Statistical Analysis of the Chemical Composition of Concrete Slurry Waste: A Case Study of Sedimentation Tanks from Concrete Batching Plants in Manaus-Brazil

Jéssica Raíssa M. Guimarães,[©]*,^a Yves Nathan M. de Faria,[©]^b Mateus F. de Oliveira,[©]^a Lizandro Manzato[©]^a and Cláudia C. Silva[®]^b

^aLaboratório de Síntese e Caracterização de Nanomateriais (LSCN), Instituto Federal de Educação, Ciência e Tecnologia do Amazonas Campus Manaus Distrito Industrial (IFAM/CMDI), Av. Gov. Danilo de Matos Areosa, s/n, Distrito Industrial, 69075-351 Manaus-AM, Brazil

^bGrupo Crowfoot de Métodos de Raios-X, Escola Superior de Tecnologia, Universidade do Estado do Amazonas (UEA), Av. Darcy Vargas, 1200, 69065-020 Manaus-AM, Brazil

Concrete slurry waste is a material removed from sedimentation tanks that receive water used to wash concrete mixer trucks and patios of concrete batching plants (CBP). The objectives of this study were to determine the chemical composition of this residue from three different producers, in three consecutive months and at different collection points using wavelength dispersive X-ray fluorescence, investigate the inconstancy of content through analysis of variance, compare the qualitative and quantitative results with the literature and discuss the possibility of inserting the material in the proposal of different authors. Twenty-two oxides and elements were identified; among them, five showed their incidence in a punctual way. The analysis of variance showed that there are significant differences in the concentration of components between batching plants, months and collection points. The comparison with the literature showed that CaO, Al₂O₃ and SiO₂ are the majority, both in the samples under study and in the samples from different authors. However, the average amounts of CaO are higher in the waste sludge from the concrete batching plants in question and, consequently, if this material were reused in the same proposals as the authors, the products would possibly present different characteristics and performance.

Keywords: concrete slurry waste, chemical composition, WD-XRF, calcium-rich residue

Introduction

Concrete batching plants (CBP) generate several types of waste, such as remnants and fresh concrete ballasts, washing water and residual sludge. The production of the latter occurs when the solids suspended in wastewater (derived from the washing of the concrete mixer trucks and the yard) are deposited at the bottom of tanks, after a sedimentation process.¹ This residue has high water content, and the resulting fines consist of hardened cement, fine aggregate and a small portion of coarse aggregate.²

Periodically, the volume of residual sludge is dredged from the sedimentation tanks, disposed of in drying bays and then sent to landfills.² However, this approach is not an environmentally sustainable option due to energy consumption and CO_2 emissions associated with transport

*e-mail: jessi_raissa@hotmail.com Editor handled this article: Maria das Graças A. Korn (Associate) and handling. In addition, studies report that dry waste can still have high levels of trace metals that do not meet the criteria for accepting inert waste in landfills.³ Indiscriminate disposal can cause harmful effects on the environment and human health due to its high alkaline content.⁴

Within the context of reusing this waste, the literature presents some research developed in different applications. Most studies are focused on the incorporation of waste in new concretes, as binders or fillers, and on the replacement of aggregates.^{2,3,5,6}

There is still research directed towards applications in glass-ceramic components;⁷ geopolymer;⁸ road bases or concrete filling;⁹⁻¹¹ absorbent of chemical products, such as CO₂ capture, phosphorus recovery, water clarification and so on.¹²⁻¹⁷

The purpose of this work is to determine the qualitative and quantitative chemical composition of waste from three different producers, in three consecutive months and at different collection points through wavelength dispersive X-ray fluorescence (WD-XRF). Then, designate the possible origins of the elements and oxides found in the residues, investigate the inconstancy of their concentrations through analysis of variance (ANOVA) and compare the data with the literature, considering the proposals of different authors. The present study also points out possible promising applications that have not yet been explored, in order to value the waste.

Experimental

The study began with the collection and preparation of samples from the sedimentation tanks of the concrete batching plants. Then, the chemical composition was determined using WD-XRF, which was divided into two aspects: qualitative and quantitative. For the first aspect, the possible origins of the identified elements and oxides were discussed. For the quantitative aspect, the concentrations of each constituent of all the samples were submitted to statistical analysis in order to investigate the inconstancy of the content. First, the significant variability of the means of elements and oxides inside the sedimentation tank of each CBP was verified, comparing the collection points with each other. The analysis continued with the comparison between the means of oxides by months of collection, again by CBP. Finally, a comparison was made between the CBP, considering the mean of all collected samples.

After the statistical analysis, the total means of the elements and oxides of each CBP were compared with the data found in the literature for the same type of residue. Finally, possible promising and unexplored applications for the residual concrete slurry were presented, considering the chemical composition studied. More details of the work steps are described in the sections below.

Sample collection and preparation

The residual slurry samples were provided by three different CBP in the city of Manaus, called CBP K, CBP S

and CBP P. The collection in the sedimentation tanks was carried out at three different points (Figure 1), once a month and for three consecutive months: July, August, and September of 2020, titled as month 7, 8, and 9, respectively.

