Metallurgy and materials Metalurgia e materiais

Blocks for civil construction made with the sediment deposited in the Candonga dam

http://dx.doi.org/10.1590/0370-44672017720198

Mavare de Souza Moura Maciel Machado¹ Ana Maria Matildes Santos^{2,3} Carolina Braccini Freire^{2,4} Ana Cláudia Pinto Dabés Guimarães^{2,5} Fernando Soares Lameiras^{2,6}

¹Universidade Federal de Ouro Preto - UFOP, Programa de Pós-Graduação em Engenharia de Materiais da REDEMAT. Ouro Preto - Minas Gerais - Brasil. mayaremaciel@gmail.com

²Centro de Desenvolvimento da Tecnologia Nuclear -CDTN, Comissão Nacional de Energia Nuclear - CNEN Belo Horizonte - Minas Gerais - Brasil

E-mails: amms@cdtn.br3, cbf@cdtn.br4, anadabes@gmail.com5, fsl@cdtn.br6

Abstract

The use of the sediment deposited in the Candonga dam after the collapse of the Fundão dam is being considered as raw material for civil construction. Blocks were obtained by adding 5 % of sodium carbonate and 13 % of water to the dried dredged sediment, which was mixed and heat treated at 700 °C and cooled slowly inside the oven to room temperature. The water absorption of the blocks was in the 12-16 % range, the loss of mass in water was (0.01 ± 0.01) %, the density was (1.95 ± 0.03) g/cm³, the compression strength was (11.05 ± 1.29) MPa (reference value ≥ 1.7 MPa), and the flexural strength was (4.42±0.69) MPa (reference value ≥ 3.5 MPa). Small cracks and pores, as well as the growth of white spots (efflorescence) were observed on the surface of the blocks, which can be avoided or attenuated by improving the compaction pressure and the heat treatment.

Keywords: Candonga, dredged sediment, blocks, civil construction, fluxing, heat treatment.

1. Introduction

After the collapse of the Fundão dam (Morgerstem et al., 2016), on November 5, 2015, in Mariana, Minas Gerais, Brazil, 9 million tons of tailings from desliming and flotation of iron ore concentration processing are estimated to be deposited in the Candonga dam. Candonga is the artificial lake of the Risoleta Neves hydroelectric power plant, located in the municipality of Rio Doce in the state of Minas Gerais. These tail-

ings are being dredged out and stored in a nearby location by the Renova Foundation (TTAC, 2016). Its use as raw material for manufacturing products for civil construction (Gama et al., 2015; Carrasco et al., 2013) is being considered to promote an alternative economic activity for the communities affected by the accident.

The use of the tailings as fine aggregate in Portland cement products is limited, due to their fine granulometry (Pereira Júnior, 2011), (Melo, 2012), (Freire, 2012), (Versieux, 2014). The manufacturing of products of clay ceramics with the addition of the dredged sediment is also limited in the region because there is no clay production or distribution in the vicinities. The dredged sediment is also contaminated with organic matter, such as tree branches, roots, and leaves. The coarser pieces of organic matter can be removed by hand, but the finer pieces require sieving, which can be problematic due to water consumption and pollution (wet sieving) or dust emission (dry sieving).

X-ray diffraction analyses showed that the dredged sediment is composed of quartz (80 wt%), iron oxide (19 wt%), and clays (1 wt%). In order to produce a monolithic block, an alternative route is

proposed with the addition of a fluxing for quartz. By heating, the surfaces of the quartz particles are melted and adhere to each other. By cooling, a vitreous phase is obtained that joins the quartz particles and gives mechanical strength to the block. The particles of iron oxide and clay are also adhered to in this vitreous phase. The organic matter is burnt during the heating. The objective of this paper is to present the factors (parameters) that influence on the manufacturing of a block like this and its characterization (water absorption, loss of mass in water, density, compression strength, flexural strength, and microstructure).

