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RESEARCH ARTICLES

UTILIZATION OF FLY ASH AND CONCRETE RESIDUE IN THE PRODUCTION OF GEOPOLYMER BRICKS

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ABSTRACT

This study presents the development of geopolymer bricks synthetized from industrial waste, including fly ash mixed with concrete residue containing aluminosilicate compound. The above two ingredients are mixed according to five ratios: 100:0, 95:5, 90:10, 85:15, and 80:20. The mixture's physico-mechanical properties, in terms of water absorption and the compressive strength of the geopolymer bricks, are investigated according to the TIS 168-2546 standard. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) analyses are used to investigate the microstructure and the elemental and phase composition of the brick specimens. The results indicate that the combination of fly ash and concrete residue represents a suitable approach to brick production, as required by the TIS 168–2546 standard.

KEYWORDS

geopolymer; facing brick; fly ash; concrete residue

1. INTRODUCTION

Ordinary Portland cement (OPC) is widely used as a binder in concrete manufacture. The process of transforming natural limestone into reactive cementitious phases in cement production requires a large amount of energy, leading to intensive carbon dioxide emissions [1-2]; the production of one ton of OPC releases one ton of carbon dioxide gases to the atmosphere [3-6]. Due to the increasing associated environmental concerns, various studies have explored alternative materials to secure the sustainability of the construction sector, including those relating to geopolymerization technology [7-12]. A geopolymer is an inorganic mineral binder produced from the reaction of aluminosilicate in a highly concentrated alkali hydroxide and/or silicate solution, which forms an amorphous to semi-crystalline polymeric structure with interconnected Si-O-Al bonds [2, 12].

In Thailand, fly ash (FA) is the rawest material used in geopolymers. FA is the waste produced from the combustion of pulverized coal in electrical power plants. According to Chindaprasirt et al. [13], 3.5 million tons of FA have been generated annually in Thailand

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since 2001. These authors showed that the use of FA that has been classified as class F FA in accordance with ASTM C618 in the production process can improve the properties of geopolymers due to its main chemical composition, namely silica (SiO2) and alumina (Al_2O_3), which are essential for geopolymers' structure [13]. Hanjitsuwan et al. [14] proved that the mechanical properties of geopolymers are enhanced by increasing the calcium in OPC, as a high amount of calcium oxide (CaO) in OPC (65.3%) forms calcium silicate hydrates (CSH) along with the aluminosilicate network that is configured in the geopolymer's matrix. The properties are further improved by the coexistence of the geopolymer gel and the calcium silicate hydrate (CSH) productivity, as enclosed in the geopolymer's structure. Similarly, Ahmari et al. [6] stated that the use of waste concrete (14.5% by weight of type II Portland cement, 31.2% sand, 44.8% coarse aggregate, and 9.5% water) as the calcium source, together with FA, can bolster the geopolymer's compressive strength. Many researchers have found that FA can be deployed as a geopolymer binder in the formation of concrete containing recycled materials, including used packaging foam [15], bottom ash [16], and recycled aggregate [17-19] as a natural aggregate of satisfactory strength, leading to the reduction of cement, natural aggregate consumption, and waste disposal. Recently, Kathirvel and Kaliyaperumal studied the influence of recycled concrete aggregates on the flexural properties of reinforced alkali-activated slag concrete, reporting that the addition of recycled concrete aggregates improves the strength characteristics of geopolymer concrete [20].

As a result, this study used concrete residue (CR) as its calcium source rather than OPC. CR is the waste that remains after cleaning the equipment for concrete production; namely, concrete that is originally prepared from Portland cement type I (12.5%), sand (37.5%), coarse aggregate (45.8%), and water (4.2%), which consists mainly of CaO (32.0%) and SiO_2 (23.1%). As not all the CR can be recycled into the system, it is disposed of in landfills, causing harmful effects on the surrounding environment and ecosystems [21]. Meanwhile, FA is the waste from the combustion of lignite coal in electrical power plants, and is classified as class C FA in accordance with ASTM C 618 [22]. Few studies have been conducted on the use of CR with FA as a partial replacement for OPC in the production of geopolymer bricks.

Accordingly, the objective of this study was to investigate the optimum ratio of CR and FA in the production of bricks through the geopolymerization process. The produced bricks were examined at various ratios of CR to FA, to test their physico-mechanical properties according to TIS 168-2546 specification [23]; their microstructure and elemental and phase composition were assessed via scanning electron microscopy (SEM) and X-ray diffraction (XRD) analyses.

2. MATERIALS AND METHODS

2.1 Materials

The materials used in this study were FA and CR. The former was obtained from the Mae Moh power plant, Lampang province, Thailand, while the latter was procured from Tratmunkong Co. Ltd., Trat province, Thailand. The CR was dried at 105°C for six hours to remove moisture content before being ground into a smaller size using a sieve No. 140 (106 μm).

The chemical characterizations of both ingredients were investigated and their elements analyzed via an X-ray Fluorescence Spectrometer (XRF, Bruker model 58 Tiger). The elemental composition of the two components is shown in Table 1, whereby it can be observed that the FA consisted mainly of SiO_2 (34.4%) and Al_2O_3 (18.7%), classifying it as class C FA [22], while the CR was chiefly comprised of CaO (32.0%) and SiO_2 (23.1%).

TABLE 1. Chemical compositions of fly ash and cement residue.

