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Contributed Paper

Effects of Water Content on Compressive Strength of Eco-friendly Light-weight Cement Blocks Using Cement-like Material Prepared from Agricultural Wastes

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ABSTRACT

Limestone quarrying process is recognized as an initial step in cement production, which produces high content of dust and creates detrimental effects on both the environment and human health. Utilization of agricultural wastes as alternative raw materials is one of the routes to solve such problem. This research is intended to produce eco-friendly light-weight cement blocks from a cement-like material. The cement-like material was synthesized by the low-energy consumption solution combustion technique, using cockleshell and rice husk ash as raw materials. Phase identification analysis of the synthesized cement-like powder indicated the expected phases of tri-calcium silicate (C_3S), di-calcium silicate (C_2S), tri-calcium aluminate (C_3A), and tetra-calcium alumino ferrite (C_4AF). To fabricate eco-friendly light-weight cement blocks, the synthesized powder was mixed with Ordinary Portland Cement (OPC), rice husk ash (RHA) and water, with the weight ratio of the synthesized powder:OPC:RHA equal to 25:25:50. The mixture was subsequently cast into light-weight cement blocks. Effects of water contents on compressive strength and bulk density of the light-weight cement blocks were examined. Experimental results revealed that compressive strength of the samples ranged between 1.1 MPa and 2.1 MPa, whereas density ranged from 1.01 g/cm^3 to 1.14 g/cm^3. The compressive strength of the samples in this study were in a comparable range with that of light-weight concrete defined by Thai Industrial Standards Institute (TISI) and American Concrete Institute (ACI 213,2001). The optimal condition to achieve the standard properties of light weight cement blocks is water-to-binder ratio of 1.3:1 by weight. Results from the current study, therefore, suggested that the eco-friendly light-weight cement blocks obtained from this study exhibited potential practical usage.

Keywords: eco-friendly light-weight cement blocks, combustion synthesis, waste, cockleshells, rice husk ash

1. Introduction

It has been generally accepted that construction industry makes a significant contribution to economic development worldwide. Researches in the field of advanced construction materials have played important roles in promoting advancement of construction technology, as well as solving problems related to social and environmental impacts. At present, key raw materials used in the production of Ordinary Portland Cement (OPC) are limestone, which is the source of calcium oxide, shale and clay, which are sources of alumino silicate. These materials are usually obtained from quarrying processes, which cause harmful effects on the environment. Utilization of agricultural wastes, such as cockleshells and rice husk ashes, as alternative raw materials is one of the routes to alleviate such problem. M. Riccardo *et al.*, 2018 revealed that 1 ton of cement produces 1 ton of CO_2, which indicated 7% from quarrying limestone and 35% from fuel. It was shown that the energy consumption was very high due to the high temperature of 1500°C used in the process. In this study the calcination temperature is 900°C, which decreases the energy consumption and can reduce the production of CO_2 up to 20% compared with conventional cement production [1].

OPC mainly consists of tri-calcium silicate (C_3S, Ca_3SiO_5), di-calcium silicate (C_2S, Ca_2SiO_4), tri-calcium aluminate (C_3A, $Ca_3Al_2O_6$,), and tetra-calcium alumino ferrite (C_4AF, $Ca_4Al_2Fe_2O_{10}$) phases. Alternative raw materials with high content of calcium and silica, therefore, can be utilized as substitute materials for limestone and clay in the production of cement-like material.

With high calcium content, cockleshells can be used as an alternative raw material for cement production. Numerous studies have reported that calcium oxide (CaO) phase ranging between 96 and 97 wt% purity was observed after calcination of cockleshells at 900°C [2][3]. Seashells calcined at 700°C, 800°C, and 900°C could also provide calcium oxide with 95.6 wt%, 98.5 wt%, and 96.3 wt% purity, respectively [4]. According to P-.L. Buasri *et al.*, 2012 mussel shells and scallop shells calcined at temperatures ranging from 700°C to 1000°C could result in formation of calcium oxide as high as 98 wt% purity [5]. Owing to high silica content, rice husk ash can also be used as an alternative raw material in the production of cement-like material. According to M. Monshizadeh *et al.*, 2009 rice husk ash calcined at 700°C contained as high as 98.14 wt% silica [6]. A decrease of silica content from 98 to 86 wt% was observed at lower calcination temperature of 450°C [7].

