

Lime-activated one-part geopolymer mortars from construction, demolition and industrial wastes

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ABSTRACT

This work focused on the production of one-part geopolymer mortars from construction and demolition wastes (CDW) blended with steel slag. Previous related studies on geopolymer production from CDW utilized conventional two-part geopolymers comprised of highly alkaline activator solutions and CDW materials. Thus, the study's significance consists in producing high-strength (≥ 35 MPa) ambient-cured mortars from CDW with predominantly concrete waste by replacing conventional highly alkaline activator solutions with an environmentally-friendly alkaline activator, $\text{Ca}(\text{OH})_2$ powder. Four mortar mixtures were produced with CDW contents ranging from 50 to 65 % dry weight, varying the brick waste content from 3 to 18 %. The effect of elevated temperature (40 °C) curing was also considered. The results showed that 55 % CDW content had optimum performance across all parameters studied such as compressive and flexural strengths, setting time, as well as changes in nuclear magnetic resonance (NMR)-determined pore structure (porosity and mean pore size) and x-ray diffraction (XRD)-determined degree of crystallinity over time. It had 28-day compressive and flexural strengths of 42 and 5.8 MPa, respectively, and initial and final setting times of 25 and 50 min. The importance of sufficient brick waste content in the geopolymer mixtures for effective mechanical performance is highlighted. The inclusion of concrete waste in powder form reduced compressive strength under ambient curing but improved performance at 40 °C curing. It is concluded that sustainable structural mortars can be produced by 'just adding water' to an optimized CDW mixture with predominantly concrete waste blended with brick waste and slag and activated by powdered $\text{Ca}(\text{OH})_2$.

1. Introduction

The construction industry contributes significantly to global greenhouse gas emissions and solid waste streams [1]. This evokes the need to develop sustainable green geopolymer concrete/mortars. Integrating such mortar mixtures with 3D concrete printing (3DCP) could lead to significant benefits including improvement in construction speed, material use efficiency, etc [2]. Geopolymers are new-generation alkali-activated binders that can be synthesized from various aluminosilicate sources [3]. They can be formed at much lower temperatures compared to ordinary Portland cement. Hence, geopolymers are more environmentally friendly, especially as about half of the CO_2 emissions from

cement production result from the calcination of limestone at high temperatures (around 1450 °C) [4].

There is a fast-growing trend in recycling construction and demolition wastes (CDW) since natural resources for use in construction materials are depleting and environmental problems and solid waste handling are gaining more attention. However, there is significant variation in the properties of concrete containing CDW between different sources and locations [5]. CDW accounts for more than 75 % of all solid waste in Qatar with concrete waste constituting the majority (about 86 %) of construction wastes [6]. CDW contain aluminosilicates although they are less reactive compared to fly ash, metakaolin and blast furnace slag usually employed for geopolymer production. Steel slag is

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Table 1Sieve analysis of the concrete waste aggregates (between 75 μ m and 4 mm).

Sieve size (mm)	<0.075	<0.15	<0.5	<1	<2	<3.15	<4.0
Percentage passing (%)	6.5	23.6	52.8	65.8	79.8	92.7	100

also generated in large quantities in Qatar without a sustainable recycling option [7]. Thus, blending CDW with steel slag for geopolymer production would be an effective valorization strategy for the waste streams. Studies have shown that optimum geopolymer mixtures can be produced with about 30–40 % slag content. This leads to phenomenal 28-day strength increase due to enhancement of the calcium silicate hydrate (C–S–H) gel [8,9].

Several studies have investigated the use of CDW for geopolymer production. However, these were mostly done with highly alkaline activator solutions and thermal curing or treatment. One such study used concrete-based demolition waste as coarse aggregate blended with metakaolin and NaOH and Na₂SiO₃ as alkaline activators. It was observed that the addition of Ca(OH)₂ up to 10 % by weight improved the mechanical and microstructural properties [10]. Another study used a mixture of brick waste (40–100 %) and concrete waste (0–60 %) activated by NaOH–Na₂SiO₃ combinations. Brick waste was observed to be more suitable than concrete waste for geopolymerization reactions. The 28-day compressive strengths ranged from 10 to 40 MPa and the maximum strength mix contained only brick waste and the aforementioned activators [11]. More recent studies used CDW-based precursors such as hollow brick, red clay brick, roof tile, and glass waste activated by different combinations of NaOH, Na₂SiO₃ and Ca(OH)₂ for production of ambient-cured 100 % CDW-based geopolymers. Maximum 28-day compressive strengths of 19.5 MPa and 36 MPa were obtained in these studies, which used highly alkaline activator solutions including 5 M – 25 M NaOH and the optimum mixtures were successfully deployed for laboratory scale 3DCP [12,13].

The foregoing shows that previous related studies utilized conventional two-part geopolymers [14] comprised of highly alkaline activator solutions and mostly CDW materials such as brick and glass wastes that are more suitable for geopolymers. The alkaline activators used in conventional two-part geopolymers involve concentrated aqueous alkali solutions that are corrosive, viscous, difficult to handle and are not user friendly compared to a one-part geopolymer mixture that involves ‘just adding water’ to a blend of solid alkaline activator and aluminosilicate precursors [14,15]. Hence, this study differs from related studies, for e.g. Refs. [12,13,16], as it sought to produce high strength (≥ 35 MPa) ambient-cured geopolymer mortars from CDW consisting of predominantly concrete waste, which is usually considered unsuitable for geopolymers [17]. Its innovation and significance consists in replacing conventional highly alkaline activator solutions with a solid environmentally friendly alkaline activator, Ca(OH)₂ powder, to produce high strength geopolymer mortars cured at ambient temperature (20 °C) from CDW with predominantly concrete waste. Furthermore, the utilization of large amounts of waste concrete aggregates would promote the development and application of green building materials leading to lower life cycle costs [18], especially as recent studies have characterized their effective structural behaviors with and without reinforcement using appropriate constitutive models [19–21]. The study investigated the optimum CDW content for maximum strength of geopolymer mixtures comprised of at least 50 % by weight CDW content since high CDW content geopolymers are often characterized by relatively low strengths. The study also evaluated how changes in the pore structure and microstructure of the mixtures over time account for observed differences in mechanical behavior through proton nuclear magnetic resonance (¹H NMR) and X-ray diffraction (XRD) analysis.

2. Experimental methodology

2.1. Waste-based aluminosilicate sources

Waste concrete and red clay solid bricks were obtained from demolished structures in Doha, Qatar and were generally stable and not highly dispersed. These were broken into granular form using a 2.25 × 3 inches model LC-27F–O363835 laboratory jaw crusher (Gilson, Ohio, USA). The concrete waste was broken down into powder (<75 μ m) and aggregate (between 75 μ m and 4 mm) forms with the aid of the jaw crusher and appropriate sieve sizes. Similarly, the crushed red clay solid brick waste (<1.4 mm based on the jaw crusher’s maximum size) was separated into powder (<75 μ m) and aggregate (between 75 μ m and 1.4 mm) portions. The <75 μ m powder form of the CDW materials were used together with steel slag as aluminosilicate sources, while portions with sizes ranging from 75 μ m to 4 mm were used as aggregates in the geopolymer mortars. Table 1 shows the sieve analysis data of the concrete waste aggregates (between 75 μ m and 4 mm). The particle size analysis of the waste materials as well as powdered alkaline activator and strength enhancement additives, determined with the Beckman Coulter LS13 320 laser-diffraction particle size analyzer (LD-PSA), is shown in Fig. 1.

2.2. Alkaline activator and additives for strength enhancement

Ca(OH)₂ with 90 % purity was used as the main alkaline activator in this work. Some other additives were added in little amounts for strength enhancement. These include Gypsum powder, microsilica and Superplasticizer Hyperplast-ES910i (H-ES910i). Specifically, the superplasticizer was added to reduce the water to cementitious materials ratio, improve mechanical behavior and make the geopolymer mixtures flowable and suitable for 3DCP.

