

Improvement, characterization and use of waste corn cob ash in cement-based materials

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Abstract. This work investigates the development of waste corn cob ash as supplementary cement replacement materials. The study focused on the effects of heat treatment on chemical composition, physical properties and engineering properties of corn cob ash. The results suggest corn cob ash that was heat treated at 600°C for 4 h shows percentage of SiO₂ + Al₂O₃ + Fe₂O₃ around 72%, which can be classified as Class N calcined natural pozzolan, as prescribed by ASTM C618. The X-ray diffraction patterns indicated that the amorphous silica phase increased with increasing calcining temperatures. The water requirement, initial setting time and final setting time of specimens increased with increasing replacement percentage of raw or treated corn cob ash. The mortar cubes which used 20% of treated corn cob ash replaced cement showed 103% of the 28 days compressive strength as compared to reference samples. The corn cob ash that was treated at 600°C for 4 h samples shows slightly higher effectiveness for improving the splitting tensile strength and compressive strength of concrete when compared to the untreated corn cob ash.

1. Introduction

Ordinary Portland Cement (OPC) is the most common type of cement used in construction applications. The current cement production rate worldwide is approximately 1.2 billion tons/year. This consumption is expected to grow to about 3.5 billion tons/year by 2015 [1]. However, it is an expensive binder due to the high cost of production associated with the high energy requirements of the manufacturing process itself [2, 3]. Nowadays, industrial wastes such as blast furnace slag, fly ash and silica fume are being used as supplementary cement replacement materials [4-7]. These materials consist of silica oxide or alumina oxide, which react with calcium hydroxide in concrete, forming the cementitious materials. The agricultural waste or industrial waste which is used for partial cement

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replacement is called pozzolanic materials [8]. The waste products which possess pozzolanic properties and which have been studied for use in blended cement are fly ash [9, 10], rice husk ash [11-13], palm oil ash [14-16] and sugarcane bagasse ash [17-19]. Corn cob is an agricultural waste product obtained from maize or corn, which are important for the food industry. Corn cob ash (CCA) waste is produced in the boilers of the animal feed industry. Adesanya, et al. [1] extensively studied the pozzolanic properties of CCA from South Africa. The sum of SiO_2 , Al_2O_3 and Fe_2O_3 in the chemical composition of CCA satisfies the requirements of ASTM C618, suggesting its pozzolanic nature. Beside strength properties, the thermal conductivity of concrete is decreased and the insulation properties are improved by blending the CCA in concrete [20].

The quality of pulverized fuel ash or biomass fuel ash, used as a supplementary cementing materials in cement and concrete, varies on account of the variations in the firing processes, plant design, engineering parameters and dust collection process. It is well known that firing conditions strongly influence the physical characteristics and the chemical compositions of burned ash. For instant, the content of amorphous silica phase is decreased and carbon fraction is eliminated from the system with an increase firing temperature. The optimum firing temperature also provides the highest amount of amorphous silica and highest specific surface area, which results in satisfactory engineering properties when the burned ash is applied in concrete. To the best of our knowledge the improvement of physical and chemical properties of CCA using the heat treatment has not been yet investigated. Therefore, the aim of this study is to investigate the chemical and physical properties of CCA produced in Thailand. The effect of firing temperatures on the chemical properties, physical properties and engineering properties of CCA-cement replacement in concrete was investigated.

2. Materials and experiments

2.1. Materials

The CCA was obtained from the thermal power plant of Charoen Pokphand Foods Public Company Limited Plant in Phitsanulok, Thailand. The CCA was weighed and ground by the ball-milling machine for 12 h and then sieved into a fine powder. The chemical reaction of CCA at high temperature was investigated by thermogravimetric analysis and differential thermal analysis (TGA-DTA) using a heating rate of $10^\circ\text{C}/\text{min}$ from room temperature to 700°C . The CCA was heated from 200 to 600°C for 4 h and the changed physical and chemical characteristics of the treated ash were observed.

