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ORIGINAL PAPER

Setting time and microstructure of Portland cement-bottom ash–sugarcane bagasse ash pastes

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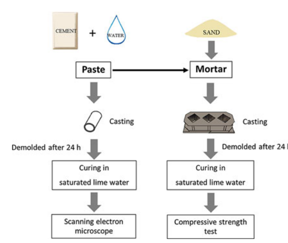
Abstract Bottom ash (BA) and sugarcane bagasse ash (SCBA) are by-products from power plants and the sugar industry and they have a great potential to be used in green-concrete structure applications. This study reports the effects of BA and SCBA on the properties of Portland cement (PC) pastes and mortars as cement replacement, by up to 20 percent by weight. All mortars had a water to binder ratio (w/PC + BA, w/PC + SCBA, and w/PC + BA + SCBA) of 0.5. Normal consistency, setting time, compressive strength, and microstructure (using scanning electron microscope, SEM) were systematically investigated. The results showed that the water requirement for a normal consistency was decreased with increasing BA content. The addition of SCBA, on the other hand, caused the water requirement to increase. The initial and final setting times of all pozzolan mixes were longer when compared to that of PC mix. The compressive strengths of all mixtures with BA were similar to that of the PC mix at 90 days. In addition, the SEM micrograph of pastes confirmed a good pozzolanic reaction between ash particles and Portland cement, resulting in an increase in the compressive strength of the mortars, especially after a period of time (more than 28 days).

Graphical abstract



Keywords Bottom ash · Sugarcane bagasse ash · Pozzolanic reaction · Hydration reaction

Introduction

Cement is the principal material used in the production of concrete, which is the most important material in the world. Concrete forms the basis of the construction industry today, but it emits CO_2 at a rate of 0.7–1.1 tons for every ton of cement produced [1]. To reduce the amount of CO_2 emissions from the cement industry, the manufacturing process has to be improved to reduce its emissions of air pollution [2]. Methods of reducing air pollution include utilizating supplementary cementitious materials such as natural pozzolans material [3–5].

Bottom ash (BA) is a by-product of the combustion of coal in power plants and is formed when ashes at the bottom of the furnace are consolidated. Bottom ash particles are physically coarse, porous, granular, and graying color. The world production of coal ash was approximately 459 million tons in 1992 [1] while the output of lignite BA at Mae Moh power plant in the north of Thailand is about

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0.8 million tons per year [6]. Many researchers have reported that BA contains a high silica and alumina contents similar to fly ash. Furthermore, it has been reported that the pozzolanic reactivity BA may improve the compressive strength of concrete [7–9]. Several researchers [10, 11] found that grinding BA can increase its pozzolanic activity in the concrete and make it suitable for use as a partial replacement in Portland cement.

Sugarcane bagasse ash (SCBA), which is a by-product of the burning of sugarcane bagasse from the sugar industry, has recently been accepted as a pozzolanic material and can be used as a supplementary material in cement-based materials [12–16]. However, the SCBA is a pozzolanic material with high silica content, which could also be used as a pozzolan [15]. This ash is generally disposed in landfills every day, leading to environmental problems in the region. Since the SCBA received from the sugar industry has a large particle size and high porosity, the concrete made with it needs higher water content that gives the concrete lower mechanical properties. However, when SCBA is ground into small particles, the properties of the cement-based materials containing ground SCBA improves significantly [17]. Ganesan et al. [15] reported that the SCBA was an effective mineral admixture, with 20% by weight of the binder as optimal replacement ratio of cement. The 28- and 90-day compressive strengths have been higher than that of the mix without SCBA.

The relationship between the microstructure and compressive strength of BA and SCBA blended cement is not well reported. Consequently, this research investigated the effect of ground BA and SCBA on the microstructure, setting time and compressive strength of pastes and mortars and compared them to the PC control mix. This knowledge could be beneficial and used commercial to incorporate these wastes products into concrete, leading to a reduction in the amount of cement used and environmental problems associated with cement production.