2.3. Preparation of geopolymer mortars

The steel slag was first ground separately using a 450-W Philips model HR2058/91 blender for about 3 min. Thereafter, the powdered materials (slag, Ca(OH)₂, gypsum, microsilica, and CDW powders), were dry-mixed together and collectively ground in the Philips blender for another 3 min. The aggregate portion (>75 μ m) of the CDW was then added to the ground powders and thoroughly mixed. Thereafter, the superplasticizer was added to the water and mixed with the aforementioned geopolymer constituents in a Hobart N50 5-Quart commercial stand mixer. The geopolymer mixtures were casted with flowable water contents as the focus is on mixtures amenable to 3DCP. The geopolymer mortars formed were then placed into molds – 50 × 50 × 50 mm for compressive strength measurements and 160 × 40 × 40 mm for flexural strength measurements. The samples were demolded after 24 h and cured at ambient temperature (20 °C). Some samples were cured at 40 °C to investigate the effect of elevated temperature curing as such temperatures are not uncommon in Qatar. The flexural strength samples were stored in water until tested in line with ASTM C-348 [22]. Samples for NMR porosity measurements were placed in 25 mm diameter by 50 mm long plastic molds and cured at ambient temperature after demoulding. They were then stored in water for 48 h to be saturated before testing. Fig. 2 shows some of the samples from different geopolymer mixtures, while Fig. 3 shows a flow chart summarizing the experimental methodology.

Table 2 shows the mix proportions of the geopolymer mortars tested. The geopolymer constituents were arbitrarily proportioned such that the CDW contents varied from 50 to 65 % (dry weight) of the mixture. Concrete waste aggregate comprised 47 % of the mixture since it is the most abundant CDW in Qatar, while red clay solid brick powder comprised 3 % of the mixture. The CDW content variation was based on increasing the solid brick aggregates content from 0 to 15 %. Gypsum and silica fume were added in small amounts to Mixes 1 and 2, which

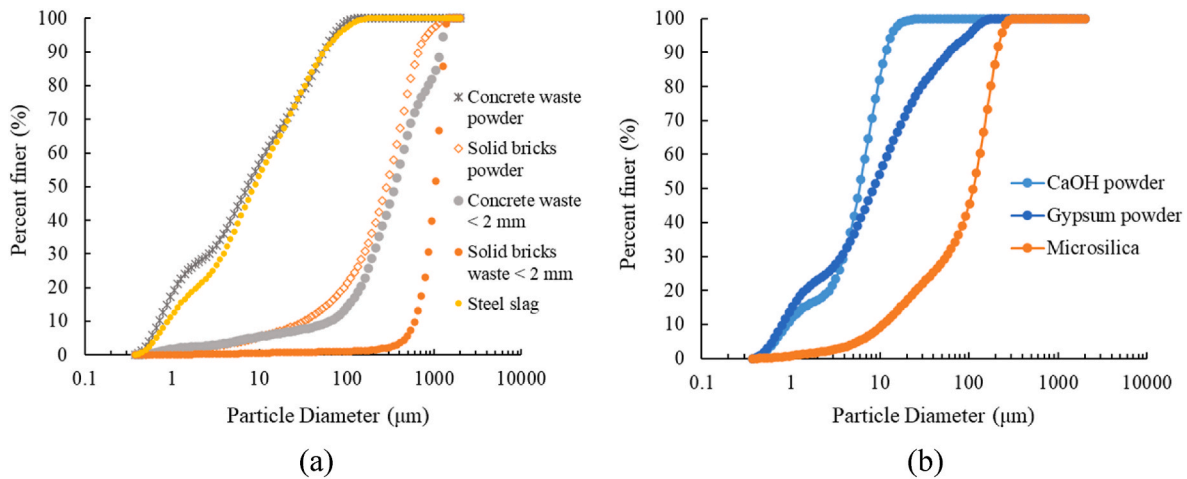


Fig. 1. Particle size analysis of the materials used in the geopolymer mortars determined with laser-diffraction particle size analyzer, (a) aluminosilicate sources, and (b) alkaline additives.



Fig. 2. Photo showing the appearance of samples of the geopolymer mixtures used for ultrasonic pulse velocity (UPV) testing.

had lower brick aggregate content, for strength enhancement (see Table 2). The slag content was kept around 23–28 %. The water-to-cementitious materials ratios were 0.36 and 0.37 as appropriate. These were kept at the lowest possible level that made the mixtures flowable and amenable for 3DCP similar to values used for actual 3D printing of geopolymer mortar in a related work [23].

A set of mixes (labelled as Mixes 1cp – 4cp) tested the effect of concrete waste granulometry. Hence, unlike Mixes 1–4, which had only 47 % concrete waste aggregate (between 75 μm and 4 mm), these mixes had 44 % concrete waste aggregate and 3 % concrete waste powder (<75 μm) (Table 2). Consequently, fewer testing was conducted on these mixes in contrast to the main mixes. Similarly, mixes cured at 40 °C had fewer testing since this work focused on developing lime-activated geopolymers suitable for ambient temperature curing.

2.4. Testing methods

2.4.1. Compressive strength

Mortars were tested for compressive strength at 3, 7, 14 and 28 days after casting, as applicable, on three replicate 50 mm cube specimens prepared for each geopolymer mixture following ASTM C109/C109 M

[24].

2.4.2. Flexural strength

The flexural strength of selected mixtures was determined on 160 $\times$ 40 $\times$ 40 mm prisms that were tested immediately after removal from the storage water using the centerpoint loading method in line with ASTM C348 [22] at 7 and 28-day curing age.

2.4.3. Setting time evaluation

The setting time of the geopolymer mixtures was determined using both the Vicat needle test, which is an ASTM standard test for the property (ASTM C191) [25], and the ultrasonic pulse velocity (UPV) test. The UPV was used to continuously monitor the geopolymerization/hydration reactions of the mixtures and assess the setting times as done in a related work [26], while the Vicat needle test was used for confirmation of the UPV setting time evaluation. The Vicat needle test was conducted using the Vicat apparatus and geopolymer mortar specimens suited for the Vicat conical mold. The initial setting time was taken as the elapsed time required to achieve a 25-mm penetration of the needle after mixing the geopolymer materials. The final setting time was taken as the total time elapsed until the needle does not

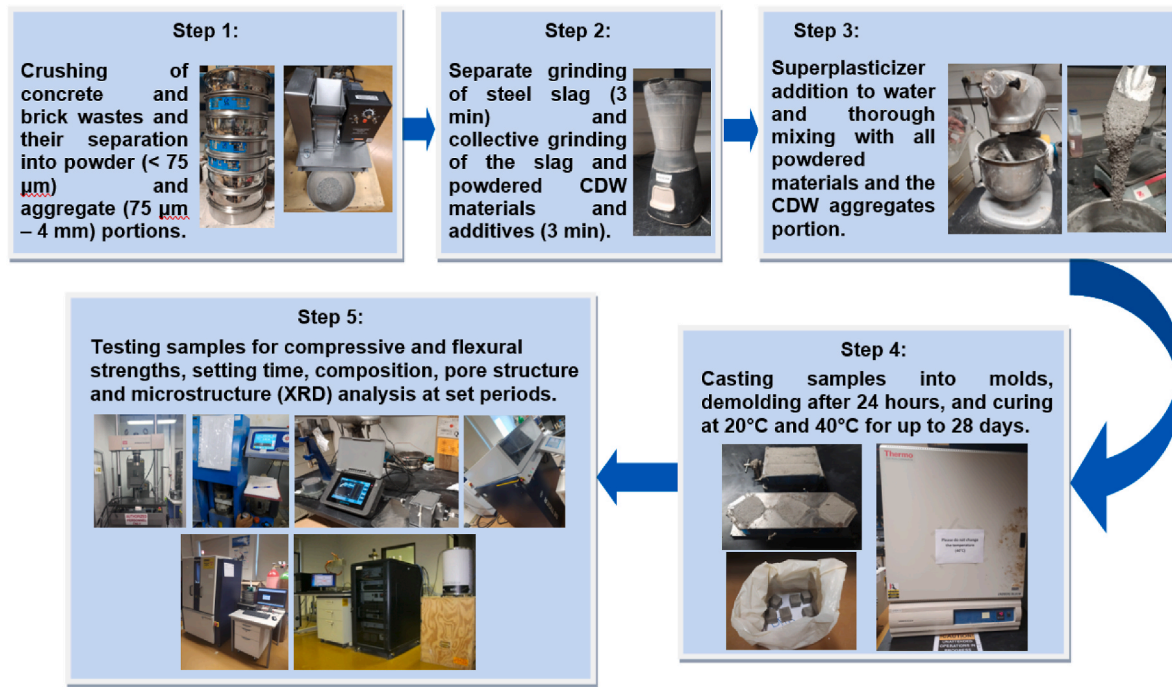


Fig. 3. Flow chart summarizing the experimental methodology.

Table 2

Components of the geopolymer mixtures (percentage by weight).

