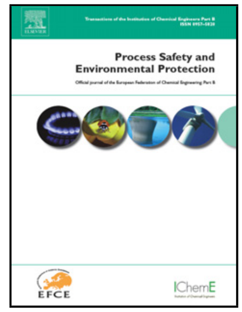

Title: Effect of the particle size range of construction and demolition waste on the fresh and hardened-state properties of fly ash-based geopolymer mortars with total replacement of sand

Authors: A. De Rossi, M.J. Ribeiro, J.A. Labrincha, R.M. Novais, D. Hotza, R.F.P.M. Moreira

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Effect of the particle size range of construction and demolition waste on the fresh and hardened-state properties of fly ash-based geopolymer mortars with total replacement of sand

A. DE ROSSI (1), M. J. RIBEIRO (2), J. A. LABRINCHA (3), R. M. NOVAIS (3), D. HOTZA (1), R. F. P. M. MOREIRA (1)*

(1) Department of Chemical Engineering (EQA), Federal University of Santa Catarina (UFSC), 88040-900 Florianópolis, Brazil

(2) Materials Science Research and Development Center (UIDM), Polytechnic Institute of Viana do Castelo, 4900-348 Viana do Castelo, Portugal

(3) Department of Materials and Ceramic Engineering, University of Aveiro, 3810-193 Aveiro, Portugal

*Corresponding author: Regina de Fatima Peralta Muniz Moreira

E-mail: regina.moreira@ufsc.br

Telefone: +55 48 3721 7238

Highlights

- The use of CDW as fine aggregate enhances the mortars' mechanical properties.
- Recycled fine aggregate completely replaces sand to produce geopolymer mortars.
- Geopolymers can be produced using 75 wt.% solid residues;
- CDW does not act as binder in the geopolymerization process.

Abstract

This study seeks the valorization of industrial residues (fly ash and construction and demolition waste (CDW)) through the production of geopolymer mortars. The effect of the sand substitution by CDW and the influence of the particle size range of CDW fine aggregates on the fresh and hardened properties of the mortars were evaluated. Geopolymer mortars were produced using biomass fly ash waste and metakaolin as a binder, CDW as fine aggregates, and an alkali solution of sodium silicate and sodium hydroxide as activator. The geopolymer mortars were characterized in fresh state by the flow table test and in the hardened state through chemical, physical/microstructural analyzes. The mortars produced with CDW showed lower flowability when compared to the ones prepared with sand. The compressive and flexural strength of hardened mortars, respectively, obtained with residues were higher when compared to sand: 40 MPa and 8.5 MPa with CDW, against 23 MPa and 3.1 MPa for sand-based samples. It was observed that mortars developed with recycled aggregate and natural aggregate present similar chemical and mineralogical compositions. The superior results obtained in the mechanical properties of mortars produced with CDW are related to the recycled aggregate-geopolymer paste interface.

Keywords: CDW; fine aggregates; geopolymer mortars.

1. Introduction

The European Commission has identified construction and demolition waste (CDW) as a priority stream for reuse, highlighting the environmental benefits of its recovery (Directive 2008/98/EC, 2008; Pacheco-Torgal et al., 2012). It is estimated that the construction industry generates about 860 million tons of waste per year in the European Union, representing 34% of all waste produced (Eutostat, 2014). In some

European countries, such as Denmark, Germany, Ireland, Netherlands and Estonia, the CDW recycling rates reach over 80% (Villoria Saez et al., 2011). In developing countries, such as Brazil, only 21% of CDW is recycled (Miranda et al., 2016). Processed CDW has been increasingly recycled as aggregate in the production of new construction products, including concrete (Raeis Samiei et al., 2015).

Alternatively, CDW has been used to produce geopolymers (Komnitsas et al., 2015; Zaharaki et al., 2016). These novel binders may present properties comparable to those of Portland cement, but with a much lower CO_2 footprint (Davidovits, 1994; Provis and Bernal, 2014). A rigid control of the particle size and chemical composition of silicates and aluminates sources is required to obtain the suitable characteristics. Usual raw materials employed in geopolymerization are fly ash, blast furnace slag, and metakaolin (calcined clay)(Khale and Chaudhary, 2007). Some studies have demonstrated that metakaolin could be completely/partially replaced by biomass fly ash (Novais et al., 2018) or powdered CDW (Komnitsas et al., 2015; Vásquez et al., 2016; Zaharaki et al., 2016).

CDW can be also used as fine aggregates to produce mortars, which is a more attractive application, since the particle size required in this case is coarser than that to corresponding to geopolymer binders. However, the mechanical properties of the mortars produced from CDW, such as flexural strength and compressive strength, decrease as the amount of recycled aggregates in the mortar increases (Silva et al., 2016).

Research targeting the use of CDW as aggregate in geopolymers is scarce (Cristelo et al., 2018; Mohammadinia et al., 2016). Nevertheless, fine sized CDW (d_{50} = 0,39 mm) was considered an adequate aggregate to be used in alkali activated fly ash, since no effect on the strength value during or after activation reactions was observed. CDW might act as filler but the finer fraction might even react with the activator (Cristelo et al., 2018), partially acting as binder in the geopolymerization process. Nevertheless, the influence of the particle size on the characteristics of geopolymer mortars was not fully evaluated, being this the main objective of the current work. We observed that is possible to produce geopolymer mortars with enhanced mechanical strength by adjusting the CDW particle size, used as alternative aggregate to natural sand.