Table 1 Normal consistency and setting time of PC with BA and SCBA pastes

Mix	Water requirement for normal consistency/g, w/b	Setting time/min	
		Initial	Final
PC	136, 0.27	127	155
20BA	133, 0.27	153	186
20SCBA	145, 0.29	134	179
5BA15SCBA	139, 0.28	138	178
10BA10SCBA	137, 0.27	141	172
15BA5SCBA	134, 0.27	146	171

Results and discussion

Normal consistency

Cement paste is considered to have normal consistency when a plunger can sink 10 ± 1 mm into the paste in 30 s. The water requirement needed to produce cement pastes that incorporate BA and SCBA is shown in Table 1 and Fig. 1. The results showed that the normal consistency of the PC 20BA and 20SCBA pastes had water requirements

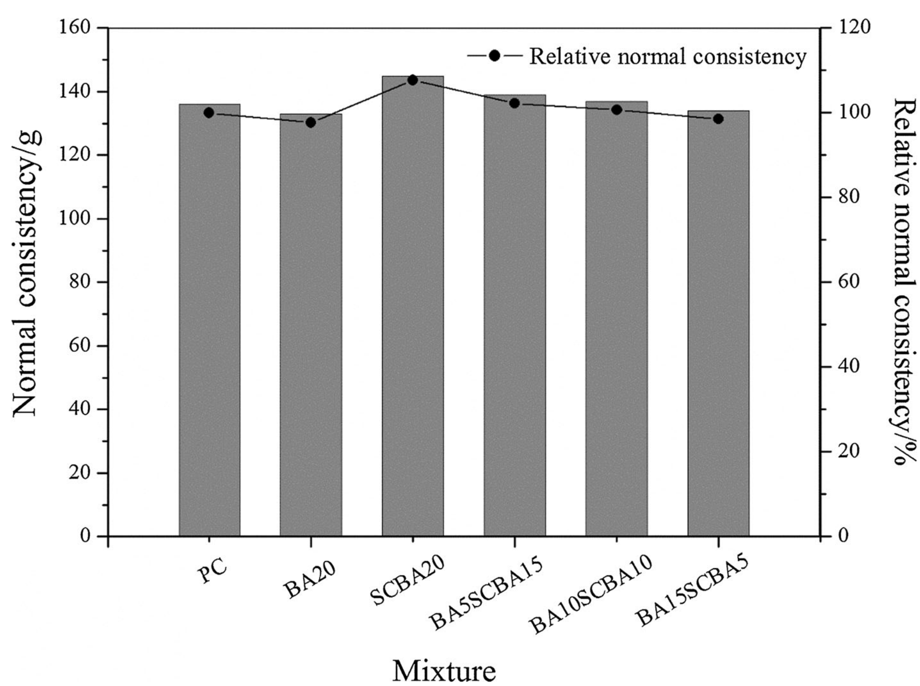

Fig. 1 Normal consistency of PC containing BA and SCBA paste

of 136, 133, and 145 g, respectively. Therefore, the water requirement of 20BA paste was lower than that of the ordinary cement paste while 20SCBA paste had a higher water requirement than that of the control cement paste. This is mainly due to the LOI content of the SCBA which was higher than that of the BA (LOI of BA and SCBA of 3.4 and 11.2%, respectively). This behavior was also found in the case of high LOI content in fly ash [18–20]. However, high LOI content had more of an effect on water demand, which was attributed to the absorption of free water by unburned carbon particle in the pozzolanic material [21]. In addition, it can be noticed that the spherical shape of the partial BA particle can increase the movement of the binders. Furthermore, it was found that the water requirements for normal consistency of cement paste with 20 wt% of a mixture of BA and SCBA (5BA15SCBA, 10BA10SCBA, and 15BA5SCBA) were 139, 137, and 134 g, respectively. This result indicated that the water required for normal consistency of the paste was decreased when the amount of SCBA decreased.

Setting time

The initial and final setting times of the control cement paste and the pastes modified with BA and SCBA (20BA, 20SCBA, 5BA15SCBA, 10BA10SCBA, and 15BA5SCBA) are shown in Fig. 2 and Table 1. It can be seen that all specimens with BA and SCBA were found to have higher initial and final setting times of the pastes than the PC control mix. It can be noted that BA caused a small delaying effect on the hydration of Portland cement paste, which has been reported by the previous studies [22–25]. The main reason for the increase in both the initial and final setting times is the reduction of the C_3A component (in the