In addition to replacing original raw materials with eco-friendly agricultural wastes, exploitation of robust cement processing techniques can also be a route to sustainability. Since it has been generally accepted that one ton of cement production can generate roughly one ton of carbon dioxide, therefore reducing cement processing temperature may contribute to minimizing the use of fossil fuel in the production of cement, consequently leading to a reduction of carbon dioxide emission. Conventionally, solid state reaction, a common firing process at the temperatures ranging from 1400 to 1600°C, is employed in the synthesis of cement with main constituent products consisting of tri-calcium silicate, di-calcium silicate, tri-calcium aluminate and tetra-calcium alumino ferrite. These aforementioned cement constituents can also be obtained by various fuel-efficient techniques with much lower processing temperatures. It has been reported that di-calcium silicate powder could be synthesized by flame spray pyrolysis and solution combustion methods at the temperatures ranging from 300 to 910 °C [8]. According to N.B. Singh *et al.*, 2006 β-di-calcium silicate (β-Ca_2SiO_4) powder could be successfully synthesized by hydrothermal method and calcined at 600 °C, using rice husk ash and fly ash as main raw materials [9]. Tri-calcium silicate powder with a minor secondary phase of calcium oxide (CaO) had also been synthesized by the sol-gel method and calcined at 1300°C [10]. R. Lanoş *et al.*, 2009 also succeeded in synthesizing tri-calcium aluminate powder by solution combustion technique. The synthesized powder, however, contained some secondary phases. Upon calcination at 900°C, the powder containing $3CaO{\cdot}Al_2O_3$, $12CaO{\cdot}7Al_2O_3$, and CaO were observed [11]. Tetra-calcium alumino ferrite powder was also achieved by self-propagating combustion reaction method [12][13]. According to a previous study from O. Jonprateep *et al.*, 2014 cockleshells and rice husk ash could be exploited as main raw materials for synthesis of di-calcium silicate and tri-calcium silicate phases by solution combustion technique [3].

Since cement hardens as a result of a chemical reaction between cement and water, another important aspect for practical uses of cement is how it is mixed with water. In general, water–to-cement ratios ranging between 0.40 and 0.76 are used in commercial concrete [14]. Nevertheless, required water content is fundamentally dependent on particle size of cement powder. As finer cementitious or cement-like materials of smaller particle size are used as cement substitutes, higher water content is required. Numerous studies indicated that an increase of water content was required when fine particles of rice husk ash was added. In the case when rice husk ash of 12.5-50 wt% was added, water/binder (W/B) ratio increased to 0.65-1.43. Excessively high water content, however, led to diminished compressive strength [15]. For this reason, determination of optimal water content is essential to ensure the compressive strength required for practical uses.

For practical uses, light-weight cement blocks are required to have compressive strength and density in a comparable range with that defined by industrial standards. According to American Concrete Institute (ACI), acceptable ranges of compressive strength and density for structural light-weight aggregate concrete (ACI 213, 2001) are 0.7-2.0 MPa, and 300 to 800 kg/m^3 (0.3 to 0.8 g/cc), respectively [16]. Compressive strength and density requirements for Type C6, C7 and C8 light-weight concrete defined by Thai Industrial Standard Institute (TISI.2601-2556) are 2.0 MPa (20.4 Kg/cm^2) and 501-800 Kg/m^3 (0.5-0.8 g/cc), respectively [17].

The aforementioned researches confirmed that agricultural wastes as well as fuel-efficient processing techniques could be exploited in cement production. Previous studies also denoted the importance of water to cement ratios. This research, therefore, intended to synthesize cement-like materials by the low-energy consumption solution combustion technique, using rice husk ash and cockleshells as alternative raw materials. The effects of water content on compressive strength and density of the eco-friendly light-weight cement block were also examined.

2. MATERIALS AND METHODS

2.1 Preparation of Cement-like Material

Preparation of cockleshells as raw material for synthesis of cement-like materials included thorough cleaning and calcining the cockleshells at 900°C for 3 hours. The cockleshells were subsequently ground by ball milling technique to obtain fine powder. Rice husk ash (DhebKaset Industrial Co., Ltd) was ground and screened using a 325-mesh sieve to obtain ash with particle size smaller than 45 micrometers.