Chemical compound	Fly ash (%)	Concrete residue (%)
SiO_2	34.4	23.1
Al_2O_3	18.7	6.70
CaO	16.7	32.0
Fe_2O_3	12.4	3.41
SO_3	4.06	1.98
K_2O	2.44	0.44
MgO	2.04	2.22
Na_2O	1.80	0.29
TiO_2	0.41	0.32

To determine the particle size distribution of both constituents, a Laser Particle Size Distribution Analyzer (Malvern Mastersizer 3000) was employed. The results denote that the mean particle sizes of FA and CR were 30.8 and 14.0 μm respectively. For approximately 90% of the former, the particles were smaller than 193 μm, while for around 90% of the latter, they were smaller than 55.9 μm, indicating that CR is much finer than FA.

The SEM micrographs of the twin components are given in Figure 1. As may be observed, the FA particles were spherical with smooth surfaces; a large proportion of them had diameters larger than 100 μm. Meanwhile, the CR particles were irregularly shaped with rough surfaces; a significant percentage had diameters smaller than 100 μm, confirming the observed particle size distribution of both ingredients as described earlier.

FIGURE 1. SEM micrographs of: (a) fly ash (FA) and (b) concrete residue (CR).

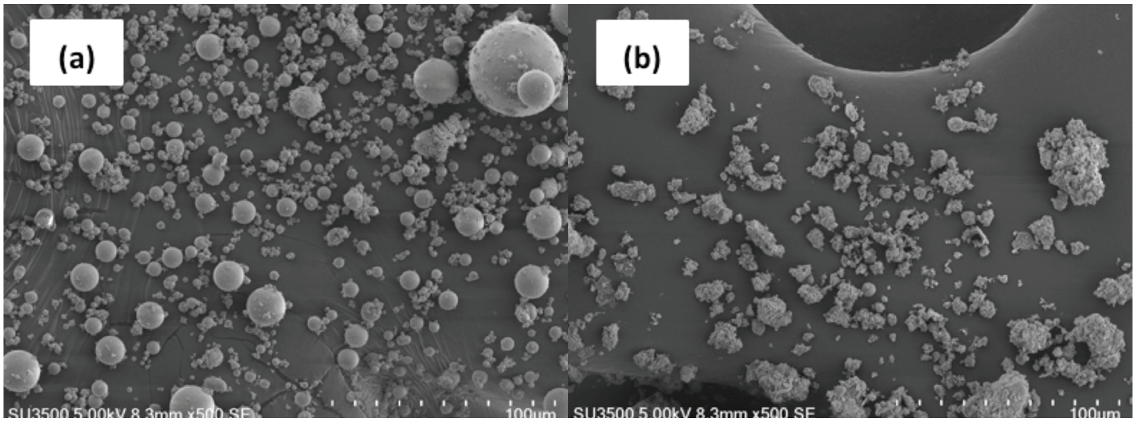
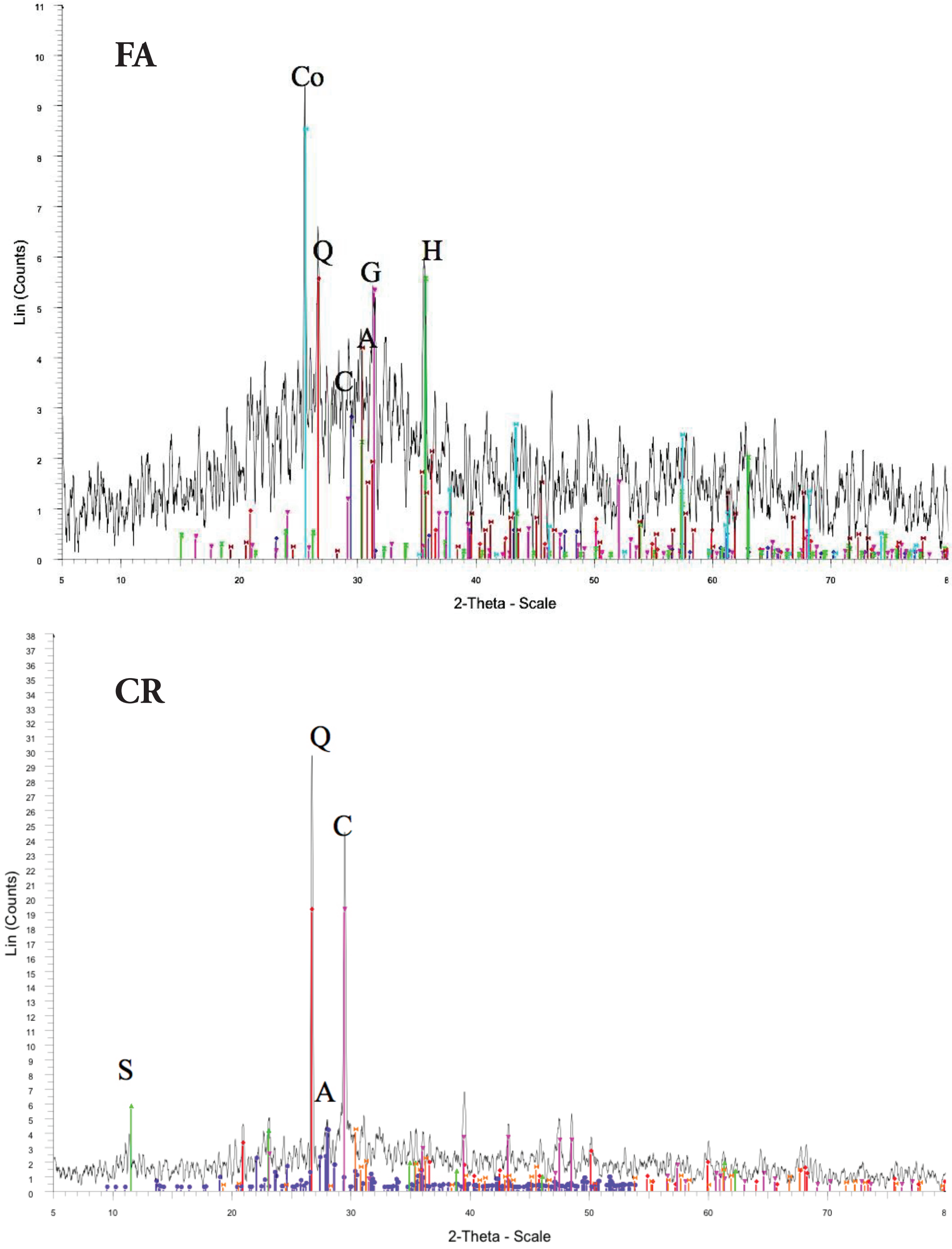


