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Thermal and mechanical study from granite and marble industry reject

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Abstract

Granite and marble residue from dimension stone companies is solid, not degradable and its composition is principally of silicates (quartz, plagioclase, orthoclase, and mica). These companies produce more than 150,000 t per year of residue, which is put in a decant lagoon or a large landfill area, thus resulting in environment pollution. The objective of this work is to investigate the possibility of using residue to obtain ceramic material. For that purpose, thermal analysis has shown to be a great tool to predict the process conditions such as sintering, temperature, heating rate and reaction to manufacture of ceramic elements. The thermal behavior was observed on a TA Instruments SDT 2960 at a heating rate of 4, 6, 8, 10 and 12 °C/min in air atmosphere and over the temperature range 25–1400 °C. Thermogravimetry (TG) curves showed two decomposition stages, the ideal heating rate was found to be 10 °C/min, as it presents less weight loss. Differential thermal analysis (DTA) exhibited endothermic and exothermic events which suggested the sintering temperature at about 1125 °C. Post-sintering samples were collected and the point load test conducted to evaluate the material strength.

Keywords: Granite; Marble; Thermal analysis

1. Introduction

Residue or reject can be material or parts of material from human activities that is discarded because it has no use in our society. Solid residue are classified by physical nature, e.g. like dry or wet, chemical composition, i.e. organic or inorganic, hazardous potential to environment and public health. The main sources of solid residues can be classified as industrial, public, household, commercial, health and transports services, agriculture and construction [1]. Granite is a natural hard igneous rock formation of visibly crystalline texture formed essentially of quartz, feldspar and mica. It is specially used for building and monuments. Marble is a limestone, more or less crystallized by metamorphism, that ranges from granular to compact in texture. It is easily polish and used especially in architecture and sculpture. Residue which comes from granite and marble can have parts from gneiss, migmatite, diorite, slate, quartzite and others. Granite and marble industry have been increased in the last years, consequently the volume of residue disposed in the

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decant lagoons or landfills increased, which results environment problems. The objective of this work is using the residue to obtain ceramic material by utilizing thermal analysis like a great tool to predict the process condition such as sintering, temperature, heating rate.

2. Experimental

2.1. Industrial granite and marble residue

This reject is obtained when these rocks are sawed in blocks or slices. It was dried in an air oven at 120 °C for 24 h and next mechanically ground and sieved. The granulometric analysis showed that 61% passed 0.053 mm, 33% in 0.125, 5% in 0.25, almost 1% in 0.5 mm mesh and small grains could not at all pass through in 0.5 mm mesh. The material had to be sieved through mesh #250 before analysis [2].

2.2. Samples preparation

The samples with ≈16 g of residue were molded (⇔≈3 cm) in a carver press at room temperature and 600 psi. They were sintered in electric furnace in the temperatures of 950, 1000, 1050, 1100 and 1125 °C, and a heating rate of 4, 6, 8, 10 and 12 °C/min in room atmosphere for 1–2 h.

2.3. TG/DTG/DTA measurements

Thermal behavior was measured on a TA instruments SDT 2960 at a heating rate of 4, 6, 8, 10 and 12° C/min in air atmosphere, over the temperature range from 25 to 1400 °C.

2.4. Determining point load strength

This parameter was measured on an Aiphageos portable point load test machine.

3. Results and discussion

The obtainment of a product depends on both material and nonmaterial factors such as the economy of the marketplace, consumer response, dimensional and surface finish tolerances, apparent quality and manufacturing productivity. Ceramic manufacture is a complex interaction of raw materials, technological processes, environment, people, and financial investment [3]. X-ray diffraction (XRD) showed the residue to consist of quartz, plagioclase, orthoclase, dolomite and mica. Chemical analysis (titration) showed almost 10% (w/w) of Fe_2O_3, but XRD did not. XR fluorescence (XRF) showed a high percentage of Si (up to 29%), a medium percentage of Al, Fe, K, Na, Ca, Mg (1–8%), and very low figures for Ti, Co, Ni, Mn, Sr, Ba, P and Zr [4].

Fig. 1 shows thermogravimetry (TG) and DTA curves for residue in air atmosphere, where the TG curve presents three decomposition stages. The water loss occurred (≈1%) in the first stage (Ti 100 °C), in the second temperature range (450–650 °C) the weight loss was 4.3%, which can be attributed to the decomposition of carbonates. The TG plot shows a composition of up to 90% which indicates the presence of silicates (quartz, plagioclase, orthoclase and mica). DTA thermogram presents four endothermic and two exothermic events. The two endothermic peaks (495 and 645 °C) are confirmed different types of carbonates. The endothermic peak at 567 °C is suggesting of a polyrmorphous transformation of α-quartz into β-quartz and the last endothermic effect near 1170 °C was caused by melting (T_m) so indicating the presence of feldspars (orthoclase and plagioclase). One exothermic events occurred at about 977, it can be due to a structural reorganization, mullite nucleation [5]. The second effects can be crystalization process. The results obtained from TG and DTA are supported by XRD and XRF studies.

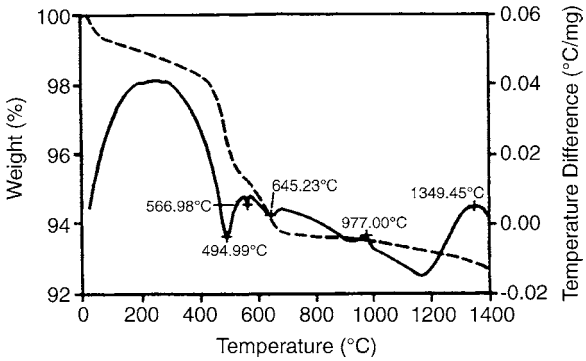

Fig. 1. TG/DTA curves of residue, under air atmosphere.

