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# CLAY BRICK WASTE: NEW FILLER TO NATURAL RUBBER COMPOSITE

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**Abstract:** The development of urban cities increases the amount of construction and demolition waste, such as ceramic materials, mainly clay bricks, and consequently, generates cost for the management and environmental disposal due to the few routes to recycle or reuse them. Here, it is proposed a new approach to reuse clay bricks waste (CB) composed by red ceramic as reinforcement filler to natural rubber (NR) composites. It was verified that the residue is mostly composed of silicon, which is a filler widely used in rubber industry as SiO2, to mechanically reinforce composites. Tensile strength showed an increment around 16% when 20 phr of CB waste was added to the natural rubber (reaching 12,4 MPa). In addition, composites with 10 phr of waste showed great abrasion resistance and the hardness property increased as CB waste was added. The results indicate that the residue can be used as a possible filler in natural rubber products.

**Keywords:** clay bricks; composites; natural rubber; reuse.



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## INTRODUCTION

The development of urban cities has generated large amounts of construction and demolition waste. Every year, it is estimated the production of 3 billion tons of construction and demolition waste around the world (Akhtar, 2018), being 85% related to concrete, ceramics and masonry wastes. Based on the classification of NBR 10004/04 (NBR standard, 2004), this kind of residue is framed on 2B class i.e. inert (Monier, 2018). However, the high amount of residues became an environmental issue inferring costs to management and landfill disposal. Thus, clay bricks waste are intensively studied for the replacement of fine aggregate (partly or totally) in cementitious (Amin, 2015), mortar (Dang, 2018) and concrete materials (Zong, 2014). Currently, several researches evaluated the effectiveness of hybrid-type binders of a red clay brick waste production, which reduced around 30% of Portland cement and showed great compressive strengths (102 MPa at 28 days) (Robayo-Salazar,2017). The substitution of fine aggregates was also studied in concrete properties, with good replacement levels (10%, 20%, and 30%). The results also revealed similar density values around 2400-2500 kg/m³ and a decrease in early-age strength (Mohammed, 2017).

As an alternative method to reuse and recycle most industrial and organic wastes, the application of polymeric composites with residues as reinforcement fillers has grown interest among the researchers. Composites using pineapple fiber (Pittayavinai, 2016), sugarcane bagasse fiber (Paiva, 2018), jute fiber (Huang, 2017), leather waste (Garcia, 2015), rice husk ash (Pongdong, 2016), blast furnace slag (Patnaik, 2018), phosphogypsum (Essabir, 2017), glass fiber (Novais, 2017), marble (Fiore, 2018), and mineral wool (Väntsi, 2014), have been widely reported. Among the polymers, research involving the use of natural rubber for artificial muscles (Hawkes, 2016), microfluidic matrix (Cabrera, 2014), antibacterial composites (Chen, 2018), green synthesis (Cabrera, 2013) and, solvent-sensitive memory materials (Dong, 2016) as well as the recycling of residues as fillers (Barrera, 2016) and (Zefeng, 2018) make the elastomer an interesting way to reuse clay brick residues. Here, we demonstrate a new approach to reuse clay bricks waste (CB), composed by red ceramic, as a reinforcement filler to natural rubber (NR) composites. The mechanical, morphological and structural properties of the composites were investigated in order to avoid incorrect disposal and to generate a new eco-friendly material.

## MATERIALS AND METHODS

Clay brick waste was collected from a construction residue in the city of Presidente Prudente, São Paulo, Brazil. Natural rubber of Crepe Brazilian Clear (CCB) type was provided by DLP Industry and commerce of rubber and artifacts, being used as the polymer matrix with Mooney viscosity higher than 98. Zinc oxide was provided by LABSYNTH Laboratory Products, Diadema, SP, Brazil and stearic acid by Jand Chemistry Industry and Commerce of Products Chemistry Ltda, Jandira, SP, Brazil, both used as activators. The accelerators dibenzothiazole disulfide was provided by Shandong Shanxian Chemical Co. Ltd., Shanxian, Shandong Province, China and tetramethylthiuram monosulfide was purchased from Zhejiang Huangyan Zhedong Rubber Auxiliary Co., Ltd., Huangyan Laobei Road, Zhejiang Province, China. The cure agent, sulfur, was supplied by Jand Chemistry Industry and Commerce of Products Chemistry Ltda, Jandira, SP, Brazil. Paraffinic oil was purchased from FRAGON Products for rubber industry.