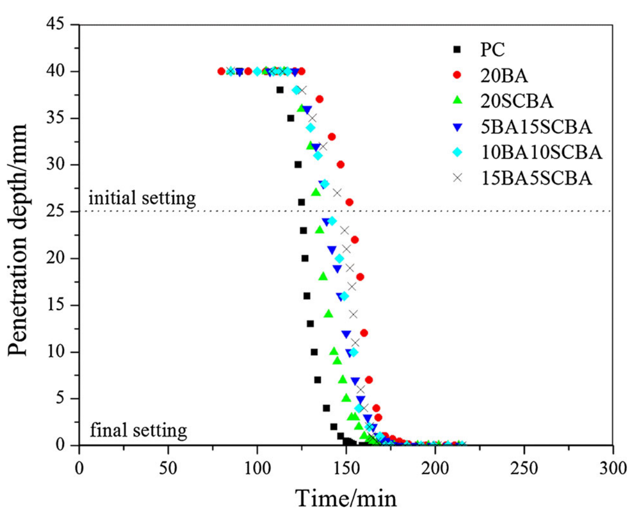

Fig. 2 Setting time of ordinary Portland cement containing BA and SCBA

case of using mineral additive) in the mixture that has an important role in improving the hydration in the early hours. By replacing cement with 20BA in certain proportions, the initial and final setting times of the cement increased 26 and 31 min in comparison with PC cement paste. Moreover, the final setting time of the 20 wt% of overall replacement by BA and SCBA tended to slightly decrease when the fraction of BA increased.

Compressive strength

Table 2 shows the compressive strength of the mortar blended with pozzolan after 7, 28, and 90 days of curing. The compressive strengths of the PC mix at 7, 28, and 90 days were 40.5 ± 2.7, 41.4 ± 1.5, and 46.3 ± 0.9 MPa, respectively. At 7 days of curing, the compressive strengths of the mortars with 20 wt% BA and 20 wt% SCBA were 26.2 ± 2.5 and 30.6 ± 0.9 MPa, respectively. The compressive strengths of the 20BA and 20SCBA mixes at 28 days were 39.6 ± 1.6 and 35.1 ± 1.9 MPa, respectively, while the compressive strengths of these mixes at 90 days were 45.1 ± 0.6 and 46.3 ± 0.4 MPa, respectively. The strength of the mortar was similar to that of mortar that used other pozzolan materials, such as the fly ash and bagasse ash in concrete, as reported by Akram and Ganesan [15, 26].

The compressive strength of the mortar with 20 wt% of the total cement replacement by BA and SCBA (5BA15SCBA, 10BA10SCBA, and 15BA5SCBA) trended to decrease when BA increased and SCBA decreased, for all ages. These compressive strength results of 7 days curing were 26.7 ± 4.7, 26.6 ± 0.9, and 23.8 ± 2.9 MPa, respectively. Furthermore, the compressive strengths at 28 days were 40.3 ± 4.2, 38.0 ± 3.8, and 37.2 ± 2.5 MPa, respectively, while the compressive strength was 46.7 ± 0.4, 46.2 ± 0.6, and 46.2 ± 2.5 MPa for 90 days. This result indicates that the compressive strength of all mixes with ashes at 28 and 90 days curing was close to the strength of the pure PC mortar. It can be interpreted from this result that the replacement of PC by both ashes at 20 wt% possibly results in lower effects on the compressive strength at later ages.

Microstructure

A comparison of the microstructure of the pastes blended with BA and SCBA using SEM analysis is presented in Fig. 3a, c, e, and g. The regions analyzed by EDS are indicated by a gray rectangle in the SEM image. Results obtained in the EDS spectra are shown with the relative intensities of each element in the SEM micrograph from Fig. 3b, d, f, and h.

Table 2 Compressive strength of mortar containing BA and SCBA after curing in saturated lime water at 7, 28, and 90 days

Mixture	Compressive strength/MPa			Relative compressive strength/%		
	7 days	28 days	90 days	7 days	28 days	90 days
PC	40.5 ± 2.7	41.4 ± 1.5	46.3 ± 0.9	100.0	100.0	100.0
20BA	26.2 ± 2.5	39.6 ± 1.6	45.1 ± 0.6	64.7	95.7	97.4
20SCBA	30.6 ± 0.9	35.1 ± 1.9	46.3 ± 0.4	75.5	84.9	99.9
5BA15SCBA	26.7 ± 4.7	40.3 ± 4.2	46.7 ± 0.4	65.9	97.3	100.9
10BA10SCBA	26.6 ± 0.9	38.0 ± 3.8	46.2 ± 0.6	65.5	91.9	99.8
15BA5SCBA	23.8 ± 2.9	37.2 ± 2.5	46.2 ± 2.5	58.8	90	99.7