Synthesis of cement-like powders by solution combustion technique involved dissolving the prepared cockleshells and rice husk ash in 70% AR grade nitric acid (HNO_3, RCI Labscan). To attain the powder containing di-calcium silicate (C_2S) and tri-calcium silicate (C_3S), the cockleshells and rice husk ash were mixed at the molar ratio of 0.85:0.39. To prepare tri-calcium aluminate (C_3A) powder, cockleshells and aluminium nitrate nonahydrate ($Al(NO_3)_3$, Daejung) were mixed at the molar ratio of 0.72:2.78. For preparation of tetra-calcium alumino ferrite (C_4AF), cockleshells, aluminium nitrate nonahydrate ($Al(NO_3)_3$, Daejung) and 95% ferric oxide red (Fe_2O_3, Lobachemie) were mixed at the molar ratio of 0.81:2.36:0.50. Glycine ($C_2H_5NO_2$, Daejung) and deionized water were added to each solution to achieve the concentration ranging between 0.1 and 0.2 M. To initiate the combustion process, the solution was heated on a hotplate at a temperature close to 400°C. After the combustion was completed, the synthesized powders were collected and calcined at 900°C for 3 hours.

2.2 Casting of Light-weight Cement Blocks

The synthesized powders containing di/tri-calcium silicate, tri-calcium aluminate and tetra-calcium alumino ferrite were mixed with the ratio of 0.79:0.13:0.09. 3 wt% of gypsum was added to the powder mixture. Rice husk ash, which acted as a pozzolanic material, making up 60 wt% of the total cementitious materials, was added to the powder mixture. Subsequently, aluminium powder, which functioned as a foaming agent, was also added. Water was added to the powder mixtures with water to cementitious material ratios of 1.2, 1.3, 1.4, 1.5, and 1.7 to obtain homogenous pastes. The pastes were cast in 2.5 × 2.5 × 2.5 cm^3 molds and cured in water for 7 days.

2.3 Characterization

Elemental compositions of the raw materials were examined by an x-ray fluorescence technique (Horiba, XGT-5200), whereas phase identification of the synthesized powders and the cast light-weight cement blocks was investigated using an x-ray diffractometer (Philips, X'Pert) over angles ranging from 10°-70° in 2 with a step size of 0.02°/min. A scanning electron microscope (SEM) (FEI, Quanta 450) was employed in microstructural examination. Compressive strength of the light-weight cement blocks was tested by a universal testing machine (Hounsfield, H50KS).

3. Results and discussion

3.1 Chemical Composition of Raw Material

Chemical compositions of rice husk ash and cockleshells were examined by x-ray fluorescence spectrometer and are shown in Table 1. The results revealed that rice husk ash consisted of 93.193 wt% silica, whereas 99.61 wt% calcium oxide was observed in cockleshells.

These results were in a comparable range with those reported in previous studies. According to the study previously conducted by O. Jonprateep *et al.*, 2014 89.7 wt% silica and 95.7 wt% calcium oxide were observed in calcined rice husk ash and cockleshells, respectively [3]. Other researchers also reported similar observations. According to Mustakimah et al, cockleshells contained relatively high amount of calcium oxide compared to other sea shells. While 86.57 wt% calcium oxide was found in cockleshells, only 50.45 and 54.53 wt% calcium oxide were evident in mussel shells and scallop shells, respectively [18]. I.R. Umasabor *et al.*, 2018 reported silica content in rice husk ash to be 87.22 wt%. [19]. Silica content ranging from 73 to 86 wt% in rice husk ashes, was reported by M. Vigneshwari *et al.*, 2018 and R.M. Shatat *et al.*, 2013 [20-21].

Table 1. Chemical composition of raw materials used in synthesis of cement-like powders.

Composition (wt %)	K_2O	CaO	FeO
Rice husk ash	3.810	1.672	0.890
Cockleshells	-	99.609	0.047

Composition (wt %)	SrO	SiO_2	MnO	Al_2O_3
Rice husk ash	-	93.193	0.433	0.002
Cockleshells	0.344	-	-	2.784

With high silicon and calcium contents, results from the current study suggested that rice husk ash and cockleshells were potential candidates for raw materials to be used in the synthesis of cement constituents.