### Composites preparation

The residue was previously milled to reduce the particle size. After, the clay brick waste was dried in an oven for 24 h at 80 °C and sieved into 170 mesh to obtain fine particles (around 88 μm). The composites were produced by mixing CB waste into the polymer matrix of natural rubber, varying residue ratios of 10-40 parts per hundred rubber (phr). To carry out the comparison, a sample of vulcanized natural rubber without filler was prepared. The mixture was prepared in two stages in an open chamber mixer (Makintec, model 379) with a friction ratio of 1.0:1.25. The curing agents are given in **Table 1**.

**Table 1.** Composites formulation (phr).

| Materials | Composites Formulation | | | | |
|---|---|---|---|---|---|
| | NR | $NR/CB_{10}$ | $NR/CB_{20}$ | $NR/CB_{30}$ | $NR/CB_{40}$ |
| Natural Rubber (NR) | 100 | 100 | 100 | 100 | 100 |
| Stearic Acid | 2.0 | 2.0 | 2.0 | 2.0 | 2.0 |
| Oxide Zinc | 5.0 | 5.0 | 5.0 | 5.0 | 5.0 |
| Paraffinic Oil | 3.5 | 3.5 | 3.5 | 3.5 | 3.5 |
| Clay Brick Waste (CB) | 0 | 10 | 20 | 30 | 40 |
| MBTS[a] | 0.17 | 0.17 | 0.17 | 0.17 | 0.17 |
| TMTM[b] | 0.46 | 0.46 | 0.46 | 0.46 | 0.46 |
| Sulfur | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |

[a] Dibenzthiazyl disulphide; [b] Tetramethylthiuram monosulfide

In the first stage, the natural rubber was processed by 10 minutes around 65 °C. Then, zinc oxide and stearic acid, the vulcanization activators, were added and stirred for approximately 15 minutes until complete homogenization. Moreover, after the first stage completion, samples were stored at room temperature for 24 hours to allow the formation of zinc stearate that promote the reaction between the accelerators and curing agent. After that, the second stage begins with the incorporation of paraffinic oil, clay brick waste, the accelerators Dibenzothiazole disulfide and Tetramethylthiuram monosulfide and sulfur. The homogenization process was carried out around 25 minutes, with 5 minutes for each additive.

## MATERIALS CHARACTERIZATION

The samples were vulcanized in a rheometer from Team Equipment Ltda, Brazil, with oscillating disk 1°and isothermal temperature at 150 °C in accordance with ASTM D 2084 (ASTM, 2012). The mechanical properties were evaluated in triplicates. The abrasion resistance test was performed with a cylindrical sample with diameter of 16.0 ± 0.2 mm and thickness of 6.0 mm in a rotating cylinder from MAQTEST, with frequency rotation of 40 cycles min-1, according to ASTM D 5963 (ASTM, 2010). The cylinder has a diameter of 150 mm with nominal distance for abrasion of 40 meters and abrasive paper with P60 grit size and aluminum oxide grain. Hardness tests were performed using a Kiltler durometer graduated in Shore A scale, according to the ASTM D 2240 standard (ASTM, 2010). The strain-stress test was investigated in type C samples using a universal testing machine EMIC model DT500 at 500 mm min-1 with load cell of 100 kgf, to analyze the strength and elongation at break according to ASTM D-412 (ASTM, 2013).

The microscopic images were obtained by a scanning electron microscope (SEM, ZEISS, Model EVO LS15). A gold deposition was previously performed on the samples by Sputter in a Quorum Equipment, Model Q150TE. The particles sizes were measured using ImageJ @ program. X-ray fluorescence was performed by Dairix company with a Rigaku equipment, model Supermini 200 and power of 200 W.

The structural characterization of the elastomers composites was studied by Fourier Transform Infrared Spectroscopy (FTIR), in the region of 4000-500 cm-1, with an accuracy of 2 cm-1 and 24 scans, in a Bruker Model Vector 22 spectrophotometer. The composite surface was analyzed directly through the attenuated total reflectance (ATR) technique.