2.2. Physical and chemical properties characterization

The chemical composition of raw CCA and treated CCA were analyzed using X-ray fluorescence (XRF) analyzer. Perkin-Elmer FTIR instrument with resolution 3 cm^{-1} was used to monitor the vibrational stretching and bending bands in silica of the samples. Mineralogical analysis was studied using the X-ray diffraction with monochromatic $\text{Cu-K}\alpha$ radiation at a scanning speed of $1.5^\circ (2\theta)\text{ min}^{-1}$ in a conventional diffractometer. The crystalline phases were identified by comparing the intensities and positions of the Bragg peaks with those listed in the JCPDS databases. The microstructure of raw CCA and treated CCA were also studied by low vacuum scanning electron microscope or SEM (Model JOEL JSM-5910LV) with the accelerating voltage of 15 kV. The specific surface area and particle size distribution of raw CCA and treated CCA were measured by Brunauer, Emmett and Teller (BET) and particle size analyzer (PSA), respectively.

2.3. Engineering properties investigations

The raw CCA and treated CCA were prepared by replacing cement over the range of 0-20% by weight (step up 5%). The 0% replacement serves as the reference specimen. The amount of water required for normal consistency of samples was carried out in accordance with the ASTM C187 standard [21]. The ASTM C191 [22] was applied to determine the initial and final setting time of the cement-CCA paste. A mortar cube of $50\times 50\times 50\text{ mm}$. was produced in accordance with ASTM C109 [23] subsequently

with the pozzolanic activity of the mortar determined, based on its compressive strength according to ASTM C311 [24]. For a concrete application, fresh concrete was tested using slump cone test followed ASTM C1611 [25] to find the workability of concrete. The compressive strength test and the splitting tensile strength test were operated. For compressive strength test, cube specimens of dimensions $150 \times 150 \times 150$ mm were cast with different proportions of cement replacement fraction similar to the compressive strength test of mortar cube. The compressive strength was tested for 7, 14, 28, and 56 days curing. To investigate the splitting tensile strength, the $150 \text{ mm} \times 300 \text{ mm}$ cylinders were also cast from each mix. Splitting tensile strength test was conducted after 28 days of moist curing.

3. Results and discussion

3.1. Chemical reaction of CCA during heat treatment

A typical graphical illustration of the TGA–DTA curves for CCA is shown in figure 1. The TGA result showed three distinct weight losses. The first weight loss occurred between 30 and 137°C corresponding to the endothermic peak at 106°C in the DTA curve. These observations related to the vaporization of light volatile matters and water. The second weight loss occurred between 137 and 382°C corresponding to the endothermic peak at 170°C in the DTA curve. This observation can be attributed to the depolymerizations of the polysaccharides [26]. The third weight loss was continuously occurred at the temperature higher than 500°C and corresponded to the endothermic peak at 520°C in the DTA curve. This may be attributed to the carbon char was burned and evaporated [27].

