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DEVELOPMENT OF A CEMENTLESS ECO-BINDER AS AN ALTERNATIVE TO TRADITIONAL PORTLAND CEMENT IN CONSTRUCTION ACTIVITIES

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Abstract

In this research, the performance of a cementless eco-binder, a mixture of waste materials including slag, circulating fluidized bed combustion ash (CFA), and rice husk ash (RHA) was investigated, in which CFA acted as an activator. One hundred and twenty paste samples were prepared by using the RHA/(slag + RHA) ratios of 0, 15, 30, 45% while keeping a constant ratio of CFA/(slag + RHA) at 25%. The setting period, compressive strength, the ultrasonic pulse velocity (UPV), and drying shrinkage of paste samples were determined at the samples' age of up to 91 days. In addition, the microstructures of all paste samples were also characterized by scanning electron microscopy (SEM). It was found that the use of cementless eco-binder significantly increased the setting times, lower compressive strength, drying shrinkage, and UPV values compared to the control OPC sample. The maximum 91-day-old compressive strength gained by the binary binder of slag and CFA (R00C25) was 90% of that of the control specimen. Incorporation of RHA with higher replacement levels up to 45% resulted in a significant decrease in compressive strength up to 50%. Moreover, the SEM analysis revealed that there was a large difference in the microstructures of the control and the cementless eco-binder samples, in which the main hydration products were C-S-H/C-A-S-H gels and ettringite (AFt) due to relatively high amount of SO_3 and SiO_2 in the CFA and RHA, respectively. Thus, it can be realized that the potential for the use of slag, CFA, and RHA as a sustainable cement-free binder is promising in the construction industry, especially for lower strength or no required early high strength structures.

Keywords: cementless eco-binder; circulating fluidized bed combustion; rice husk ash; slag; microstructure; compressive strength; drying shrinkage; setting time; ultrasonic pulse velocity.

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1. Introduction

At present, concrete is recognized as the second most-consumed material (about 3 tons/person/year) in the world just behind water. And an important constituent of concrete is cement, typically ordinary Portland cement (OPC), which is considered as high energy consumption and significant contribution to global carbon dioxide emissions (around 5% and is the third-highest, man-made producer of

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CO_2, after transportation and energy) [1]. In developing countries, especially in Vietnam, with such extensive use of this material for further infrastructure improvement and rapid industrialization/ urbanization process comes a hefty increase in environmental problems. Recently, due to increasing concerns with global climate change and sustainable growth, there have been huge efforts on the use of waste products in construction materials. Various types of alternative binders instead of the conventional OPC, so-called 'cementless eco-binder', has been proposed and developed with the help of supplementary cementitious materials (SCMs) and chemical admixtures as activator agents. It is well-known for a long time that the use of SCMs in concrete, either as addition or as partial replacement of cement, not only significantly improves its workability, strength, and durability, reduces the cost but also attains the environmental benefits due to pozzolanic/ hydraulic activities and filling effect [2]. Among the SCMs, slag, circulating fluidized bed combustion ash (CFA), rice husk ash (RHA), fly ash, silica fume, etc. can be used individually or/and combined to create an innovatively green binder. In Vietnam, the blast furnace slag containing mainly of SiO_2, CaO, and Al_2O_3, a by-product from the steel manufacturing process, is discharged with gradually increasing volume in recent years and the following years. It is forecasted that by 2020, the amount of slag generated may reach 5-7 million tons, and by 2025 it may reach 10 million tons [3]. Therefore, it is essential to have solutions for promoting treatment, recycling, and limiting the landfill storage, and restraining negative effects on the environment. On the other hand, the CFA with a high content of CaO and SO_3 resulted from the clean-coal combustion in the power generation or coal plant industry. In fact, due to the mixing of fly ash and gypsum generated from factories using this technology, it is difficult to separate gypsum from fly ash, and thus further restrain to use it as a raw material for construction material production due to its excessive expansion [4, 5]. In 2016, the total ash volume generated in Vietnam was about 15,784,357 tons/year, of which CFA production was 5,102,461 tons/year, accounting for about 32%. Currently, the total amount of existing ash is around 22,705,558 tons [6]. Moreover, RHA mainly composed of SiO_2 is the by-product of rice husk after burning. In Vietnam, around 43 to 45 million tons of paddy rice are produced annually. The volume of rice husk accounts for 20% of the grain composition resulting in about 9 million tons of rice husks each year. And then once burning 10 million tons of rice husk, it yields about 1.8 million tons of rice husk ash every year [7]. Some previous studies revealed that the application of RHA as an SCM in manufacturing concrete and cement provides several advantages, such as improving the strength and durability properties [8, 9]. However, little research has been done to investigate the use of RHA in cement and concrete productions in Vietnam. As a result, until now there were no potential and alternative uses of RHA except for treating it as waste disposal resulting in environmental pollutions.