The crosslink density of the composites was evaluated by the swelling technique. First, the samples were weighed approximately 0.25 ± 0.05 g and immersed in toluene for five days. Then, the samples were removed from toluene, surface-dried on absorbent paper, and weighed. Finally, the samples were dried in an oven at 60 °C for 24 h and weighed again. The crosslink density was calculated according to Equation (1), by Flory-Rehner method (Vieyres, 2013) and (Flory, 1943).

$$\frac{1}{(2\mathrm{Mc})} = \frac{-(\ln(1-Vb)+Vb+X(Vb)^2)}{(pb)(V_0)(Vb^{\frac{1}{3}}-\frac{Vb}{2})} \quad (1)$$

where Vb is the volume fraction of the polymer in the swollen gel at equilibrium, X is the polymer-solvent interaction parameter, pb is the density of the polymer, and V0 is the molar volume of the solvent.

## RESULTS AND DISCUSSIONS

**Table 2** presents the chemical components of CB waste analyzed by X-Ray Fluorescence. A typical composition of sedimentary clay which is used to produce red ceramics is observed. In accordance with the literature, there is a predominance of silicon and aluminum, in addition, the presence of calcium, iron, titanium and potassium (Pinheiro, 2010). **Figure 1** shows SEM images of CB waste with 170 mesh where it is observed an agglomerate of fine particles with irregular profile and rough surface.

**Table 3** shows the rheometric results. The increase of CB waste leads to a slight increase of the minimum (ML) and maximum torques (MH), but to a significant decrease in ts2 and t90.

**Table 2.** Chemical components of CB waste analyzed by X-Ray Fluorescence.

| X-Ray Fluorescence | |
|---|---|
| Component | % m/m |
| Si | 55.90 |
| Al | 25.08 |
| Fe | 9.60 |
| Ca | 3.25 |
| Ti | 2.42 |
| K | 2.07 |
| Others | 1.68 |

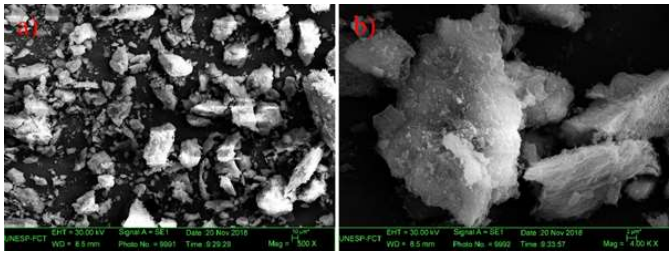

**Fig. 1** Scanning electronic microscope (SEM) images of CB waste at amplifications of (a) 500X and (b) 4000X.

**Table 3.** Rheometric parameters of NR compared to composites of NR with Clay Brick Waste (CB).

| Samples | $M_L$ (dN.m) | $M_H$ (dN.m) | ΔM (dN.m) | $t_{s2}$ (min) | $t_{90}$ (min) |
|---|---|---|---|---|---|
| NR | 1.00 | 14.50 | 13.30 | 4.51 | 6.13 |
| 10 phr | 1.00 | 13.10 | 12.10 | 3.25 | 4.50 |
| 20 phr | 1.20 | 14.80 | 13.80 | 2.57 | 4.25 |
| 30 phr | 1.20 | 15.10 | 13.90 | 2.34 | 4.01 |
| 40 phr | 1.20 | 16.30 | 15.10 | 2.22 | 3.50 |

Minimum torque presents a slight increment with the increase of filler loading, mainly for 20 phr, which indicates that the processability of the compounds becomes a little more difficult due to the enhanced viscosity and stiffness. In addition, this increase may be attributed to the agglomeration of filler particles in the polymeric matrix. For low loading mass, such as 10 phr, a better dispersion of the filler occurs, and the probability of aggregates formation is lower when compared to higher amounts of filler (Sareena, 2012).