The samples were collected in a humid state and dried in an oven (CIENLAB, model CE 220/81, Campinas, Brazil), at 105 °C for 24 h, according to the literature.^{1,2,17} After drying, the material was macerated, with the aid of a porcelain pestle, and manually sieved, separating for use the volume passing through the ABNT No. 100 sieve (mesh opening of 150 μ m), in order to ensure a narrow distribution particle size for the characterization analysis.

For data control, the samples received unique identifications that included the initial of the concrete batching plant (K, S or P), the month of collection (7, 8 or 9) and the collection point in the sedimentation tank (A, B or C). For each point, 8 microcentrifuge tubes were reserved, totaling 216 samples.

For each sample, with the aid of a 10,000 kgf press, pellets of fine powder were prepared with uniform granulometry, whose composition presents 1.000 g of the sample, homogenized in an agate mortar and pestle with 4.000 g of high purity H_3BO_3 (Merck > 99%, São Paulo, Brazil). All samples were analyzed in eight replicates to provide more consistent response repeatability and application in the analysis of variance between measurements.

It should be noted that the samples were used dry and in powder form because the moisture that was presented could influence the WD-XRF analysis. In addition, heterogeneous samples with inadequate surfaces are obligatorily pretreated and analyzed in pellet format.¹⁸

Data collection by WD-XRF

The analyses were carried out in a XRF instrument (Rigaku, supermini model, Wilmington, USA) using wave dispersion (WD-XRF) and a palladium tube, exposure time of 200 s, with a power of 200 W. The conditions were adjusted, considering the sample matrix, the sample holder,



Figure 1. Sedimentation tanks, (a) CBP K; (b) CBP P and (c) CBP S. Collection points: (A) tank inlet, (B) tank middle and (C) tank outlet.

and the sample support. All elements were identified by their K α and/or K β energies.¹⁹ Analyzer crystals LIF 200, PET and RX25 were used. In this equipment, scintillation counter and detectors are available, both used in the analysis of these samples.

Geological reference standards such as GBW 3125, 7105 and 7113 were used to calibrate the equipment. Through mathematical software, the emission peaks are related to the respective concentrations of a certain element.

For each element found, quantification was carried out using external standards of salts of known purity, diluted in boric acid, also of known purity, in at least six predetermined concentrations, which were submitted to the same sample analysis conditions. In the end, their quantifications were performed based on their intensities (counts *per* second (cps) / uA) using the ZSX program-Spectrometer Status.²⁰

Statistical analysis

The Grubbs's test was used for outliers' exclusion, and the comparison of mean concentrations among collection points, months and CBPs was performed using one-way analysis of variance (ANOVA). Both tests were performed in Microsoft Excel and a 95% significance level was adopted for all comparisons. For the ANOVA test, if the *F* ratio exceeded critical *F*, at least one of the group means is different from the others.

Graphs illustrate concentrations in percentages from tabulated data. For the means of the three major oxides of the three CBP, the 95% confidence interval for each component was also calculated to verify if the total means overlap in their intervals and to verify if there were two equal percentages and a different one.

Results and Discussion

Qualitative results

The chemical composition is divided into two groups

Table 1. Specific constituents of collected waste sludge

of oxides and elements: those common to the samples from the three CBP, in their three collection points in the tank, in the three months of collection: Na₂O, MgO, Al₂O₃, SiO₂, P₂O₅, SO₃, Cl, K₂O, CaO, TiO₂, V₂O₅, Cr₂O₃, MnO, Fe₂O₃, CuO, ZnO and SrO. And the second group refers to punctual constituents, which were found in only two CBP and/or did not occur in all months or collection points in each CBP (Table 1).

Discussion of qualitative results

According to Schoon *et al.*,²¹ the chemical composition of the sludge is mainly influenced by the raw materials used in the production of concrete. This mixture is composed of Portland cement, aggregates, water, among other options, according to the required product.²²

The content of CaO, SiO₂, Al₂O₃ and Fe₂O₃ possibly has Portland cement as its main source, as these oxides are essential for this binder. Other smaller components can also be derived from this material, such as MgO, SO₃, Na₂O and K₂O.²³

Common and specific oxides can also come from natural aggregates. The presence of SiO₂ is explained by the natural sand or gneiss aggregates used in concrete.²⁴ Other studies²⁵⁻²⁹ indicate the incidence of TiO₂, Al₂O₃, Fe₂O₃, MnO, MgO, CaO, Na₂O, K₂O, P₂O₅ as the main soil constituents in the region, and As, Ba, Cl, Cu, Ni, Sb, Sr, V and Zn in lower concentrations.

Chemical species Cl, CaO and I can also originate from water. Since this resource comes from the city public supply system and undergoes potability treatment, there are additions of coagulants consisting of the first two constituents. Iodine occurs naturally in river waters, predominantly in the form of iodide ion (I^-) .^{30,31}

The occasional occurrence of some oxides may be related to the variation of batches and typology of Portland Cement (type I, II, III and IV) in the concrete mixes and with the origin of the aggregates used in the north and northeast region of Brazil, which can be originated from crystalline massifs in Amapá, Roraima, Amazonas and Maranhão.^{32,33}

Oxide/element	СВР К				CBP P		CBP S			
	K7	K8	К9	P7	P8	P9	S7	S8	S9	
NiO	×	×	$X^{a,b}$	×	×	×	-	× ^{b,c}	×	
As_2O_3	-	×	×	-	-	-	×	-	×	
Sb ₂ O ₃	-	-	-	×	×	×	×	×	×	
I	×	×	×	×	×	-	×	×	×	
BaO	×	×	×	×	-	×	×	×	×	

^{a,b}Identified only at collection points A and B; ^{b,c}identified only at collection points B and C.