Figure 2 shows the XRD pattern of both appurtenances. It is observed that FA contained a higher content of amorphous phase than did CR. In the former, the main crystalline phases were identified as corundum (Al_2O_3), quartz (SiO_2), hematite (Fe_2O_3), and gehlenite ($Ca_2Al(AlSiO_7)$; meanwhile, peaks of anorthite ($CaAl_2Si_2O_8$) and calcite ($CaCO_3$) were observed. For the latter, the main peaks were quartz (SiO_2) and calcite ($CaCO_3$); a few minor peaks of spinel ($MgAl_2O_4$) and anorthite ($CaAl_2Si_2O_8$) were also reported.

FIGURE 2. XRD patterns of FA and CR: [A: Anorthite, C: Calcite, Q: Quartz, G: Gehlenite, H: Hematite, S: Spinel, Co: Corundum].

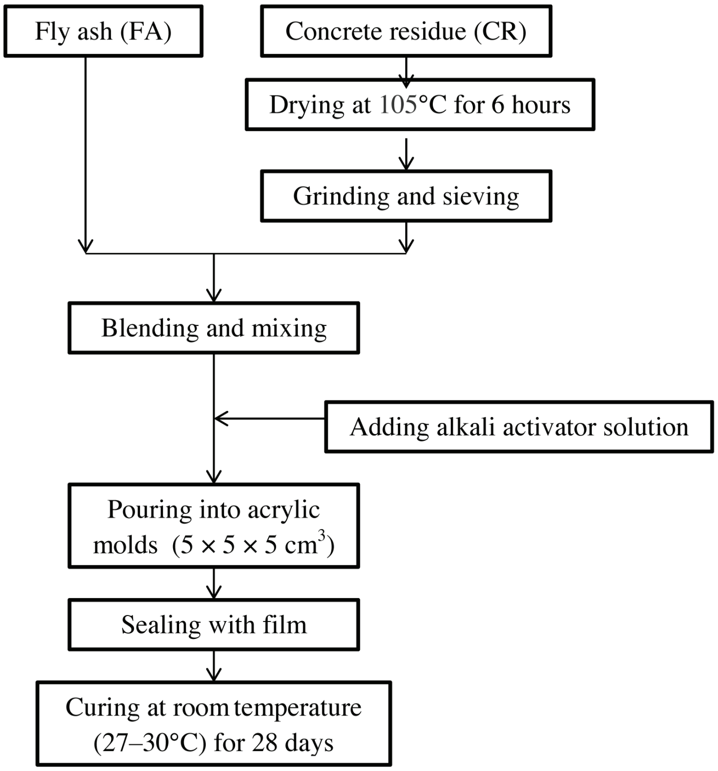


2.2 Experimental methods

Sodium hydroxide concentration of 10 molar (10M NaOH) was prepared at least 24 hours before use by dissolving analytical grade sodium hydroxide in flake form (98% purity) in distilled water. Meanwhile, sodium silicate (Na_2SiO_3) solution was obtained from industrial grade sodium silicate in liquid form with 14.25% of Na_2O, 31.25% of SiO_2, and 54.50% of water by weight. In another step, alkaline solution was prepared by mixing 10M NaOH with Na_2SiO_3. The ratio of Na_2SiO_3 solution to NaOH solution was fixed at 16:24 by weight, as per the research of Hanjitsuwan [14]. The alkaline solution was cooled down at room temperature before use.

FA and CR were mixed according to the following five ratios: 100:0 (FA100CR0), 95:5 (FA95CR5), 90:10 (FA90CR10), 85:15 (FA85CR15), and 80:20 (FA80CR20) by weight. The mixtures were then commingled with an alkali activator solution in a blender at a liquid-to-solid ratio of 40:100. The geopolymer paste was then poured into 5 cm × 5 cm × 5 cm acrylic molds. Consequently, the molded samples were sealed with film wrap to prevent moisture evaporation during curing at room temperature (27-30°C) for 28 days. The specimen preparation process is summarized in Figure 3.

FIGURE 3. The process of geopolymer brick production.



2.3 Test program

The general appearance, compressive strength, and water absorption of the geopolymer bricks were determined according to the Thai Industrial Standard (TIS) 168-2546 specification. As mentioned above, SEM (Jeol JSM-6480LV) and XRD analysis were used to identify the microstructure characterization and mineralogical phase of the bricks, respectively. Finally, the

IBM SPSS Statistics program was used for the mean and standard deviation calculation of the data in each parameter, while a one-way ANOVA was conducted to compare the effects of varying amounts of concrete residue on the water absorption and compressive strength.