Geopolymer mortar component		Main mixes				Mixes testing effect of concrete waste granulometry			
		Mix 1: 50 % CDW	Mix 2: 55 % CDW	Mix 3: 60 % CDW	Mix 4: 65 % CDW	Mix 1cp: 50 % CDW	Mix 2cp: 55 % CDW	Mix 3cp: 60 % CDW	Mix 4cp: 65 % CDW
Alkaline activator or additives	CaOH powder	14.0	12.5	13.2	11.5	14.0	12.5	13.2	11.5
	Gypsum	2.2	3.0	–	–	2.2	3.0	–	–
	Silica fume	5.5	4.1	–	–	5.5	4.1	–	–
Waste-based aluminosilicate sources	Steel slag	27.9	25.0	26.4	23.1	27.9	25.0	26.4	23.1
	Red clay solid brick powder	3.0	3.0	3.0	3.0	3.0	3.0	3.0	3.0
	Concrete waste powder	–	–	–	–	3.0	3.0	3.0	3.0
	Concrete aggregate (> 75 μm but < 4 mm)	47.0	47.0	47.0	47.0	44.0	44.0	44.0	44.0
	Red clay solid brick aggregate (> 75 μm but < 1.4 mm)	–	5.0	10.0	15.0	–	5.0	10.0	15.0
Liquids	Superplasticizer	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4
	TOTAL	100	100	100	100	100	100	100	100
	Water/Cementitious materials (powder) ratio	0.36	0.36	0.37	0.36	0.37	0.37	0.37	0.37

Note: Mix 1cp – Mix 4cp are mixes in which concrete waste powder (<75 μm) was included alongside the aggregates.

sink visibly into the paste in accordance with ASTM C191 [25]. A Pundit PL-200 with a 54-kHz transducer/receiver system (Proceq SA, Switzerland) was employed to measure and monitor the evolution of the UPV of the geopolymer specimens in line with BS EN 12504-4 [27]. The fresh geopolymer mortar was poured into the UPV mold and the UPV recorded at 15 s intervals via the transducer and receiver modules, which has direct contact with the mortar specimen. The initial and final setting times were then determined from the inflection points on the UPV curve in line with previous related studies [26,28].

2.4.4. Elemental composition

X-ray fluorescence (XRF) was used to detect elements in the form of oxides in the geopolymer constituents and mixtures. A homogenised sample of the material tested was ground to powder form. The sample was then loaded onto a 40 mm diameter aluminium cup and a powder pellet was prepared using a 20T power press. The powdered samples

were analyzed using an S8 Tiger Series 2 wavelength-dispersive XRF spectrometer (Bruker, Germany).

2.4.5. Mineralogical composition

X-ray diffraction analysis (XRD) is a microstructural analysis method used for the identification of crystallinity of polymers and crystalline phases present. Analysis of the mineralogical composition of the geopolymer constituents and mixtures was conducted using a Rigaku Ultima IV multipurpose X-ray diffractometer (Rigaku Corporation, Tokyo, Japan). The XRD radiation source (Cu-Kα) was operated at 40 kV and 30 mA. XRD data collection was performed using continuous scan mode in the 2θ range of 5–70° with 0.02° step width. The phase analysis and quantification were done via the reference intensity ratio (RIR) method using the Match! software (version 3.15, Crystal Impact, Bonn, Germany). The degree of crystallinity (DOC) of the materials was also outputted by Match! software after subtraction of the instrumental

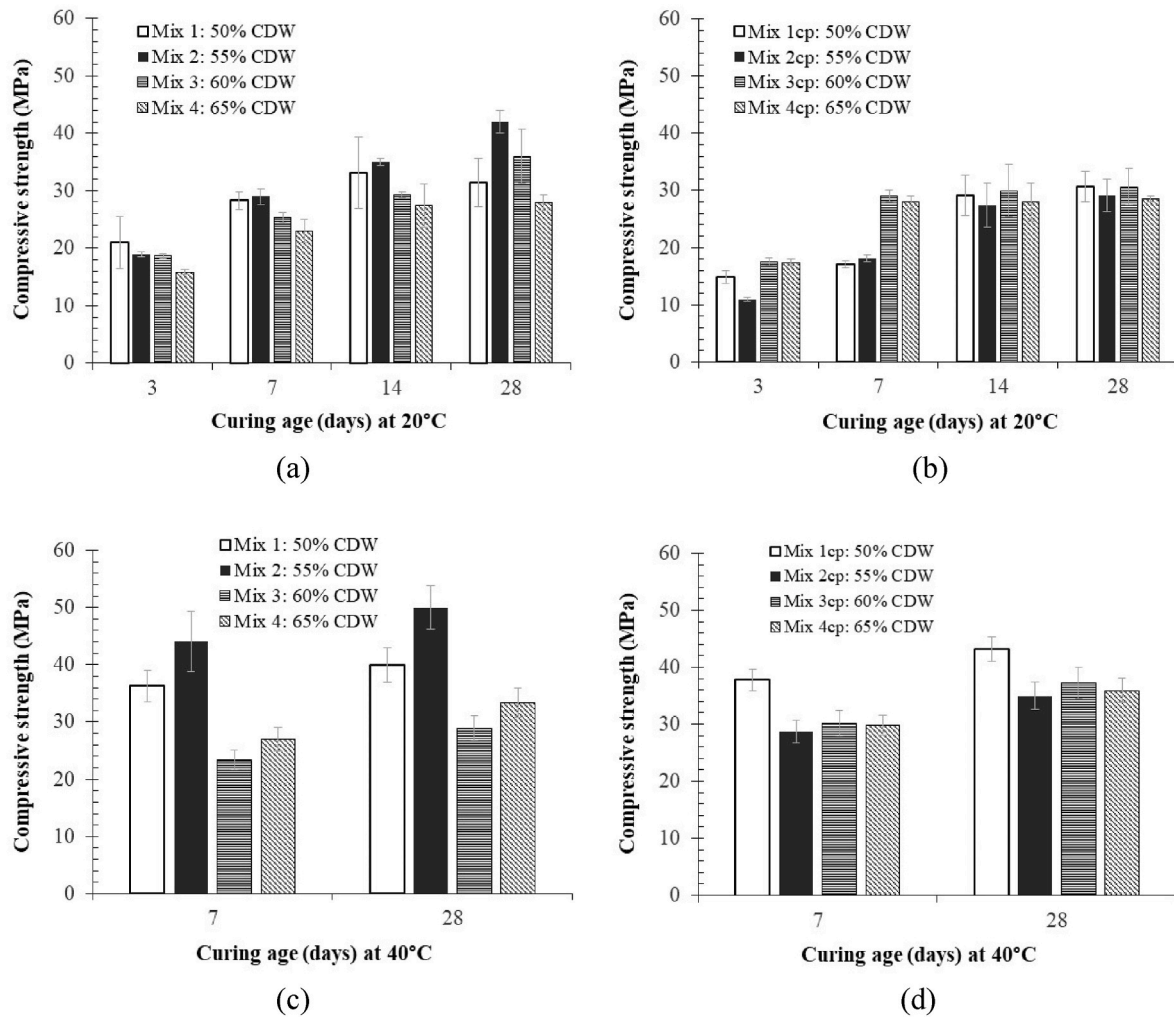


Fig. 4. Compressive strength behavior of the geopolymer mixtures, (a) and (c) main mixes without concrete waste powder, (b) and (d) mixes testing effect of concrete waste granulometry, at ambient and elevated temperatures, respectively.

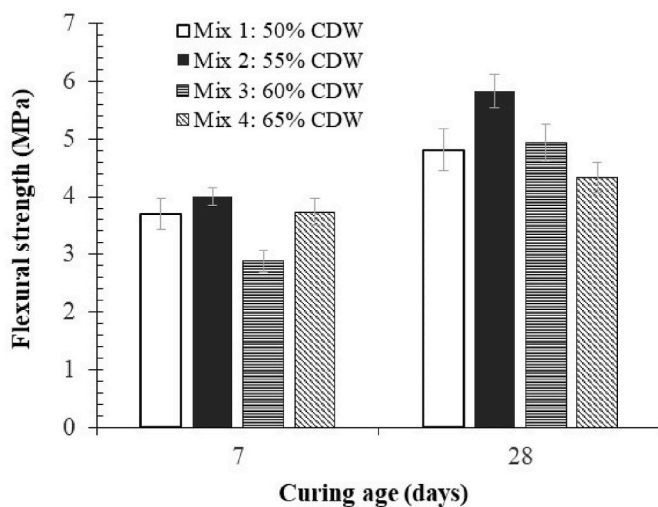


Fig. 5. Flexural strength of the main geopolymer mixtures cured at ambient temperature (20 °C).

background (empty sample holder) from the sample's diffraction pattern.