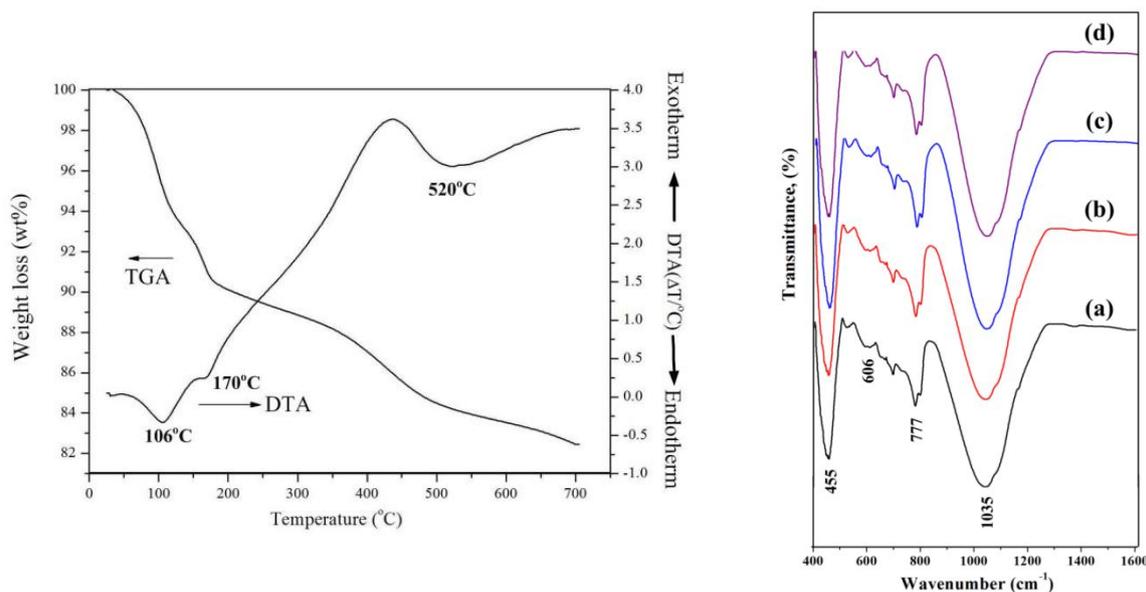


Figure 1. TGA–DTA curves for the CCA.

Figure 2. FTIR spectra of the corn cob ash and treated corn cob ash at various temperatures: (a) corn cob ash, (b) corn cob ash calcined at 200°C for 4 h, (c) corn cob ash calcined at 400°C for 4 h and (d) corn cob ash calcined at 600°C for 4 h.

3.2. Physical and chemical analysis of CCA and treated CCA

The FTIR spectrum of CCA, calcined corn cob ash at 200°C , 400°C and 600°C are presented in figure 2. The infrared spectrum of CCA is shown in figure 2(a) and three main signals are observed. The first

one is a strong broad band at 1035 cm^{-1} is assigned to the asymmetric stretching vibration of tetrahedral SiO_4 . The next signal is a sharp medium band at 777 cm^{-1} . This band is due to symmetric stretching vibrations of the same units. The third signal is a strong band at 455 cm^{-1} . This point is assigned to the Si-O bending mode. In addition, a very weak band at 606 cm^{-1} is observed due to the characteristic of the crystalline cristobalite [28, 29]. The spectrum patterns are insignificantly changed when the CCA was treated at various temperatures.

Figure 3 illustrates the X-ray diffraction patterns of CCA, calcined corn cob ash at 200°C , 400°C and 600°C for 4 h. In the case of CCA, the diffraction patterns correspond to quartz (JCPDS No: 33-1161). After heat treatments at 200, 400 and 600°C , the intensity of the observed peaks tends to decrease. This indicates that silica in the ash is transformed from crystalline to amorphous phase. This result corresponds to the thermal analysis investigation and the suggesting by Jal, et al. [30]. According to their report, naturally occurring silica is crystalline whereas synthetically obtained silica is amorphous in nature. Moreover, a broad hump at $2\theta = 22^\circ$ to 36° observed in the CCA calcined at 600°C sample indicates the Si-O band structure is disordered [27].

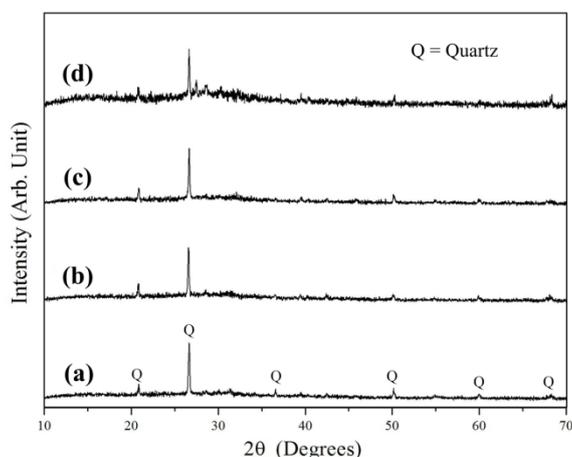


Figure 3. XRD patterns of: (a) corn cob ash, (b) corn cob ash calcined at 200°C for 4 h, (c) corn cob ash calcined at 400°C for 4 h and (d) corn cob ash calcined at 600°C for 4 h.