Maximum torque increases with the increase of CB residue, mainly for 20 phr. The increase of maximum torque by the addition of the residue is an expected effect and is related to the increase of the stiffness of the polymeric matrix after vulcanization. This higher stiffness may be attributed to the presence of the residue and the formation of a higher number of cross-links (chemical or physical) (Escócio, 2003).

Crosslink density increases as the amount of clay brick waste is added to the samples, Fig. 2. The reduction in swelling when compared to natural rubber without waste evidence the interaction between rubber and the CB waste particles. However, compositions with 30 and 40 phr of CB waste exhibit a decrease in properties, such as tensile strength and abrasion resistance, while crosslink density continuously increases. In this case, the enhancement of crosslink density with the increase of CB waste content has also been attributed to the restriction of rubber swelling caused by the filler and not effectively by the increase of crosslinking (Oliveira, 2014). The variation between the maximum and minimum torques (ΔM) indicates that the degree of reinforcement increases proportionally to the incorporation of the filler CB waste (Santos, 2014).

The reduction of ts2 and t90 can be related to the fact that CB waste behaved as a vulcanizing agent in natural rubber leading to an increase of the elastomer vulcanization rate. It may contribute to an earlier and faster process of natural rubber vulcanization when compared to natural rubber without waste. It means that in the industry, less energy is spent in the vulcanization process, saving costs (López-Manchado, 2003).

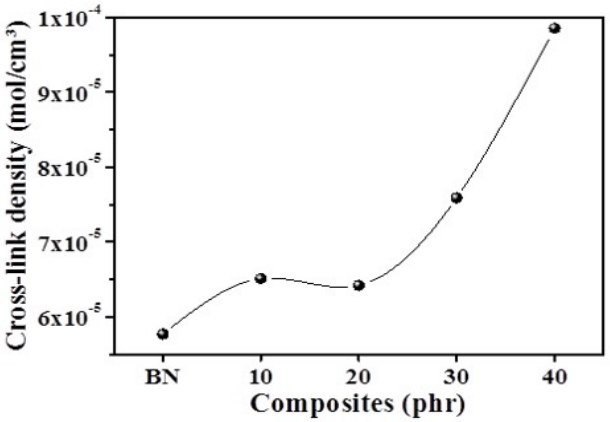


**Fig. 2.** Crosslink density of composites with Clay Brick waste.

**Table 4** presents the values of tensile strength and strain at break of natural rubber and composites with CB residue. It is observed that natural rubber presents tensile strength value around 10.39 MPa while composites with 10 and 20 phr of residue show a reinforcement of the polymeric matrix reaching values around 11.23 and 12.41 MPa, respectively. This result indicates a suitable dispersion of the filler in the polymeric matrix. Composites with 30 and 40 phr exhibit similar values to the natural rubber, 10.71 and 9.85 MPa, respectively. It can be attributed to the formation of aggregates, where the particle-particle interaction force, i.e., cohesion force, is greater than the interaction force between particle-polymer, also called cohesion (Navarro, 1997). A slight increase of the strain values at break is observed as an amount of waste is added until 30 phr. This is attributed to charge-load interactions, which cause uneven surface and volume in the composites and, consequently, increase hardness. On the other hand, it reduces tensile strength and deform the matrix.

**Figure 3** shows the values of abrasion loss of natural rubber and composites with Clay Brick waste. Abrasion loss decreases with the addition of CB waste until 10 phr (168.22 mm3), increasing the abrasion resistance of the composites. This result can be attributed to the mechanical reinforcement of the filler and a well

**Table 4.** Tensile strength and strain values at break of natural rubber and composites with CB waste.

| Composites | Tensile strength (MPa) | Strain at break (%) |
|---|---|---|
| NR | 10.39 ± 0.59 | 681.00 ± 37.00 |
| 10 phr | 11.23 ± 0.80 | 687.50 ± 12.50 |
| 20 phr | 12.41 ± 0.07 | 712.50 ± 12.50 |
| 30 phr | 10.71 ± 0.05 | 732.50 ± 7.50 |
| 40 phr | 9.85 ± 0.17 | 725.00 ± 0.00 |

