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Influence of brick waste microparticles as reinforcement of a bioepoxy based composite

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Abstract

This research proposes the use of micrometric sintered clay particles as reinforcement for composite materials with a renewable thermosetting polymeric matrix. For this purpose, H10 brick shards were collected in the municipality of Pamplona - Norte de Santander. Then, a reduction of their size was carried out until the sintered clay microparticles and their distribution of grain sizes was obtained. For the manufacture of the composite material, the cast moulding technique was used. Samples of it were obtained, using SUPER SAP CLR bioepoxy resin as matrix and microparticles smaller than 75 microns were used as reinforcement. Reinforcement percentages at 0 and 10% by weight were used. For comparison purposes, the material was manufactured with a commercial resin matrix designated P-2002. Subsequently, tensile and bending tests were carried out under the conditions of the ASTM D638 and ASTM D790 standards, respectively. The destructive tests showed a linear-elastic behaviour and a favourable influence by the sintered clay reinforcement on the mechanical properties of both materials. Finally, micrographs of the composites were taken using the scanning electron microscopy technique to determine the material's failure mechanism. In conclusion, the reinforcement of sintered clay microparticles makes for a promising option to reinforce renewable thermoset matrices.

Keywords: Composites, thermoset resins, sintered clay, mechanical properties.

I. INTRODUCTION

The applications of polymeric matrix composites are growing exponentially, together with the exorbitant expectations from customers [1]. This is mainly due to its improved properties that provide superior performance in terms of specific strength, toughness, rigidity, corrosion resistance, and resistance to chemical attacks [2, 3]. In addition, the manufacturing methods of this type of materials provide production lines with low complexity procedures and greater profitability margins in contrast to the manufacturing processes of traditional materials [4].

On one hand, thermosetting resins are a very important component in a wide range of manufacture applications such as: plasticizers, coatings, adhesives, electronics, home construction, and in composite material matrices. However, most resins use petroleum as a raw material, including epoxies and unsaturated polyesters [5, 6]. Therefore, these resins are not biodegradable and cause dangerous emissions. In 50-60 years they will no longer be synthesized due to oil shortage, also their derivative products are very difficult to manage or recycle, they will remain in landfills for decades [7, 8].

Consequently, alternative and ecological thermoset resins made from renewable resources have caught the attention of researchers and have risen as a viable substitute for polymers based on petrochemical products. [7]. In the case of this research, one of its purposes is to compare two commercial thermosetting resins (traditional one and a biodegradable one): the P-2002 polyester resin from Industria de Resinas SAS, and the SUPER SAP® CLR bioepoxy system from Entropy Resins.

On the other hand, with the acceleration of urban construction and the improvement of buildings large amounts of clay brick waste have been produced, these are considered the second most used construction material after concrete. Clay bricks that do not meet quality standards at the end of the production line as well as shards collected in construction and demolition sites are treated as waste [9, 10]. As a result, millions of tons of waste are produced which represent a serious environmental problem because disposal of these is carried out in open fields, this causes pollution in the form of accumulation and particulate waste dispersion problems [11].

Therefore, it is of great importance to explore the potential of recycling and reusing brick waste not only because it is considered an environmentally friendly alternative, but because it helps to reduce dependence on natural raw materials, the harmful effects on the environment, and the high amount of waste while promoting sustainable development for the construction industry in the Norte de Santander region. This is why this research seeks to propose the use of micrometric sintered clay particles obtained from the collection and processing of H10 brick shards accumulated in the material deposits of the Pamplona municipality as thermosetting polymer matrix composite materials reinforcement.

II. METHODOLOGY

II.I Materials

As proposed material a bioepoxy resin was used, reference Super Sap® CLR and hardener Super Sap® CLF,

commercialized by the Entropy Resins company. This resin is an ultraviolet stabilized epoxy system and has an ideal viscosity for a wide range of applications.