2. Experimental

2.1. Materials

Geopolymer mortars were prepared using a mixture of 75 wt.% biomass fly ash (FA) and 25 wt.% metakaolin (MK) (Argical M 1200 S, Univar) as a source of aluminosilicates (De Rossi et al., 2018, 2017). FA was collected from burned Eucalyptus biomass in the bubbling fluidized combustor of a paper and pulp plant in Portugal.

CDW was collected from a demolition site of a concrete-based construction and classified as 17.01.01 (concrete) and 17.01.02 (bricks) according to the European List of Wastes (EU, 2014). Before drying at 110 °C for 24 h and crushing, wood residues, plastic and metals were separated from the CDW. A double comminution process was then carried out: jaw crushing (BB2 Retsch) followed by hammer milling (5657 Retsch), to obtain particles ≤ 4 mm in diameter. The CDW samples used in this work were sieved in two fractions: 0.5-1.0 mm, 1.0-2.0 mm; one third fraction (0.5-2.0 mm) was obtained by mixing (1:1 wt.%) the two sieved fractions. The particle size distribution is shown in Figure 1. Normalized sand used as fine aggregate was commercially supplied (Mibal, Barqueiros, Portugal) with similar particles size distribution of the prepared CDW.

A mixture of sodium hydroxide (SH, 97 wt.% purity, Sigma Aldrich) and sodium silicate (SS, 9.13 wt.% Na_2O, 28.77 wt.% SiO_2, 62.1 wt.% H_2O; Quimiamel) were used (weight ratio SS:SH = 1.5) as activator. The NaOH solution (10 M) was prepared by dissolving sodium hydroxide beads in distilled water.

2.2. Mortar preparation and flow characterization

The geopolymer mortars were prepared using binder: aggregate weight ratio = 1:3, the mix design of samples prepared in this study is presented in Table 1. The binder (FA and MK), aggregates (CDW or sand) and alkaline activators (SS and SH) were added to the mixer, following the steps: i) initial homogenization of sodium silicate and NaOH solution at 60 rpm for 5 min; ii) mixture at the same speed of the alkaline solution with solids materials for 10 min, and iii) homogenization and mixture for another 5 min at 95 rpm.

The mixture was maintained under agitation until the materials were completely impregnated. The alkaline solution was the only liquid component in all mixtures (no water addition).

The effect of the replacement of natural by recycled aggregate and of different particle sizes, 0.5-1.0 mm, 1.0-2.0 mm and 0.5-2.0 mm, on the rheological behavior of mortars was evaluated. Flow measurements of fresh mortars were performed according to ASTM C 1437 (2007) and the results were expressed as spread diameter.

Table 1. Mix design of geopolymer mortars with CDW or sand (wt.%).

Mortar	CDW	Sand
Binder	20	20
Alkaline activator	20	20

Aggregate	60	60

2.3. Chemical, physical and mechanical characterization

CDW 0.5-1.0 mm and 1.0-2.0 mm fractions were visually selected in brick and concrete particles for characterization. The mineralogical compositions of MK, FA and CDW were evaluated by X-ray diffraction (XRD, D8 Advance Bruker) using Cu K α radiation, in the range of 5-80°, with a 0.02° step-scan and 10 s/step. Phase identification was carried out through the use of EVA Bruker software.

The chemical compositions of raw materials, including CDW 0.5-1.0 and 1.0-2.0 mm fractions, were obtained by X-ray fluorescence (XRF, X'Pert PRO MPD Philips), while the loss on ignition (LOI) at 1000 °C was also determined. The CDW aggregates and alkali-activated pastes were characterized by scanning electron microscopy (SEM, SU1510 Hitachi) equipped with energy dispersion spectroscopy (EDS, Bruker).

Compressive and flexural strength measurements were performed according to EN 1015-11 (1999) from samples obtained by filling prismatic molds with the mortar, after compaction by manual vibration. The prismatic samples were removed from the molds after 24 h and subsequently cured in room conditions (20 °C and 68 % relative humidity) for 28 days. A universal testing machine (Lloyd, LR 30 K) was used, running at a displacement rate of 0.5 mm/min. Three prismatic samples (40 x 40 x 160 mm^3) were tested for the flexural strength, resulting into two broken parts, which, according to the standard procedure, were then used five cubic samples (40 x 40 x 40 mm^3) for the compressive strength determinations (Raeis Samiei et al., 2015). Average and standard deviation data is reported.

Physical characteristics of the hardened mortars, such as water absorption, bulk density and porosity, were estimated, according to the Archimedes principle using water as immersion fluid. The bulk density of the aggregates, CDW and sand, was measured according to ASTM C29/C29M, (2009). The thermal conductivity was measured according to ASTM C518 (2004) on three samples of each formulation using cubic specimens (40 × 40 ×40 mm^3).

3. Results and discussion

3.1. Chemical and phase composition of raw materials

Materials used as sources of aluminosilicates (FA and MK) and as fine aggregates (CDW and sand) have the chemical compositions shown in Table 2. As expected, in sand the dominant oxide was SiO_2, and in MK were SiO_2 and Al_2O_3. In the FA and CDW, the main components were SiO_2, Al_2O_3 and CaO. The SiO_2/Al_2O_3 molar ratio of binder was 4.56. The composition of CDW is variable, as expected, but falls reasonably within the weight ranges referred in the literature: 40-70 wt.% SiO_2, 5-20 wt.% Al_2O_3, 10-25 wt.% CaO, and 0.5-8 wt.% Fe_2O_3 (Contreras et al., 2016; Saiz Martínez et al., 2016; Vásquez et al., 2016).

