 27.3% of the control, respectively. Mortars with 12.5% RHA-T (12.5MT) and 17.5% RHA-T (17.5MT) also increased in strength by 15.5% and 3.6% of the control, respectively. After 120 curing days, similar trend to 28 days is observed where 12.5MO, 17.5MO, 12.5MT, and 17.5MT showed strength increase equal to 23.0%, 14.0%, 17.9%, and 1.6% of the control, respectively. It is also evident that mortars with 12.5% RHA-O and RHA-T performed slightly better than mortars with 17.5%. Habeeb and Mahmud [15] reported that CH from the hydration of Portland cement may be insufficient to react with the available silica in RHA, thus leading to the incomplete pozzolanic reaction.

Researchers have reported that the compressive strength increase of mortars with RHA may be partially due to its high fineness and pozzolanic reaction [11], particularly the presence of highly amorphous silica in RHA enhancing its reactivity [4].

5.3.2 Microstructure

Prismatic CH and dense C-S-H gel were visible on the surface of control mortar (CM) at 120 curing days as seen in Fig. 5a. The prismatic CH is abundant and embedded on the C-S-H matrix since it is one of the main products from the hydration of Portland cement. In Fig. 5b, mortar containing 12.5% RHA-O showed a denser C-S-H than CM. The presence of CH also diminished. Production of secondary C-S-H from the pozzolanic reaction of RHA-O contributed to a denser C-S-H matrix and less CH when compared to the control. Pore

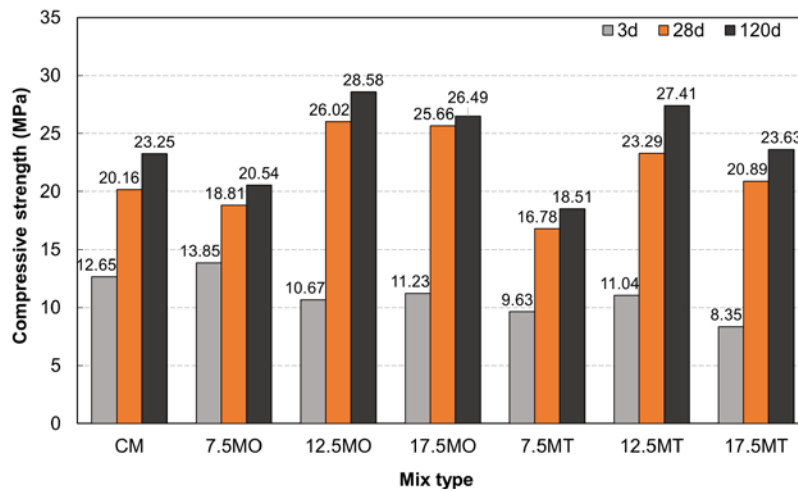


Fig. 4 Compressive strength development of RHA-blended mortars after 3, 28 and 120 days of curing.

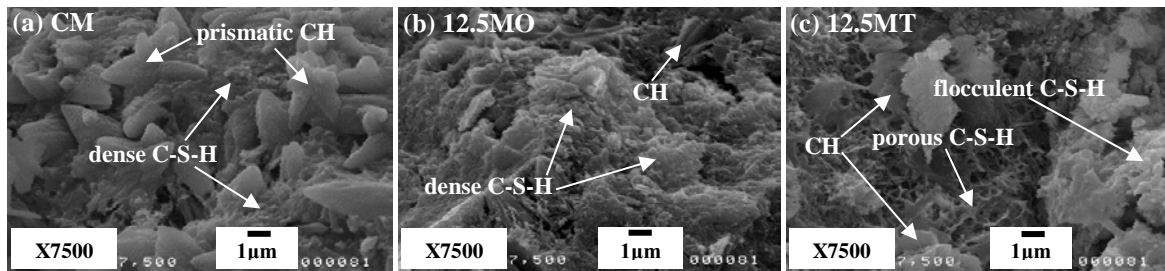


Fig. 5 Microstructure of (a) control mortar (CM), (b) mortar with 12.5% RHA-O (12.5MO) and (c) mortar with 12.5% RHA-T (12.5MT) after 120 days of curing

refinement of the microstructure of the mortar with 12.5% RHA-O resulted to the highest compressive strength. On the other hand, the microstructure of mortar with 12.5% RHA-T in Fig. 5c manifested porous and flocculent C-S-H gel with the presence of more voids and CH crystals.