As aforementioned, it is easy to infer that the application of slag, CFA, and RHA with large quantity and sufficient quality in manufacturing construction materials in Vietnam is limited except for treating it as waste disposal resulting in environmental pollutions and lack of landfill sites. In addition, there is not much significant research on the utilization of SCMs in mortar and concrete as either an addition or a partial replacement of traditional OPC, especially slag, CFA, and RHA. It is because of its slow reaction rate and other issues related to its properties, such as toxic leachates, large ranges in nature, and the quality when being used as a main component in the composite binder. It is common that lime and anhydrite can be used respectively or combined together as the alkaline and sulfate activators of slag, stimulating the formation of C-S-H and ettringite (AFt), which will develop the slag strength during the hydration process. Additionally, self-cementitious properties of CFA are possibly the most useful for the synthesis of a hydraulic binder and it is also identified that the optimum utilization of CFA in the blended binder of slag and CFA was in a range of 15-25%

[10]. In previous work, the authors studied the engineering and durability properties of eco-friendly mortar developed from slag, CFA, and RHA, in which CFA acted as an activator [11, 12]. However, the leaching potential of heavy metals from these waste materials in Vietnam and the characterization of hydration products contributing to strength development and its properties of eco-binder still have not been yet examined and verified. To address the gap and extend the understanding on the sustainable green binder, this research was only to conduct paste samples incorporating various mixtures of three available by-products: slag and RHA in various ratios while CFA used at a constant ratio from the point of view of the setting times, compressive strength, the ultrasonic pulse velocity (UPV) and drying shrinkage. Especially, the microstructures of all paste samples were also characterized by scanning electron microscopy (SEM) and the leaching concentrations of heavy metals from the used materials were determined.

The typical characteristics of the starting materials were first described. And then, the results drawn from the experimental study, obtained by testing several eco-paste samples containing blends of slag, RHA, and CFA in comparison with the control OPC pastes, were presented and discussed. The significance of this research is to broaden the application of slag, RHA, and CFA by totally replacing traditional OPC as a binder in the construction industry, and thus significantly reduce the cost, save the natural resources, and offer further greenhouse benefits.

2. Experimental details

2.1. Characteristics of starting materials

A mixture of slag, rice husk ash (RHA), and circulating fluidized bed combustion fly ash (CFA), which were provided by local companies in Vietnam, was prepared as the cementless eco-binder in the research. In order to investigate the performance of this green type of binder containing various ratios of component materials, OPC with a density of 3.15 g/cm^3 was used as the control mixture. Table 1 shows the physicochemical properties of materials used while Figs. 1, 2, and 3 show the particle size distribution, the SEM images, and X-ray diffraction (XRD) patterns of these materials, respectively. It can be seen in Table 1 that the CFA contains mainly CaO and SO_3 (53.5 and 40.6% by weight, respectively) while the RHA composes predominantly SiO_2 (95.6% by weight), which can be realized as a source of pozzolan in compliance with ASTM C618 [13]. Fig. 1 reveals that the particle sizes of slag, RHA, and CFA were comparatively similar to the OPC particles. It was confirmed in Figs. 2 and 3, the RHA comprises primarily crystalline silica in form of cristobalite with a microporous structure and the CFA comprises various phases of quartz, portlandite, anhydrite, and lime. Although RHA is not fully an amorphous material (Fig. 3), it is believed that some of the very

Table 1. Physical-chemical properties of starting materials

Materials	Density (g/cm^3)	LOI (%)	Chemical compositions (% by weight)						
			SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Others
OPC	3.15	1.77	20.0	4.24	3.12	62.4	4.17	2.97	1.38
Slag	2.92	4.72	39.1	13.00	0.23	37.5	7.12	1.99	0.23
RHA	2.18	2.67	95.6	-	0.24	0.7	-	0.15	0.54
CFA	2.71	-	2.59	0.77	0.48	53.5	1.42	40.6	0.41

Note: LOI – Loss on ignition.

fine and active RHA particles may act as SCMs while the less active ones may act as the micro-fillers in the cementless eco-binder matrix. Furthermore, it was also observed that slag was an amorphous cementitious material, containing mainly of SiO_2 (39.1%), CaO (37.5%), a small amount of Al_2O_3 (13%), and the particle size of slag was smaller than that of the other components. It is worth noting that among these waste products, RHA possessed the smallest specific gravity due to its highly porous structure [14]. Furthermore, the heavy metal concentration in leachates of the starting materials was also examined by using the toxicity characteristic leaching procedure (TCLP) as shown in Table 2.