2. Materials and methods

Five hundred kilograms of dredged sediment was collected in the Candonga dam. They were exposed to the sun during five days to dry. The coarser organic matter was removed by hand. The homogenization of the material was done by the quartering technique with the long pile method and the homogenized dredged sediment was packed in 7 kg plastic bags. The composition of the dredged sediment was measured by X-rays diffraction and the particle size distribution was determined by laser diffraction (CILAS, 1190). An image of the particle morphology was also obtained by field emission scanning electron microscopy (FE-SEM, JEOL, JSM-5610).

Sodium carbonate usually employed in the glass and detergent industries, manufacturing other chemicals and pH control of swimming pools was used as the fluxing agent dissolved in water. The sodium carbonate solution was poured on the dredged sediment and mixed to obtain a paste. The paste was put in a metallic mould and pressed in a manual hydraulic press (SKAY, 98 kN). After demoulding, the pressed blocks were put in an oven for heat treatment.

Preliminary studies were performed to establish the factors for obtaining the blocks and their range of values. Based on a given mass of dredged sediment, it was observed that the quantity of sodium carbonate should be in the 1 – 5 wt% range and of water in the 10 – 15 wt% range. Compaction pressures at 1.2 and 2.4 MPa were tested. The pressing at 2.4 MPa produced blocks with uniform surface by visual inspection. The heat treatment was performed at a heating rate of 10 °C/min, 2 hours at the specified temperature, and

natural cooling of the oven. The blocks were removed from the oven at room temperature. With the heat treatments at 600 °C and 650 °C the blocks presented efflorescence (white spots on the block surface). At temperatures higher than 800 °C no efflorescence was observed. Based on these results, a 2³ factorial design with two replicates were performed according to Table 1. The compaction pressure was fixed at 2.4 MPa. The experiments were performed in random order. Sixteen blocks were produced and characterized (water absorption, density, and compression strength).

The factors were optimized based on the results of this factorial design and 30 blocks were produced and characterized (water absorption, loss of mass under water, density, compression strength, flexural strength, and microstructure).

| Factor | Levels | Values | | |
|----------------------------------------|--------|--------|--|--|
| Codi | -1 | 2 | | |
| Sodium carbonate content (wt%) | +1 | 5 | | |
| \\\\at\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\ | -1 | 13 | | |
| Water content (wt%) | +1 | 15 | | |
| Town out to us (0C) | -1 | 800 | | |
| Temperature (°C) | +1 | 900 | | |

Water absorption was measured by drying the blocks in a kiln at 120 °C during 24 hours, weighing the dried blocks at room temperature, immersing them in water at room temperature during 24 hours, removing excess water with a flannel, and weighing the block to calculate the mass gain (ABNT NBR 8491). The mass loss was measured by drying the blocks under the same conditions after the measurement

of water absorption and comparing the

weights before and after the immersion

in water. The density was calculated with

the dried mass of the samples and by taking their dimensions with a ruler (for the preliminary tests) or a pachymeter. For the measurement of the compression strength, the samples were capped with sulfur to get uniform and parallel faces. The compression was performed in a press with a charge cell of 294 kN and 9.8 N resolution (Emic, PC 150/200I). The flexural strength was measured in a universal press machine of 100 kN (Instron, 5882) according to the three-point bend test. Samples were cut from the blocks produced with the opti-

Table 1
2³ factorial design to study
the obtaining of blocks made with
the dredged sediment of Candonga dam.

mized factors to measure the compression strength (two pieces for the first 15 blocks) and flexural strength (one piece for the last 15 blocks). Microstructure images of the blocks were taken with SEM and material compositions at the different regions were identified by Energy-Dispersive X-ray Spectrometry-EDS coupled to FE-SEM. The white spots on the block surface were collected by careful scraping and analyzed by Fourier Transform Infrared (FTIR) dispersed in KBr (Bomem, MB102, resolution of 4cm⁻¹, transmittance mode).

3. Results and discussion

Figure 1 shows the particle size distribution of the Candonga dredged sedi-

ment. It is finer than the specified ranges of sand for Portland cement products.

Figure 2 shows the morphologies of the particles of the dredged sediment.