The SEM photograph of the PC paste at 28 days is shown in Fig. 3a. The microstructure of the PC paste presented several shapes such as a plate-like crystals, condensed phase and needle-like crystals. Such large plate-like crystals are referred to an unhydrated cement phase and the needle-like crystals have been described as CSH phases inside pores or cracks [27] as reported by other studies [28]. Furthermore, the SEM image relates to the high amount of Ca, Si, and O elements by the EDS analysis (Fig. 3b), having 40.96, 8.39, and 36.97 wt%, respectively. This result confirms that the element Ca is generally found in ordinary Portland cement [29, 30].

Figure 3c shows an SEM micrograph for the paste of 20 wt% BA after curing in water for 28 days. The BA was obtained with burning procedure of coal and they are often contaminated with fly ash particles (spherical shape). It can be seen that the BA particle has semi-spherical chunks with a rough surface, where a particle size was approximately 25 µm. The chunks are bonded with the paste surface (as seen in the interface around the BA particles) and some spherical BA particles came off when crushing samples. The SEM image shows that a hydration gel surrounds the large BA particle and their surfaces were a little rough, indicating the pozzolanic reaction occurred by 28 days. An investigation of elements of the BA paste by EDS is shown in Fig. 3d. The elements of the BA mix (overall area) showed high Si and Ca, which are the main elements detected for the normal pozzolan material usage [31, 32].

Figure 3e shows the morphology of the 20SCBA paste at 28 days of curing. It can be seen that the SCBA particle inserted into hydration products of PC and had a size of approximately 70 µm. It should be note that the cement dilution can be effected by the hydration degree. However, the microstructure seen in the SEM results of the SCBA paste was related to the compressive strength of the SCBA mix that was found to be less than that of the PC mix at 28 days curing. The EDS analysis of the paste with 20 wt% SCBA is shown in Fig. 3f. Si and Ca of the SCBA mix detected by the EDS analysis were similarly reported in other pozzolan usage [33–35]. Moreover, it can be noticed by EDS analysis that the SCBA paste shows Si and Si/Ca ratio higher than that of the PC paste.

The microstructure of 10BA10SCBA is shown in Fig. 3g. The SEM image showed the BA and SCBA particles in the matrix and that the large BA particles were surrounded by a hydration gel and their surfaces appeared to be rougher than that of the SCBA surfaces. This seems to be caused by that the BA consuming $Ca(OH)_2$ (from the hydration reaction), leading to an increase in C–S–H formation via the pozzolanic reactions. However, both ashes were not only used for the development of the pozzolanic reaction [21], but also affect the paste by the microfiller effect [36–39]. In addition, the EDS analysis of such areas (Fig. 3h) showed Si and Ca elements of 12 and 24 wt%, respectively.

Conclusion

Based on the present experimental results, the following conclusions can be drawn:

1. The water requirement for normal consistency of the paste was found to decrease with increasing BA content while increased water was required when the SCBA content increased. The initial and final setting times were increased with added BA and SCBA content up to 20 wt%, indicating that BA and SCBA had lowered the hydration reaction than that of the PC phases due to the pozzolanic reaction.
2. The highest compressive strength of the mortar samples was observed in the 5BA15SCBA mortar at 28 and 90 days, with values of 40.3 and 46.7 MPa, respectively. Furthermore, the BA and SCBA had a positive influence on the compressive strength of the mortars, which made them suitable for use as a mineral addition.
3. It can be observed at boundaries of the pozzolan particles that the utilization of BA and SCBA into PC mortar showed a fairly dense matrix (from SEM analysis) via the pozzolanic reaction. This results in a significant gain in the compressive strength.