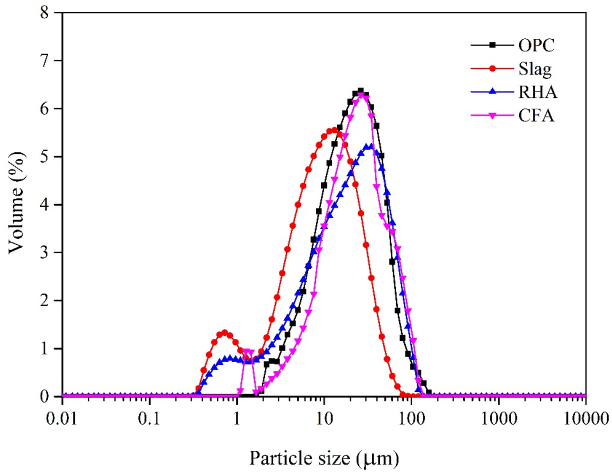
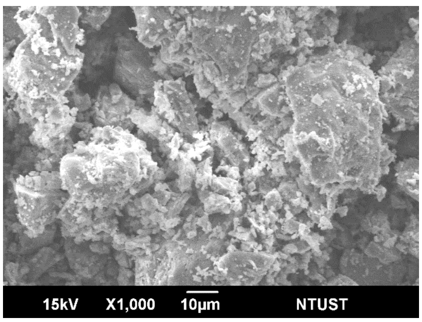
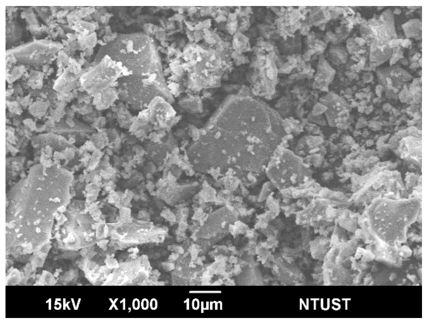


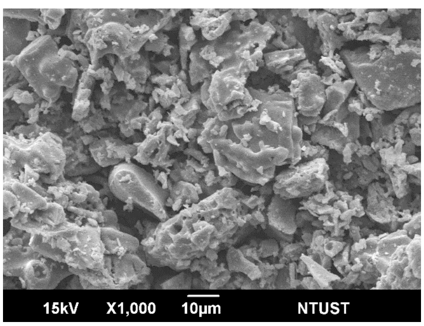
Figure 1. The particle size distribution of initial materials