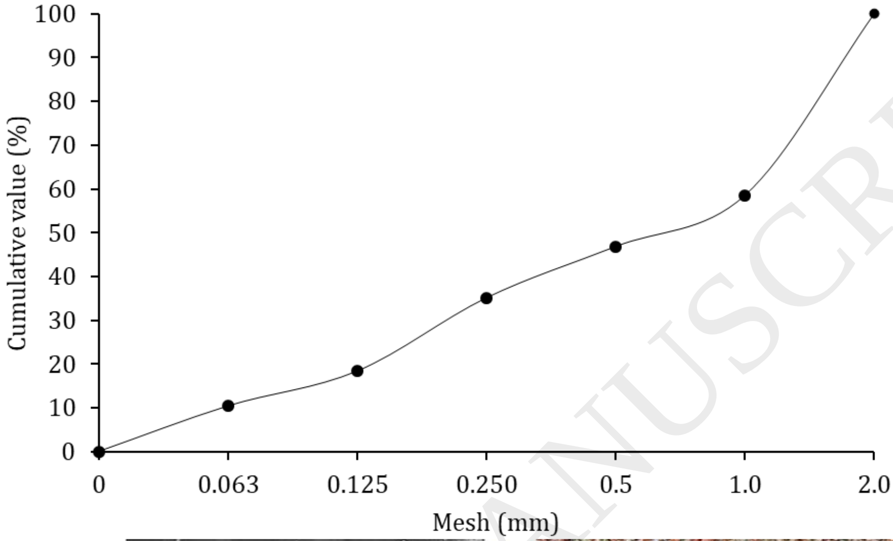
Table 2. Chemical composition (wt.%) of FA, MK, sand and different fractions of CDW.

Oxides		SiO_2	Al_2O_3	MgO	CaO	Na_2O	K_2O	Fe_2O_3	TiO_2	SO_3	LOI
FA		34.00	13.50	3.10	16.50	1.50	5.50	5.00	0.60	2.80	14.30
MK		54.40	39.40	0.14	0.10	-	1.03	1.75	1.55	-	2.66
Sand		98.90	0.10	0.11	0.22	0.15	0.35	0.02	0.06	0.09	-
CDW	0.5-1.0 mm	70.51	8.01	1.11	9.62	0.16	1.80	2.27	0.32	0.41	5.48
	1.0-2.0 mm	69.48	8.67	1.19	8.73	0.16	1.84	2.54	0.34	0.38	6.44
	brick	58.53	19.73	2.44	3.29	0.28	4.16	6.45	0.78	0.11	3.87
	concrete 0.5-1.0 mm	75.40	0.64	0.64	10.37	0.13	0.99	0.93	0.17	0.46	6.48
	concrete 1.0-2.0 mm	69.51	0.75	0.75	15.41	0.14	0.99	1.07	0.20	0.71	6.35

LOI = Loss on Ignition.

The particle size distribution of CDW can be seen in Figure 1a. After milling, ∽65 wt.% of particles are between 0.5 and 2.0 mm. Figure 1b shows SEM and optical photographs of CDW particles. In the Figure 1b, it is possible to visually observe the CWD composition from the optical photos and SEM micrographs, consisting basically of concrete and clay brick particles in the size ranges of 0.5-1.0 and 1.0-2.0 mm.

(a)



(b)

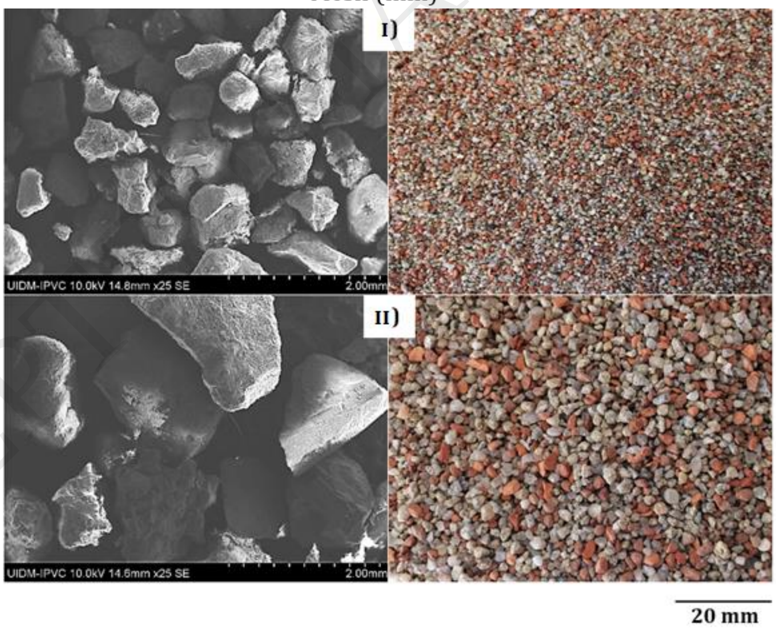


Figure 1. Characteristics of CDW: a) Particle size distribution; b) SEM micrographs (left) and optical photos (right) of CDW samples: I) 0.5-1.0 mm and II) 1.0-2.0 mm.

They also reveal the irregular shape of the grains, which causes ∽ 20% reduction in the bulk density of the recycled aggregates compared to sand (Table 3), this effect is related to the greater packing of the spherical particles of the sand.

Table 3. Physical properties of aggregates and geopolymer mortars.