3.2 Phase Identification of the Synthesized Powders

After calcination, the phases of the synthesized powders were identified by x-ray diffraction technique. The powder prepared from initial reagents containing calcium (cockleshells) and silicon (rice husk ash) showed a diffraction pattern with prominent peaks corresponding to tri-calcium silicate (Ca_3SiO_5, JCPDS 00-031-0301) and di-calcium silicate (Ca_2SiO_4, JCPDS 00-029-0369), as shown in Figure 1. Compared with the previous study conducted by O. Jonprateep *et al.*, 2014 [3] the current study exhibited superior chemical composition with lower content of secondary phases. Results from the current study showed subtle peaks at 37°, 54°, 64° and 69° 2θ suggesting the presence of only a small content of calcium oxide (CaO, JCPDS 01-077-2010).

The powder prepared from initial reagents containing calcium and aluminium showed an x-ray diffraction pattern with major peaks corresponding perfectly to tri-calcium aluminate ($Ca_3Al_2O_6$, JCPDS 00-008-0005) as shown in Figure 2. It was noted that only diffracted peaks of the main phase were evident.

As shown in Figure 3, an x-ray diffraction pattern containing prominent peaks corresponding to tetra-calcium alumino ferrite ($Ca_4Al_2Fe_2O_{10}$, JCPDS 00-011-0124) was observed in the powder prepared from initial reagents containing calcium, aluminium and iron. Low intensity peaks identified as calcium oxide (CaO, JCPDS 01-077-2010) were also evident. The presence of minor calcium oxide secondary phase might be attributed to incomplete combustion process.

A semi-quantitative analysis was employed to estimate the content of calcium oxide secondary phase, according to the following Klugg's equation:

$$f = 1 - \frac{[\frac{I_1}{I_1+I_2}]A_2}{A_1 - [\frac{I_1}{I_1+I_2}][A_1 - A_2]} \quad (1)$$

where f is the weight faction of the secondary phase (calcium oxide), I_1+I_2 is the integrated intensity of primary phase (tetra-calcium alumino ferrite) and secondary phase (calcium oxide), I_1 is the integrated intensity of primary phase, A_2

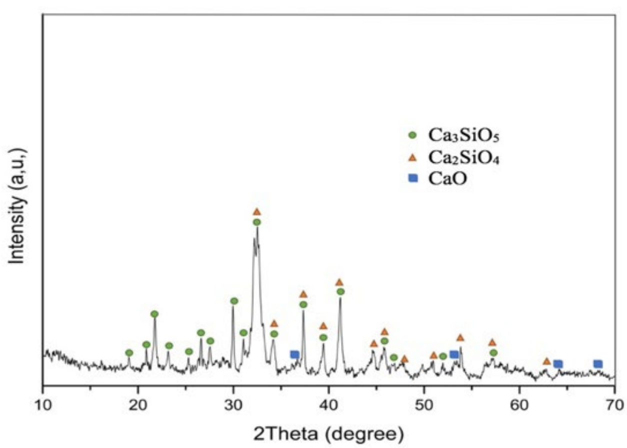

Figure 1. X-ray diffraction pattern of the synthesized powder containing tri-calcium silicate (Ca_3SiO_5) and di-calcium silicate (Ca_2SiO_4)

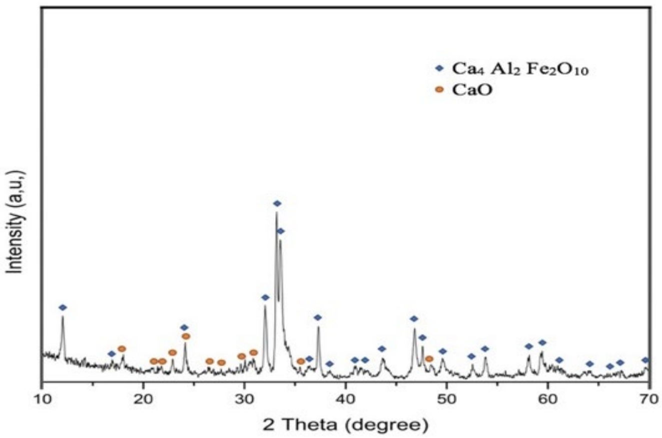

Figure 2. X-ray diffraction pattern of the synthesized powder containing tri-calcium aluminate ($Ca_3Al_2O_6$).