Table 1. Chemical composition, mean grain size and specific surface area of OPC, CCA and CCA600.

Chemical constituents (%)	OPC	CCA	CCA600
Silicon dioxide (SiO_2)	20.71	60.09	63.91
Aluminum oxide (Al_2O_3)	5.16	3.99	4.01
Iron oxide (Fe_2O_3)	3.22	4.03	3.95
Calcium oxide (CaO)	66.23	5.18	4.13
Magnesium oxide (MgO)	0.95	3.12	2.91
Potassium oxide (K_2O)	0.48	14.73	12.12
Sodium oxide (Na_2O)	0.19	-	-
Sulfur trioxide (SO_3)	2.14	0.82	0.88
Phosphorus pentoxide (P_2O_5)	-	6.72	6.49
$\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$	-	68.11	71.87
Mean grain size (μm)	15.37	23.56	28.45
BET specific surface area (m^2/kg)	3,200	9,050	7,441

The chemical composition and physical properties of OPC, CCA and corn cob ash calcined at 600°C for 4 h (CCA600) are compared in table 1. Chemical composition analysis of CCA indicates that CCA have $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ equal to 68.11%, which is lower than chemical requirements of the standard specification for natural pozzolan Class N according to ASTM C618 (summary of SiO_2 ,

Al_2O_3 and Fe_2O_3 higher than 70%) [31]. However, after heat treatment at 600°C for 4 h (CCA600), the percentage of $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ was increased to 71.87% with less than 4% of SO_3 , which can be classified as Class N calcined natural pozzolan as prescribed by ASTM C618. An increase of $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ fraction in CCA600 ash is attributed to the vaporization of typical elements such as carbon, calcium or magnesium during heat treatment.

In terms of physical characteristics, heat treatment of CCA at 600°C for 4 h slightly increased mean grain size from 23.56 to 28.45 μm compared with the CCA (table 1). The specific surface area was determined by the Brunauer, Emmett and Teller (BET) theory as listed in table 1. The study results revealed CCA exhibits higher specific surface area compared with OPC. After heat treatment at 600°C for 4 h, specific surface area slightly decreased. This result corresponds with an increase in particle size. However, the higher means particle size with higher specific surface suggested the CCA and calcined CCA at 600°C samples show higher porosity than OPC.

The surface microstructural photographs of CCA and CCA calcining at different temperature are shown in figure 4. The CCA exhibits a polygonal shape and a wide range of particle size distribution (figure 4(a)). A higher narrow range of particle size distribution and formation of larger ash agglomerates appeared when the CCA was heat treated at 200°C as shown in figure 4(b). Increasing the calcining temperature to 400°C brought about an increase in the particle size due to smaller particles melting and merging into larger grains (figure 4(c)). An increase of the particle size and an agglomerate effect were obviously observed when the calcining temperature was increased to 600°C (figure 4(d)). The increase in mean particle size that accompanied the heat treatment process, observed from the SEM results, corresponds to the particle size analysis which was observed from the particle size analyzer.