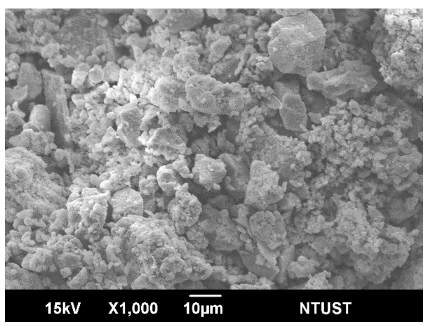
(a) OPC



(b) Slag



(c) RHA



(d) CFA

Figure 2. SEM images of starting materials

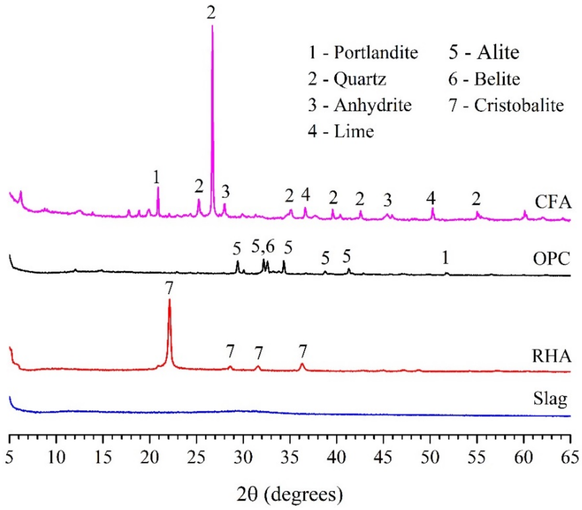


Figure 3. XRD patterns of initial materials

Table 2. Heavy metal concentration in TCLP leachates for the starting materials

Materials	Level of toxicity leached of heavy metals (mg/L)					
	Cu	Cr	Cd	Pb	Ni	Zn
Slag	N.D.	N.D.	0.013	0.023	N.D.	N.D.
RHA	N.D.	N.D.	0.010	0.033	N.D.	0.013
CFA	N.D.	N.D.	0.017	0.057	N.D.	N.D.
QCVN 07:2009/BTNMT	-	≤ 5	≤ 0.5	≤ 15	≤ 70	≤ 250
EPA (Taiwan)	≤ 15	≤ 5	≤ 1	≤ 0.05	-	-

Note: N.D. – None detection (< 0.005 mg/L).

The purpose of this study was to apply the industrial and agricultural wastes in the production of the cementless binder. As a result, it is essential to examine the leaching potential of heavy metals from these by-products to protect the environment from the negative effect of leached elements. Based on the TCLP test results, it can be considered that the slag, RHA, and CFA can be reused in the cementitious composite or eco-friendly binder manufacture as non-toxic wastes as per National Technical Regulation on hazardous waste thresholds enforced in Vietnam, namely QCVN 07:2009/BTNMT [15] as well as satisfying the Environmental Protection Administration (EPA) in Taiwan.

2.2. Mixture proportions

Table 3 shows the mix proportions used for preparing various cementless eco-binder paste samples, in which OPC100 mix is considered as the reference sample. In this study, four different ratios of RHA/(slag + RHA) were used to prepare the cementless eco-binder mixtures as 0, 15, 30, and 45%, respectively. Moreover, according to previous research, it can be stated that the optimum utilization of CFA in the blended binder of slag and CFA was in the range of 15-25% [10]. Therefore, a constant ratio of CFA/(slag + RHA) of 25% was used in this research. The water-to-binder ratio was kept constant at 0.4 for all mixtures. Local tap water was used as the mixing water for all mixtures in this experiment.

Table 3. Paste mixture proportions of cementless eco-binders

Mix designation	RHA (wt.%)	OPC (kg/m^3)	Slag (kg/m^3)	RHA (kg/m^3)	CFA (kg/m^3)	Water (kg/m^3)
OPC100	-	1394	0	0	0	558
R00C25	0	0	1070	0	267	535
R15C25	15	0	893	158	263	525
R30C25	30	0	722	309	258	516
R45C25	45	0	557	456	253	507

2.3. Test programs and sample preparation

Setting time is one of the important properties that need to be considered once placing the cementitious composite in position. Therefore, to examine the influence of industrial waste types on the fresh properties, different paste samples were proportioned and mixed as mentioned above, then the period of setting: initial and final setting times of paste samples were determined by the Vicat method according to ASTM C191 specification [16].

The compressive strength test was performed to evaluate the strength development of the paste samples incorporating various types and ratios of waste materials. The control sample (OPC100) was also tested for data in comparison with other eco-binder samples. The test was performed at 3, 7, 14, 28, 56, and 91 days of curing time using cubes of size 50 × 50 × 50 mm in compliance with ASTM C109/109M [17]. Prior to testing, these samples were air-cured at a temperature of 27 ± 2°C and relative humidity (RH) of 65 ± 5%. The average test of three specimens for each group of samples was assumed as the compressive strength for different curing times up to 91 days.

Drying shrinkage is considered as an important feature of hardened cement pastes that affecting the durability. For this purpose, the prismatic paste samples of 25 × 25 × 285 mm were prepared for testing the shrinkage behavior. After de-molding, the samples were placed in open-air conditions (27 ± 2°C temperature and 65 ± 5% relative humidity). The drying shrinkage of the paste samples was monitored for up to 91 days in accordance with the measurement procedures described in ASTM C596 [18].

Ultrasonic pulse velocity (UPV): In order to assess the quality and uniformity of paste samples in terms of the internal structure or an indirect indicator for mechanical properties, the UPV test, kind of non-destructive tests, was applied for all samples in this study. Several cylindrical paste samples of size 100 mm diameter × 200 mm height were cast for the UPV test. After casting, these samples were air-cured at a temperature of 27 ± 2°C and relative humidity (RH) of 65 ± 5%. The test was conducted at designed testing ages of 28, 56, and 91 days in compliance with the standard test procedure ASTM C597 [19].