A commercial polyester resin was used to study the proposed material's behaviour. The selected matrix was a pre-accelerated orthophthalic resin marketed under reference P-2002 by the Industria de Resinas SAS company. The manufacturer recommends the use of MEK peroxide (methyl ethyl ketone), as a catalyst.

As reinforcement, sintered clay microparticles were used in percentages of 0 and 10% by weight for the mixture with each thermosetting matrix. The grain size used was less than 75 microns (<200 mesh sieve). To obtain the waste, brick shards were collected in the municipality of Pamplona - Norte de Santander. Then, a mechanical grain size reduction was performed. Finally, the #40, #60, #80, #100, #140, and #200 mesh sieves were used under the ASTM C136 / 136M [12] standard conditions to obtain the sintered clay microparticles (Fig. 1).

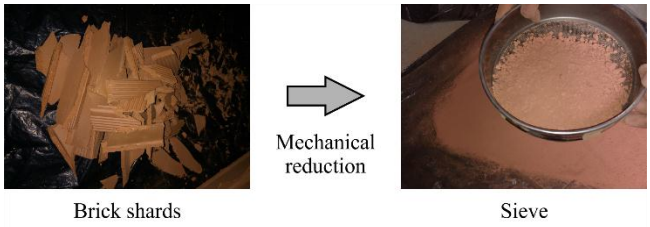

Fig.1 BRICK WASTE SIZE REDUCTION PROCESS.

II.II 2.2. Manufacturing process

Samples of the composite materials were manufactured using percentages of 0 and 10% by reinforcement weight and the cast moulding technique. In general, the following configurations were manufactured:

- **BER:** 100/33 CLR bioepoxy resin/CLF hardener with 0% sintered clay by weight.
- **BER-SC:** 100/33 CLR bioepoxy resin/CLF hardener with 10% sintered clay by weight <75 microns.
- **PR:** 99/1 P-2002/MEK peroxide resin with 0% sintered clay by weight.
- **PR-SC:** 99/1 of P-2002/MEK peroxide resin with 10% sintered clay by weight <75 microns.

Importantly, the sintered clay microparticles were dried at 100°C for 24 hours before the casting process. Then, the resin was heated to a temperature of 50°C, the microparticles were added, and a homogeneous mixture was obtained. Finally, the mixture was moulded at 30°C and left to reticulate for 7 days under room pressure and temperature conditions before the destructive tests (Fig. 2).

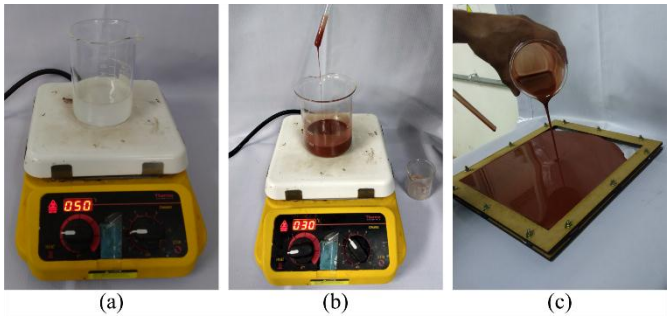

Fig.2 MANUFACTURING PROCESS: (a) HEATING THE RESIN. (b) ADDING AND MIXING THE SINTERED CLAY. (c) CAST MOULDING.

II.III Destructive tests

The tensile test was carried out under the ASTM D638 standard [13]. Default values were used for Type I specimens with a gauge length of 50mm, test section width of 13mm, and thickness of 5mm. The speed of load application was 5 mm/min.

The three-point bending test was carried out following the ASTM D790 standard conditions [14]. The dimensions of a flat specimen of 150x13x5 mm (Length x Width x Thickness) were used, with a distance between supports or span of 80 mm. Similarly, the speed on which the load was applied was established at a value of 2.13 mm/min due to the geometry of the piece and the use of procedure A of the aforementioned standard. Both tests were carried out using the SHIMADZU UH-600KN universal testing machine.