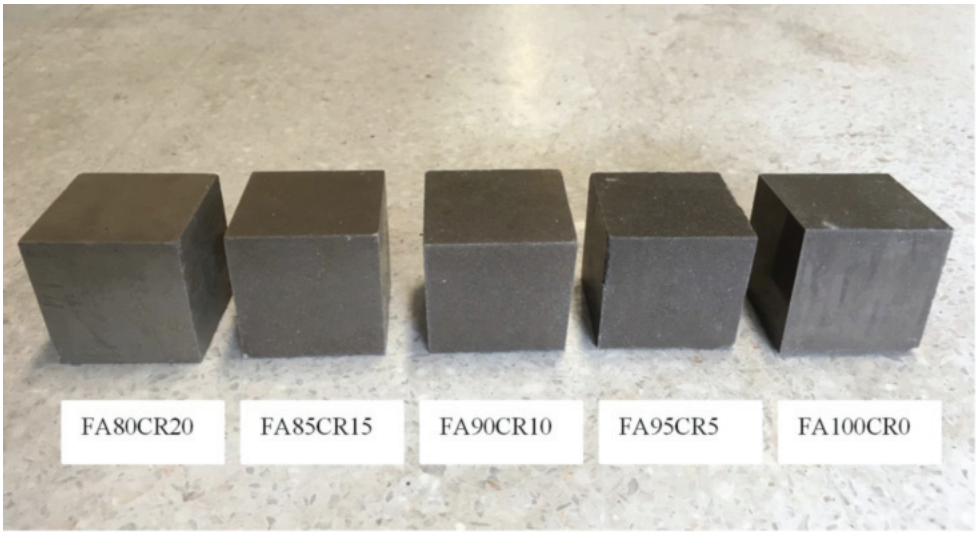
3. RESULTS AND DISCUSSION

3.1 Physical and mechanical properties

The characteristics of geopolymer bricks of all series (FA100CR0, FA95CR5, FA90CR10, FA85CR15, and FA80CR20), including general appearance, compressive strength, and water absorption, were compliant with the TIS168-2546 specification.

All bricks were dark brown in appearance, with cubic solid shapes, as shown in Figure 4. They were all the same size and had smooth surfaces with no cracks.

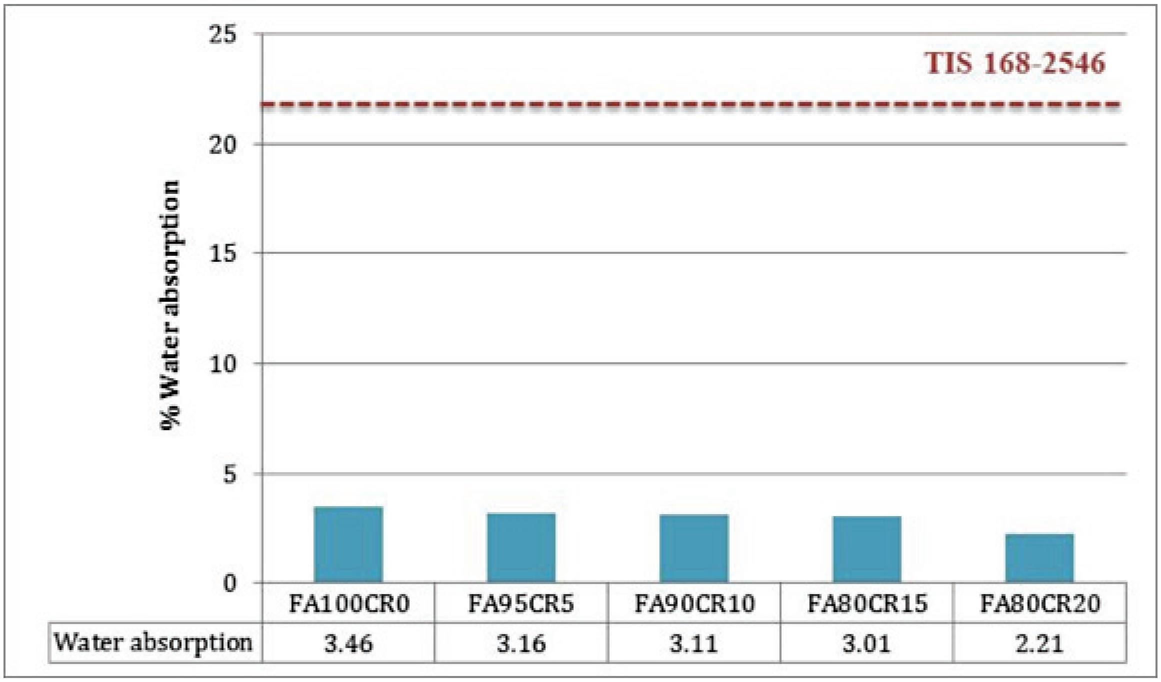
FIGURE 4. General appearance of geopolymer bricks.



Water absorption is a significant parameter for the structure of geopolymer bricks, as it demonstrates their level of permeability. In this regard, there were statistically significant differences between all series ($p < 0.0005$, $N = 25$). Figure 5 shows that the water absorption of bricks with 0% (FA100CR0), 5% (FA95CR5), 10% (FA90CR10), 15% (FA85CR15), and 20% (FA80CR20) of concrete residue was 3.46%, 3.16%, 3.11%, 3.01%, and 2.21% respectively. Therefore, the bricks with the highest proportion of CR had the lowest level of water absorption. The latter decreased when increasing the concrete residue replacement, as concrete residue particles act as filler in pores [6]. With regards to the results obtained, all bricks satisfied the water absorption requirement (limit of 22%) prescribed by the TIS 168-2546 standard.