Quantitative results

Specific oxides were not exposed in the graphic data because the analysis of variance was not applied to them in all proposed situations. However, it is worth adding that these oxides did not obtain concentrations greater than 0.500%, with BaO being the most expressive within this group. Means with significant differences were observed only for NiO and BaO in samples from CBP K and only NiO in samples from month seven from CBP P.

Collect point

In CBP K, the oxides Na₂O, V₂O₅ and iodide had similar mean concentrations *per* point during the three months of collection. In addition to these, P₂O₅, MnO, ZnO, NiO, As₂O₃ and Cr₂O₃ also showed up in this condition, but in just one or two months and with contents lower than 0.500%.

According to Figures 2a-2i, most of the other oxides and elements, whose means *per* point differ from each other, exhibited lower amounts at point C, except for CaO, SO₃, SrO and Cr₂O₃ as shown in Figures 2a-2c. The highest

percentages are given to CaO (60-75%) and SiO₂ (12-20%), in all collection periods.

The chemical composition of the CBP P samples showed only CuO and Sb_2O_3 with equivalent averages by points in months 7, 8 and 9. However, other oxides and elements also obtained their local concentrations similar, but not in all months of collection, namely: I, BaO, V_2O_5 , NiO, TiO₂, Na₂O, Cl, V_2O_5 , Cr₂O₃, MnO, Fe₂O₃ and SrO. The amount of each component of this group is less than 1.000%.

As shown in Figures 3a-3i, the set of oxides and elements with means of different contents did not show position predominance (A, B or C) in terms of greater or lesser concentration. As with CBP K, the most expressive percentages are CaO (48-56%) followed by SiO₂ (25-30%) as shown in Figures 2a, 2d, 2g.

CBP S had the highest number of oxides with similar averages of concentration *per* point. The incident components in the three months of the collection were Cr_2O_3 , MnO, Sb_2O_3 , I and BaO; and with similarities in fewer months of collection, there are Al_2O_3 , Cl, TiO_2 , V_2O_5 , Cr_2O_3 , Fe_2O_3 , CuO, As_2O_3 , NiO, SrO, Na₂O.



Figure 2. Average concentration of elements and oxides *per* collection point at CBP K: (a), (b) and (c) in month 7; (d), (e) and (f) at month 8; (g), (h) and (i) in month 9.



Figure 3. Average concentration of elements and oxides *per* collection point at CBP P: (a), (b) and (c) in month 7; (d), (e) and (f) at month 8; (g), (h) and (i) in month 9.

There was no domain of any collection point referring to a greater or lesser percentage of oxides and elements, considering the entire composition. However, by restricting the set to oxides with different means, it was observed that most of the highest concentrations are at point C as shown in Figures 4a-4i. CaO and SiO₂ exhibited the majority contents (Figures 4a, 4d, 4g), as observed in CBP K and CBP P.

Collection month

The monthly averages reaffirm that the highest concentrations of the material in the three CBP belong to CaO, SiO_2 and Al_2O_3 , in decreasing order. However, when comparing the monthly quantities, it was observed that these do not have different means in all CBP, as occurred in the analysis by collection point.

The oxides with similar monthly averages in the three months of collection for the three CBP are TiO_2 , V_2O_5 and MnO. There are also P_2O_5 , MgO, Cr_2O_3 , NiO, CuO, ZnO, SrO, I, BaO and Sb₂O₃ that occur for CBP pairs; and Al₂O₃, SiO₂, CaO and Fe₂O₃ only for CBP K, which presented the highest number of constituents with equal monthly means.

As for the components with significant differences in the means (Figures 5a-5h), only SO₃, K_2O and Cl are repeated for the three CBP (Figures 5a, 5b, 5d, 5e, 5g, 5h), whose largest quantities are found in CBP K. The greatest number of oxides and elements in this group are attributed to CBP P.

Dosing center

Only two constituents have means without significant differences between CBP: Na_2O and V_2O_5 . The three plants presented CaO as the majority, whose most expressive concentration is found in CBP K, which was the only one that presented statistically equivalent monthly averages of this oxide.

According to Figures 6a-6c, there are 15 constituents with significative differences between CBP. The amount of SiO₂ in the CBP K material, unlike CaO, is the lowest among the three collection sites, as well as Al_2O_3 (Figure 6a). The higher percentages of the other oxides with significant differences were divided, for the most part, between CBP K and CBP P. P_2O_5 is the only component that has a higher content attributed to CBP S (Figure 6b).



Figure 4. Average concentration of elements and oxides *per* collection point in CBP S: (a), (b) and (c) in month 7; (d), (e) and (f) at month 8; (g), (h) and (i) in month 9.

When comparing the averages of the three largest quantities with their respective confidence intervals, it was noted that they do not overlap (Figures 7a-7c), so it can be stated that the residual concrete sludge produced in the three CBP is different, considering the averages of all contents of these oxides in all collected samples.