2.4.6. Porosity and pore size distribution

The porosity and T_2 distribution (a proxy for pore size distribution) of selected geopolymer mixtures were determined by nuclear magnetic resonance (NMR) using a 2 MHz NMR rock core analyser (Magritek, New Zealand). The T_2 relaxation data was determined on a water-saturated cylindrical geopolymer sample. The Carr-Purcell-Meiboom-Gill (CPMG) sequence with 100 ms echo time, an inter-experimental delay time of 6500 ms and 200 scans was employed for the analysis as done for concrete specimens in a previous work [29], which contains the details of the test method. The CPMG decay was analyzed using the Prospa software (Magritek, New Zealand), which outputs the cumulative porosity of the sample, the T_2 distribution and the T_2 log-mean – a proxy for the mean pore size.

2.4.7. Statistics

Simple descriptive statistics such as mean and standard deviation were employed for data analysis. Analysis of variance (ANOVA) was also used to test for significant differences between the different geopolymer mixtures at the 5 % probability level.

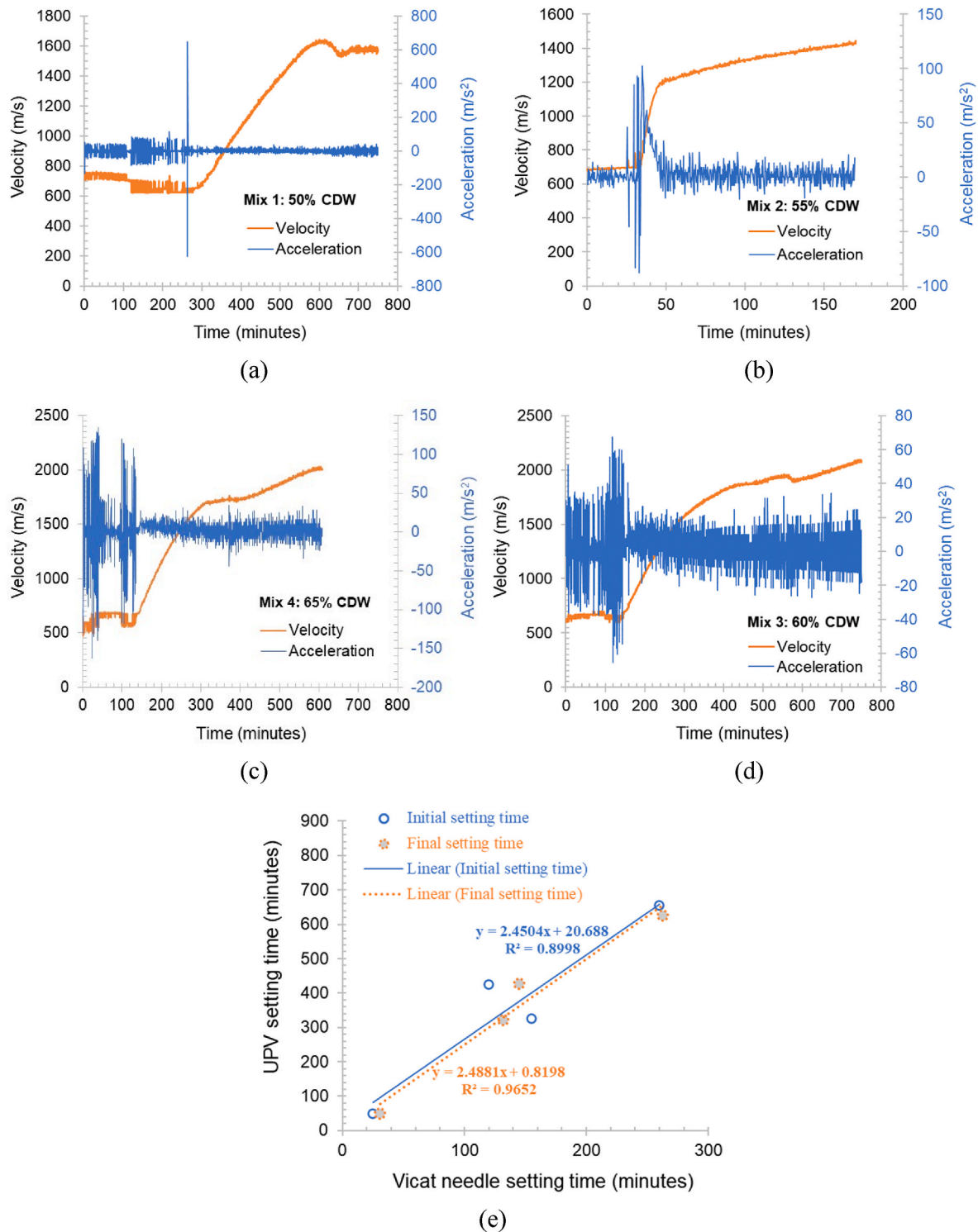


Fig. 6. Pulse velocity and pulse velocity rate (acceleration) evolution for the main geopolymer mixtures cured at ambient temperature, (a)–(d) Mixes 1–4, respectively, and (e) correlation between UPV and Vicat needle setting times.

3. Results and discussion

3.1. General comments

As mentioned previously, separate grinding of the steel slag followed by collective grinding of the powdered materials was employed in preparation of the geopolymer mortars. This was based on compressive strength observations from preliminary investigations with a range of

geopolymer mixtures including a related publication that used nano-materials to develop a fast-setting 3D printable mixture along the lines of this study [23]. The two-step grinding process increased led to an increase of up to 25 % in the LD-PSA-measured specific surface area of both the slag and the combined powders. This in turn reduced the typical water/cementitious materials ratio for mixture flowability after super-plasticizer addition from 0.42 to a maximum of 0.37, which led to 25 % compressive strength increase. This corroborates previous findings that

Table 3

Vicat needle and UPV setting times of the main geopolymers cured at 20 °C.

Test	Main mixes			
	Mix 1: 50 % CDW	Mix 2: 55 % CDW	Mix 3: 60 % CDW	Mix 4: 65 % CDW
Vicat needle initial setting time (mins)	260	25	120	155
Vicat needle final setting time (mins)	655	50	425	325
UPV initial setting time (mins)	263	31	145	132
UPV final setting time (mins)	626	48.5	427	322.5
UPV at the initial setting time (m/s)	642	694	637	585
UPV at the final setting time (m/s)	1612	1191	1852	1707

Note: UPV: Ultrasonic pulse velocity; CDW: Construction and demolition waste.

slag fineness improves hydration activity and collective grinding of cementitious materials improves compressive strength and cementitious activity [30–32].

3.2. Compressive and flexural strength behavior

Fig. 4 shows the compressive strength evolution of the geopolymer mixtures. At ambient curing, the compressive strength of the main mixes without concrete waste powder (Mixes 1–4) initially decreased with increasing CDW content after 3 days. However, at later curing ages, Mix 2 with 55 % CDW content emerged as the optimum with a 28-day compressive strength of 42 MPa (Fig. 4a). Mix 1 showed a slight compressive strength decrease between 14 and 28 days. In contrast, Mixes 1cp – 4 cp with concrete waste powder showed a different behavior as the 60 and 65 % CDW content mixes had significantly higher ($p < 0.001$) 7-day strengths. However, no statistically significant difference ($p = 0.20$) existed between the different mixtures after 28 days (Fig. 4b).

Table 4

X-ray fluorescence (XRF) analysis of the geopolymer constituents and mixtures determined as oxides.