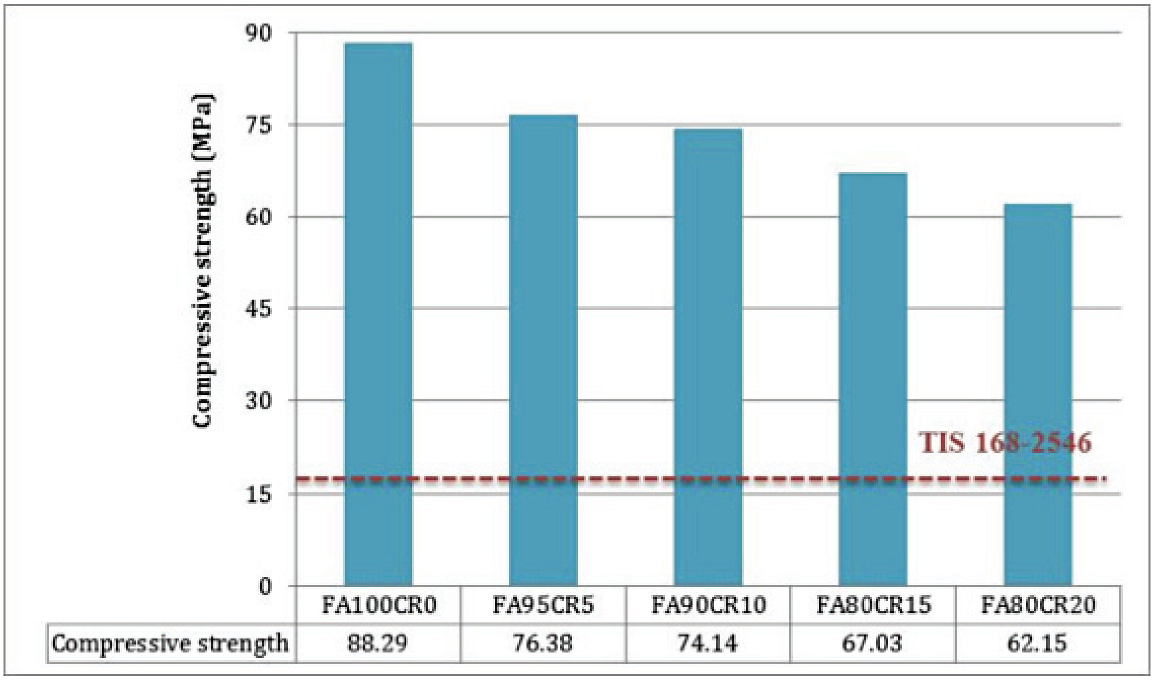
Compressive strength is a significant parameter for the construction materials. Looking at this parameter for all series of geopolymer bricks, FA and CR had statistically significant differences ($p < 0.0005$, $N = 25$). Figure 6 shows that the compressive strength of bricks with 0% (FA100CR0), 5% (FA95CR5), 10% (FA90CR10), 15% (FA85CR15), and 20% (FA80CR20) of CR cured for 28 days was 88.29, 76.38, 74.14, 67.03, and 62.15 MPa

FIGURE 5. Water absorption of geopolymer bricks.



respectively, which are impressive values when stacked up against the TIS 168-2546 standard (17 MPa). As Lloyd and Ragnan [24] observed, compressive strength will be higher when there is more curing time allocated to complete the geopolymerization process.

FIGURE 6. Compressive strength of geopolymer bricks.



As expected, the use of CR in the geopolymer system led to a reduction of water absorption, which positively reflected on the compressive strength for geopolymer specimens, due to the additional calcium oxide sourced from CR forms' calcium silicate hydrates (CSH) in the geopolymer matrix, as shown in Figure 7, and the incorporation of calcium into the geopolymer network as a charge-balancing cation [6]. Diaz et al. [25] and Yip, Lukey and Deventer [26] found that increasing the calcium oxide content in a geopolymer matrix's composition can improve the compressive strength. Aliabdo, Elmoaty and Salem [27] also found that adding Portland cement type I can improve fly ash Class F based geopolymer properties including compressive strength, tensile strength, modulus of elasticity, water absorption and porosity. Ahmari et al. [6] explained that the contribution of Ca to the geopolymerization can be explained via two mechanisms. In the first state, Ca^{2+} as a charge-balancing agent is added to the geopolymeric network as follows:

$$\equiv \mathrm{Si\ O^{-}} + \mathrm{Ca^{2+}} \rightarrow \equiv \mathrm{Si\ O^{-}Ca+} + \mathrm{H_2O}$$

$$\equiv \mathrm{Si\ O^{-}Ca^{2+}} + \mathrm{O^{-}\ Al} + \mathrm{H_2O} \rightarrow [\equiv \mathrm{Si\ O\ Ca\ O\ Al} \equiv] \rightarrow \equiv \mathrm{Si\ O\ Al} \equiv + \mathrm{Ca^{2+}} + 2\mathrm{OH^{-}} \qquad (1)$$