Aggregate	Particle size (mm)	Aggregate bulk density (kg/m^3)	Water absorption (%)	Bulk density (kg/m^3)	Porosity (%)
CDW	0.5 - 1.0	1250 ± 0.04	12.1 ± 0.1	1780 ± 0.02	21.5 ± 0.19
	1.0 - 2.0	1210 ± 0.04	12.3 ± 0.2	1830 ± 0.01	22.5 ± 0.29
	0.5 - 2.0	1290 ± 0.07	12.3 ± 0.1	1820 ± 0.01	22.3 ± 0.15
Sand	0.5 - 1.0	1520 ± 0.05	11.7 ± 0.1	1830 ± 0.01	21.5 ± 0.18
	1.0 - 2.0	1460 ± 0.05	11.6 ± 0.2	1910 ± 0.01	21.8 ± 0.20
	0.5 - 2.0	1540 ± 0.06	12.2 ± 0.2	1890 ± 0.01	22.7 ± 0.17

XRF results of CDW 0.5-1.0 and 1.0-2.0 mm fractions are similar, revealing that the grain size fractioning does not significantly alter the chemical composition. However, by segregating the brick and concrete fraction of the CDW, compositional changes were observed (Fig. 2).

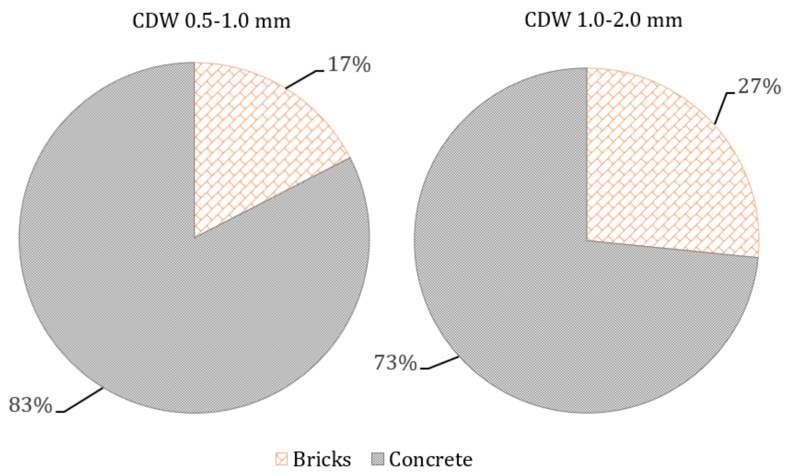


Figure 2. Composition (wt.%) of bricks and concrete in the CDW aggregates of sizes 0.5-1.0 and 1.0-2.0 mm, respectively.

Initially, in the 1.0-2.0 mm fraction, brick and concrete components correspond to 27 wt.% and 73 wt.%, respectively. In the 0.5-1.0 mm fraction, the brick amount decreased to 17.4 wt.%. This is expected, since the concrete is composed of finer elements (cement and sand) than bricks, being segregated upon grinding. This is confirmed by FRX results, revealing a ~6% increase in the SiO_2 content in the concrete fraction in the particle size of 0.5-1.0 mm, as shown in Table 2.

The mineralogical compositions of the raw materials used are given in Figure 3. The identified crystalline phases in CDW were quartz (PDF 00 046 1045), calcite (PDF 04 012 0489) and muscovite (PDF 00 058 2035). In the XRD patterns of FA were identified quartz and calcite, and quartz and muscovite in the MK, as presented in previous work (De Rossi et al., 2018). Similar composition was found elsewhere for fly ash (Mehta and Siddique, 2017; Novais et al., 2016a) and CDW (Arenas et al., 2017; Contreras et al., 2016; Vásquez et al., 2016).

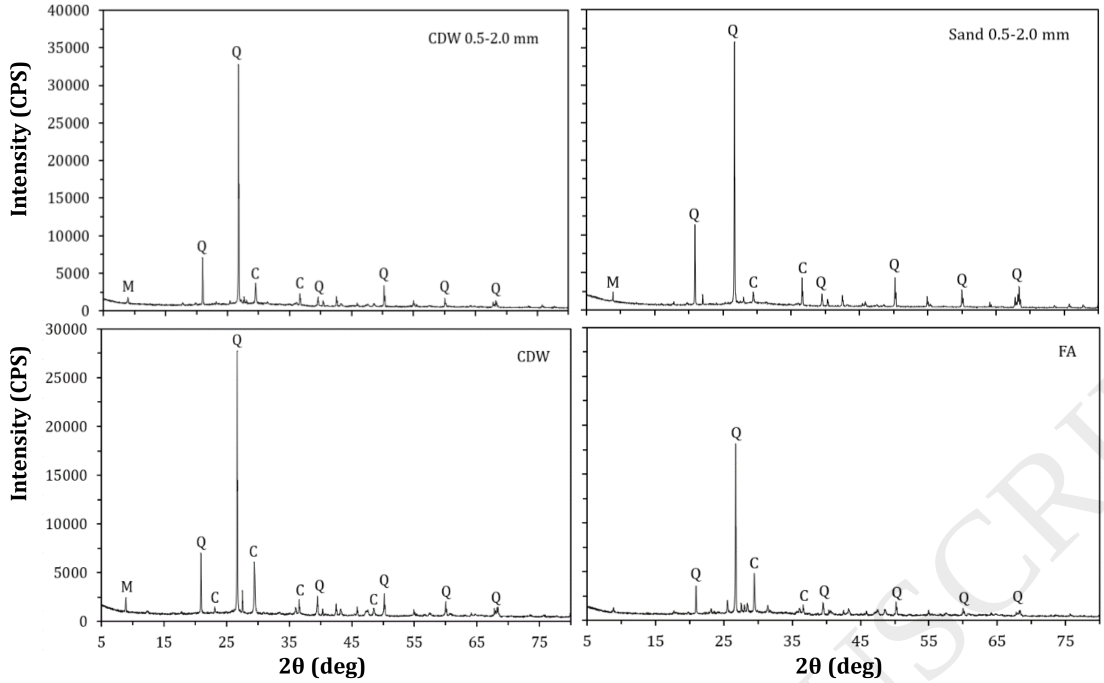


Figure 3. XRD patterns of raw materials (CDW and FA) and the geopolymer mortars with CDW 0.5-2.0 mm and sand 0.5-2.0 mm.