II.IV Scanning electron microscopy (SEM)

The surface fracture obtained in the bending test of the PR-SCP sample was analysed using a TESCAN scanning electron microscope, MIRA 3 FEG-SEM model. The sample was plated with a gold to improve electrical conductivity.

III. RESULTS AND DISCUSSION

III.I Granulometric distribution of sintered clay

In Fig. 3 the granulometric distribution of the sintered clay is shown. As can be seen, there was a greater quantity of particles with a size greater than 425 microns (51.35%) with a high number of impurities. Even in particles with sizes greater than 180 microns, significant amounts of them were detected. Consequently, almost 53% of the sintered clay powder was unsuitable for use as reinforcement from a macro point of view. On the other hand, 2.82% of particles smaller than 75 microns were obtained. Therefore, the particles with a size smaller than 75 microns were third with regarding their concentration and respecting grain sizes smaller than 180 microns that apparently did not have any impurities.

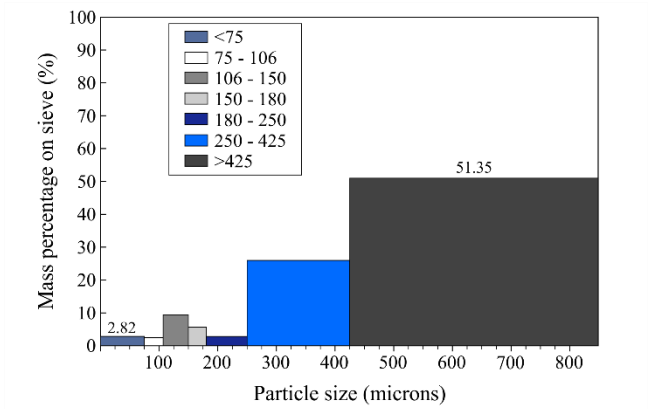

Fig.3 PARTICLE SIZE DISTRIBUTION OF SINTERES CLAY.

III.II Tensile test properties

Table 1 shows the results obtained in the tensile test comparison. The sintered clay reinforced polyester resin obtained a Young's modulus of 0.36 ± 0.15 GPa, meaning a decrease in this property respecting the unreinforced polyester resin. However, the tensile strength and deformation increased to 40.34 ± 5.64 MPa and 11.37 ± 1.60% respectively. This shows an improvement in the mechanical properties of the polyester resin when reinforced with sintered clay, but a decrease in its rigidity.

Table 1 COMPARISON OF TENSILE TEST RESULTS.

Material	Young's modulus (GPa)	Tensile strength (MPa)	Tesnile strain (%)
PR	0.53±0.12	33.19±3.97	7.68±1.60
PR-SC	0.36±0.15	40.34±5.64	11.37±1.53
BER	0.45±0.14	16.15±2.72	3.15±0.74
BER-SC	0.45±0.26	32.55±4.95	6.11±2.22

On the other hand, the sintered clay reinforced bioepoxy resin obtained a Young's modulus of 0.45 ± 0.26 GPa whose value is similar to that of the unreinforced bioepoxy resin. Likewise, the tensile strength and deformation of the reinforced bioepoxy resin was higher than that of the unreinforced resin with values of 32.55 ± 4.95 MPa and 6.11 ± 2.22% respectively. This shows that the sintered clay reinforcement contributed positively to the increase of all mechanical properties in this particular case.

In Fig. 4 the tensile mechanical behaviour comparison is shown and described in the stress-strain graph obtained from a sample from each of the tested materials. In the case of materials with bioepoxy resin, the line's slope is very similar, which allows to verify the stiffness' quantitative results. In the case of materials with polyester resin, the change in the slope between the standard resin and the reinforced one is evident, and therefore, there is an increase in its rigidity when adding the particulate reinforcement.