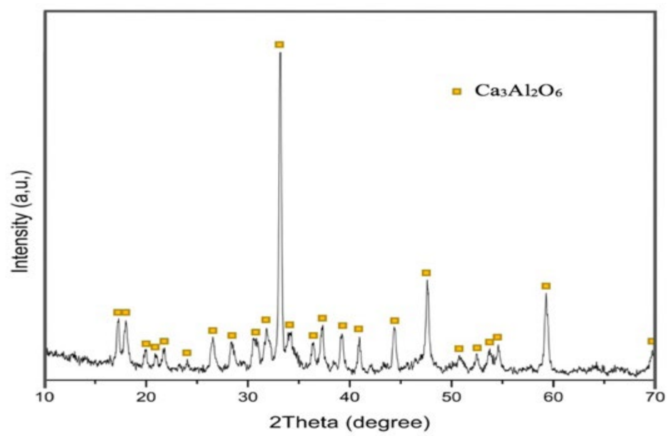

Figure 3. X-ray diffraction pattern of the synthesized powder containing of tetra-calcium alumino ferrite ($Ca_4Al_2Fe_2O_{10}$) and calcium oxide (CaO).

is the mass attenuation factor of calcium oxide, which is 182.61, and A_1 is the mass attenuation factor of tetra-calcium alumino ferrite, which is 1105.76.

Results from the semi-quantitation analysis indicated that the amount of calcium oxide phase present was only 0.417 wt%. With the minor calcium oxide content of less than 1 wt%, effects of calcium oxide on mechanical properties might be negligible.

3.3 Phase Identification of Light-weight Cement Blocks

The synthesized di-calcium silicate, tri-calcium silicate, tri-calcium aluminate and tetra-calcium alumino ferrite powders were mixed with gypsum, rice husk ash, and water. The pastes were subsequently cast, cured and characterized. Results from x-ray diffraction analysis showed that the light-weight cement blocks contained calcium silicate hydrate (CSH, ($Ca_2SiO_4 \cdot xH_2O$), JCPDS

00-003-0248), calcium hydroxide (CH, ($Ca(OH)_2$), JCPDS 01-084-1268) and calcium sulfoaluminate or ettringite (Et, ($Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$), JCPDS 00-013-0350).

Calcium silicate hydrate and calcium hydroxide are common products formed as a result of hydration reactions, shown in the following equations:

Hydration reactions of di-calcium silicate and tri-calcium silicate

$$3CaO\cdot SiO_2 + (x+1)H_2O \Rightarrow Ca_2SiO_4\cdot xH_2O + Ca(OH)_2 \quad (2)$$

$$2CaO\cdot SiO_2 + xH_2O \Rightarrow Ca_2SiO_4\cdot xH_2O \quad (3)$$

Hydration reaction of tri-calcium aluminate

$$3CaO\cdot Al_2O_3 + 6H_2O \Rightarrow Ca_3Al_2(OH)_{12} \quad (4)$$

It is generally accepted that calcium silicate hydrate (CSH), which occurred as a result of the hydration reaction, plays a key role in the enhancement of cement's compressive strength.

According to the XRD patterns shown in Figure 4, all light-weight cement blocks with various water-to-binder ratios (W/B) showed prominent diffracted peaks corresponding to calcium silicate hydrate. The highest intensity of CSH peaks, however, was observed in the specimen prepared with W/B of 1.3. The results suggested that such specimen contained a greater content of strengthening phase, which might consequently lead to superior compressive strength.

It should be addressed that the water to binder ratios employed in the current experiment were higher than those of ordinary concrete containing aggregates. It is commonly accepted that particle size of cementitious materials and the presence of aggregate affect water to cement ratio. In general, water to cement ratios lower than 1 are used in concrete. Higher water content is required for the system with fine particles of cementitious materials. In the current experiment, the synthesized cement-like powders and rice husk ash contained much finer particles compared to those of commercial grade OPC. The particle sizes of the ash and cement-like materials ranged between 2.67±1.9 μm