Q = Quartz (PDF-00-046-1045); M = Muscovite (PDF-00-05-2035); C = Calcite (PDF-04-012-0489).

The fly ash presents an halo visible approximately 2θ between 17° and 33°, revealing its amorphous or vitreous character (Cristelo et al., 2018). In this fly ash, no halo was identified, evidencing low potential for geopolymerization. However, it is supposed that if formulated correctly with MK, it could be used as a binder for geopolymers, as demonstrated by Novais et al., [18] and De Rossi et al., [28,29]. The mortars produced with CDW and sand with particle size of 0.5-2.0 mm present the same minerals of the raw materials, as can be also observed.

3.2. Flow measurements of fresh geopolymer mortars

The results of the flow table test presented in Figure 4 show that mortars developed with CDW and sand presented similar scattering diameters as error ranges overlap, with exception in 0.5-1.0 mm. The lowest flowability values were registered on mortars prepared with aggregates of broader size distribution (0.5-2.0 mm): 126 mm and 133 mm, respectively for CDW and sand. The extended particle size range will increase the packing density of the fresh mortars, and more compact systems reach higher yield stress values. Similar results were found in the literature using fine CDW aggregates for cement mortars (Fan et al., 2015; Topçu and Bilir, 2010; Torkittikul and Chaipanich, 2010). Thus, the results show the possibility of producing mortars with total replacement of the natural aggregate by the recycled aggregate, maintaining the fresh state of the geopolymer mortars.

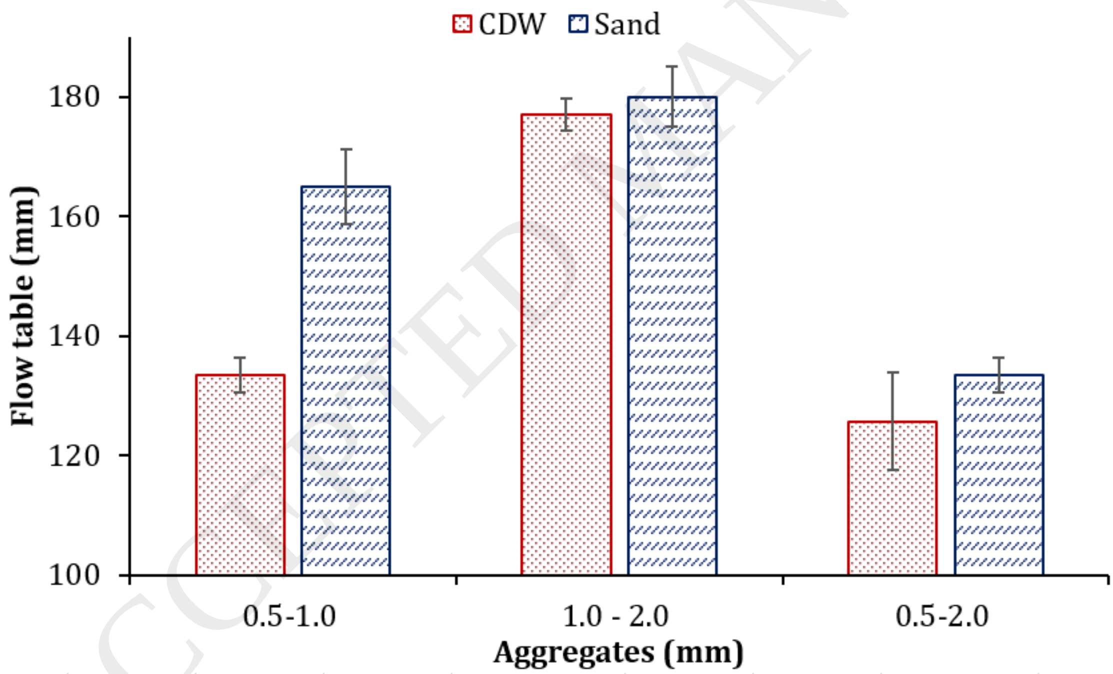


Figure 4. Flow diameter of geopolymer mortars with CDW and sand aggregates.

3.3. Physical properties and mechanical behavior of hardened geopolymer mortars

The properties of hardened mortars samples are listed in Table 3. The water absorption results were similar for all mortars produced (∽12%), which is in accordance with the results obtained by Mermerdaş et al., (2017a), using natural sand as an aggregate for geopolymer mortars. Nonetheless, CDW and sand mortars have a similar porosity (∽22%) after 28 days of curing period, showing the viability of CDW reuse as fine aggregate. The results of the mortars bulk density show that the natural aggregate replacement by the recycled aggregate, in all size ranges, influences the obtained results. Thus, the mortars produced with CDW presented a lower density compared to the sand mortars, which are in agreement with several works showing that, by increasing the amount of recycled aggregate, the density of mortars decreases (Hwang et al., 2008; Silva et al., 2016).