Microstructure analysis: The microstructural characteristics of the paste samples were observed by using SEM analysis. After the 28-day compression test, some sample pieces were collected and soaked in methyl alcohol to stop further binder hydration and then prepared for SEM analysis. Before starting the test, the samples were covered with a platinum-palladium alloy by using an auto-fine coater and then vacuum-dried using a 15kV beam. SEM observations were performed using an SEM model JEOL JSM-6390LV in the laboratory.

3. Results and discussion

3.1. Setting time

The setting times of the eco-binders and especially the final setting of OPC also were far beyond the maximum specified limit of 420 min mentioned in the ASTM standard [19, 20]. This is most likely due to the relatively high water-to-binder ratio used and the inherent slow reactivity of pozzolanic materials for the synthesis of cementless eco-binder. As can be seen in Fig. 4, the initial and final setting times of OPC100 were 387 and 1434 min, respectively. Whereas, the initial and final setting times of the R00C25, R15C25, R30C25, and R45C25 samples were 1456, 1560, 1669, and 1735 min and 1643, 1692, 1781, and 1869 min, respectively. Moreover, while the setting period of paste sample using 100% OPC was 1047 min, the period between initial and final setting times for samples 0, 15, 30, 45% replacement ratios of RHA were 187, 132, 112, and 134 min, respectively. Thus, the use of SCMs as binders instead of OPC significantly prolong the initial setting time and shorten the setting period of paste samples. As above mentioned, the delay in the initial setting of the eco-binders is due to the combined effect of a high water-to-binder ratio and slow reactivity (e.g. stable crystal phase of silica in RHA, as shown in Fig. 3) of the SCMs in the system [21].

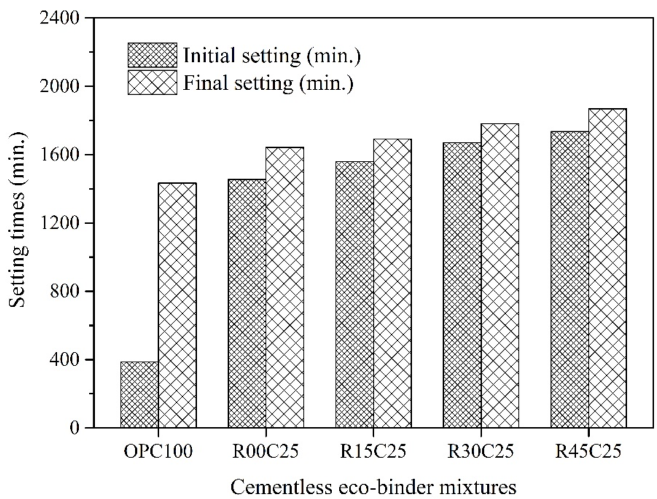


Figure 4. Setting times of cementless eco-binders

Moreover, it can be observed that the setting periods of the mixture containing SCMs were less than that of the control one independent of dosage used and even lesser when the replacement ratios of RHA increased up to 45%. Among all of the eco-binders, the initial and final setting times of the R45C25 sample was the longest, while the shortest setting duration was observed in the R00C25 sample (just accounted for 11% compared with the control sample). This obtained result can be explained that due to the nature of high porosity structure (Fig. 2(c)) and lower density of RHA (Table 1) in comparison with slag. The more slag replacement ratio by RHA is, the more water-to-binder ratio reduces because of the increment in volume of the binder, as a result, the setting duration of paste sample decreases.

3.2. Compressive strength

The results of compressive strength for all mixtures at 3, 7, 14, 28, 56, and 91 days were given in Fig. 5. The compressive strength of cement-free binders was less than the control OPC100 mixture regardless of the types of waste materials used, replacement levels of RHA, and the testing ages. For instance, the 91-day compressive strength values of SCM samples ranged from 25.4 to 46.7 MPa (accounted for 50-90%) in comparison to the corresponding reference paste of 53.4 MPa. This reduction trend observed in the cementless binder may be explained by the slower reaction rate of SCMs in the blended system [11, 21].