Discussion of quantitative results

The global average of the oxides identified in the samples showed that the sedimented material is different among the CBP. As explained above, this fact may occur due to the different origins of the raw materials used in each place for the manufacture of concrete. In addition, the CBP meet the different demands of the initial product, this includes changes in characteristic strengths and modifications in dosage, changing amounts of aggregates and binders, which in turn may contain variations in chemical composition by classification and/or manufacturing batch.

Statistically divergent monthly averages can also be justified by requests for different concretes within a single CBP, as it can produce in different strength classes. Regarding the difference *per* collection point, this is possibly associated with the geometry of the sedimentation tanks and the particle size of the material.

The tanks enable the separation of suspended particles in wastewater through the action of gravity and a difference in density of the components involved. Thus, the performance of this treatment system is influenced by the characteristics of the suspended solids and by the geometry and dimensions of the tank.³⁴

The three CBP have rectangular tanks, but with different internal subdivisions. According to Al-Sammarraee and Chan,³⁵ the greater the number of internal walls, the more intense the vertical movements and the smaller the horizontal movements of the fluid. The wall increases hydraulic retention time, resulting in significantly increased sedimentation.

This effect may explain the means of higher concentrations at point A, observed in months 7 and 9 of CBP K, as the tank has, in this region (Figure 1a), the greatest number of walls and, consequently, the lowest level of the oxides occurred at point C in the three months of collection. That is, there was a greater sedimentation of



Figure 5. Average concentration of elements and oxides per month of collection: (a), (b) in CBP K; (c), (d) and (e) in CBP P; (f), (g) and (h) in the CBP S.



Figure 6. Average concentration of elements and oxides of the three CBP: (a) CaO, SiO₂, Al₂O₃ and Fe₂O₃ in the CBP K, P and S; (b) SO₃, MgO, K₂O, TiO₂ and P₂O₅ in the CBP K, P and S; (c) SrO, ZnO, MnO, Cl, CuO, Cr₂O₃ in the CBP K, P and S.

particles in the area related to the entrance and the center of the tank, probably associated with the location of the walls in greater number.

Another factor that influences sedimentation efficiency is the decrease in particle size. Larger particles are expected to settle quickly along the tank inlet because of their mass and inertia. As particle size decreases, the effect of gravity decreases.³⁶

CBP P and CBP S, as mentioned above, do not have a predominance of collection points with greater or lesser concentrations of oxides, in general. This occurrence may be related to a narrow particle size range of sediment particles, which may lead to similar deposition along with the tank, in addition to the layout of the inner walls, which are spaced at similar distances (Figures 1b and 1c).

Table 2 shows the chemical composition in oxides and elements of the same type of waste under study presented by different authors. The material collected comes from CBP from different countries: Brazil, Thailand, Japan, Belgium, France and China. In Figures 8a-8d, the average of the concentrations in the literature is found, together with the averages of the samples presented above, by CBP.

Literature, in general, presented 11 of the 17 oxides common to CBP K, P and S. The components not identified



Figure 7. Average concentration and confidence interval of major oxides among CBP: (a) CaO; (b) SiO₂; (c) Al₂O₃.

Table 2. Chemical composition of residual concrete sludge in literature

Reference	Concentration of elements and oxides / %											
	CaO	SiO ₂	Al_2O_3	Fe ₂ O ₃	SO ₃	MgO	K ₂ O	Na ₂ O	TiO ₂	P_2O_5	Cl	Others
Chatveera et al.37	32	26.87	6.91	3.17	3.92	1.51	0.98	0.18	_	-	_	24.46
Iizuka et al. ¹²	17.2	3.2	0.6	1.9	_	-	-	-	-	-	-	77.1
Schoon et al.21	23.695	49.198	4.597	1.956	1.292	2.558	0.564	0.298	0.314	0.154	0.057	15.317
Audo et al. ³⁸	34.458	53.545	1.925	1.525	0.77	-	_	-	-	_	-	7.778
Xuan <i>et al.</i> ⁵	35.41	33.146	8.633	6.585	2.956	1.377	1.652	-	0.526	-	-	9.715
Tang <i>et al</i> . ⁶	35.065	26.895	9.76	4.645	2.6	1.85	1.15	0.83	-	0.25	0.015	16.94
Tang et al.39	36	28	9.4	7	3.7	1.6	1.5	1	-	0.3	0.1	11.4
Martins et al.40	38.167	26.233	6.03	2.753	1.113	1.293	0.817	0.637	0.28	0.17	-	22.507
Martins et al. ²⁴	22.703	50.722	6.581	2.2	_	0.873	1.407	1.243	0.211	0.091	-	13.968
Mean	30.522	33.09	6.048	3.526	2.336	1.58	1.153	0.698	0.333	0.193	0.057	22.132
Standard deviation	7.381	15.938	3.192	2.07	1.514	0.832	0.611	0.476	0.194	0.117	0.036	21.338







Literature Mean CBP K Mean CBP P Mean CBP S Mean

so,

Figure 8. Average of the contents of oxides and elements common between CBP and literature.

in the references were the following metal oxides: V_2O_5 , Cr_2O_3 , MnO, CuO, ZnO and SrO, which exhibited chemical concentrations lower than 1.000% in the samples analyzed in this work.