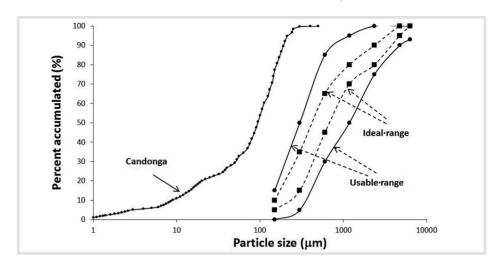


Figure 1
Particle size distribution of the
Candonga dredged sediment compared
to the size ranges of sand for Portland
cement products (ABNT NBR 7211).

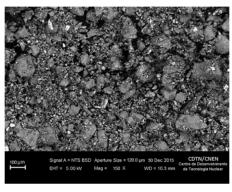


Figure 2 SEM image of the Candonga dredged sediment. The coarser particles are quartz and the finer particles are iron oxides and clays.

The results of the 2³ factorial design are shown in Table 2. Due to problems during the execution of the experiments, the run (+1,-1,-1) was performed with three replicates for the water absorption and

density and two replicates for the compression strength, and the (-1,+1,-1) run was performed with only one replicate for the water absorption and no results for the density and compression strength. This design

is not orthogonal, so that attention should be given to the confoundments between the effects and the interactions. Table 3 shows the calculation of the effects and interactions obtained with Minitab® 17.

| SCC | WC | Temperature | W _{abs} (%) | | | | Density (g/cm³) | | | | CS (MPa) | | | | |
|-----|----|-------------|----------------------|-------|-------|-------|-----------------|------|------|------|----------|-----|-----|-----|--|
| -1 | -1 | -1 | 26. | 26.49 | | 25.86 | | 1.41 | | 1.48 | | 1.8 | | 0.7 | |
| +1 | -1 | -1 | 27.66 | 19 | .45 | 21.26 | 1.44 | 1. | 57 | 1.53 | - | 5. | .6 | 6.8 | |
| -1 | +1 | -1 | 15. | .90 | | - | - | | - | | - | | - | | |
| +1 | +1 | -1 | 15.99 | | 14.95 | | 1.60 | | 2.02 | | 4.8 | | 7.1 | | |
| -1 | -1 | +1 | 26.80 | | 26.74 | | 1.52 | | 1.51 | | 0.7 | | 0.4 | | |
| +1 | -1 | +1 | 15.48 | | 19 | 9.81 | 1.68 | | 1.60 | | 3.4 | | 4.6 | | |
| -1 | +1 | +1 | 18.39 | | 14 | 1.51 | 1.87 | | 1.99 | | 1. | 4 | 7 | 7.8 | |
| +1 | +1 | +1 | 17.91 | | 17 | 7.71 | 1.70 | | 2.05 | | 3. | .5 | 8 | 3.1 | |

Table 2 Results of the 2³ factorial design of blocks made with the Candonga dredged sediment.

SCC: sodium carbonate content; WC: water content; W_{abs} : water absorption; CS: compression strength

| Factor or | W _{abs} | | | | Density | | CS | | | |
|-------------|------------------|------|-------|-------|----------|-------|-------|------|-------|--|
| Interaction | Coef. | SD | Р | Coef. | SD | Р | Coef. | SD | Р | |
| Constant | 19.88 | 0.68 | 0.000 | 1.71 | 0.05 | 0.000 | 3.95 | 0.80 | 0.002 | |
| SCC | -1.45 | | 0.066 | 0.00 | | 0.948 | 1.54 | | 0.096 | |
| WC | -3.47 | | 0.001 | 0.18 | | 0.007 | 0.95 | | 0.273 | |
| Temperature | -0.21 | | 0.769 | 0.03 | | 0.532 | -0.21 | | 0.798 | |
| SCC*WC | 1.68 | | 0.039 | -0.05 | | 0.410 | -0.56 | | 0.504 | |
| SCC*Temp | -0.49 | | 0.489 | 0.01 | | 0.782 | -0.38 | | 0.653 | |
| WC*Temp | 0.93 | | 0.210 | -0.01 | | 0.764 | 0.51 | | 0.542 | |
| SC*WCC*Temp | 0.94 | | 0.205 | c | onfounde | d | c | d | | |

Table 3 The factors effects of the Table 2 design calculated with Minitab® 17.