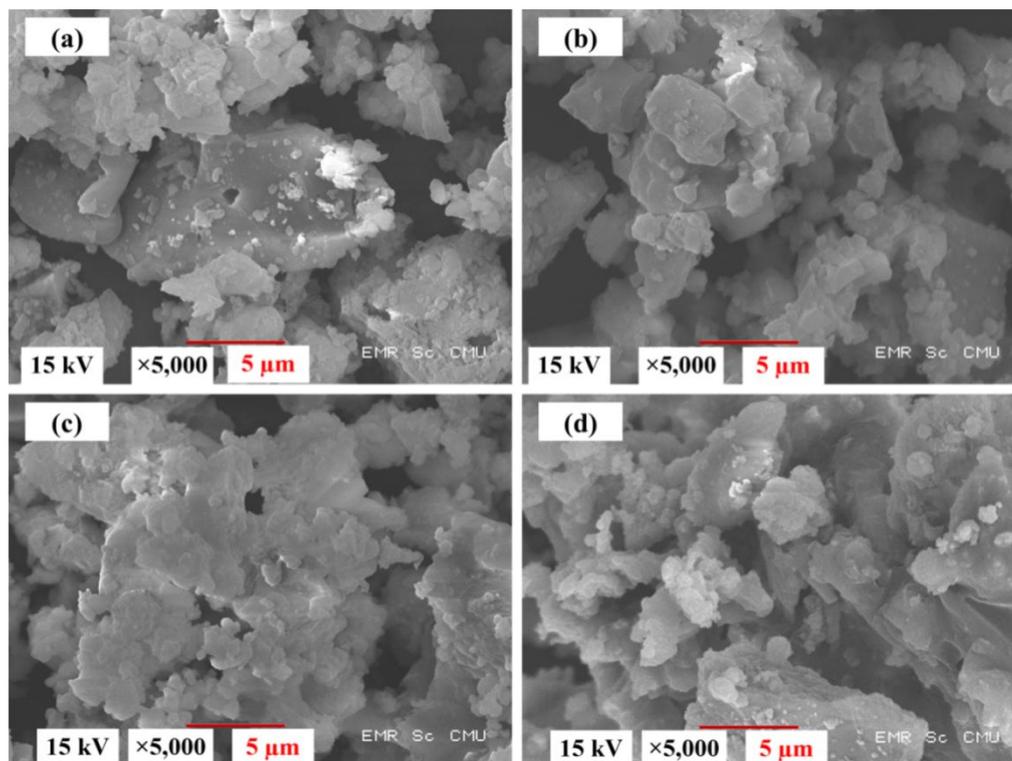


Figure 4. SEM photographs of corn cob ash and calcined corn cob ash; (a) corn cob ash, (b) corn cob ash calcined at 200°C for 4 h, (c) corn cob ash calcined at 400°C for 4 h and (d) corn cob ash calcined at 600°C for 4 h.

From the XRF and XRD analyzes, the CCA600 showed higher amounts of $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ fraction and amorphous silica phase compared with raw CCA. These factors suggest the CCA600

probably exhibits more appropriate behaviors of pozzolanic material than raw CCA. Therefore, the CCA600 was selected to investigate the engineering properties for applied as supplementary cementing material when compared with the raw CCA material.

3.3. Consistency and setting time of CCA and CCA600 blended cement

The standard test method following ASTM C187 [21] is used to determine the amount of water required in preparing hydraulic cement pastes and cement-ash replacement with normal consistency. The results are listed in table 2. Replacing a fraction of OPC with CCA or CCA600 caused an increase in water requirement in a paste. This observation can be attributed to the internal porosity and specific surface area of CCA and CCA600 particles being higher than OPC particles. The water requirements of cement pastes with CCA and CCA600 replacement are slightly different. However, the standard specification for raw or calcined natural pozzolan for use in concrete (ASTM C618) [31] limits water requirements at 115% of reference specimen (water/binder ~30.87%). Hence, this is a problem in the application of CCA or CCA600 as a supplementary cementing material.

Table 2. Water/binder ratio, initial setting time and final setting time of cement paste, CCA-blended cement at various percentage and CCA600-blended cement at various percentage.

Samples	% replacement	W/B ratio at normal consistency (%)	Initial setting time (min.)	Final setting time (min.)
Reference	0	26.84	115	223
CCA	5	28.80	183	314
	10	30.98	222	349
	15	34.05	252	387
	20	35.77	271	405
CCA600	5	28.44	175	306
	10	32.48	229	370
	15	34.26	261	377
	20	36.43	270	414

The water/binder ratios are giving the paste normal consistency were selected to prepare specimens for initial and final setting time testing as shown in table 2. In the case of CCA, the initial and final setting times increased from 115 to 271 min. and 223 to 405 min., respectively when the replaced CCA percentage was enhanced from 0 to 20%. An increment of setting times with the increasing of replaced CCA ratio can be explained by two reasons. The first one is an increase of CCA content reduces the surface area of cement. As a result of this, the hydration process is slowed down, thus causing the setting times to increase. Another one reason is replacing of CCA caused the rate of the hydration process to slow down. Hence, the heat released from the hydration reaction is decreased when the evaporation of water slows which delays the setting times. The variation of setting times of the samples with CCA600 replacement shows a similar trend to CCA replacement. It can be observed that all the sample satisfy the NIS 439:2000 requirement of 45 minutes minimum initial setting time and maximum of 10 h final setting time [1].