Regarding the percentage of oxides, it was observed that the predominance of CaO and SiO₂ (Table 2) occurred both in the literature samples and in the samples from the visited CBP. However, only in the quantifications presented by Schoon *et al.*,²¹ Audo *et al.*,³⁸ and Martins *et al.*²⁴ SiO₂ concentrations were higher than CaO, which did not occur in any of the measures presented in this study. On the contrary, in the average of the results obtained, the CaO content was around twice as much SiO₂ (Figure 8a).

The average percentage of SiO₂, K₂O, Na₂O and Cl in the literature surpasses all the percentages presented by the samples of CBP K, P and S, as well as their respective standard deviations, denoting a considerable variability among the published chemical compositions (Figures 8a, 8c, 8d, 8e).

The qualitative and quantitative differences between the research present in the literature and this work can also be elucidated for the reasons already detailed: different origins and chemical compositions of the raw materials of the original product (concrete), variations in cement batches, dosage changes and/or mineral additions.

Chatveera *et al.*³⁷ used the water from the waste sludge in the manufacture of Portland cement concrete, replacing public water supply. By submitting the specimens to acid attacks, they observed that the concrete mixed with the wastewater was more vulnerable and showed a greater percentage of weight loss concerning the control concrete. This is because wastewater contains suspended solids with additional CaO content that make the concrete matrix more porous and less resistant to acids. The percentage of CaO in the sludge powder, presented by the authors, is 32.00%. The average of the same oxide in this research is 59.48% and, if the water from these samples were used in concrete, possibly the resistance to acids would be more affected.

The work by Iizuka *et al.*¹² used concrete sludge as a source of calcium to prepare a solid adsorbent for phosphorus recovery by hydroxyapatite. The material was previously submitted to different dilution ratios (5,10 and 15) for the formation of calcium ions, influencing the results of the process. The effect was unclear, but the authors stated that a dilution ratio of 10 performed best. The CaO content in the chemical composition of this literature study for the investigated samples is about 40.00% lower. Therefore, it can be deduced that by using the same dilution ratios for the same purposes, consequently, the adsorbents would present different performances. Schoon *et al.*²¹ investigated the chemical composition and variations of 87 dry waste sludge samples collected over two years at five different CBP in Belgium. The purpose of the research was to value a fraction of the residue as an alternative raw material to produce Portland clinker. The authors found high variability in the constitution of the samples, which makes the intended use unfeasible, due to the recommended limits for the final clinker. Likewise, the samples collected in Manaus would also be unfeasible for the same purpose, as they present significant differences in chemical composition.

Audo *et al.*³⁸ collected concrete waste sludge from four different CBP located in France. The chemical compositions showed weak variability, as, according to the authors, the CBP produced mainly the same concrete. The research concerned the incorporation of the residue as limestone filler in replacement of 25.00% of cement in concrete. The results showed a decrease in mechanical strength and change in the rheology of the mortar in its fresh state, making it more plastic. The residue may also have contributed to the acceleration of hydration kinetics because of its Ca content. Thus, in a similar application, the samples of CBP K, P or S could enhance the final characteristics obtained by the authors, due to the higher content of Ca.

The research by Xuan *et al.*⁵ investigated the potential for sequestering CO₂ from waste sludge and sought its valorization as a building material. The authors applied the CaO, SO₃, MgO and K₂O content in an equation to obtain a theoretical extension of CO₂ sequestration. Although the experimental results indicate lower CO₂ absorption, the performance of the carbonated products surpassed the reference products. The averages of the quantities of the oxides mentioned, presented by the authors, are below the averages of the samples collected in CBP K, P and S, except for K₂O. This suggests that the absorption of CO₂ from these CBP samples may present a higher percentage, both theoretical and experimental, and, consequently, products with better performance.

The studies by Tang *et al.*⁶ and Tang *et al.*³⁹ used concrete waste sludge as a fresh cement binder, and combined with fine bottom ash, to produce cold-bonded lightweight aggregates, evaluated by different curing methods. Curing with CO_2 resulted in denser products with lower absorptions, due to the Ca content, in the form of CaCO₃, forming a dense layer on the product surface. The authors also demonstrated the reactive potential of the residual sludge with a pozzolan and Portland cement by obtaining results compatible with mixtures without this combination. The collected samples, if used for the same product in the same arrangements as the studies, could present the aforementioned properties more markedly, due to the higher Ca content, in addition to good combination compatibility with the pozzolan and the binder used in research.

The study by Martins et al.⁴⁰ proposed replacing 25% of Portland cement with fine fractions (< 150 μ m) of concrete slurry waste in the manufacture of structural mortar. The authors collected the waste in three different concrete batching plants in the metropolitan region of Belo Horizonte, Brazil. The chemical composition of the three samples showed CaO content lower than 45%, SiO₂ ranging from 15-42% and Al₂O₃ from 3-10%, approximately. The mortars made with the residue samples that contained the highest CaO contents showed test results closer to those recorded for the mortars produced with the addition of limestone filler and were within the limits established by the standard for composite cement with filler. Therefore, it can be suggested that more promising results would be achieved with the CBP K, P or S samples, as they showed CaO concentrations greater than 50%.