It is important to highlight that the behaviour of all the materials was linear elastic, with an increase in mechanical properties due to the addition of 10% micrometric particles of sintered clay by weight. In addition, the best attraction properties obtained were those obtained by the polyester resin composite material reinforced with micrometric particles of sintered clay.

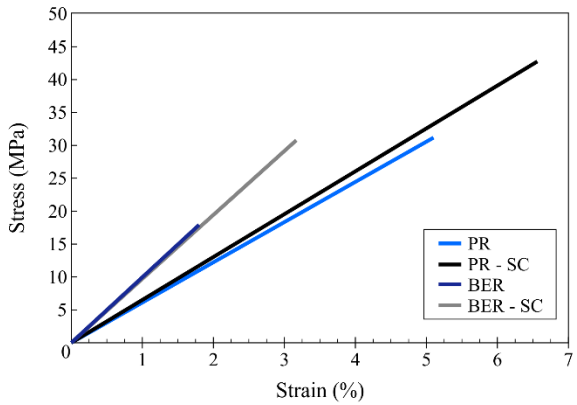

Fig.4 TENSILE MECHANICAL BEHAVIOUR.

III.III Flexural test properties

Table 2 shows the results obtained in the bending test comparison. The polyester resin reinforced with sintered clay obtained a flexural modulus of 1.20 ± 0.56 GPa and a deformation of 5.49 ± 1.29%, meaning a decrease in these two properties respecting the unreinforced polyester resin. However, the flexural strength increased to 58,545.34 ± 37.41 MPa. There is considerable results dispersion in these tests, demonstrated by the exorbitant values in the standard deviation for each of the three bending mechanical properties obtained in the destructive tests.

On the other hand, the sintered clay reinforced bioepoxy resin obtained flexural modulus of 5.46 ± 1.86 GPa showing an increase in this property respecting the unreinforced bioepoxy resin. The flexural strength and deformation of the BER-SC composite decreased to 23.32 ± 4.63 MPa and 0.50 ± 0.04%, respectively. In this case, the particulate reinforcement impaired the bending mechanical properties values, this conclusion was made taking the data obtained by the bioepoxy resin without reinforcement as reference.

Table 2 COMPARISON OF FLEXURAL TEST RESULTS.

Material	Flexural modulus (GPa)	Flexural strength (MPa)	Flexural strain (%)
PR	1.61±0.67	55.79±18.90	6.23±0.34
PR-SC	1.20±0.56	58.55±37.40	5.49±1.29
BER	4.68±0.62	26.78±5.51	0.67±0.29
BER-SC	5.46±1.86	23.32±4.63	0.50±0.04

In Fig. 5 the bending mechanical behaviour comparison is shown and described in the stress-strain graph obtained from a representative sample for each of the materials tested. For both composite materials (PR-SC and BER-SC), there was a clear decrease in flexural performance due to the addition of the particulate reinforcement. It is important to highlight that the

behaviour of all the materials was of a linear elastic type, and that the best properties obtained in flexion were those obtained by the polyester resin without reinforcement.

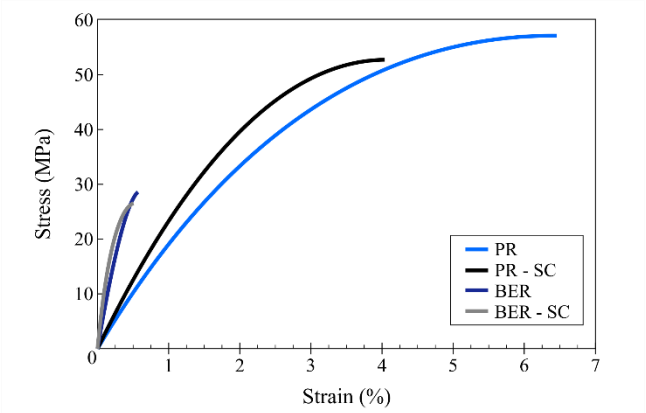

Fig.5 FLEXURAL MECHANICAL BEHAVIOUR.