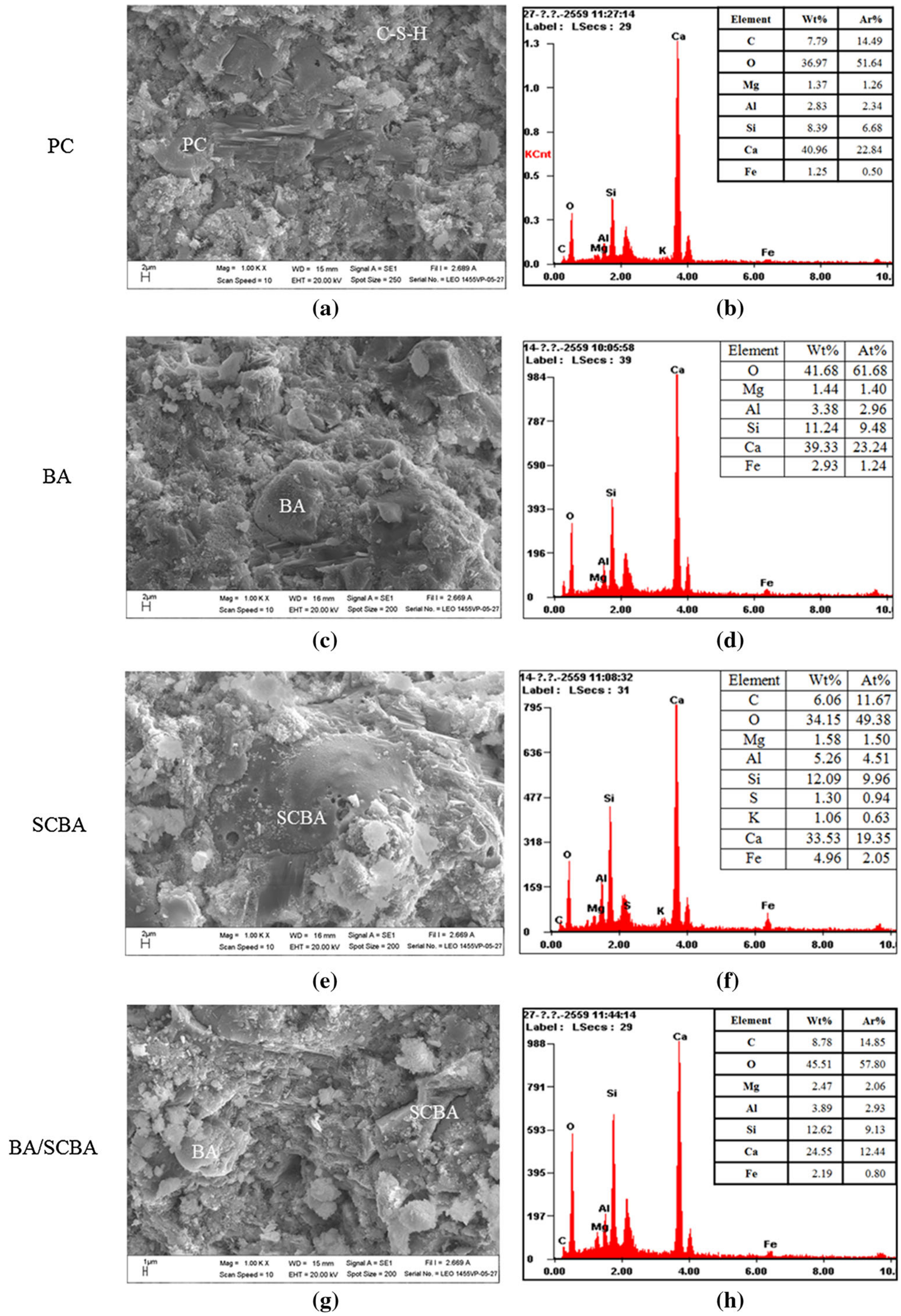

Fig. 3 SEM photographs and EDS analyses of the pastes blended pozzolan; **a** PC, **b** PC-EDS, **c** 20BA, **d** 20BA-EDS, **e** 20SCBA, **f** 20SCBA-EDS, **g** 10BA10SCBA, and **h** 10BA10SCBA-EDS at 28 days