5.4 Effect of RHA and CWRT on Concrete

5.4.1 Compressive strength

It can be observed in Fig. 6, R17.5C0 recorded the highest compressive strength improvement equal to 12.4% when compared to the control (R0C0). Comparing the results of sample concrete with CWRT only (R0C10) and samples with both RHA-O and CWRT (R17.5C5, R17.5C10, R17.5C15), it can be inferred that the incorporation of RHA-O can increase the compressive strength up to 10% CWRT. The compressive strength of R17.5C5 is comparable to the control with a slight decrease of 3.4%. The enhancement in strength by RHA-O is attributed to its pozzolanic effect. It is also observed that replacing fine aggregates with rubber decreases the compressive strength.

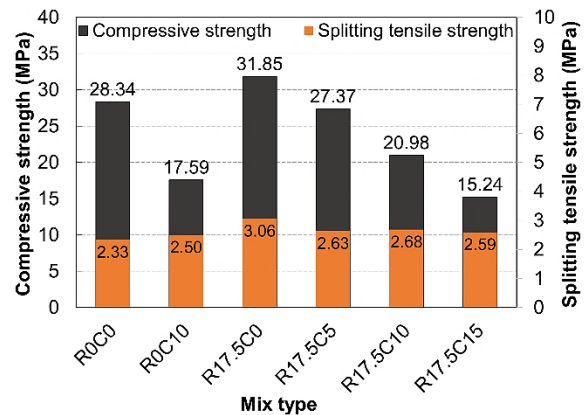


Fig. 6 Compressive and splitting tensile strength of concrete with RHA-O and CWRT after 28 days

5.4.2 Splitting tensile strength

In terms of splitting tensile strength, R17.5C0 gained the highest strength at 3.06 MPa with an increase of 31.2% of the control as shown in Fig. 6. It is also noted that additions of both RHA-O and CWRT showed remarkable tensile strength increases ranging from 7.2% up to 15.1% of the

control unlike in compressive strength where strength enhancement is only due to RHA-O. When both RHA-O and CWRT are incorporated, the concrete is seen to perform better than concrete with CWRT only (R0C10) but still lower strength than concrete with RHA-O only (R17.5C0).

6. CONCLUSION

Based on the analysis of results presented in the study, the following conclusions can be derived:

1. RHA- RHA-O and RHA-T exhibited high silica (SiO_2) contents equal to 93.38% and 82.73%, respectively. X-ray diffraction (XRD) analysis revealed that silica in RHA-O is mainly amorphous in form, whereas silica in RHA-T is partly amorphous and partly crystalline.
2. Simultaneous thermogravimetric and differential-thermal analysis (TG-DTA) can be used to quantify C-S-H and CH and examine the extent of pozzolanic reaction in blended cement pastes.
3. The amount of C-S-H gel is higher in RHA cement pastes than plain cement pastes after 120 days. C-S-H gel does not vary significantly with the RHA replacement level. Reductions in CH are observed on RHA-blended cement pastes at 120 days of curing. Increasing RHA content resulted in decreasing CH content confirming the pozzolanic activity of RHA.
4. Mortars prepared with 12.5% and 17.5% RHA-O showed excellent compressive strength gain after 28 and 120 days. To maximize RHA content, 17.5% RHA-O is used in concrete since their strengths are comparable.
5. Concrete with 17.5% RHA-O only yielded the highest compressive and splitting tensile strength after 28 days among all mix types. Increasing CWRT content decreases compressive strength. Additions of both RHA-O and CWRT resulted in higher splitting tensile strength than the control. Adding 17.5% RHA-O and 5% CWRT provided comparable compressive strength and higher splitting tensile strength than normal concrete which signifies the compensation of strength loss of CWRT by RHA.

7. ACKNOWLEDGMENTS

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