III.IV Analysis of morphology

Fig. 6 shows the surface fracture micrographs in the specimens of Polyester Resin and Bio-Epoxy Resin with sintered clay (PR-SC and BER-SC, respectively) reinforcements. The polyester resin-based composite presented cavities caused by trapped gases from the reticulation process. However, it presented good particle dispersion with uniform sizes. On the other hand, the bioepoxy resin-based composite exhibited variable particle sizes embedded in areas with acceptable dispersion.

Consequently, it is appreciated that the bioepoxy resin generates fewer surface defects than the polyester resin, and therefore, a lower amount of stress accumulators. However, the variable sizes present in the composite with bioepoxy resin, which can be agglomerate formations due to the higher viscosity of this resin, also behave as stress accumulators. In any case, these defects only explain the possible fracture starting and propagating zones. Furthermore, regardless of the defects present, the mechanical properties of both resins increased with the addition of sintered clay microparticles.

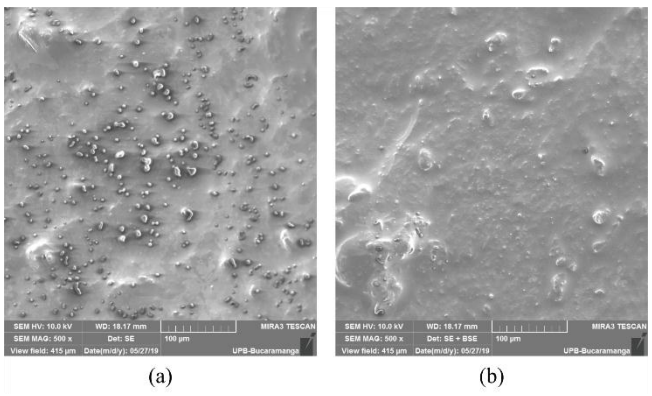

Fig.6 SEM MICROGRAPH FOR FRACTURE SURFACE: (a) PR - CS (b) BER - SC.

IV. CONCLUSIONS

The micrometric particles of sintered clay were obtained, by means of a brick shard reduction process. From this process, a granulometric distribution was obtained, here 51.35% of the sieved samples showed impurities at the macro level, the use of particles with a size greater than 425 microns was immediately ruled out. The grain size used as reinforcement for the thermoset matrices (<75 microns) is the third-highest concentration, with 2.82%.

The tensile tests results showed low dispersion results. Similarly, the addition of sintered clay reinforcement increased the mechanical properties of the thermosetting resins under study. In addition, the mechanical behaviour of composite material remained linear elastic, which is why it is directly influenced by thermosetting matrices. Finally, the material that obtained the best tensile mechanical properties was polyester resin reinforced with micrometric sintered clay particles.

The bending tests results showed a high results dispersion, particularly in materials based on polyester resin. On the other hand, the addition of the reinforcement led to a decrease in the mechanical properties of the unreinforced thermosetting resins. Therefore, the material with the best flexural properties was unreinforced polyester resin. Once more, the linear elastic behaviour was kept regardless of the particulate reinforcement.

The SEM micrographs reflected the presence of stress accumulators in both composites: polyester resin and bioepoxy. In the polyester resin, cavities were presented as a result of the gases trapped in the reticulation process, and in the bioepoxy resin, particle agglomerates with larger sizes than those observed in the rest of the surface appeared. This helps explain the fractures in the materials studied. In addition, the increase in mechanical properties after adding 10% of sintered clay microparticles is noteworthy.

Bioepoxy resin is makes for an acceptable candidate for sintered clay particle reinforcement. It is recommended to improve the manufacturing process and decrease the amount of catalyst to explore higher values in mechanical properties. It should be noted that the viscosity of the resin is higher, compared to polyester resin. Therefore, caution regarding the processing temperatures must be exercised to bring it to an acceptable viscosity that may allow the sintered clay microparticles to be mixed with the material.

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