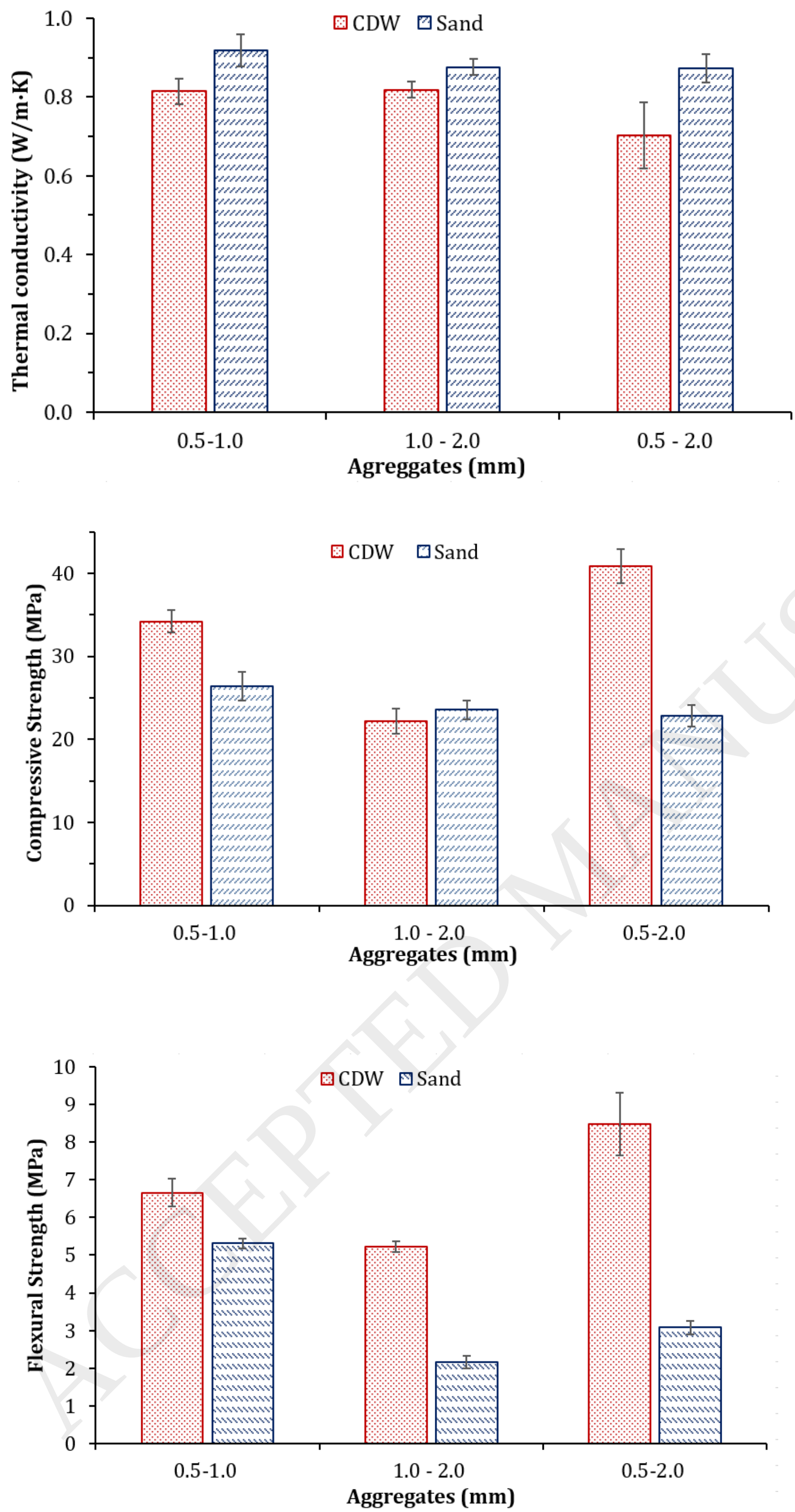


Figure 5. Physical properties of hardened geopolymer mortars: a) Thermal conductivity; b) Compressive strength; c) Flexural strength.

In the thermal conductivity tests (Figure 5a), mortars produced with CDW aggregate presented values between 0.70 and 0.81 W/m·K, lower than those obtained when using sand as aggregate (0.87 and 0.91 W/m·K). These results are related to the density of the studied aggregates, higher aggregates' density increases solid conduction and consequently thermal conductivity increases (Gomes et al., 2017) of mortar with sand. For CDW mortars, the lower density of the residues associated with the porosity of the residues influenced the lower values of thermal conductivity, as shown in Table 3. Similar thermal conductivity results were obtained by Narayanan and Shanmugasundaram, (2017), when investigating geopolymer mortars with natural sand, 0.91 W/m·K.

The compressive strength values for geopolymer mortars with CDW and sand are shown in Figure 5b. The highest strength was achieved when CDW was used as aggregate, except for the mortar produced with the particles of 1.0-2.0 mm, when higher strength was obtained with sand. The obtained results for CDW-geopolymer mortars were ∽21 MPa (1.0-2.0 mm), ∽34 MPa (0.5-1.0 mm) and ∽40 MPa (0.5-2.0 mm). The highest strength values were obtained for the mixed fraction, due to the highest packing density (Reig et al., 2017; Sohn and Moreland, 1968).

Considering EN 998-2 (2010) standard, they can be classified as M20 (>20 MPa) and Md (>25MPa), for masonry mortars have compressive strength, after 28 days. However, it is important to note that there are still no standards for the mortars produced by geopolymerization (only for geopolymer concrete standard PAS 8820, (2016)) so that the use of the standard EN 998-2, (2010) was only for comparison with commercial cement-based mortars.

Figure 5c presents the flexural strength results of the mortars cured during 28 days at room conditions. When comparing to sand used as aggregate, the best performance was obtained again with the mortars produced with CDW as aggregate. When the extended range of particles was used (0.5-2.0 mm), the highest values of flexural strength were obtained (8.5 ± 0.8 MPa), following the tendency of the compression strength results. Thus, the larger distribution of aggregate sizes increases the packing density of mortars, as observed elsewhere (Contreras et al., 2016; Mermerdaş et al., 2017b; Reig et al., 2017; Sohn and Moreland, 1968).