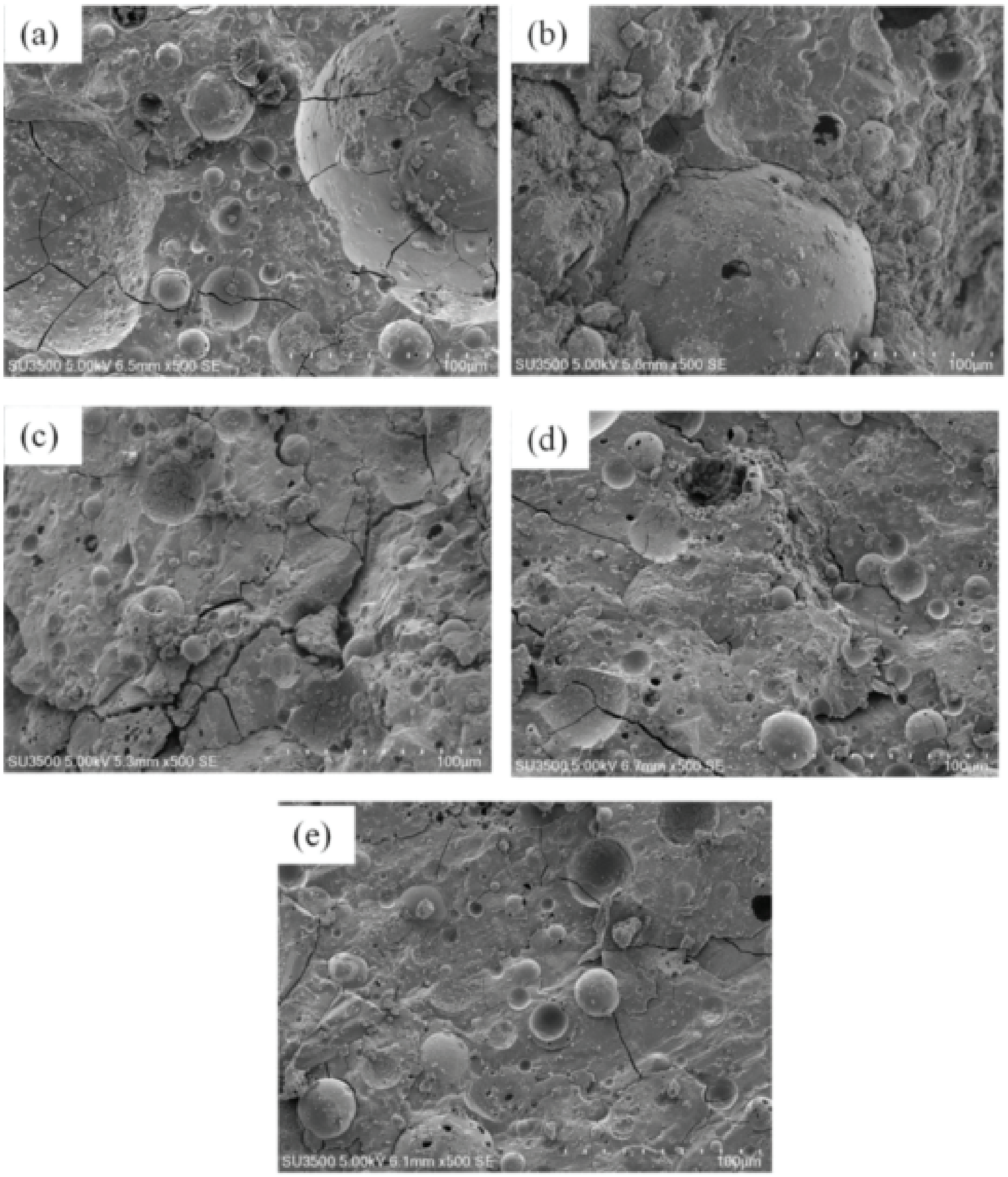
In the second state, Ca forms calcium silicate hydrate (CSH) gel, a product of a geopolymeric or pozzolanic reaction that coexists with the geopolymeric gel [6, 28].

As shown in Figure 6, the compressive strength is reduced when increasing the CR replacement, as the excess calcium from the bolstered CR content causes precipitation of calcium hydroxide ($Ca(OH)_2$), inhibiting the dissolution of silica and alumina in geopolymerization and obstructing calcium species from participating in the formation of CSH [6, 26]; all of this works to degrade the strength of the geopolymer. Based on the experiment shown in Figure 6, all series of geopolymer bricks satisfied the compressive strength requirement (more than 17 MPa) prescribed by the TIS 168-2546 standard.

3.2 SEM observations

The SEM images shown in Figure 7 clearly indicate the formation of the new phase of the geopolymer, which is entirely different in its morphology than the starting material shown in Figure 1. The SEM micrographs of the geopolymer-facing bricks with 0%, 5%, 10%, 15%, and 20% CR, as shown in Figure 7, indicate that the porosity and cracks were reduced when increasing the CR replacement, because the calcium oxide (CaO) in CR formed a product of a geopolymeric or pozzolanic reaction that coexisted with the geopolymeric gel to form a dense structure and fill up the pore space. It is evident from the images that the microstructure of the samples, especially in Figure 7 (c), (d), and (e), was homogenous with some of the crystals embedded in the gel matrix. As shown in the micrographs, the results indicate that the particle size distribution plays a major role in completing the geopolymerization process. Some large particles of FA that were embedded in the matrix can be seen in Figure 7 (a) and (b) with a partial reaction during the alkaline activation process, which means that during the geopolymerization process, smaller particles were completely reacted and dissolved while their larger counterparts were only partially so [29, 30, 31]. As per Zhang et al. [32], the particle sizes and specific surface area of the initial raw materials are the key factors for the geopolymerization reaction rate and extent. In addition, by increasing the CR with smaller-sized particles than those of FA, the porosity and pore size in the microstructure decreased, which supported the lower water absorption of the geopolymer bricks (Figure 5).

