Structure, mechanical and thermal behaviour of mixtures of polyester resin and dental ceramic waste

G Peña Rodríguez¹, I Martínez Maldonado¹ and H J Dulce Moreno¹
¹ Universidad Francisco de Paula Santander, San José de Cúcuta, Colombia.

E-mail: gpenaro@ufps.edu.co, lore_3612@hotmail.com

Abstract. The tensile strength and bending strength, structure and thermal behaviour of mixtures of polyester resin (P-2000) and powders (ASTM sieve 200, <75μm) of dental ceramic wastes (dentals impressions, alginate and gypsum) was reported. The samples consisted of mixtures with percentage weights of 50-50%, 60-40%, 70-30%, 80-20%, 90-10%, where the resin was the majority phase, the Mekp (4% wt) was used as catalyst. The structure was studied using SEM and XRD, the thermal behaviour using DSC, TGA and DMA, while the mechanical strength was tested using standards ASTM D790 and D638. Irregular morphology and presence of small agglomerations was observed, with particle sizes between 29.63 and 38.67μm, the presence of different phases of calcium sulphate was found, and that to the increasing the concentration of the powder, the materials becomes more crystalline, increasing its density. An average service temperature of 69.15±4.60°C was found. Vickers hardness values are reported in ranges from 18.65 to 27.96. Considering the elastic modules was established that the materials become more rigid by having more powder concentration.

1. Introduction
In the manufacture of dental prosthesis, dentists use alginate and gypsum for dental impressions of patients, which once used is discarded following the protocols manual integrated waste management in each country. In Colombia, there is no knowledge of the recycling of these dental impressions, while in countries such as Greece, Brazil and Jordan among others, there are studies of the composition of dental solid waste, with the aim of recycling potential [1-3].

Thermosetting resin products are some of the most adaptive materials in the marketplace and hundreds of industries, for its easy handling and shaping. In recent years it has been used as filling material recycled silicon oxide powders, aluminium oxide, rice husk, wood powders, among others, in order to find new materials for technological applications [4-6].

Therefore, the structural, thermal and mechanical characterization, of mixtures of polyester resins and waste of alginate powders and gypsum used in dentistry are reported.

2. Materials and methods
The pre-accelerated unsaturated polyester resin (P-2000) was supplied by Industries of Resins S.A.S (Medellin Colombia), which is commonly used in non-aggressive environments, pink colour, with approximately 65% of polyester. For curing, Mekp catalyst was used in concentration of 4% by weight, being gelation time at room temperature of 20 minutes, with a maximum exothermic temperature of approximately 165°C.

After obtaining dental impressions discarded of gypsum and alginate (see Figure 1), were subjected to autoclaving at pressure of 15 pounds, temperature of 121±2°C for one hour, then manually grinding...
was performed, followed by trituration using hammer mill then sieved on a 200 ASTM sieve (≤75 microns). With the above powders, a mixture was prepared with 50% by weight of gypsum and 50% alginate, which was used as filler in mixtures with polyester resin, the following mixtures defined percentage by weight: 50-50, 60-40, 70-30, 80-20 and 90-10, where the majority phase was the resin and the minority the powder mixture of gypsum and alginate. There was established a time of 10 minutes of constant mixing without the catalyst and then a time frame of 2 minutes of constant mixing with the catalyst. The moulding process was then strained in an aluminium mould with the dimensions according to the standards used.

**Figure 1.** a) Dental gypsum impressions, (b) Dental alginate impressions, (c) Powders gypsum, (d) Powders alginate. Fount: authors.

The morphology and structure of the samples were studied using the scanning electron microscope (SEM) FEI Quanta 650 FEG, and the X-ray diffractometer (XRD) Bruker D8 Discover with DaVinc geometry. Thermal behaviour is performed using thermogravimetric balance Discovery series TA Instruments under nitrogen purge gas flow of 50ml/min, temperature range 30°C to 1000°C and heating rate 10°C/min, while the differential scanning calorimetry (DSC) was performed according to ASTM E2160 for which was used a DSC of the same series and marks in a temperature range of 25°C to 500°C with 10°C/min temperature ramp. For the dynamic mechanical analysis there was used a DMA Q800 TA Instruments equipment, with temperature ranges between -145°C and 600°C and ramps up to 20°C/min. The effective thermal conductivity ($K$) at room temperature was determined using the KD2 Pro® system, and the SH-1 sensor, system operating with the physical principle of linear transient heat flow.