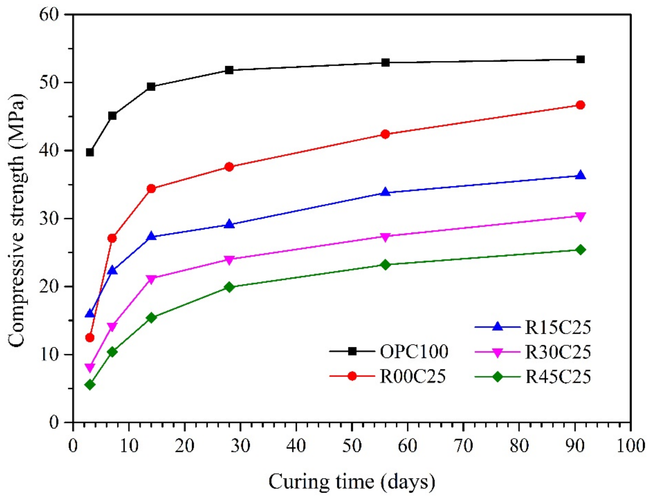


Figure 5. Compressive strength development of cementless eco-binders

In addition, the compressive strength development of all samples increased with hydration periods, and especially the rate of strength increment was significant at the later period of the eco-binders in contrast to the low strength development in the reference sample. It was easy to observe from Fig. 5, the increase in strengths at 91-day testing compared to 28-day strength for eco-binder samples ranged from 24-28% while the increment in the corresponding reference OPC100 sample was only 3%. The further strength increase in paste samples containing SCMs can be attributed to the continuous formation of C-S-H/C-A-S-H gels and AFt during the pozzolanic reaction process as mentioned in previous research [12].

However, with a varying amount of RHA in the mixture, it was clearly found that the compressive strength of cementless binder samples significantly affected. As shown in Fig. 5, the compressive strength of these samples was smaller than that of the reference R00C25 mixture (without RHA), especially once increasing the dosage of RHA in eco-binder. Typically, at 91-day, the compressive strengths of cementless paste samples containing 0, 15, 30, and 45% RHA content were 46.7, 36.3, 30.4, and 25.4 MPa, respectively. As above mentioned, the CFA used in this study, characterized with a high amount of CaO and SO_3 (Table 1), was performed as an activator for the hydration of cementless binder, in which the main products composed of C-S-H/C-A-S-H gels and AFt, contributing to the strength development of the eco-binder [11, 21]. Therefore, the 91-day compressive strength value of the R00C25 sample was slightly decreased (accounted for 90%) compared to the OPC100 sample. On the other hand, increasing RHA content was associated with incorporating less slag in the paste samples. As a result, there was a reduction in both amounts of portlandite and alumina, which contributed to the formation of C-S-H/C-A-S-H during the hydration process [12]. Therefore, the more slag replacement level by RHA content uses, the more strength reduction occurs as clearly seen in Fig. 5.

3.3. Drying shrinkage

Fig. 6 presents the development of drying shrinkage strain in all paste samples over curing time up to 91 days. In general, the shrinkage strain increased with time exposure, especially during the early age period. The result in this figure showed that the eco-binder samples displayed smaller values of dry shrinkage among all mixes, at all replacement ratios and exposure periods. For example, up to 7 days, the drying shrinkage strains of the SCM samples were comparable (just slightly smaller up to 20%) to that of the reference ones, except for the R45C25 mixture. Beyond 7 day-exposure, while the obtained values of OPC100 and R00C25 samples were just less than 20% difference, there was a significant difference between samples containing RHA and remaining ones. The more content of RHA incorporates in the blended system, the more reduction in drying shrinkage it is. For instance, the 91-day-age samples incorporating 25% CFA and RHA of 0, 15, 30, and 45% had a relative shrinkage of 90, 70, 60, and 50% compared to that of OPC100 samples. In this experiment, RHA has a significant positive effect on the shrinkage property of the eco-binder that could be attributed to slower hydration reactivity, indicating by its stable crystal form (Fig. 3), and higher microspores, resulting in the internal curing effect, reduced heat of hydration [21–23]. Additionally, with the optimal ratio of CFA in the blended system, it was believed that the internal structure of paste sample was dense enough to restrain the free water penetration due to the increase in the volume of AFt that reduced the shrinkage [11].