FIGURE 7. Scanning electron micrographs of geopolymer bricks at 100x: (a) FA100CR0 (b) FA95CR5 (c) FA90CR10 (d) FA85CR15 (e) FA80CR20.

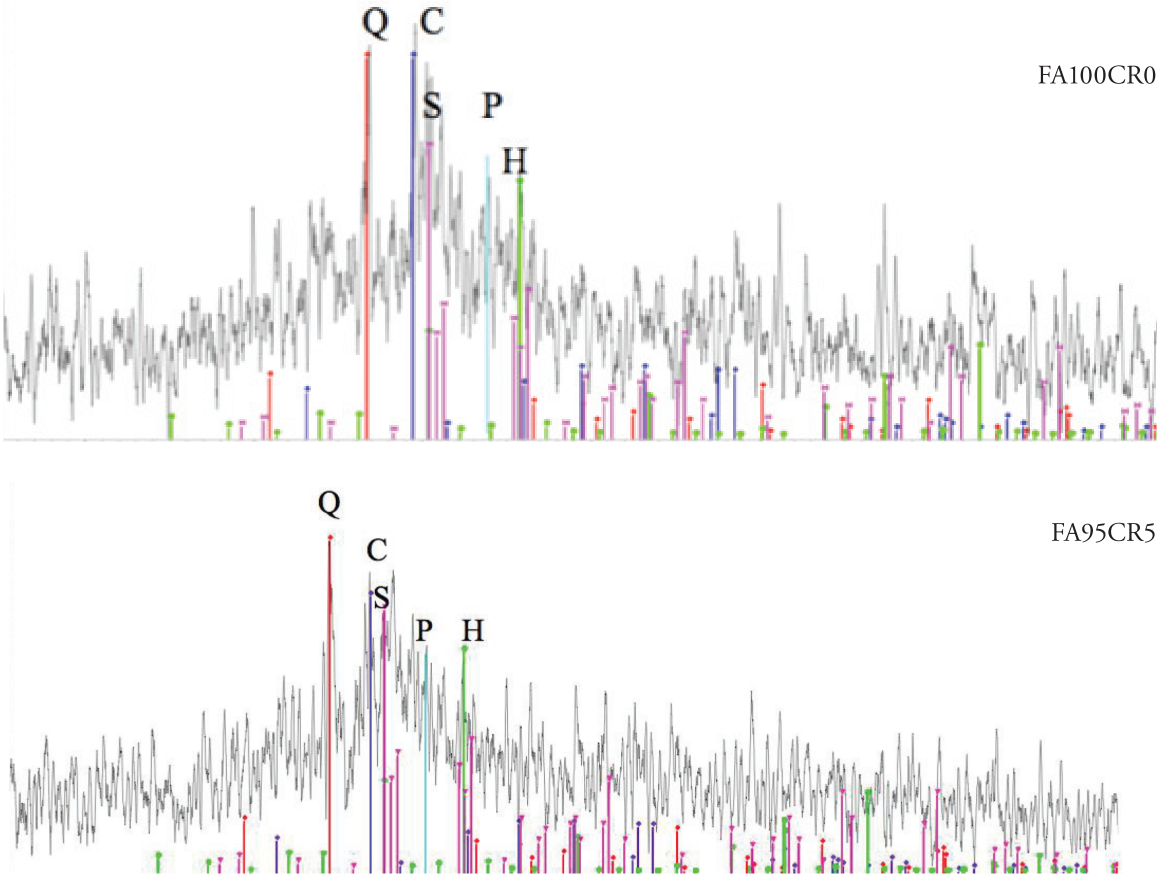


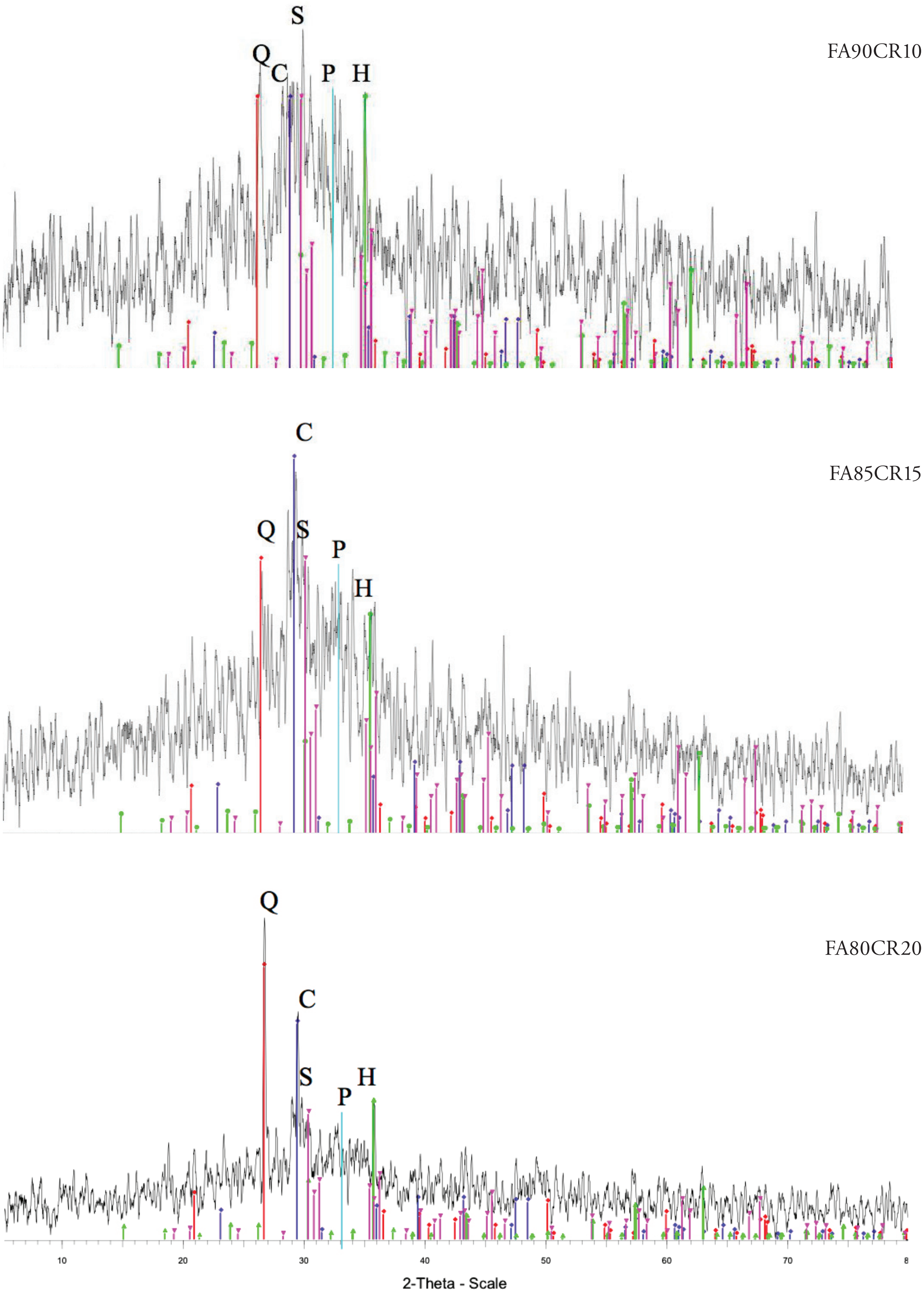
3.3 XRD analysis

The mineralogical composition of the geopolymer bricks after curing at room temperature for 28 days was determined by the XRD analytical technique. Despite the fact that an amorphous to semi-crystalline state is the normal form of geopolymer after the alkali activation processes, some minor crystalline phases were found. Figure 8 illustrates the presence of semi-crystalline phases, which caused the formation of new phases in the alkali activation process, or as unreacted remaining crystalline phases from the initial raw materials in the geopolymer specimens. The XRD patterns of geopolymer bricks with 0%, 5%, 10%, 15%, and 20% CR are presented in Fig. 8. The results indicate that in the region of 25° (2θ) to 35° (2θ), the XRD patterns of the bricks with 5%, 10%, 15%, and 20% CR are evidently similar, due to the partial replacement of CR and the use of FA in the geopolymer matrix system; a new hydration product, which formed in the specimens, was mainly composed of coexisting alkali three-dimensional amorphous aluminosilicate nets or geopolymer gel and types of calcium silicate hydrate phases (CSH) gel [2, 6, 27, 33].

The results of this research will constitute an alternative for the management of solid waste from industrial sectors, including the use of FA and CR as raw materials for the production of geopolymer bricks. According to Rloyd and Rangan [24], FA and waste from coal-fired power generation can contribute to geopolymer concrete that helps sustainable development, as FA can be used as an alternative binder to Portland cement in concrete production, with less energy consumption, low carbon dioxide emissions, and lower cost than conventional concrete. The high strength characteristic of geopolymer concrete