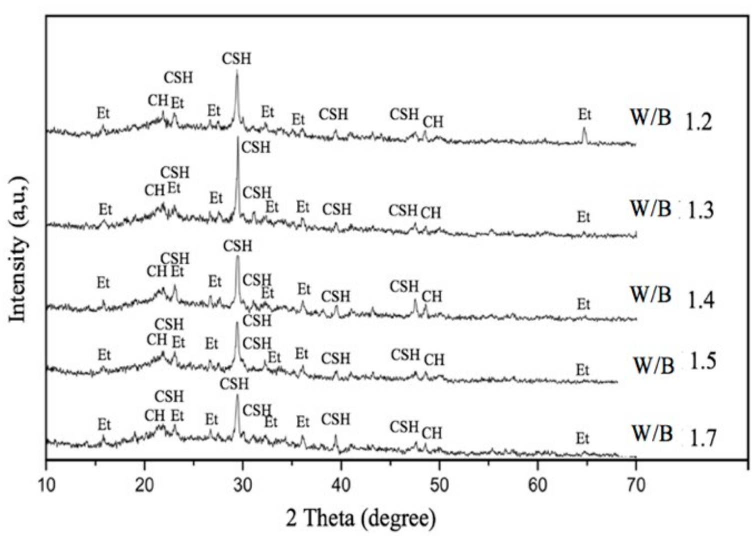

Figure 4. X-ray diffraction patterns showing light-weight cement block with W/B ratios ranging from 1.2:1 to 1.7:1.

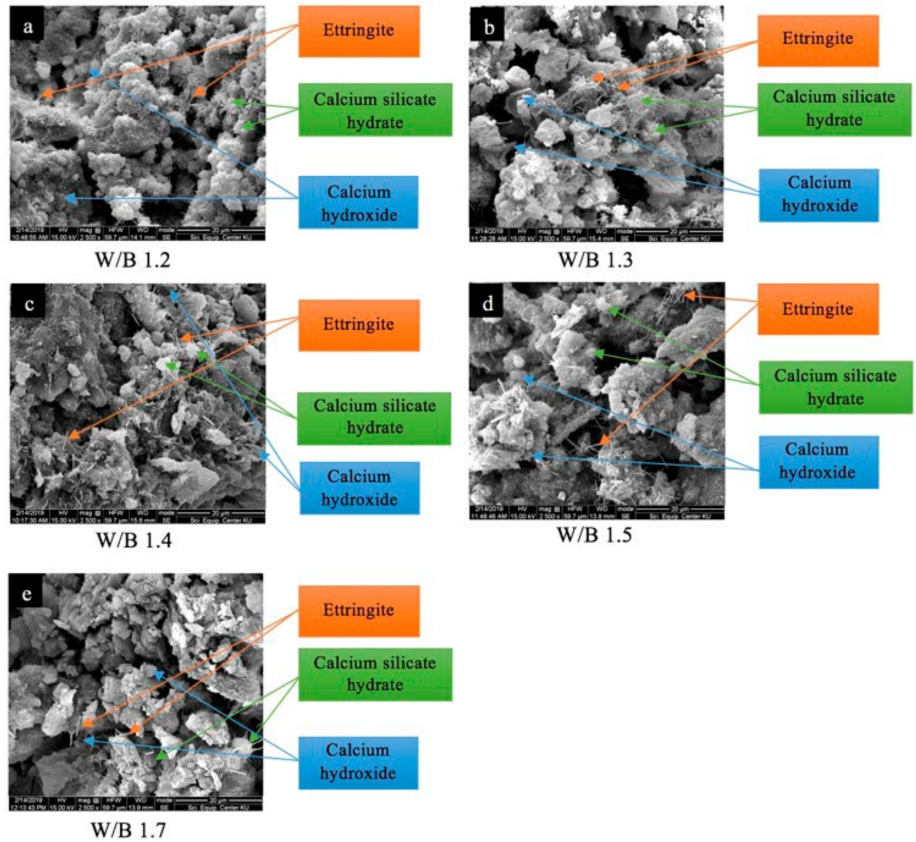

Figure 5. Scanning electron micrographs light-weight cement block specimens cured for 7 days, prepared from various water to mixture (W/B) ratios: a) W/B = 1.2, b) W/B = 1.3, c) W/B = 1.4, d) W/B = 1.5, and e) W/B= 1.7.

and 4.73±4.0 μm on average, which might result in the high consumption of water.