Oxides (%)	Geopolymer Constituent/Mixture									
	Concrete waste	Solid brick waste	Steel slag	Microsilica	Gypsum	Ca(OH) ₂	Mix 1	Mix 2	Mix 3	Mix 4
Al ₂ O ₃	10.70	5.85	14.10	0.56	0.10	0.0273	7.97	9.81	10.80	9.70
As ₂ O ₃	–	0.0019	–	–	–	–	–	–	–	–
BaO	–	–	0.0321	–	–	–	–	–	–	–
CaO	37.10	38.40	50.30	0.52	45.00	98.60	50.90	34.50	57.40	56.60
CeO ₂	–	0.0218	0.0378	–	–	–	–	–	–	–
Cl	0.0382	0.16	–	0.059	0.0174	–	0.0576	0.0396	0.0561	0.0639
Cr ₂ O ₃	0.0402	0.0342	–	–	–	0.0723	0.0078	0.0645	0.0073	0.0100
CuO	0.0097	0.0052	0.0033	0.0071	0.0060	0.0040	0.0035	0.0040	0.0050	0.0049
Fe ₂ O ₃	3.43	4.82	0.51	0.0831	0.0602	0.0147	0.83	1.75	1.12	1.09
Ga ₂ O ₃	–	–	–	0.0021	–	–	–	–	–	–
K ₂ O	0.47	0.54	0.34	0.70	0.0395	–	0.33	0.65	0.28	0.26
MgO	6.75	3.13	5.30	0.40	0.30	0.54	2.54	3.75	3.35	3.18
MnO	0.055	0.0511	0.15	–	–	–	0.0675	0.0617	0.0905	0.0790
MoO ₃	–	–	–	–	–	–	–	0.0020	–	–
Na ₂ O	1.04	0.90	0.22	0.20	–	0.0732	0.30	0.57	0.33	0.28
NiO	0.0161	0.0196	0.0036	0.0032	0.0049	–	0.0071	0.0085	0.0115	0.0083
P ₂ O ₅	0.0369	0.0452	0.0088	0.0856	–	–	0.0305	0.0346	0.0238	0.0375
PbO	–	–	–	0.0448	–	–	0.0045	–	–	–
Rb ₂ O	0.0011	0.0019	–	0.0027	–	–	–	0.0015	–	–
SiO ₂	39.10	44.30	26.90	96.30	0.37	0.14	33.20	45.80	24.70	26.40
SO ₃	0.82	1.41	1.50	1.01	53.90	0.52	3.44	2.68	1.43	1.93
SrO	0.092	0.0514	0.0318	0.0025	0.20	0.0126	0.0531	0.0464	0.0601	0.0615
TiO ₂	0.25	0.22	0.54	–	–	–	0.24	0.20	0.30	0.26
V ₂ O ₅	0.0219	0.0176	–	–	–	–	–	–	–	–
WO ₃	0.0116	–	–	–	–	–	–	–	–	–
ZnO	0.0048	0.0049	–	0.0165	–	–	0.0092	0.0069	0.0085	0.0132
ZrO ₂	0.0088	0.0111	0.0218	0.0011	–	–	0.0129	0.0194	0.0222	0.0228

Note: The standard deviations between replicates were on average within 15 % of the mean values. Mixes 1–4 were analyzed only after 28-day ambient temperature curing.

The strength behavior trend at 40 °C curing was fairly similar to that at ambient temperature for mixes without concrete waste powder as the 55 % CDW optimum mix had the maximum 28-day compressive strength of 50 MPa (Fig. 4c). However, mixes with concrete waste powder showed a different trend. The mix with the lowest CDW content had the highest 28-day strength (43 MPa), while the strength of the other three mixes ranged from 35 to 37 MPa (Fig. 4d) in contrast to 29–31 MPa strength at ambient curing. This highlights the relevance of elevated temperature curing if concrete waste is to be utilized in powder form. Generally, increase in curing temperature led to strength improvement, especially in the mixes incorporating concrete waste powder (compare Fig. 4c and d). The above strength values compares favorably with conventionally activated geopolymer mortars and concrete in previous studies [33–35]. The results corroborate the position that concrete waste powder can have detrimental impact on geopolymer mortar strength, especially considering the ambient-cured samples [17]. It also shows that the presence of a small amount of concrete waste powder in a one-part geopolymer could lead to better compressive strength at elevated temperatures than its absence.

Fig. 5 shows the flexural strength behavior at 7- and 28-day curing age. It was determined on only the main mixtures without concrete waste powder at ambient temperature. The flexural strengths increased in all mixtures between 7 and 28 days and showed a similar trend to the compressive strength. Mix 2 with 55 % CDW content had the highest 28-day flexural strength of 5.8 MPa while Mixes 1 and 3 with 50 and 60 % CDW had similar flexural strengths of 4.8 and 4.9 MPa, respectively, akin to their corresponding compressive strength behavior. Mix 4 with 65 % CDW content had 4.3 MPa flexural strength (Fig. 5). These values are higher than typical 28-day values (4 MPa) acceptable for construction applications and are similar to those obtained for NaOH/Na₂SiO₃-activated geopolymers [36,37].

3.3. Setting time from UPV and Vicat needle tests

The UPV and Vicat needle tests were conducted on only the main

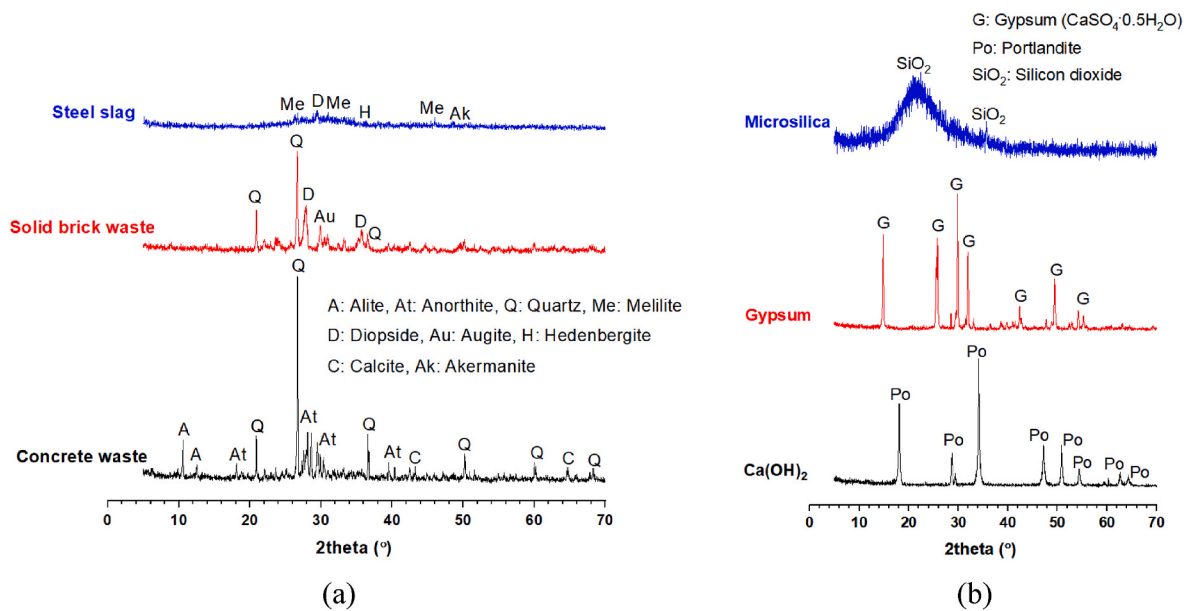


Fig. 7. XRD diffractograms showing the major mineral phases in the (a) waste-based aluminosilicate sources, and (b) alkaline activator or additives.