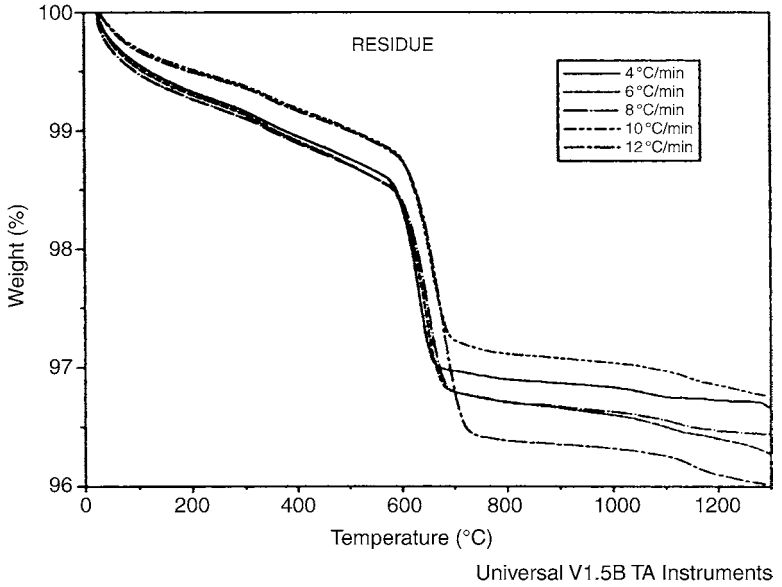

Fig. 2. Comparison of TG curves of residue, with different heating rate.

Fig. 2 illustrates a comparison of TG curves for residue at a heating rate of 4, 6, 8, 10 and 12 °C/min. The curve shows a greater weight loss to have occurred at 12 °C/min and minor was at 10 °C/min. The change in weight loss with heating rate and temperature are presented and illustrated in Table 1.

3.1. Strength characterization

The point load test was carried out in order to evaluate the material strength. This kind of test is intended to be an index for strength classification and has been frequently used in rock mechanics to predict other strength parameters with which it is correlated, for example uniaxial compressive strength.

The testing apparatus consists of a loading system, typically comprising a loading frame, pump, ram and conical platens, a system for measuring the load (F) required to break the specimen, and a system for measuring the distance (D) between the two loading platens contact points.

As mentioned before, the specimens were prepared at heating rate of 12 °C/min from room to the temperatures of 950, 1000, 1050, 1075, 1090, 1100, 1110, 1150 and 1125 °C. The specimens had a cylindrical geometry with length/diameter ratio of 0.5 and the

Table 1
Weight loss (%) with temperature and heating rate

Temperature (°C)	Heating rate (°C/min)				
	4	6	8	10	12
800	3.04	3.29	3.25	2.88	3.60
900	3.04	3.33	3.30	2.91	3.64
1000	3.10	3.39	3.34	2.95	3.68
1100	3.19	3.49	3.41	3.02	3.74
1125	3.19	3.53	3.44	3.05	3.78
1150	3.20	3.55	3.47	3.08	3.81

Table 2
Average strength of samples tested and temperature

Temperature (°C)	Strength (MPa)
950	0.30
1000	0.66
1050	1.58
1075	5.02
1090	7.30
1100	11.74
1110	15.41
1115	16.08
1125	10.39

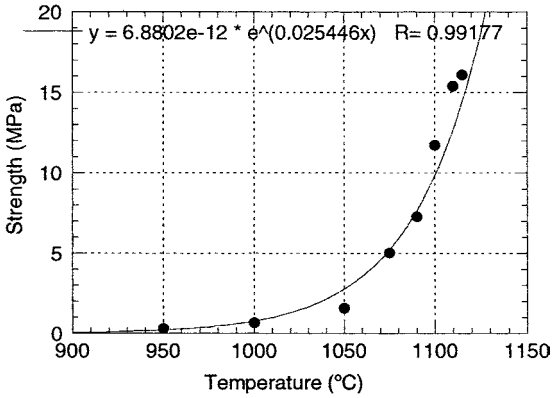

Fig. 3. Exponential curve from temperature × strength.

tests were performed applying loads in the axial direction. The strength (F/D^2) was calculated and size corrected for a 50 mm diameter to obtain a unique strength value which can be used for purposes of material classification, as suggested by commission on testing methods of the International Society for Rock Mechanics—ISRM [6].

It can be ascertained from data presented that the temperature has expressive influence over the strength, so that the average values are virtually represented by exponential curve (Table 2, Fig. 3). According to Bieniawski [7], the material can be classified as one having a extreme high resistance, comparable to the rocks like granite and quartzite.

4. Conclusion

DTA and TG can be considered as a great tool to predict the process condition to obtain ceramic material from granite and marble industry residue. DTA thermogram exhibited endothermic and exothermic events. It showed types of carbonates, silicates and a endothermic peak (567 °C) is suggesting the polymorphous transformation of α-quartz into β-quartz and also indicating the sintering (solid–liquid) at 1125 °C (near melting, T_m). TG curves showed that the minor weight loss was at 10 °C/min of 800–1150 °C. The uniaxial compressive strength increase to 16.08 MPa with temperature like exponential form until 1115 °C, after that begin to appear liquid material (melt) and the strength decrease to 10.39 MPa. Ceramic material obtained can be classified as one having a extreme high resistance, comparable to the rocks like granite and quartzite.

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