has seen it put to use in railway sleepers, sewer pipes, and other pre-stressed concrete building materials [24]. Gourley and Johnson [34] proved that geopolymer concrete sewer pipes exhibit higher performance on a commercial scale (in a simulated aggressive sewer environment) than Portland cement concrete pipes; they also reported the high quality of reinforced geopolymer concrete railway sleepers and the fire resistance of geopolymer mortar wall panels [24]. Siddiqui [35] and Cheema et al. [36] confirmed that manufactured reinforced geopolymer concrete culverts display high performance that is compliant with Australian Standards, AS 1597.1-1974 [24]. In addition, this can be an alternative for solid waste management in industrial sectors.

FIGURE 8. X-ray diffraction pattern of geopolymer bricks: [S: CSH, C: Calcite, Q: Quartz, H: Hematite, P: Portlandite].




S
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4. CONCLUSIONS

This study focused on the feasibility of using FA and CR in the production of geopolymer bricks. Based on the results of the laboratory investigation, it may be concluded that these items can be used as raw materials for the production of bricks through the geopolymerization process, as they consist of SiO_2 and Al_2O_3, which are the essential components of a geopolymer structure. Although CR mainly consists of calcium, which can act as a filler in pores, forming a dense structure and decreasing the water absorption of geopolymer bricks, the adverse effect of Ca^{2+} is precipitation of $Ca(OH)_2$ that can coexist with the geopolymeric gel, causing the reduction of geopolymer bricks' compressive strength. That said, all series of geopolymer bricks, with 0% (FA100CR0), 5% (FA95CR5), 10% (FA90CR10), 15% (FA85CR15), and 20% (FA80CR20) of CR gained low water absorption and had a high compressive strength, satisfying the requirements prescribed in the TIS 168-2546 standard.

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