Martins *et al.*²⁴ evaluated the forms of waste generation within nine CBP also located in Belo Horizonte, Brazil. After collection, the residues were characterized and the nine samples of residual concrete slurry showed a predominant composition of SiO₂ (37-68%), followed by CaO (7-33%) and Al₂O₃ (2-12%). The authors explained that the difference in chemical composition is related to the origin of the aggregates. The predominance of SiO₂ suggests the use mainly of gneiss aggregates and, when the CaO concentration increases, it corresponds to a greater introduction of limestone aggregates, in addition to also being present in hydrated cement compounds. Thus, considering that CBP K, P and S have CaO as the main constituent, it can be inferred that the aggregates used in the original concrete are composed of limestone.

Unexplored applications

Song *et al.*⁴¹ point out the great potential of calciumbased solid waste in the microbiologically induced calcium carbonate precipitation (MICP). This technology is a type of biologically induced mineralization and has been extensively investigated in different applications such as wastewater treatment, removal and immobilization of toxic metals and radionuclides, soil remediation, restoration of building materials, fabrication of bioconcrete materials and carbon sequestration.⁴²⁻⁴⁵

Based on reviews of studies carried out with the MICP technology, Song *et al.*⁴¹ concluded that the use of different solid wastes rich in calcium can result in products with different properties and this is related, among other factors, to differences in calcium content. The higher the amount of calcium, the better the contribution to the MICP process. As

an example, the authors cited class C fly ash, which, due to their calcium content higher than class F (above 20% and lower than 20%, respectively), generate a MICP material with better compressive strength. Thus, based on the notes raised by the studies, it can be said that the cements sludges studied are promising materials in this technology and can lead to better results, compared to the waste used so far.

The wide availability of Ca-rich residues also draws the researchers' attention to the production of biodiesel, as they have high catalytic activity in the transesterification of oil, are renewable, non-toxic and safe to handle and store.⁴⁵ According to Zul *et al.*,⁴⁶ most studies focus on the reuse of eggshells and shells from different animals and the calcium content represents an influential characteristic in the biodiesel conversion rate. Correia *et al.*⁴⁷ obtained a conversion rate of 97.75% from calcined eggshells with a calcium content of 32.19%, in contrast to calcined crab residues (24.60% Ca) and 83.10% conversion process, using sunflower oil as raw material. Therefore, cementitious sludge can also be studied for this application, requiring an investigation regarding its crystalline structure and probable processing to obtain maximum CaO.

Bakshi *et al.*⁴⁸ presented another application for calcium-rich waste: reinforcement of polymeric composites for use in the construction sector. The authors used gypsum powder and marble powder in different concentrations in the manufacture of polypropylene and polyethylene composites. The materials contained, respectively, 29 and 30.8% of calcium in their composition and the samples that were composed of gypsum or marble showed less photodegradation and greater flexural strength with up to 30% incorporation. Considering that cementitious sludges have a composition that is even richer in calcium, the potential of the material for this application is highlighted, and may present superior performances with lower concentrations of incorporation.

Other promising applications are found in the literature, such as making ceramics with a high CaO content,⁴⁹ composite pigments,^{50,51} production of ecological fertilizers,^{52,53} reinforcement in metal matrix composites,⁵⁴ bone regeneration,^{55,56} among others. Therefore, the material explored in this study has attractive characteristics for different applications not yet explored, in addition to requiring further research in works already carried out, enabling the reinsertion of waste in industrial chains, saving natural resources and creating new sustainable materials.

Conclusions

The chemical compositions of the studied concrete slurry waste presented CaO, SiO_2 and Al_2O_3 as major

constituents and present in all analyzed samples. The highest average concentration of CaO was identified in CBP K, while the highest concentrations of SiO₂ and Al₂O₃ were found in CBP P and, in CBP S, only the P_2O_5 content was higher among the three CBP. Oxides and punctual elements were also identified, but in concentrations lower than 0.500% and correspond to NiO, As₂O₃, Sb₂O₃, I and BaO.

The analysis of variance showed that there are significant differences in the concentration of components between batching plants, months and collection points. This fact may occur due to the use of different materials in the manufacture of concrete (aggregates and cement) and also due to the geometric characteristics of the sedimentation tanks.

Comparison of the chemical composition of the collected residues with data from the literature revealed a similarity: CaO, SiO₂ and Al₂O₃ are predominant. However, the average concentration of CaO is higher in the waste slurry of the concrete plants in question and, consequently, if this material were reused in the same proposals by the authors, the products would possibly have different characteristics and performance.

The promising applications for residual concrete sludge showed that this material is attractive to different unexplored uses, in addition to the possibility of reuse in products within the CBP itself. The greater the amount of CaO, the better the contribution to the use in microbial precipitation of calcium carbonate, catalytic activity, reinforcement of polymeric composites, among other possibilities. Therefore, the valorization of this waste can still be promoted, filling gaps in the studies already carried out and expanding the reuse alternatives.

Acknowledgments

The authors would like to thank the Universidade Federal do Amazonas (UFAM), Fundação de Amparo à Pesquisa do Amazonas (FAPEAM) and Sistema Nacional de Laboratórios em Nanotecnologia (SisNANO), CNPq 442601/2019-0 for infrastructure and financial support. The authors also would like to thank the Laboratório de Síntese e Caracterização de Nanomateriais (LSCN), Ilum Laboratory belonging to the HUB research center (EST/UEA) and the Crowfoot group of X-Ray Methods for the use of infrastructure, equipment and materials.