3.4. Compressive strength of mortar cube

The compressive strengths of mortar cube at various percentage replacements of CCA or CCA600 are shown in figures 5 and 6, respectively. In the case of CCA replacement, an increasing of replacement percentage led the short term compressive strength, such as 24 h or 3 days, decreased. This is due to an increase of CCA decreased the OPC content, which induces lower hydration reaction than those of the samples with low CCA fraction. However, a differential in compressive strength between the CCA replacement and the 0% CCA replacement samples tended to decrease by increasing a soaked time to 28 days. An increment of compressive strength in the samples with CCA replacement can be attributed to the cement gel was produced by pozzolanic reaction between the amorphous silica from CCA and the calcium hydroxide ($\text{Ca}(\text{OH})_2$) in paste. The mechanisms of cement hydration with CCA are shown

in equations (1) and (2).

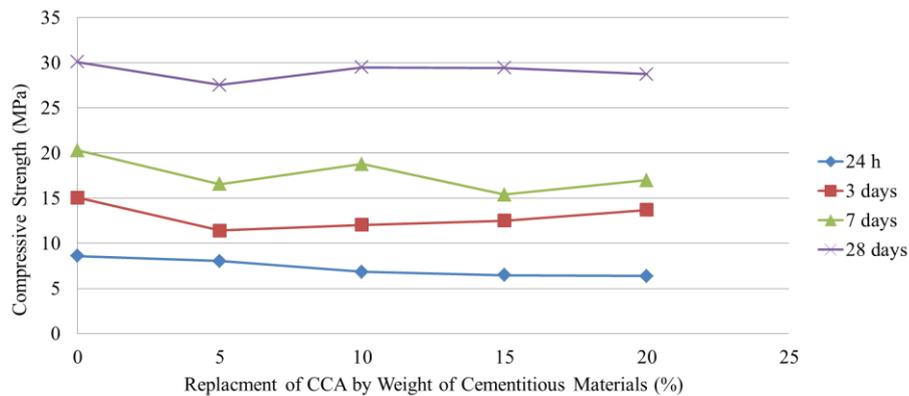


Figure 5. Effect of CCA replacement on the compressive strength of mortar cubes.

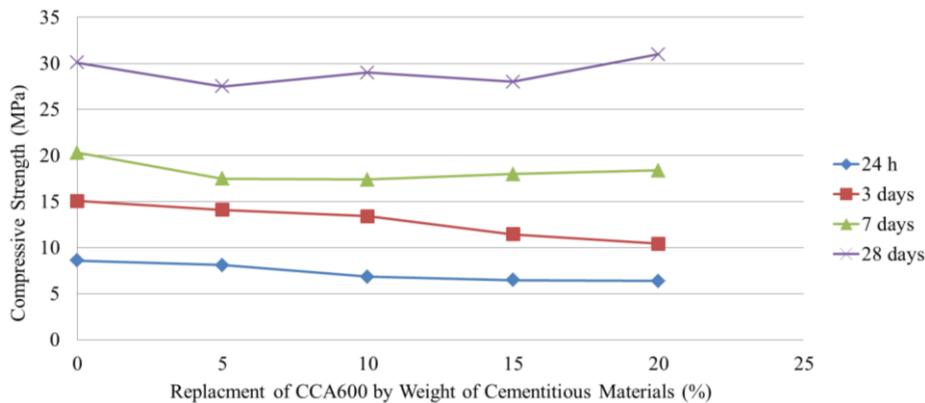


Figure 6. Effect of CCA600 replacement on the compressive strength of mortar cubes.