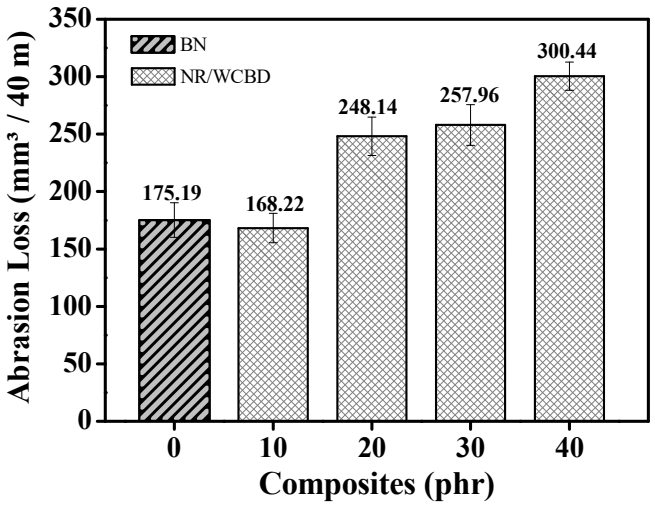


**Fig. 3.** Abrasion loss of NR and composites with Clay Brick waste.

dispersion of the residue particles that are embedded in the polymeric matrix. However, the enhancement of mass loss for 20 phr (248.14 mm3) is due to the generation of aggregates that create a tension surface on the composites and, consequently, decreases the abrasion resistance (Mousa, 2013).

**Figure 4** shows the hardness values of natural rubber and composites. It is observed that the hardness values increase proportionally to the addition of CB residue. This is associated to the increase of cross-links formed during the vulcanization process (Santos, 2015) and the presence of inorganic compound in the CB waste residue with a rigid nature (Hundiwale, 2002).

**Figure 5** presents the SEM images of composites incorporated with CB waste. As the waste concentration increases, the amount of dispersed particles of waste also increases, when compared to natural rubber without residue. FT-IR spectra of vulcanized natural rubber and the composite with 40 phr of CB are demonstrated in Fig. 6. There is no evidence of new peaks that characteristic of composites with physical interaction.