Similar results were also obtained in other studies, where it is suggested that the cement mortars produced with CDW fine fractions developed higher mechanical strength, when compared with natural aggregates (Neno et al., 2014; Topçu and Bilir, 2010). This increase in the mechanical strength may be associated with chemical reactions of the non-hydrated cement particles present in CDW (Braga et al., 2014) or to the pozzolanic reactions between alumina, silica and calcium hydroxide available in cement (Vieira et al., 2016) and also because the concrete particles have a higher specific surface and more porous than sand, promoting the better bond with the cement paste (Neno et al., 2014). Thus, other factors that generally affect the mechanical properties of mortars, such as porosity and water absorption, did not influence the results obtained in this study.

Photos of CDW and sand hardened mortars samples are compared in Figure 6. It is possible to observe that the aggregates used, CDW and sand, were totally integrated into the geopolymer paste, forming a homogeneous and compacted microstructure. Thus, no preferential paths are visible, which facilitate the rupture of the hardened mortar. The content of brick particles in the CDW aggregate and the presence of some porosity can be also observed in all samples. The residual porosity is probably due to the absence of vibration in the fresh mortar, but it did not compromise the good performance in the mechanical behavior as seen previously.

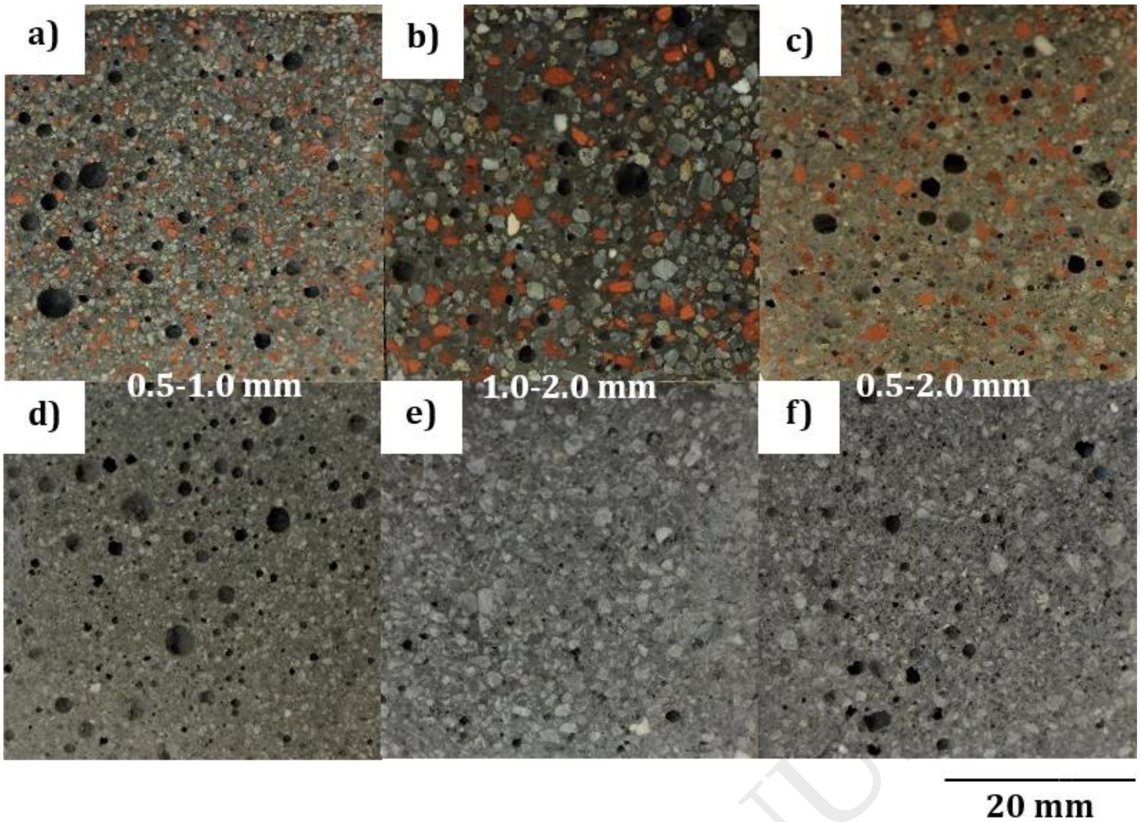


Figure 6. Photos of geopolymer mortar samples with CDW (a, b, c) and sand (d, e, f) as fine aggregates.

In Fig. 7 presented the semiquantitative chemical composition by EDS of geopolymer mortars with recycled and natural aggregate, for a particle size of 0.5-2.0 mm. It is observed that the chemical elements identified were Si, Al, Na, K and Ca in both mortars analyzed. It was detected that in the mortar produced with sand, an increase in the silicon peak is observed, due the sand presence (with 98 wt.% of SiO_2).