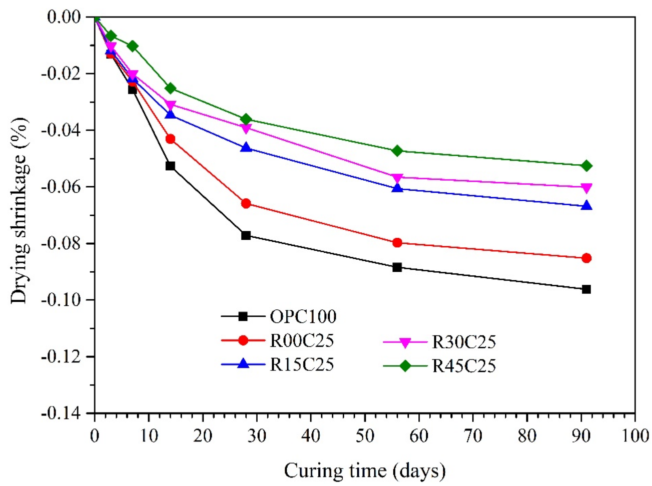


Figure 6. Drying shrinkage of cementless eco-binders

3.4. Ultrasonic pulse velocity

The UPV test was performed to obtain preliminary information on the internal structure or the compressive strength of the materials. It is the non-destructive test method, which is used to assess the quality of the solid materials [24]. In addition, the UPV values vary depending on both density and compositions of the materials. The UPV values for all test samples obtained in this experiment were reported at various curing ages of 28, 56, and 91 days as seen in Fig. 7. Compared with the control paste OPC100, it is clear that there is a reduction in the UPV observed in eco-binder samples, especially in samples containing higher RHA content and it was observed that the UPV increases with the time exposure of curing. For example, at 28-day, the sample without RHA content obtained the highest UPV value of 3276 m/s (reduced by 7% compared to that of OPC100) among all eco-binder series, while the 45% RHA sample presented a significant decrease in UPV value (reduced by 17% compared to that of OPC100). This could be explained through the decrease in the volume of hydration products (Fig. 8(e)) and higher porosity of RHA particles (Fig. 2(c)), due to changes in mineralogical compositions of cementless eco-binders. Thus, in the samples incorporating waste materials, the increased number of pores and C-S-H/pore interfaces delayed propagation of the ultrasonic pulse, leading to reduced velocity [25, 26]. The obtained results mostly confirm the compression results. There was a similar tendency in Figs. 5 and 7, in which the sample obtained higher UPV value corresponding with good quality or higher compressive strength of paste sample.

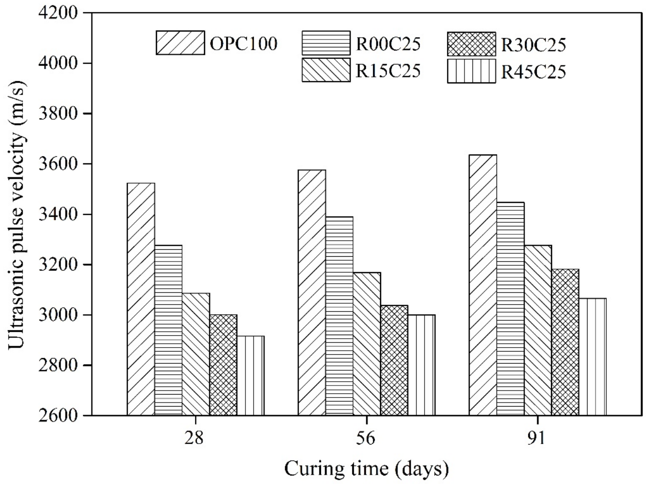


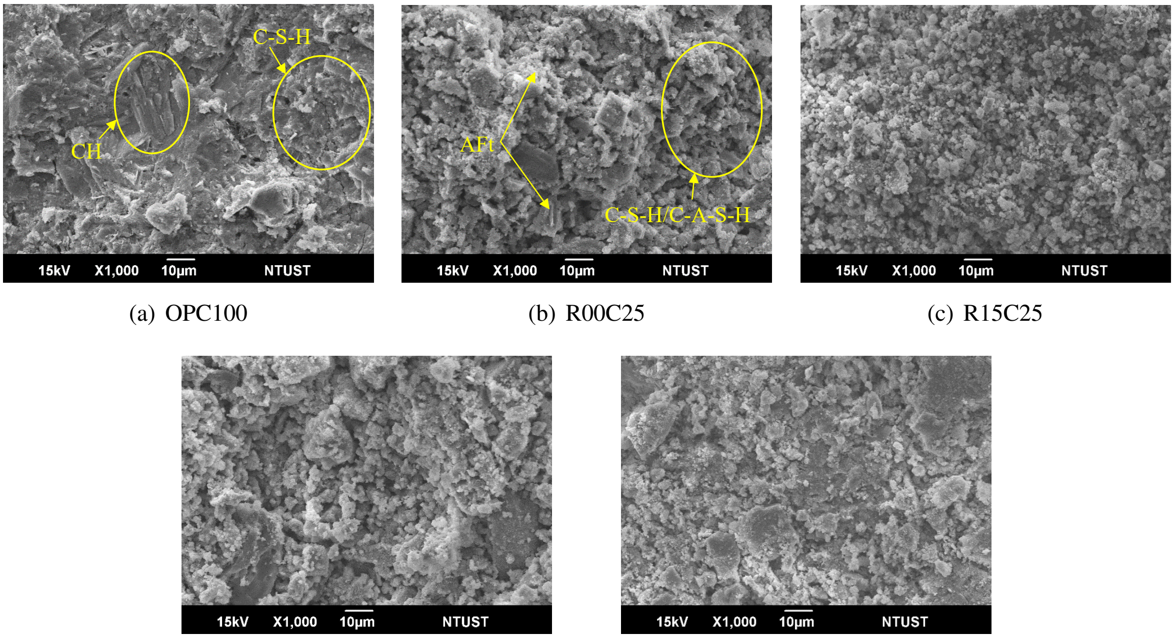
Figure 7. Ultrasonic pulse velocity through cementless eco-binders