The relationship between the compressive strength of mortar and replacement of CCA600 showed a similar trend with the CCA replacement. Moreover, at the age of 28 days, the compressive strength of CCA600 with 20% replacement was higher than the sample with 0% replacement. This may be due to strong pozzolanic reaction between amorphous silica and Ca(OH)_2 and results on the higher compressive strength. The compressive strengths at 28 days were used to calculate the pozzolanic activity index. The samples of 20% replacing by CCA and the 20% replacing by CCA600 samples showed 95% and 103% of the pozzolanic activity index, respectively. These values are higher than minimum requirements of 75% as referred by ASTM C618 [31], which indicate that CCA and CCA600 are suitable pozzolanic materials to be used for replacing OPC for concrete.

3.5. Compressive strength and splitting tensile strength of concrete

Five different replacement percentages of concrete mixes (replacement ranging from 5% to 20% by weight of cement) including the control mix were prepared with a water binder ratio of 0.53 for a design cube compressive strength of 25 MPa. The slump test results indicate that the concrete slump decreases as the CCA or CCA600 content increases (table 3). These results indicate that concrete becomes less of their workable as the CCA or CCA600 replacement percentage increases and meaning that more water is required to make the mixes more workable. The high demand for water as the CCA or CCA600 content increases shows similar trend when compared with the W/B ratio at a

normal consistency of cement paste results. These can be attributed to the silica (from CCA or CCA600) – lime (from hydrated cement) reaction requires more water in addition to a water required during hydration of cement [32-34].

Table 3. Compressive strength and splitting tensile strength at 28 days of CCA and CCA600 mixed concrete.

Sample	% replacement	W/B ratio	Slump (mm)	28 days compressive strength (MPa)	28 days splitting tensile strength (MPa)
Reference	0	0.53	102	38	4.2
CCA	5	0.53	104	34	4.2
	10	0.53	98	42	4.1
	15	0.53	90	41	4.3
	20	0.53	87	37	3.8
CCA600	5	0.53	97	41	4.0
	10	0.53	97	45	4.6
	15	0.53	94	39	4.1
	20	0.53	90	39	3.7

The compressive strength and the splitting tensile strength values of CCA and CCA600 blended concretes after 28 days of curing are listed in table 3. It can be clearly observed that the compressive strength reached highest value for the 10 replacement percentage of CCA or CCA600 samples. The 28 days compressive strength of the sample with 10% replacement of CCA and CCA600 are increased by 11% and 18%, respectively as compared to the control sample. The 28 days splitting tensile strength of the CCA or CCA600 replacement samples slightly changed with the increase of replacement fraction. The highest values of splitting tensile strength can be observed from 15% replacement percentage of CCA and 10% replacement percentage of CCA600. However, the splitting tensile strength tended to decrease when the CCA or CCA600 replacement fraction was increased to 20%.

The compressive strength of CCA and CCA600 blended concretes specimens for 7, 14, 28 and 50 days of curing times are shown in figures 7 and 8, respectively. It can be observed that the short-term strength (7 days and 14 days curing times) of the CCA and CCA600 replacement slightly changed with increasing replacement percentage. However, it was found that as CCA or CCA600 fraction increased, long-term strength (28 days and 50 days curing times) tended to increase and reached maximum value in the samples with 10% replacement percentage thereafter, it decreased for the samples with 15 and 20% replacement fraction. A decrease of the 28 days compressive strength of concrete with 15 and 20% of CCA replacement is not a similar trend when compare to a mortar cube, which shows constant strength with increase CCA fraction. This can be attributed to the water added affect. In the previous analysis, an increase of CCA caused increase in water requirement in a paste. The water added in mortar mixed was adjusted for a normal consistency. On the other hand, the W/B ratio of concrete mixed was fixed to 0.53 and may be caused an ineffectiveness of cement hydration reaction due to insufficient water.