### 3. Analysis and discussion of results

From analysis using SEM images, for alginate, an irregular morphology was found with the presence of diatomic particles of circular shape with diameters between 15.4±0.8 and 17.06±0.76 microns, which are found in Chrysophyta and correspond to algae, Bacillariophyceae class, which are part of inorganic composition of alginate [8], while gypsum powder, consists of very fine particles of different geometries. Microanalysis, showed that sodium alginate is kind, moreover, gypsum main elements were found as calcium and sulphur, results that match those reported in references [9-10].

In Figure 2 the SEM micrograph at 500X for 70-30% mixture is presented, the morphology of alginate and gypsum particles is irregular with average sizes between 11.5 and 74.83µm, immersed in the resin matrix. Microanalysis by EDS-SEM, shows that as the concentration of filler in the sample increases, the weight percentage of carbon and oxygen decrease, while the sodium, aluminium, silicon and calcium increase.

Qualitative analysis using XRD (see Figure 3), evidence the presence of hydrated calcium sulfate, Cristobalite low, Anhydrita, Quartz, Chantalita, Basinita and Dolomite. Also, it is observed that as concentration of the filler (powdered gypsum and alginate) to the sample increases, the crystallinity increases, which is evidenced by the decrease of the curvature at the base of the diffraction patterns [11].

The decrease in curves thermogravimetry (TG), allow to infer a sample decomposition in a simple process, *ie*, the material undergoes mass loss, where the first drop in the curve corresponds to evaporation catalyst [12], a proportional relationship between the amount of resin present in the
composition and weight loss is also shown, since at these temperatures the matrix phase is tending to undergo changes [13]. It was found that the glass transition temperature (Tg) increases with increasing concentration of filler [14], reporting an average value of 69.15±4.596°C Tg, very close to the value reported in the literature [15], the lack of significant variability of Tg, is based on that it is a function of the degree of cure. In Figure 4, the behaviour of the heat capacity of the sample 70-30wt%, wherein the observed endothermic processes to 108.30°C and 364.50°C respectively is presented. With regard to dynamic mechanical analysis (DMA) increased elastic modulus with increasing concentration of filler, from 1917MPa for the sample with 0% filler, to 4845MPa for the sample with 50% wt of plaster dust is evident and alginate, showing a higher thermo-mechanical added to the filler material [16].

Figure 2. SEM micrograph at 500X using the sample 70-30% wt.

Figure 3. Patterns XRD of different mixtures.

Figure 4. DSC graph corresponding to 70-30% mix.

Table 1. Thermal conductivity, hardness, elastic modulus and maximum stress of the samples.
| Mixture | $K$ (W/m°C) | Hardness Vickers | Elastic Modulus (MPa) | Maximum Stress (MPa) |
|---------|-------------|------------------|----------------------|--------------------|
| 100     | 0.1635 ± 0.001 | 22.35 ± 1.29     | 1534.22              | 110.000            |
| 90-10   | 0.1887 ± 0.004 | 18.65 ± 1.84     | 1751.74              | 63.893             |
| 80-20   | 0.2132 ± 0.009 | 21.21 ± 0.78     | 2168.18              | 57.494             |
| 70-30   | 0.2506 ± 0.016 | 21.15 ± 1.62     | 2193.71              | 47.952             |
| 60-40   | 0.2810 ± 0.006 | 26.26 ± 1.33     | 2818.14              | 45.912             |
| 50-50   | 0.3105 ± 0.004 | 27.96 ± 0.98     | 2595.96              | 42.802             |

From the results of Table 1, an increase in the effective thermal conductivity ($K$), the hardness and elastic modulus in tension is seen, with increasing proportion of powders, while the maximum stress in bending decreases with increasing material filler, which can be attributed to the decrease in the proportion of resin in the material, since it is responsible for granting flexibility compound.

4. Conclusions

It was developed and characterized a material based on polyester resin, which was used as material powder filling dental waste of dental impressions, which are added in weight percentage of 50, 40, 30, 20 and 10% to resin matrix. The morphology and structure of the sample reported a heterogeneous size distribution of particles with average sizes ranging between 11.50 and 74.83 microns, with the presence of hydrated calcium sulphate, Cristobalite low Anhydrita, Quartz, Chantalita and basanita, the crystallinity increases with increasing filler concentration. The thermal behaviour was found that the average temperature was $69.15 ± 4.60 ºC$, also the thermal conductivity, the hardness and elastic modulus in tension is increased with increasing concentration of filler, while the maximum stress in bending decreases. This work contributes to the mitigation of environmental impact by recycling dental solid waste, to be used as fillers in composite materials based on polyester resins.

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