Furthermore, the UPV values of the 91-day-old paste samples containing 0-45% RHA ranged from 3066 to 3477 m/s, demonstrating that the quality of paste mixtures incorporating waste materials as an alternative to OPC can be graded as medium according to BIS: 13311-92 and Neville [27]. Therefore, this proves its use for various construction activities, especially for low-strength or no required early high strength structures.

3.5. Microstructure analysis

SEM analysis was conducted to verify the effect of SCMs on the microstructure of the cementless pastes. After 28 days, the SEM images of all samples were shown in Fig. 8.

It can be seen in Fig. 8 that the main hydration products of the control paste OPC100 having cement as the only binder were amorphous C-S-H gel and portlandite (CH) (Fig. 8(a)), while the main ones of eco-binder samples were C-S-H/C-A-S-H and AFt (in form of crystal-like) (as pointed out in Fig. 8(b)) due to relatively high amount of SO_3 and SiO_2 in the CFA and RHA, respectively. In addition, the existence of CH was observed in the SEM image of the OPC100 sample, whereas CH was less in the SEM images of paste samples incorporating cement eco-binders, it contained a sponge-type gel structure. Therefore, it can be inferred that there was a large difference in the microstructures of control and alternative samples. In this study, CFA was considered as an important activator for stimulating the chemical reaction during the hydration process of the cement-free binder [10, 28]. The formation of C-S-H/C-A-S-H and AFt resulted in the strength development of eco-binder samples with an increase in curing duration [29]. However, it can be observed in Figs. 8(b)–8(e) that with



(a) OPC100 (b) R00C25 (c) R15C25

(d) R30C25 (e) R45C25

Figure 8. SEM images of resulting cementless eco-binders

the increasing of the amount of RHA content in eco-binder, the porosity of paste samples were also increased due to its nature of microspore structure as well as its pozzolanic properties. As a result, the eco-binder samples containing the higher volume of RHA possessed lower compressive strength as seen in Fig. 5. This finding was in good agreement with the results of the previous study [12].

4. Conclusions

The following conclusions can be drawn based on the experimental results obtained in this research:

- Based on the TCLP test results for the starting materials used in this study, it can be concluded that the slag, RHA, and CFA can be reused in the cementitious composite manufacture for construction activities as non-toxic wastes.

- The 91-day-old compressive strength of all cementless eco-binder paste mixtures ranged from 25.4 to 46.7 MPa that accounted for 50-90% that of reference paste. At a curing age of 91 days, the paste samples incorporating 25% CFA and without RHA content exhibited the maximum compressive strength among all of the cementless samples while the strength value was lower in samples containing higher replacement ratios of slag by RHA materials.

- Varying RHA content up to 45% in the blended eco-binder system showed a significant and negative influence on the setting times and UPV values compared to the control OPC sample. In contrast, with a constant ratio of CFA at 25%, the more content of RHA incorporates in the blended system, the more the reduction in drying shrinkage it is.

- There was a reduction in the UPV observed in eco-binder samples, especially in samples containing higher RHA contents. On the other hand, SEM image analysis revealed that the main hydration products of the eco-binders were C-S-H/C-A-S-H gels and AFt due to the presence of the relatively high amount of SO_3 and SiO_2 in the CFA and RHA, respectively.

- The results of this study reveal the promising potential for the use of slag, CFA, and RHA as a sustainable cement-free eco-binder in the construction industry, especially for structures or construction materials that no require relatively high and early compressive strength.

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