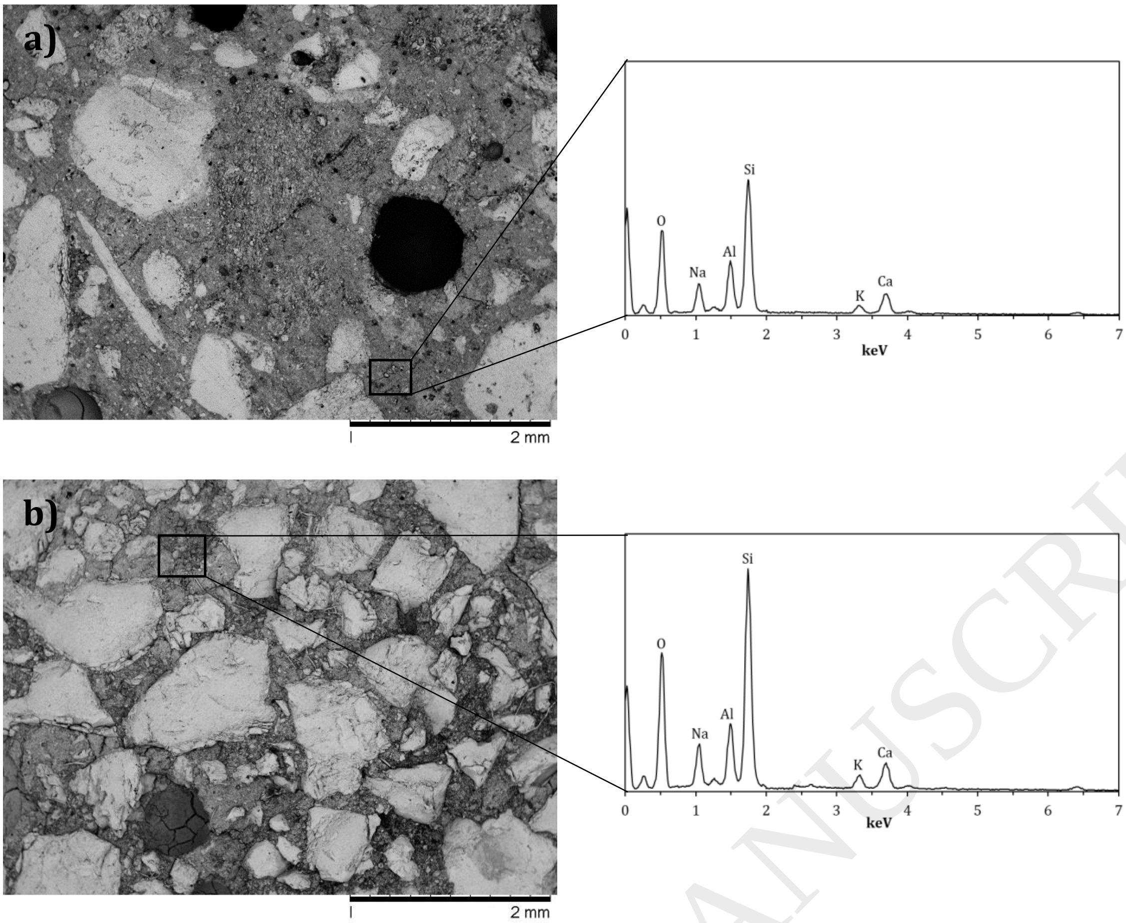


Figure 7. SEM micrographs and EDS spectra of mortar paste with: a) CDW 0.5-2.0 mm and b) sand 0.5-2.0 mm.

The lower results obtained with the mortar developed with natural aggregate can be attested by SEM micrographs (Fig. 7b). Here microcracks are observed in the interface region of the natural aggregate and the geopolymer paste, which induce the easily rupture of the specimens and reduce the compressive and flexural strength of hardened mortars. These microcracks were not observed in the microstructure of mortars produced with CDW, thus reiterating the advantages of the recycled fine aggregate using, in the production of geopolymer mortars.

4. Conclusions

In this work, the geopolymer mortars produced with biomass fly ash as a binder and CDW as fine aggregate and cured in environmental conditions exhibited enhanced mechanical and flexural strength, exhibited increase 78% in mechanical strength and 175% flexural strength, in comparison with the sand as fine aggregate (0.5-2.0 mm).

In the fresh state, mortars produced with CDW showed less dispersion compared to the sand aggregate samples, possibly due to the higher absorption of water by this industrial waste. However, in the hardened state, the highest mechanical strength results were obtained with CDW with a wide range of particles (0.5-2.0 mm), due to the higher packaging of the particles and microcracks absence. The results obtained for porosity, density and water absorption show total compatibility between mortars produced with CDW in substitution of sand as aggregate. Thus, the possibility of adding CDW as a fine aggregate in the production of geopolymer mortars has been proven, with excellent mechanical properties and potential for several applications in building, replacing conventional mortars.

In addition, 75 wt.% of the materials used in the production of geopolymer mortar are industrial wastes (FA and CDW). The use of wastes as raw materials reduces the exploitation of natural resources and highlights the environmental and economic advantages of recycling.

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Figure Captions

Figure 1. Characteristics of CDW: a) Particle size distribution; b) SEM micrographs (left) and optical photos (right) of CDW samples: I) 0.5-1.0 mm and II) 1.0-2.0 mm.

Figure 2. Composition (wt.%) of bricks and concrete in the CDW aggregates of sizes 0.5-1.0 and 1.0-2.0 mm, respectively.

Figure 3. XRD patterns of raw materials (CDW and FA) and the geopolymer mortars with CDW 0.5-2.0 mm and sand 0.5-2.0 mm.

Figure 4. Flow diameter of geopolymer mortars with CDW and sand aggregates.

Figure 5. Physical properties of hardened geopolymer mortars: a) Thermal conductivity; b) Compressive strength; c) Flexural strength

Figure 6. Photos of geopolymer mortar samples with CDW (a, b, c) and sand (d, e, f) as fine aggregates.

Figure 7. SEM micrographs and EDS spectra of mortar paste with: a) CDW 0.5-2.0 mm and b) sand 0.5-2.0 mm.

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Table Captions

Table 1. Chemical composition (wt.%) of FA, MK, sand and different fractions of the CDW.

Table 2. Physical properties of aggregates and geopolymer mortars.