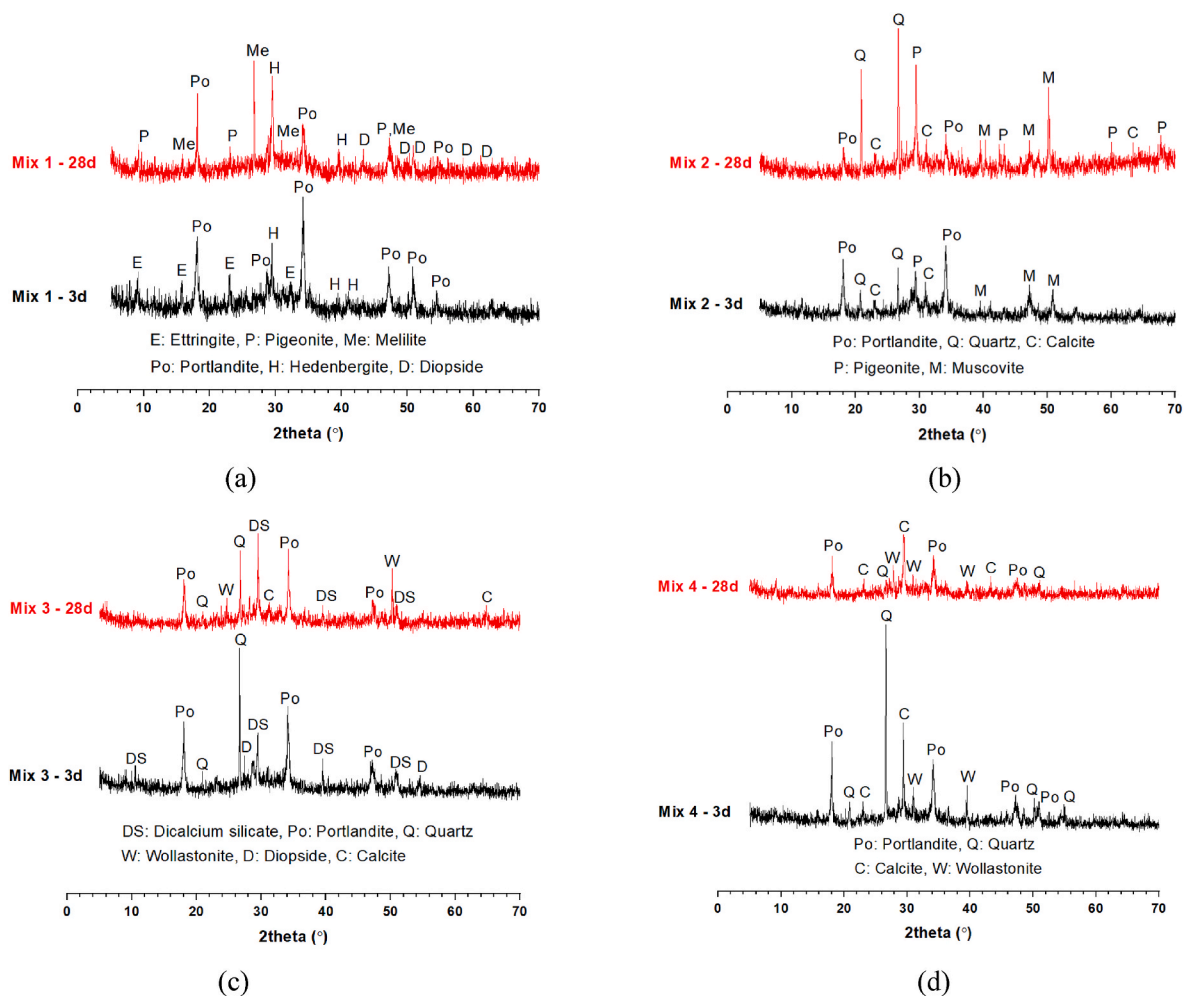


Fig. 8. XRD diffractograms and identification of the major minerals in the main geopolymer mixtures after 3- and 28-days curing at ambient temperature, (a)–(d) Mixes 1–4, respectively.

Table 5

X-ray diffraction (XRD) quantification of the main minerals in the geopolymer constituents and mixtures by Match! software.

Mineral	Percent (%) by weight of geopolymer constituent/mixture													
	Concrete waste	Solid brick waste	Steel slag	Microsilica	Gypsum	Ca (OH) ₂	Mix 1: 50 % CDW		Mix 2: 55 % CDW		Mix 3: 60 % CDW		Mix 4: 65 % CDW	
							3d	28d	3d	28d	3d	28d	3d	28d
Akermanite [Ca ₂ Mg(Si ₂ O ₇)]	–	–	10.0	–	–	–	–	–	–	–	–	–	–	–
Alite (Ca ₃ O ₅ Si)	7.8	–	–	–	–	–	–	–	–	–	–	–	–	–
Anorthite (CaAl ₂ Si ₂ O ₈)	48.0	–	–	–	–	–	–	–	–	–	–	–	–	–
Augite (Al _{0.7} CaFe _{0.2} Mg _{0.6} O ₆ Si _{1.5})	–	12.9	–	–	–	–	–	–	–	–	–	–	–	–
Calcite (CaCO ₃)	6.5	–	–	–	–	–	–	–	18.9	16.6	–	10.6	22.9	51.1
Dicalcium silicate (Ca ₂ SiO ₄)	–	–	–	–	–	–	–	–	–	–	28.7	22.2	–	–
Diopside (MgCaSi ₂ O ₆)	–	28.9	27.4	–	–	–	–	21.2	–	–	20.4	–	–	–
Ettringite [Ca ₆ Al ₂ (SO ₄) ₃ (OH) ₁₂ ·26H ₂ O]	–	–	–	–	–	–	30.1	–	–	–	–	–	–	–
Gypsum hemihydrate (CaSO ₃ ·0.5H ₂ O)	–	–	–	–	100	–	–	–	–	–	–	–	–	–
Hedenbergite (CaFeSi ₂ O ₆)	–	–	11.1	–	–	–	30.9	11.1	–	–	–	–	–	–
Mellilite (Ca _{5.95} Na _{2.05} O ₁₅ Si ₄)	–	–	51.5	–	–	–	–	38.0	–	–	–	–	–	–
Muscovite (Al ₃ H ₂ KO ₁₂ Si ₃)	–	–	–	–	–	–	–	–	29.1	16.9	–	–	–	–
Pigeonite (Ca _{0.107} , Fe _{0.417} Mg _{0.462} Mn _{0.014} O ₃ Si)	–	–	–	–	–	–	–	18.2	14.2	20.4	–	–	–	–
Portlandite [Ca(OH) ₂]	–	–	–	–	–	100	39.0	11.5	27.9	6.2	36	20.2	16.6	17.9
Quartz (SiO ₂)	37.7	58.2	–	100 (s)	–	–	–	–	9.9	39.9	14.9	15	51.9	20.2
Wollastonite (CaSiO ₃)	–	–	–	–	–	–	–	–	–	–	–	32.0	8.6	10.8
TOTAL	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Degree of crystallinity (DOC) (%)	52.8	40.1	32.2	36.0	64.9	68.8	42.3	44.2	49.9	38.0	44.5	38.3	45.6	40.0
Percentage decrease in DOC (%)	–	–	–	–	–	–	–4.5	–	23.8	–	13.9	–	12.3	–

Note: The (s) for microsilica indicates that the peaks were specifically identified as silica (SiO₂) rather than quartz. As shown by the degree of crystallinity values, all of the geopolymer constituents/mixtures had different amounts of amorphous materials, not identifiable by XRD. Hence, the weight percentages in this table only shows the crystalline portions of the sample.

mixes cured at ambient temperature without concrete waste powder inclusion. The UPV pulse velocity and pulse velocity rate evolution are shown in Fig. 6a–d. The pulse velocity rate is the first derivative of the UPV curve (i.e., acceleration) and is meant to better highlight inflection points on the curve. The derivative at a point is calculated by taking the average of four slopes at the point's immediate vicinity [38]. The initial and final setting times derived from the UPV curves are shown in Table 3 together with the Vicat needle setting times. The initial setting time was taken as the first turning point on the UPV curve, which corresponded with the end of the dormant stage, where there is a rapid increase in the pulse velocity as observed in previous studies [28,39]. The dormant stage involves dissolution of the source materials, hydrolysis of silicate and aluminate oligomers or monomers in the solution, and their complexation with calcium [28,38]. The UPV initial setting times of the four mixes coincided with points where the acceleration curve showed both local minimum and maximum values (Fig. 6a–d). The final setting time was chosen as the time taken for the curve to reach a plateau (Fig. 6a–d and Table 3). There were very strong correlations between the chosen initial ($R^2 = 0.90$) and final ($R^2 = 0.97$) setting times of the UPV and the Vicat needle test (Fig. 6e). There was no clear trend in the acceleration curves of all four mixes as observed in a previous study [38]. Hence, the initial or final setting times cannot be associated with the maximum acceleration point for the mixtures studied.

The pulse velocity values of the mixes ranged from 585 to 694 m/s for the initial setting time and 1191–1852 m/s for the final setting time (Table 3). These values were similar to those of some slag-fly ash geopolymer mixes in a previous work [39]. They, however, differ from those of Tekle et al. [38], thus corroborating their position that the use of certain (or range of) pulse velocity values to estimate setting times is impracticable. With both the UPV and Vicat needle tests, the optimum 55 % CDW mix for strength with 5 % brick aggregate content showed the lowest initial and final setting times. It was followed by Mixes 3 and 4 with higher (10 and 15 %) brick aggregate content (Table 3). Mix 1 without brick aggregate content but with 3 % brick powder content like the other mixes had the longest setting times. This corroborates the position that waste bricks are more reactive during geopolymerization

than waste concrete [17]. The optimum mix (Mix 2) probably had better strength and faster setting as it also included small amounts of gypsum and microsilica unlike Mixes 3 and 4, which could lead to enhanced geopolymerization or hydration [40,41]. The recorded setting times are within ranges reported for geopolymers in previous studies [9,34].