3.4 Microstructural Analysis of Light-weight Cement Blocks

In addition to phase identification, the microstructure of the light-weight cement blocks were also examined using a scanning electron microscope (SEM). Microstructural analysis revealed phases with morphological characteristics corresponding to calcium silicate hydrate (CSH), calcium hydroxide (CH), and ettringite.

As shown in Figure 5, diminutive coral-shaped or fine-branched particles were ubiquitously dispersed throughout the specimens. This distinctive morphology was commonly reported to be that corresponding to calcium silicate hydrate (CSH) [22-23-24]. Small crystallites or platelet-shaped particles with sizes ranging between 2.255±2.0 micrometers were reported to match with the microstructure of calcium hydroxide (CH) phase [24-25-26]. Ettringite phase has always been reported as needle-like particles with sizes in range between 2.5 and 7 micrometers [3]. Results from microstructural analysis, therefore, corresponded well with phase identification analysis reported in the previous section.

Considering results from microstructural analysis by SEM and phase identification by XRD, the results were consistent. All main

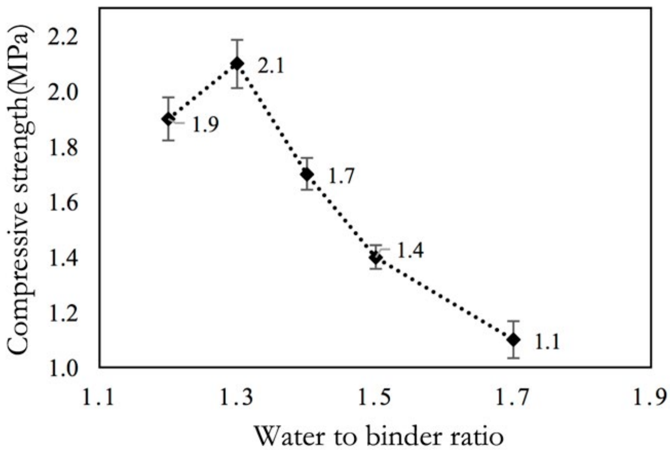

Figure 6. Relationship between compressive strength and water-to-binder ratio.

phases shown in x-ray diffraction patterns of the hydration products: calcium silicate hydrate, calcium hydroxide, and ettringite were all also evident in the scanning electron micrographs.

3.5 Relationship between Water Content and Compressive Strength of Light-weight Cement Blocks

Results from compression tests indicated that the average compressive strength of light-weight cement blocks ranged from 1.1 to 2.1 MPa, as shown in Figure 6. The compressive strength results were in an acceptable range for light-weight concrete according to Thai Industrial Standards Institute (TISI 2601-2556) Type C8 and American concrete institute (ACI 213,2001).

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The results also revealed that the specimens prepared at water-to-binder (W/B) ratio of 1.3 exhibited the greatest strength. As mentioned in the previous section, phase identification and microstructural analysis indicated that the specimens with W/B ratio of 1.3 contained high content of calcium silicate hydrate, which is the phase generally accepted as the strengthening phase. The experimental results from various analysis techniques were all in agreement.

3.6 Relationship between Water Content and Bulk Density of Light-weight Cement Blocks

As shown in Figure 7, density of light-weight cement blocks with various water to binder ratios ranged from 1.01 to 1.14 g/cm^3 on average. The results revealed that high water content generally led to a diminished bulk density of the light-weight cement blocks. This might be attributed to the dilution of cement paste and pore creation at high water to binder.

Density values obtained from the current experiment were in comparable ranges with the values obtained from the study reported by A. Chaipanich *et al.*, 2015 and P. Chindaprasirt *et al.*, 2016 [27,28]. Low density of the cement block offers a key advantage in terms of a dead load reduction. According to Z. Guo *et al.*, 2014, the structures using light weight concrete with density ranging between 0.5 and 1.9 g/cm^3 as replacement

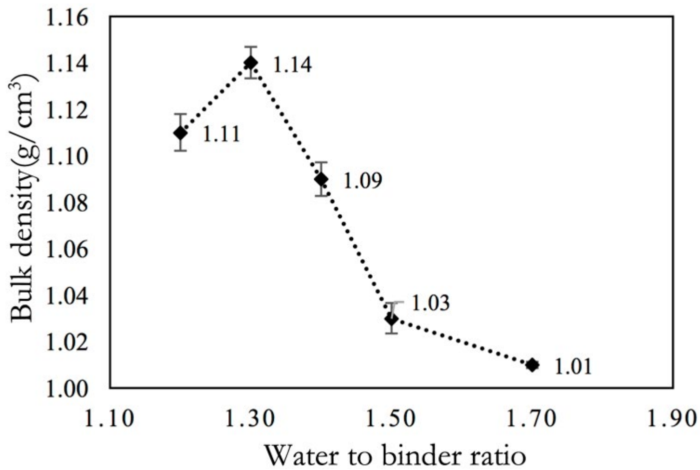

Figure 7. Relationship between bulk density and water-to-binder ratio.