References

- Xuan, D.; Zhan, B.; Poon, C. S.; Zheng, W.; J. Hazard. Mater. 2016, 312, 65. [Crossref]
- 2. Silva, D. O. F.: Reaproveitamento de Lamas Residuais do Processo de Fabricação do Concreto; MSc. Dissertation,

University of São Paulo, São Paulo, Brazil, 2016. [Link] accessed in February 2023

- Audo, M.; Mahieux, P.-Y.; Turcry, P.; Chateau, L.; Churlaud, C.; J. Cleaner Prod. 2018, 183, 153. [Crossref]
- Hossain, M. U.; Xuan, D.; Poon, C. S.; *Waste Manage*. 2017, 61, 397. [Crossref]
- Xuan, D.; Zhan, B.; Poon, C. S.; Zheng, W.; Constr. Build. Mater. 2016, 113, 664. [Crossref]
- Tang, P.; Xuan, D.; Cheng, H. W.; Poon, C. S.; Tsang, D. C. W.; *J. Hazard. Mater.* 2020, *381*, 120951. [Crossref]
- Tian, Q. B.; Wang, Y.; Xu, L. N.; Wang, X. H.; Gao, H.; *Rare Met. Mater. Eng.* 2007, *36*, 979. [Link] accessed in March 2023
- Yang, Z. X.; Ha, N. R.; Jang, M. S.; Hwang, K. H.; Jun, B. S.; Lee, J. K.; *Ceram. Trans.* 2010, 210, 221. [Crossref]
- Zhang, J.; Fujiwara, T.; *Transp. Res. Rec.* 2007, 2026, 54. [Crossref]
- Correia, S. L.; Souza, F. L.; Dienstmann, G.; Segadães, A. M.; Waste Manage. 2009, 29, 2886. [Crossref]
- Zervaki, M.; Leptokaridis, C.; Tsimas, S.; J. Sustain. Dev. Energy, Water Environ. Syst. 2013, 1, 152. [Crossref]
- Iizuka, A.; Sasaki, T.; Hongo, T.; Honma, M.; Hayakawa, Y.; Yamasaki, A.; Yanagisawa, Y.; *Ind. Eng. Chem. Res.* 2012, *51*, 11266. [Crossref]
- Iizuka, A.; Honma, M.; Hayakawa, Y.; Yamasaki, A.; Yanagisawa, Y.; *Kagaku kōgaku ronbunshū* 2012, 38, 129. [Crossref]
- Iizuka, A.; Sakai, Y.; Yamasaki, A.; Honma, M.; Hayakawa, Y.; Yanagisawa, Y.; *Ind. Eng. Chem. Res.* 2012, 51, 6099. [Crossref]
- Tsunashima, Y.; Iizuka, A.; Akimoto, J.; Hongo, T.; Yamasaki, A.; *Chem. Eng. J.* **2012**, 200-202, 338. [Crossref]
- Iizuka, A.; Takahashi, M.; Nakamura, T.; Yamasaki, A.; *Ind. Eng. Chem. Res.* 2014, *53*, 4046. [Crossref]
- 17. Yoo, J.; Shin, H.; Ji, S.; Metals 2018, 8, 666. [Crossref]
- Marguí, E.; Queralt, I.; Van Grieken, R. In *Encyclopedia of Analytical Chemistry*; John Wiley & Sons: Chichester, 2016, p. 1-25. [Crossref]
- Janssens, K.; van Grieken, R.; Non-Destructive Microanalysis of Cultural Heritage Materials, 1st ed.; Elsevier: Amsterdam, 2004.
- ZSX Spectrometer Status, version 5.74; Rigaku Corporation, Wilmington, USA, 2008.
- Schoon, J.; de Buysser, K.; van Driessche, I.; de Belie, N.; J. Mater. Civ. Eng. 2015, 27, 04014272. [Crossref]
- Zhao, C.; Liu, Y.; Ren, S.; Quan, J.; *Key Eng. Mater.* 2018, 768, 293. [Crossref]
- Marchon, D.; Flatt, R. J. In Science and Technology of Concrete Admixtures; Aïtcin, P.-C.; Flatt, R. J., eds.; Elsevier: Cambridge, 2016, p. 129. [Crossref]
- Martins, J. V.; Aguilar, M. T. P.; Garcia, D. C. S.; dos Santos, W. J.; *J. Mater. Res. Technol.* 2022, 20, 1157. [Crossref]

Guimarães et al.