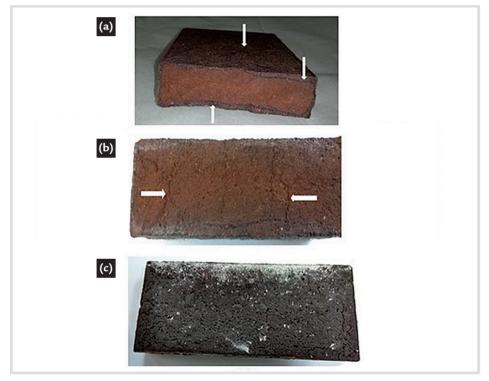
"*" means interaction between the factors;

the value of the factors effects is the double of the coefficient; SD: standard deviation; p: p-value

Considering a significance level of 0.10, the sodium carbonate content decreases the water absorption, has no significant effect on the density, and increases the compression strength. The water content also decreases the water absorption, but increases the density, and has no significant effect on the compression strength. The temperature has no significant effect on the water absorption, density, and compression strength. Tests in the 600 °C - 900 °C range are necessary to confirm the influence of the temperature. The interaction between the sodium carbonate and water contents is significant for the water absorption. The interaction between the sodium carbonate content and the temperature has no significance, as well as the interaction between the water content and the temperature. The three factor interaction could only be calculated for the water absorption and it is not significant.

The effect of the water content can be explained by the greater easiness of mixing to obtain the paste caused by water, which wets the surface of a larger number of quartz particles with the sodium carbonate solution and produces a more glass phase with less pores. With a more concentrated sodium carbonate solution, it is expected that a more glass phase be formed. But the lack of significance of the temperature was not expected. By observing a fractured cross section of a typical block in the

Figure 3a, a glassy layer around the block can be visualized (see the arrows), whereas the inner region (Figure 3a and Figure 3b) has no glassy aspect. Cracks can be seen in the inner region (Figure 3b). The outer layer and the inner regions of the blocks were analyzed by X-ray diffraction. The composition of these regions is practically the same, but there is a more amorphous phase in the outer layer. Being less dense, the glassy phase seems to move to the surface and the crystalline phases to the inner region. This effect could be an explanation for the lack of influence of the temperature on the results of Table 2. The efflorescence (white spots on the surface) is shown in the Figure 3c.



legal standard for the manufacture of the block developed here, in this table reference values are placed based on already existing standards.

rescence, an intermediate temperature of 700 °C for the heat treatment was chosen. The content of sodium carbon-The water absorption data are not ate was fixed at 5 % to obtain a more normally distributed according to the glassy phase and higher mechanical Anderson-Darling test. Another factor strength. The water content was fixed besides the random experimental errors at 13 % to obtain lower densities. The should be influencing the absorption of water absorption would in principle be water. By comparison, with the visual higher at this level of water content, inspection results, it can be seen that but with less segregation of the glass the more pores and cracks are observed, phase, it is expected that the water the greater the water absorption. These absorption should decrease. Moreover, blocks also have the characteristics the demoulding of the pressed block shown in Figures 3a and 3b, with an is easier at a water content of 13 %. outer layer, which is darker and more Table 4 shows the results of the characcohesive, and an inner layer of lighter terization of the 30 blocks made under coloration, which can be partially the optimized conditions. As there is no scrapped by friction with the finger. It

(efflorescence) on the block surface.

seems that this less cohesive region contributes more to the absorption of water. If there are more pores and cracks, it can be more connected to the surface of the block, contributing to the increase of

Some aspects of a typical block: a) cross section showing a glassy layer; b) internal region with small cracks and c) white spots

Figure 3

block, contributing to the increase of water absorption. The water absorption was in the range of 12 to 16 %, which is less than the range observed in Table 2.