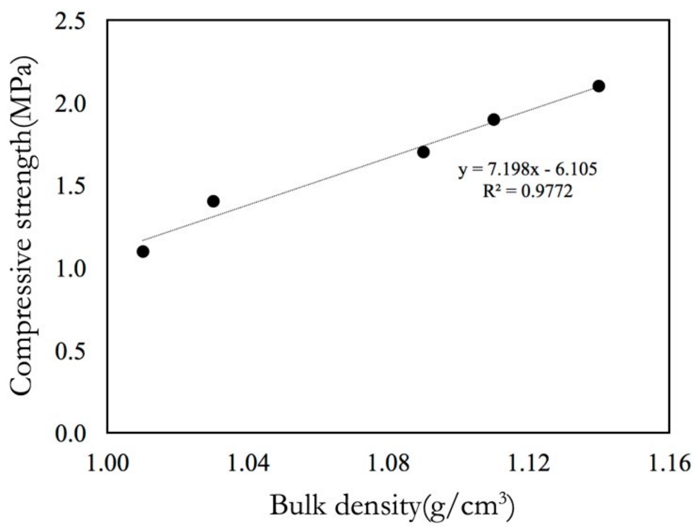

Figure 8. Relationship between compressive strength and bulk density.

of ordinary concrete showed reduction of the dead load as high as 20-40%. Since the intrinsic weight of a structure is a part of the dead load, reduction of overall structural weight results in minimization of the dead load. As a result, beams, columns, piers, and foundations are required to carry smaller load, which is advantageous in terms of structural design—reduction of size and numbers of load-carrying structural components is then possible.

Being porous, light-weight cement blocks are also beneficial with respect to their relatively low thermal expansion, low heat conduction, high temperature resistance, high thermal conservation and enhanced fire endurance [29].

3.7 Relationship between Compressive Strength and Density

The relationship between compressive strength and density of the eco-friendly light-weight cement blocks were shown in Figure 8. The results indicated that compressive strength increased with density. A linear relationship between the strength and density was demonstrated with a good correlation of R^2=0.9772.

It is generally accepted that porosity had an adverse effect on compressive strength and that the porosity increases with water to cement ratios. Excessively high water content, therefore, depresses compressive strength. The linear relationship between compressive strength and density was also observed by the study conducted by J.Zh. Xiao *et al.*, 2006 [30].

4. Conclusions

With high silica and calcium contents, rice husk ash and cockleshells were exploited in the synthesis of cement-like materials by solution combustion technique. Phase identification of the synthesized powders contained main phases of tri-calcium silicate, di-calcium silicate, tri-calcium aluminate, and tetra-calcium alumino ferrite, which are key constituents in Portland cement. Upon casting, light-weight cement blocks prepared from the synthesized cement-like materials revealed formation of calcium silicate hydrate, calcium hydroxide, and calcium sulfoaluminate or ettringite, which are common products obtained from hydration reactions. In addition to chemical compositions, microstructural analysis of light-weight cement blocks indicated similar characteristics compared with typical hardened cement paste made from OPC.

Effects of water contents on compressive strength and bulk density of the light-weight cement blocks were examined. Experimental results indicated that excessively high water content led to diminished compressive strength and density. The relationship between compressive strength and density revealed linear relationship with good correlation of R^2=0.98.

The optimal water-to-binder ratio of 1.3 was used to cast light-weight cement blocks that achieved the compressive strength value of 2.1 MPa and an average bulk density of 1.14 g/cm^3. The compressive strength was in comparable range to that of the light-weight concrete defined by Thai Industrial Standards Institute (TISI 2601-2556) Type C8 and American Concrete Institute (ACI 213,2001).

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