- Matschullat, J.; Martins, G. C.; Enzweiler, J.; von Fromm, S. F.; van Leeuwen, J.; de Lima, R. M. B.; Schneider, M.; Zurba, K.; *J. Geochemical Explor.* 2020, *211*, 106433. [Crossref]
- da Costa, M. L.; Kern, D. C.; *J. Geochemical Explor.* 1999, 66, 369. [Crossref]
- Konhauser, K. O.; Fyfe, W. S.; Kronberg, B. I.; *Chem. Geol.* 1994, 111, 155. [Crossref]
- de Souza, J. J. L. L.; Fontes, M. P. F.; Gilkes, R.; *Rev. Bras. Ciênc. Solo* 2018, 42, 170192. [Crossref]
- Horbe, A. M. C.; de Paiva, M. R. P.; Motta, M. B.; Horbe, M. A.; *Acta Amazonica* 2007, *37*, 81. [Crossref]
- Shetaya, W. H.; Young, S. D.; Watts, M. J.; Ander, E. L.; Bailey, E. H.; *Geochim. Cosmochim. Acta* 2012, 77, 457. [Crossref]
- Pichel, N.; Vivar, M.; J. Trace Elem. Med. Biol. 2017, 42, 32. [Crossref]
- Hoppee Filho, J.; Gobbi, A.; Pereira, E.; Quarcioni, V. A.; de Medeiros, M. H. F.; *Matéria* 2017, 22, e11872. [Crossref]
- 33. La Serna, H. A.; Rezende, M. M.; Agregados para a Construção Civil, https://www.gov.br/anm/pt-br/centrais-de-conteudo/ publicacoes/serie-estatisticas-e-economia-mineral/outraspublicacoes-1/8-1-2013-agregados-minerais, accessed in March 2023.
- 34. de Luna, F. D. T.; da Silva, A. G.; Vianna Jr., A. S.; Open J. Fluid Dyn. 2020, 10, 164. [Crossref]
- Al-Sammarraee, M.; Chan, A.; *Chem. Eng. J.* 2009, *152*, 315. [Crossref]
- Al-Sammarraee, M.; Chan, A.; Salim, S. M.; Mahabaleswar, U. S.; *Chem. Eng. J.* 2009, *152*, 307. [Crossref]
- Chatveera, B.; Lertwattanaruk, P.; Makul, N.; Cem. Concr. Compos. 2006, 28, 441. [Crossref]
- Audo, M.; Mahieux, P.-Y.; Turcry, P.; Constr. Build. Mater. 2016, 112, 790. [Crossref]
- Tang, P.; Xuan, D.; Poon, C. S.; Tsang, D. C. W.; J. Hazard. Mater. 2019, 368, 689. [Crossref]
- Martins, J. V.; Garcia, D. C. S.; Aguilar, M. T. P.; dos Santos, W. J.; *Constr. Build. Mater.* 2021, 303, 124519. [Crossref]

- Song, M.; Ju, T.; Meng, Y.; Han, S.; Lin, L.; Jiang, J.; Chemosphere 2022, 290, 133229. [Crossref]
- Seifan, M.; Samani, A. K.; Berenjian, A.; Appl. Microbiol. Biotechnol. 2016, 100, 2591. [Crossref]
- Kumari, D.; Qian, X.-Y.; Pan, X.; Achal, V.; Li, Q.; Gadd, G. M.; *Adv. Appl. Microbiol.* **2016**, *94*, 79. [Crossref]
- Xu, J.; Wang, X.; Wang, B.; *Appl. Microbiol. Biotechnol.* 2018, 102, 3121. [Crossref]
- Marwaha, A.; Rosha, P.; Mohapatra, S. K.; Mahla, S. K.; Dhir, A.; *Fuel Process. Technol.* **2018**, *181*, 175. [Crossref]
- Zul, N. A.; Ganesan, S.; Hamidon, T. S.; Oh, W.-D.; Hussin, M. H.; J. Environ. Chem. Eng. 2021, 9, 105741. [Crossref]
- Correia, L. M.; Saboya, R. M. A.; de Sousa Campelo, N.; Cecilia, J. A.; Rodríguez-Castellón, E.; Cavalcante, C. L.; Vieira, R. S.; *Bioresour. Technol.* 2014, *151*, 207. [Crossref]
- Bakshi, P.; Pappu, A.; Bharti, D. K.; Patidar, R.; *Polym. Degrad. Stab.* **2021**, *192*, 109694. [Crossref]
- Zhao, L.; Li, Y.; Zhou, Y.; Cang, D.; *Mater. Des.* 2014, 64, 608. [Crossref]
- Ahmed, N. M.; Fathi, A. M.; Mohamed, M. G.; Abd El-Gawad, W. M.; *Prog. Org. Coatings* **2020**, *140*, 105530. [Crossref]
- Zamani, A.; Eavani, S.; Rafiee, E.; J. Cleaner Prod. 2021, 304, 127103. [Crossref]
- Borges, R.; Soares Giroto, A.; Klaic, R.; Wypych, F.; Ribeiro, C.; *Adv. Powder Technol.* 2021, *32*, 4070. [Crossref]
- Amine Laadila, M.; LeBihan, Y.; Caron, R. F.; Vaneeckhaute, C.; Waste Manage. 2021, 120, 125. [Crossref]
- Ror, C. K.; Tejyan, S.; Kumar, N.; *Mater. Today Proc.* 2022, 60, 1120. [Crossref]
- Li, X.; Yang, X.; Liu, X.; He, W.; Huang, Q.; Li, S.; Feng, Q.; Prog. Nat. Sci.: Mater. Int. 2018, 28, 598. [Crossref]
- Dejob, L.; Toury, B.; Tadier, S.; Grémillard, L.; Gaillard, C.; Salles, V.; *Acta Biomater.* 2021, *123*, 123. [Crossref]

Submitted: July 27, 2022 Published online: March 14, 2023