3.4. Elemental and mineralogical composition

Table 4 shows the XRF analysis data, while Figs. 7 and 8, and Table 5 show the XRD diffractograms and crystalline phases present in the geopolymer constituents and main mixtures cured at ambient temperature. The XRF analysis show that the main elements (Si, Al, Ca) involved in the geopolymerization/hydration process are present in the concrete waste in significant amounts with values close to those in the steel slag and brick waste (Table 4). Hence, it could be used as the majority ingredient to produce high-strength geopolymer mortars. The XRF analysis of the geopolymer mixtures show how slight changes in mix proportions could lead to large differences in elemental composition. The majority of the mixes were composed of over 50 % CaO. However, the optimum mix (Mix 2) had lesser CaO (35 %) and more SiO₂ (46 %) content (see Table 4). Nevertheless, there is no direct relationship between the elemental composition and mechanical behavior of the mixes.

The XRD analyses shows that the main mineral phases in the concrete waste are anorthite - a calcium aluminosilicate (48 %), and quartz (38 %), with alite and calcite as minor phases. Quartz and mellilite are the predominant mineral phases comprising over 50 % of the brick waste and steel slag, respectively (Fig. 7 and Table 5). The XRD diffractograms of the geopolymer mixtures show a broadened amorphous hump or dome within the 20–40° 2θ range (Fig. 8). This suggests the formation of amorphous geopolymer gels of mixed composition (for e.g., amorphous aluminosilicate gel and calcium silicate gel) from the geopolymerization of the steel slag and the CDW materials after reaction with Ca(OH)₂, which is typical of calcium-containing geopolymer systems [42–44]. Specifically, alkali-activation of slag facilitates the creation of the calcium aluminum silicate hydrate (C-A-S-H) gel after dissolution of Si–O–Si, Al–O–Si, Ca–O, and Al–O–Al bonds followed by their

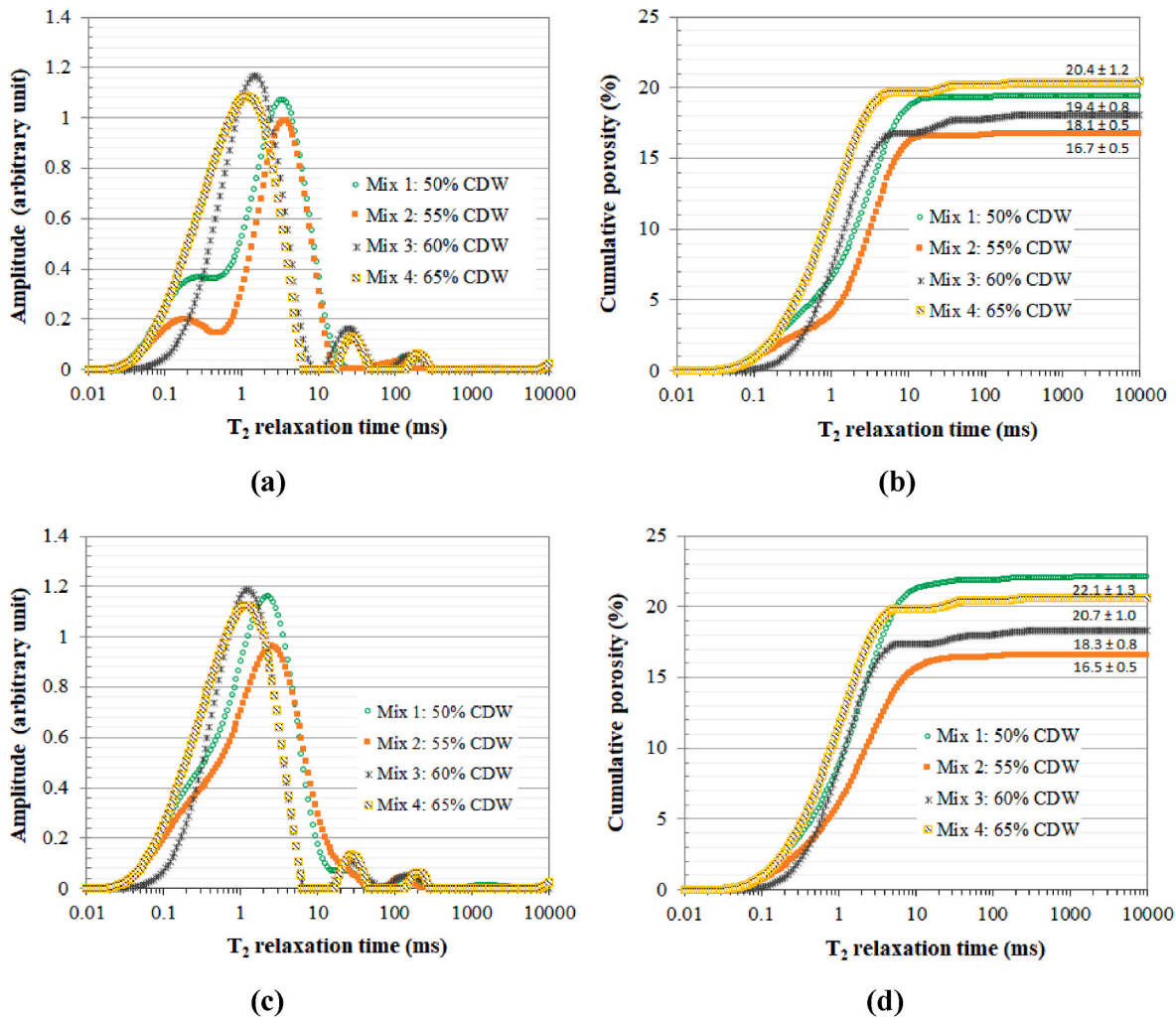


Fig. 9. NMR T_2 (proxy for pore size) distribution of the main geopolymer mixtures at (a) and (c) 7- and 28-day curing age, and cumulative porosity at (b) and (d) 7- and 28-day curing age, respectively, at ambient temperature (20 °C).

polycondensation [45].

The mixes also showed well defined peaks on the amorphous humps. The major crystalline phase present in all mixes is portlandite due to the alkaline activator used. Quartz was also present, except in Mix 1, which had the lowest (3 %) brick waste content. Notably, Mix 1 showed a high amount (38 %) of the main phase in the constituent slag, mullite, after 28 days, and had ettringite at early curing age. Its degree of crystallinity (DOC) increased by 4.5 % between 3 and 28 days, in contrast to 12–24 % decreases in the other mixes (Table 5), suggesting inhibited geopolymerization. Geopolymer formation is usually associated with decreasing DOC [46], and the best mixes in this work (Mixes 2 and 3) showed the largest DOC decrease. There were also a variety of other mineral phases in the mixes, differing from one mix to another as shown in Fig. 8 and Table 5. Generally, the crystalline minerals observed in the raw materials were transformed into different crystals. This is expected as the minerals are used in the dissolution and reorganization steps of geopolymerization reactions [47].

3.5. NMR pore structure evaluation

The NMR T_2 distributions and cumulative porosity of the main geopolymer mixtures cured at ambient and elevated temperatures are shown in Figs. 9 and 10, respectively. The T_2 log-mean of all samples, is shown in Fig. 10c. The NMR T_2 distribution is used as a proxy, for pore size distribution, and the T_2 log-mean for mean pore size without

conversion to actual pore sizes. This is because paramagnetic species such as Fe^{3+} , which is present in the mixtures as confirmed by the XRF analysis, affects relaxation times and the surface relaxivity constant required for conversion to actual pore sizes leading to inaccuracies [29]. In the T_2 distributions, the pore sizes are directly proportional to the relaxation times. The peaks represent pores of different sizes, while the amplitude relates to pore abundance. The NMR can measure micropores, mesopores and macropores with T_2 relaxation times less than 30 ms separating micropores and mesopores and relaxation times greater than 300 ms corresponding to macropores [48], although there is no unified standard for these boundary conditions.