The loss of mass was measured to

The loss of mass was measured to verify if there is dissolution of the glass phase in the water. The data is normally distributed, if one considers the "not detected" cases as zero. One calculates that the mean loss of mass is (0.01±0.01) % with a significance level of 0.05. The density data is normally distributed and one calculates a mean density of (1.95±0.03) g/cm³ with a significance level of 0.05.

In order to attenuate the segrega-

tion of the glass phase and the efflo-

Table 4 Characterization of 30 blocks made under optimal conditions.

| Run | | Visual inspection | | | | | Loss of mass (%) | Density (g/cm³) | CS (I | FS (MPa) | |
|-----|----|-------------------|----|----|---|-------|------------------|-----------------|--------|----------|--------|
| | Cs | Ef | Fp | Мр | Р | * ≤22 | - | - | * >1.7 | | **≥3.5 |
| 1 | х | х | | | | 13.64 | n.d. | 1.94 | 8.61 | 10.87 | - |
| 2 | х | х | | | | 12.60 | n.d. | 2.08 | 8.15 | 11.45 | - |
| 3 | х | х | | х | | 12.47 | n.d. | 1.94 | 12.34 | 6.45 | - |
| 4 | х | х | | х | | 12.63 | n.d. | 2.04 | 5.90 | 7.88 | - |
| 5 | х | | | | х | 12.33 | n.d. | 1.95 | 7.81 | 14.15 | - |
| 6 | х | х | | | х | 12.13 | n.d. | 1.97 | 12.20 | 11.99 | - |
| 7 | х | | | | х | 11.89 | n.d. | 2.01 | 15.85 | 12.38 | - |
| 8 | | x(spots) | х | | | 11.82 | n.d. | 2.00 | 18.89 | 18.33 | - |
| 9 | х | х | х | | | 12.24 | 0.04 | 2.03 | 8.62 | 11.60 | - |
| 10 | х | х | х | | | 12.76 | 0.01 | 2.07 | 7.62 | 6.05 | - |
| 11 | х | | | х | | 13.33 | n.d. | 1.95 | 11.86 | 10.82 | - |
| 12 | х | x | х | | | 13.72 | 0.03 | 1.90 | 12.17 | 12.69 | - |
| 13 | х | х | | | х | 13.76 | n.d. | 1.95 | 9.18 | 12.49 | - |
| 14 | х | х | | х | | 12.47 | 0.07 | 2.02 | 12.35 | 9.28 | - |
| 15 | х | х | х | | | 14.57 | -0.01 | 1.88 | 10.90 | 12.54 | - |
| 16 | х | х | х | | | 14.21 | 0.01 | 1.86 | - | - | 5.39 |
| 17 | | | х | | | 11.78 | 0.03 | 1.96 | - | - | 7.46 |
| 18 | х | х | х | | | 12.25 | 0.01 | 1.97 | - | - | 5.81 |
| 19 | х | | | х | | 13.50 | -0.01 | 1.90 | - | - | 4.85 |
| 20 | х | х | | х | | 14.80 | 0.01 | 1.84 | - | - | 5.14 |
| 21 | х | х | х | | | 14.33 | 0.01 | 1.87 | - | - | 5.63 |
| 22 | х | х | х | | | 14.53 | -0.01 | 1.86 | - | - | 5.90 |
| 23 | х | х | х | | | 15.17 | 0.01 | 1.82 | - | - | 4.32 |
| 24 | х | х | | х | | 14.07 | 0.01 | 1.92 | - | - | 4.65 |
| 25 | х | х | | х | | 16.33 | 0.03 | 1.80 | - | - | 3.37 |
| 26 | х | х | х | | | 12.12 | -0.01 | 1.95 | - | - | 3.26 |
| 27 | х | х | | | х | 12.09 | -0.04 | 1.99 | - | - | 2.45 |
| 28 | | | х | | | 11.85 | 0.03 | 1.99 | - | - | 2.40 |
| 29 | х | х | | х | | 13.02 | 0.01 | 1.96 | - | - | 2.32 |
| 30 | х | х | х | | | 12.61 | 0.03 | 2.01 | - | - | 3.40 |