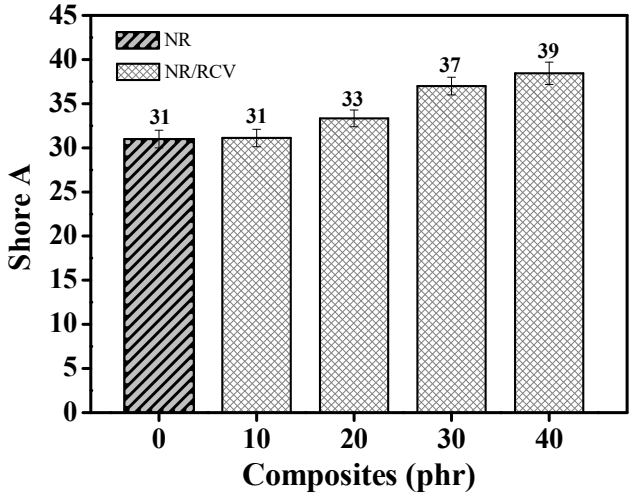


**Fig. 4.** Hardness of NR and composites with Clay Brick waste.

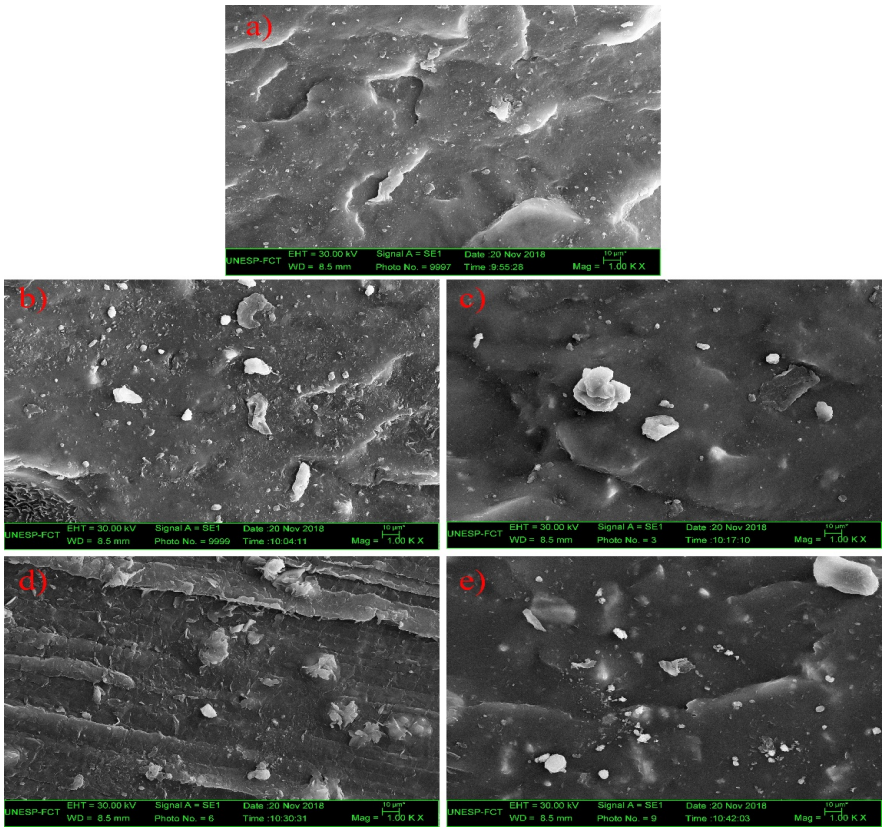


**Fig. 5**. Transversal section of SEM images of NR (a) and NR/CB 10 phr (a), 20 phr (b), 30 phr (c) and 40 phr (d).

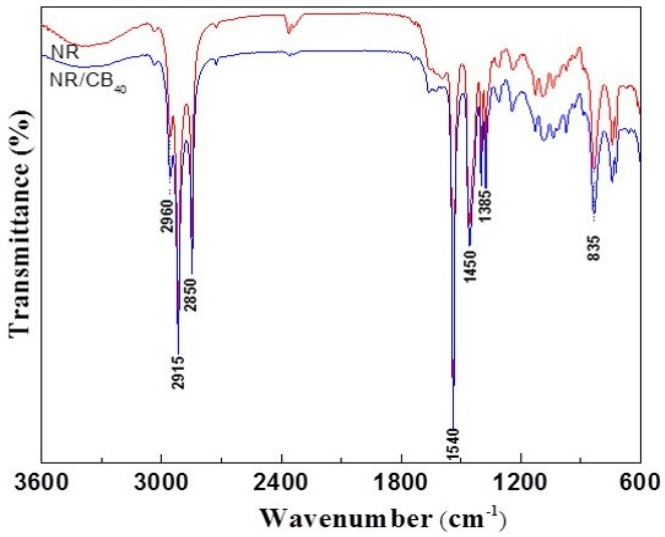


**Fig. 6.** FT-IR of NR and $NR/CB_{40}$.

FT-IR spectra show three bands at 2960 cm-1, 2915 cm-1 and 2850 cm-1 attributed to CH3 asymmetric stretching modes, asymmetric stretching and symmetric vibrations of methyl groups, respectively (Agostini, 2008). The stretching of C=C bonds is seen at 1540 cm-1 and the band at 1450 cm-1 is assigned to CH3 deformation mode. The functional groups of the rubber present bands between 1385 and 950 cm-1. The region between 1385-1150 cm-1 is attributed to the twisting and wagging of CH2 and the region between 1150 cm-1 and 950 cm-1 is assigned to CH3 rocking vibration of the unsaturated hydrocarbons. The presence of a band at 835 cm-1 is attributed to CH out-of-plane bending (Dall'Antonia, 2009) and (Nallasamy, 2004).

## CONCLUSIONS

The influence of CB waste addition in natural rubber was verified and demonstrated that clay bricks can be used as a filler. The decrease of ts2 and t90 shows that CB waste contributes to the natural rubber vulcanization which starts earlier and it is faster when compared to the natural rubber without waste. The waste presents high concentration of silicon and its incorporation in the natural rubber contributes to the increase of tensile strength in 16% when 20 phr is added. In addition, composites with 10 phr of waste showed abrasion resistance higher than the natural rubber.

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