The NMR cumulative porosities of the specimens support the higher compressive and flexural strength and setting time performance of the optimum mix (Mix 2 with 55 % CDW content). The mix had the lowest porosity (around 16.5 %) as well as the least pore abundance after 28 days at both ambient and elevated temperatures (Figs. 9 and 10). Further, Mixes 2 and 3 with 55 and 60 % CDW contents, respectively, which had the best 28-day compressive and flexural strengths, showed no significant change in the cumulative porosity between 7 and 28 days unlike Mix 1 (see Fig. 9). It is suspected that the slight porosity increase in Mix 1 may probably account for the previously mentioned slight strength decrease between 14 and 28 days. Abdulkareem et al. [49] observed a similar porosity increase leading to compressive strength reduction in biomass wood-ash-fly-ash-based geopolymer mortar with higher biomass wood ash content. The strength reduction was attributed

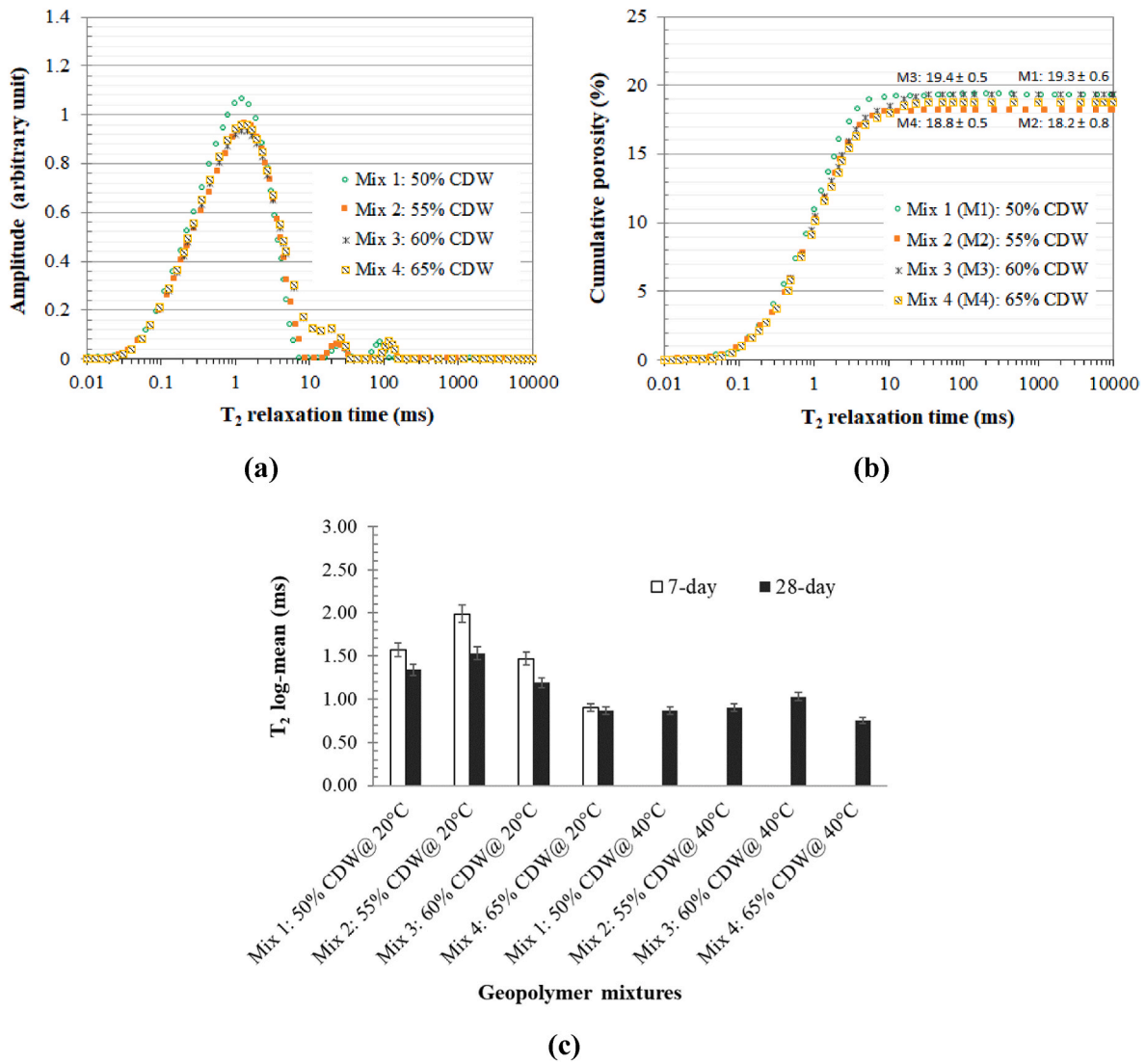


Fig. 10. NMR (a) T₂ (proxy for pore size) distribution and (b) cumulative porosity of the main geopolymer mixtures cured at 40 °C at 28-day curing age, and (c) T₂ log-mean (proxy for mean pore size) of the 7- and 28-day old geopolymer samples cured at 20 and 40 °C, as applicable.

to deficiency of dissolved Si and Al species in the reaction due to fly ash dilution. Thus, the low amount of brick waste in Mix 1 compared to the other mixtures that did not show significant porosity increase and strength reduction may lead to deficiency of dissolved Si and Al species and inhibit geopolymerization as observed in the XRD DOC measurements. Besides, the XRD mineralogical data show that the brick waste was richer in quartz content than the concrete waste, and quartz or silica content positively influences the mechanical performance of geopolymers [50].

Comparison of the ambient and elevated temperature data for 28-day old specimens shows that elevated temperature curing led to decrease in the micropores (first peaks in Figs. 9c and 10a) in all mixtures. It also led to lower T₂ log-mean values (mean pore sizes) as the pore size distribution slightly shifted towards smaller pores. Since the micropores dominate the pore sizes, this led to the higher compressive strengths observed with elevated temperature curing. This corroborates the position that the porosity decreases with increase in curing temperature up to a certain extent [51]. The decrease in the micropores (consisting of gel and capillary pores) with elevated temperature curing support the position that curing at high temperatures and mixing with a low W/C reduces the gel pore volume and increase the density of gel products like CSH [52].

4. Conclusions and future work

The innovation and significance of this work consists in replacing conventional highly alkaline activator solutions with a solid environmentally friendly alkaline activator, Ca(OH)₂ powder, to produce high strength ambient-cured one-part geopolymer mortars from CDW with predominantly concrete waste blended with steel slag that can be utilized for 3D concrete printing. Four geopolymer mixtures with CDW contents of 50, 55, 60 and 65 % that consist predominantly of concrete waste (47 %) with brick waste content varied from 3 to 18 %, and roughly 25 % slag content were investigated. The following are the major conclusions from the study.

1. Mix 2 (with 55 % CDW content) exhibited optimum performance across all parameters studied such as compressive and flexural strengths, setting time, as well as changes in NMR-determined pore structure (porosity and mean pore size) and XRD-determined degree of crystallinity (DOC) over time. It had 28-day compressive and flexural strengths of 42 and 5.8 MPa, respectively, and initial and final setting times of 25 and 50 min with ambient temperature (20 °C) curing.

- Mix 3 (with 60 % CDW content) also met the 35 MPa target compressive strength at ambient curing, and had 5 MPa flexural strength but much longer (>120 min) setting times. Mixes 1 and 4 (with 50 and 65 % CDW contents) did not meet the target strength.
- The superior mechanical performance of Mixes 2 and 3 at ambient curing was supported by a higher (24 and 14 %, respectively) decrease in the XRD-determined DOC over the 28-day period, thus reflecting better geopolymerization levels.
- Inclusion of powdered concrete waste in the geopolymer mixtures in contrast to its use as an aggregate reduced strength under ambient curing but led to better performance at 40 °C curing, with 28-day compressive strengths of all four mixtures ranging from 35 to 43 MPa.
- There was a strong correlation ($R^2 \geq 0.90$) between the initial and final setting times of the geopolymer mixtures determined in the Vicat needle and UPV measurements.
- NMR and XRD DOC measurements show that porosity increase resulting from inhibited geopolymerization could cause slight strength reduction over time as in Mix 1 due to insufficient brick waste content in the geopolymer mixture.

It is concluded that sustainable structural mortars can be produced by 'just adding water' to an optimized CDW mixture with predominantly concrete waste blended with brick waste and slag and activated by powdered $\text{Ca}(\text{OH})_2$. Future studies may consider establishing quantitative relationship models and conducting predictive analysis between mix design variables and performance parameters. Such studies may also consider research on the application of CDW in concrete, investigating strength, deformation, constitutive behavior, and structural performance to fully reflect its engineering application value. In addition, several additives to improve upon relevant performance properties for the mixtures developed, and structural reinforcement mechanisms to make them more amenable for 3D printing will also be investigated.

CRedit authorship contribution statement

Reginald B. Kogbara: Conceptualization, Formal analysis, Investigation, Methodology, Writing – original draft. **Abdelrahman Al-Zubi:** Formal analysis, Investigation, Methodology, Writing – review & editing. **Youssef Mortada:** Formal analysis, Investigation, Methodology, Writing – original draft. **Ahmad Hammoud:** Formal analysis, Investigation, Methodology, Writing – review & editing. **Eyad A. Masad:** Conceptualization, Project administration, Supervision, Writing – review & editing. **Marwan K. Khraisheh:** Conceptualization, Supervision, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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