Cs: cracks on the surface; P: pores; Fp: few pores; Mp: many pores; Ef: efflorescence in one side; n.d.: not detected;

 $W_{abs}; water \, absorption; \, CS: \, compression \, strength; \, FS: \, flexural \, strength. \, *reference \, value \, (ABNT \, NBR \, 8491); \, **reference \, value \, (ABNT \, NBR \, 9457). \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457). \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, value \, (ABNT \, NBR \, 9457); \, **reference \, val$

The Anderson-Darling test revealed that the data for the compressive strength are not strictly normally distributed. However, considering the value of p = 0.097 obtained in this test, the mean value for the compressive strength of (11.05 ± 1.29) MPa was calculated with a significance level of 0.10. The data for the flexural strength are normally distributed, so that one calculates a mean value of (4.42 ± 0.69) MPa

with a significance level of 0.10. These values were higher than those required by legal standards for solid ceramic bricks and hydraulic tiles, respectively, ABNT NBR 8491 and ABNT NBR 9457. In these standards, the compressive strength should be at least equal to 1.7 MPa and the flexural strength greater than or equal to 3.5 MPa.

Figure 4 shows a SEM image of a typical microstructure of the blocks.

Four regions can be seen. According to EDS measurements, the white particles are hematite, the light grey phase around the quartz particles are the glass phase (composed of Si, O, Fe, Al, and Na), the gray particles is quartz, and the dark grey regions are pores, that were filled with resin in the preparation of the polished cross section. As expected, a glass phase was formed around the quartz particles joining them together.

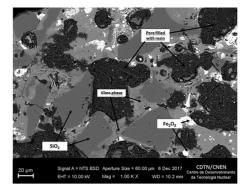


Figure 4
SEM image of the microstructure of a typical block.

Figure 5 shows the FTIR spectra of the material collected from the white spots of the blocks. It seems to be a mixture of sodium carbonate and

sodium metasilicate pentahydrate. Sodium carbonate is formed by the exposure of sodium ions to humidity and CO₂ from the atmosphere. Sodium metasilicate pentahydrate is formed by the exposition of sodium silicate to atmospheric humidity.

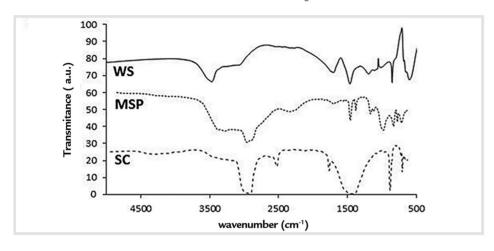


Figure 5
FTIR spectrum of white
spots (WS) on the surface of the
blocks (resolution 4 cm⁻¹) compared
to the ones of sodium carbonate (SC)
and sodium metasilicate pentahydrate
(SMP) from the NIST Standard Reference
Database 69: NIST Chemistry WebBook.

4. Conclusion

Blocks can be obtained with the Condonga dam dredged sediment using a quartz flux dissolved in water, mixing, pressing, heating and cooled to room temperature. A glass layer forms around the quartz particles that give sufficient mechanical strength to the block to be used in civil construction. The residual organic

matter is burnt during the heating. Blocks with 5 % sodium carbonate and 13 % water added to the dry dredged sediment, with heating at 700 °C, were obtained. The water absorption is in the 12-16 % range, the loss of mass in water is (0.01 ± 0.01) %, the density is (1.95 ± 0.03) g/cm³, the compression resistance is (11.05 ± 1.29) MPa,

and the flexural resistance is (4.42±0.69) MPa. Small cracks, pores and growth of white spots (efflorescence) on the surface of the blocks were observed. The improvement of the moulding, compactation pressure, and demoulding, as well as the adjustment of the heat treatment program can avoid or attenuate these defects.

Acknowledgments

To FAPEMIG, for the financial support, to CDTN for the laboratorial

infrastructure, to REDEMAT/UFOP for the academic support, and to FUNDEP/

UFMG for the support to Mayare S. M. M. Machado.

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Received: 28 December 2017 - Accepted: 16 July 2018.