Experimental

Chemical compositions of Portland cement type I (PC), BA, and SCBA are given in Table 3. Portland cement consists of 65.6% of CaO, 18.4% of Si_2O, 3.7 of Al_2O_3, and 3.4 of Fe_2O_3. The loss on ignition (LOI) of the PC particles was 2.2%, with the median particle size (d_{50}) being 36.78 µm. In this study, bottom ash from Mae Moh power plant in the north of Thailand was used. BA was ground by a ball mill for 20 h and had a median particle size of 23.21 µm. The microstructure of raw BA by SEM was presented spherical, semi-spherical chunk and irregular shapes with a particle size of about 1–50 µm, as shown in Fig. 4a. The main chemical composition of BA was SiO_2, Al_2O_3, Fe_2O_3, CaO and LOI of 41.1, 17.1, 13.8, 17.1, and 3.4%, respectively. The sum percentage of SiO_2,

Table 3 Chemical composition of PC, BA, and SCBA

Materials	Oxide content/%									
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	TiO_2	SO_3	P_2O_5	LOI
PC	18.4	3.7	3.4	65.6	1.5	0.6	0.2	4.5	–	2.2
BA	41.1	17.1	13.8	17.4	2.0	2.2	0.5	2.4	–	3.4
SCBA	74.0	3.7	1.8	1.7	0.7	4.4	0.4	0.3	1.8	11.2

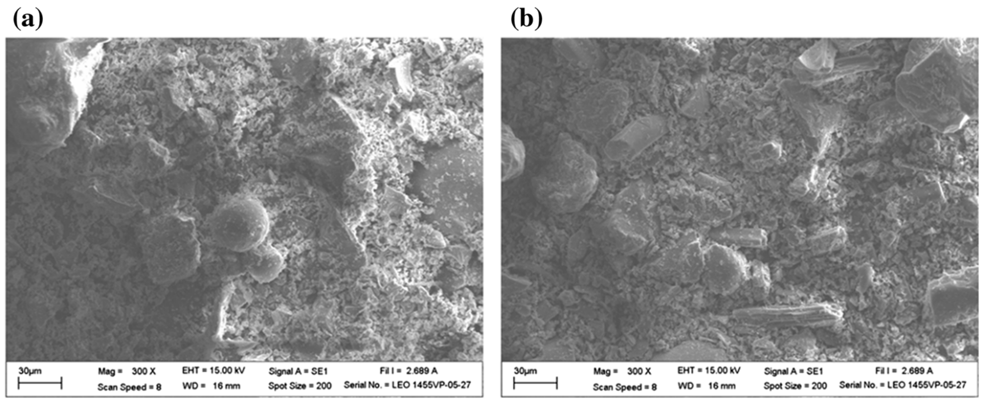

Fig. 4 SEM photographs of raw materials: **a** grinding BA and **b** grinding SCBA

Table 4 Mix proportions of PC with BA and SCBA pastes

Mix	w/b	Mix proportion/wt% of PC			
		PC	BA	SCBA	Sand*
PC	0.5	100	–	–	250
20BA	0.5	80	20	–	250
20SCBA	0.5	80	–	20	250
5BA15SCBA	0.5	80	5	15	250
10BA10SCBA	0.5	80	10	10	250
15BA5SCBA	0.5	80	15	5	250

* For mortar mixture

Al_2O_3, and Fe_2O_3 was 72%, which is higher than the 70% requirement for natural pozzolan according to ASTM C618 standard [40]. Sugarcane bagasse ash (SCBA) was obtained from the sugar industry in Phitsanulok province, Thailand. It was ground by a ball mill for 12 h to reduce the particle size (d_{50} of 27.61 μm). The morphology of raw SCBA particles presented semi-spherical, cylindrical and irregular shapes with the size of about 1–70 μm, as shown in Fig. 4b. SCBA has a sum percentage of SiO_2, Al_2O_3, and Fe_2O_3 of 79.5%, while SO_3 and LOI were 0.3 and 11.2%, respectively. The BA and SCBA were used as a cement replacement up to 20 wt%. The mix proportion of Portland cement with BA and SCBA pastes and mortars is given in Table 4.

For the normal consistency test, the binders (PC, BA, and SCBA) were firstly mixed in a mixer, after that the pastes were approximately shaped to a ball with gloved hands. The paste was tossed six times through a free path of about 150 mm and then inserted into the Vicat mold. The excess paste was removed and the surface was made smooth. After that, the center of the Vicat mold sample was placed under the center of a rod (plunger) and then the plunger was touched the paste surface. Finally, the rod was released. A normal consistency was accepted when the plunger sunk to a point 10 ± 1 mm below the original surface in 30 s in accordance with ASTM C 187 standard [41].