An increase of long-term strength with increasing in CCA or CCA600 replacement ratio may be attributed to the pozzolanic reaction between the amorphous silica from CCA or CCA600 and the calcium hydroxide in a paste as shown in equation (2). As the CCA or CCA600 content increases, more silica is available to react with the calcium hydroxide produced during the hydration of cement, thus producing more cementitious materials, which contribute to the higher compressive strength. A distinct increase of long-term strength suggests that CCA or CCA600 blended cement gain strength slowly at early curing age. This result corresponds with the findings from previous studies [35, 36], which state that pozzolanic reaction is a slow rate, thus a longer curing time is needed to observe its positive effects [37]. A decrease in compressive strength of concrete is initially observed when the CCA or CCA600 replaced cement higher than 10% by weight. This result suggests that the presence

of CCA or CCA600 above 10% is not advantageous in improving the compressive strength of concrete. Moreover, the compressive strength of the CCA600 replacement samples is slightly higher than the samples with CCA replacement. This result corresponds with the chemical and mineralogical analyzes. The compressive strength of concrete at 50 days curing period of CCA and CCA600 replaced cement shows higher values when compared with the control sample. This demonstrates the potential for the CCA and CCA600 for used as a cement replacement materials.

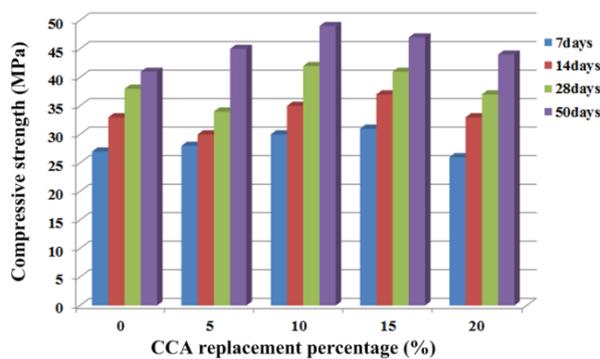


Figure 7. Compressive strength of CCA blended concretes.

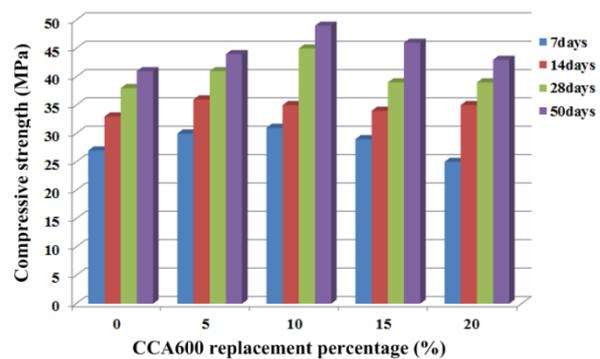


Figure 8. Compressive strength of CCA600 blended concretes.

4. Conclusion

From the results of various tests performed, the following conclusions can be drawn:

- The summary of SiO_2 , Al_2O_3 and Fe_2O_3 and the amorphous silica phase tended to increase with increasing the calcined temperatures.
- The corn cob ash which treated at 600°C for 4 h exhibits satisfied chemical and physical properties for applied as pozzolanic material.
- An increment of the replacement fraction of CCA or CCA600 caused the water requirements, initial setting time and final setting time of the cement paste increased.
- The pozzolanic activity index, which calculated from the compressive strengths of mortar at 28 days of the 20% replacing by CCA and the 20% replacing by CCA600 samples are 95% and 103%, respectively.
- The CCA600 samples show slightly higher effectiveness for improvement the splitting tensile strength and compressive strength of concrete when compared with CCA.
- CCA and CCA600 can be used as a cement replacement material in concrete to improve the compressive strength at later ages. Due to the long-term strength development.

Finally, the test results showed that the CCA or CCA which treated at 600°C for 4 h have a potential to be used as alternative pozzolanic materials.

Acknowledgments

This work was financially supported by the Naresuan University. Thanks also to Department of Civil Engineering, Faculty of Engineering, Naresuan University for supporting facilities. Acknowledgments are also to Mr. Don Hindle, for helpful comments and corrections of the manuscript.

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