The setting time, an indication of the cement paste solidification time [42], was measured using a Vicat apparatus following the ASTM C 191 standard [43]. The paste samples with and without BA and SCBA were mixed with the water to binder ratio following the water requirement of the normal consistency test, as given in Table 1. A 1 mm diameter needle was allowed to penetrate into the pastes every 15 min, and the penetration depth was measured. The time from initial contact of cement and water to the time when the penetration depth of 25 mm was reached by the needle was used to calculate the "Initial setting" time using Eq. (1):

$$\text{Initial setting} = \left(\left(\frac{H-E}{C-D}\right)(C-25)\right) + E, \tag{1}$$

where E is the time in minutes of the last penetration greater than 25 mm, H is time in minutes of first penetration less than 25 mm, C is the penetration reading at time E, and D is the penetration reading at time H. The time from the initial contact of cement and water until the

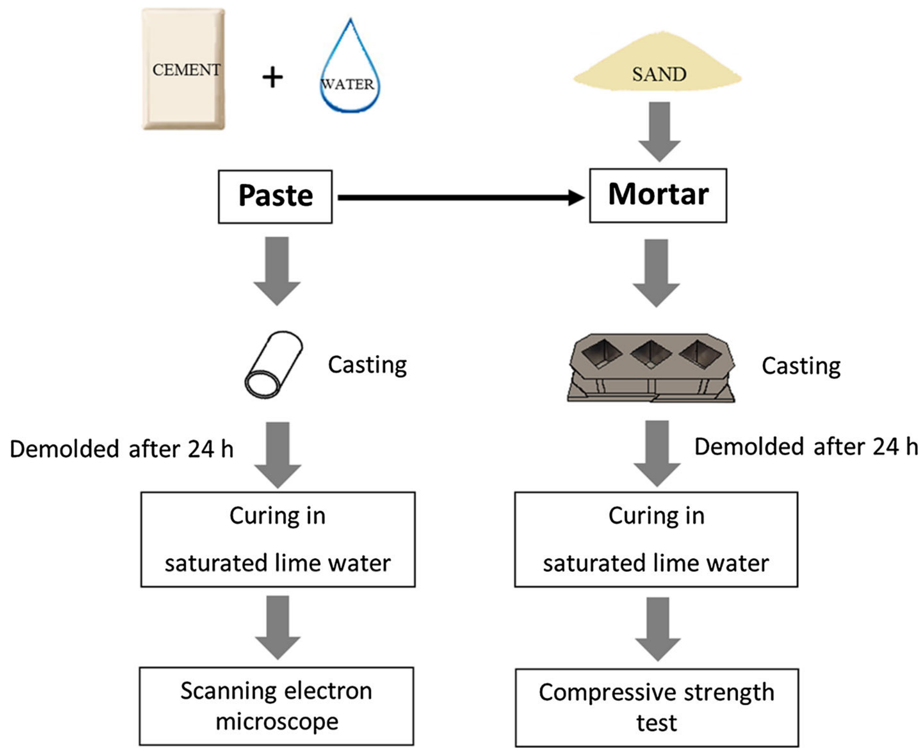

Fig. 5 Flow chart of experimental program of this research

penetration of the needle did not sink visibly into the paste surface was defined as the "Final setting" time.

For compressive strength tests, the water/binder ratio and the sand/binder ratio of the cement mortars were 0.5 and 2.5, respectively. To help homogeneity, the binders (PC, BA, and SCBA) were first mixed in the mixer and, then the water was added and mixed for approximately 2 min in accordance with the ASTM C 305 standard [44]. The pastes were then poured into cube molds (50 × 50 × 50 mm^3) and then compacted in accordance with the ASTM C 109 standard [45]. The specimens were surface smoothed, and covered with plastic film. After 24 h of casting, all specimens were removed from the molds. Thereafter, they were cured in water at 25 ± 2 °C. Three specimens of each mix were tested for compressive strength at 7, 28, and 90 days after curing and the process of mortar preparation, as followed in Fig. 5.

The microstructure of the paste samples was examined by an LEO 1455VP scanning electron microscope (SEM) coupled with an energy dispersive spectrometry (EDS). The paste samples were soaked in acetone to stop the hydration and then treated in an oven maintained at 100 °C before the analysis. The procedure of paste preparation is shown in Fig. 5.

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