Article

Optimization of performance of building paints using granite and marble waste

Otimização do desempenho de tintas imobiliárias usando resíduos de granito e mármore

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Abstract

The production of paints using waste from granite and marble processing is a new alternative to contribute to sustainable development in civil construction. However, the lack of studies on the composition and performance of these paints makes the dissemination of this new construction material unfeasible. Therefore, this study aims to investigate the composition of these paints using statistical tools to obtain products with performance compatible with technical regulations and commercial paints. The paint formulations were defined through a quaternary mixture planning. The solids content, pH, viscosity, hiding power, and abrasion resistance were determined for all samples. The paints produced with granite waste showed better hiding power and abrasion resistance, as granite waste has a finer granulometry, in addition to being predominantly composed of silica. Finally, the paints produced in this study and the commercial paints showed similar behavior, which supports the use of waste from the processing of ornamental rocks in paint production.

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Keywords: Granite waste. Marble waste. Building paint. Quaternary mixture design. Industrial residue. Recycling.

Resumo

A produção de tintas utilizando resíduos de beneficiamento de granito e mármore é uma nova alternativa para contribuir com o desenvolvimento sustentável na construção civil. Entretanto, a carência de estudos sobre a composição e o desempenho dessas tintas inviabiliza a disseminação desse novo material de construção. Logo, este estudo tem como objetivo investigar a composição dessas tintas utilizando ferramentas estatísticas para obter produtos com desempenho compatível com regulamentações técnicas e tintas comerciais. As formulações de tintas foram definidas através de um planejamento de misturas quaternárias. O teor de sólidos, pH, viscosidade, poder de cobertura e resistência à abrasão foram determinados para todas as amostras. As tintas produzidas com resíduos de granito apresentaram melhor poder de cobertura e resistência à abrasão, pois o resíduo de granito apresenta granulometria mais fina, além de ser predominantemente composto por sílica. Por fim, as tintas produzidas neste estudo e as tintas comerciais apresentaram comportamento semelhante, o que corrobora com o aproveitamento do resíduo do processamento de rochas ornamentais na produção de tintas.

Palavras-chave: *Resíduos de granito. Resíduos de mármore. Tintas para construção civil. Projeto de mistura quaternária. Resíduos industriais. Reciclagem.*

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Introduction

Due to the accelerated growth of the civil construction sector, the global market for paints and coatings was valued at around 137,100 million dollars in 2021, with an annual growth rate of more than 4% expected for the coming years (Mordor Intelligence, 2022). According to the Brazilian Association of Paint Manufacturers (ABRAFATI, 2022), Brazil ranks among the world's top five largest paint markets, along with China, the United States, India, and Japan. In 2021, Brazil's paint production exceeded 1.7 billion liters, with architectural paints constituting over 80% of the total volume. These countries collectively represent major players in the global paint industry, showcasing substantial production and consumption levels.

Paints are complex mixtures comprising organic and inorganic pigments, binding vehicles, thinner vehicles and additives. The production of paints involves the use of various chemicals, resulting in high concentrations of organic compounds, suspended solids, colored materials, and hazardous pollutants such as heavy metals (Aniyikaiye *et al*., 2019; Mandal *et al*., 2020). The environmental impacts associated with the production, use, and disposal of paints necessitate the exploration of more sustainable alternatives to improve their technical performance and develop coatings that are environmentally acceptable.

Previous experimental studies by Myint, Zakaria and Ahmed (2010), Alam and Aladis (2011), Moreno *et al.* (2014) and Bal *et al.* (2017) reported some examples of experimental studies that sought to use more sustainable resins for the production of paints. Saxena and Dhimole (2006), Legodi and de Wall (2007), Ahmed *et al.* (2015), Cardoso *et al.* (2016), Galvão *et al.* (2018), Lopes *et al.* (2019), Tressmann *et al.* (2020) and Silva (2017) studied more sustainable alternatives for replacing active and inert pigments conventionally used in paint production. In most of these studies, the solvent of the paints was water rather than organic solvents to reduce the emission of volatile organic compounds (VOCs) (Ou *et al.*, 2022).

Lopes *et al.* (2021a, 2021b, 2019) used granite waste as pigments for low-cost latex paints, and Tressmann *et al.* (2020) developed similar research using marble processing waste instead of granite waste. Promising results were obtained in these studies regarding the technical performance of the paints produced with these types of wastes.

Waste generated during ornamental stone processing consists of fine particles obtained from cutting and polishing rock blocks (Almada; Melo; Dutra, 2020; Ghannam; Najm; Vasconez, 2016; Nascimento *et al.*, 2021). China, India and Brazil are major generators of granite and marble waste. Brazil generates around 2.5 million tons of fine waste per year, with the majority consisting of granite and marble waste (ABIROCHAS, 2023; de Almeida *et al*., 2023). Similarly, China and India have thriving industries in the extraction and processing of these stones, leading to substantial waste generation. However, when not disposed of correctly, granite and marble waste can cause significant environmental impacts such as soil and water contamination. It is crucial to prioritize proper waste management practices, including recycling, reuse, and responsible disposal, to mitigate these environmental effects. By implementing sustainable approaches, these countries can minimize the adverse impacts associated with granite and marble waste and promote a more environmentally friendly stone processing industry (Bacarji *et al.*, 2013; Souza *et al.*, 2017).

Granite waste, in particular, contains a high percentage of silica (SiO_2), which enhances the mechanical properties of paints, such as abrasion and scratch resistance (Lopes *et al.*, 2019) while also imparting superhydrophobic properties and chemical durability to coatings (Ahmed *et al.*, 2015; Bozorg and Ramezani, 2017). Marble waste, on the other hand, contains calcium carbonate ($CaCO_3$), contributing to the hiding power of coatings (Tressmann *et al.*, 2020). These characteristics make these waste materials attractive for use in paint production.

However, the combined effects of granite and marble waste on the technical performance of sustainable paints have not been investigated in previous literature. Consequently, the potential synergistic effects between these waste types on the optical and mechanical properties of paints remain unknown. Furthermore, there is a need to optimize the composition of these sustainable paints to ensure they meet technical regulations.

In this context, optimization techniques play a crucial role in enhancing the characteristics and efficiency of products, processes, or methods to make them more sustainable. Variables influencing experimental performance can be categorized as process variables or mixture variables (Ong *et al*., 2019). Mixture design methods enable comprehensive and systematic studies of how different mixture components' proportions affect performance, offering significant potential for optimization (Novaes; Yamaki; de Paula, 2018; Li; Lu; Gao, 2021). Despite this potential, mixture design methods are infrequently employed in optimization applications due to their complexity in visualizing and interpreting high-dimensional systems (Falleh *et al*., 2019; Syafitri; Sartono; Goos, 2015).

Therefore, the main objective of this research is to use a quaternary mixture design to optimize the composition of paints produced with distinct types of wastes from ornamental stone processing to obtain a performance compatible with technical regulations and with commercial paints. The following original contributions were provided by this work to the state-of-the-art:

(a) the study proposes the use of accessible and cost-effective materials for paint production. These materials encompass water as a solvent, polyvinyl acetate resin as a binder, in addition to granite and marble waste as pigments/mineral fillers;

(b) it offers new insights into the combined use of granite and marble waste in paint production, an area that was not previously explored in sustainable construction;

(c) for the first time, systematic comparisons between paints made of granite waste and marble waste were provided;

(d) it presents a practical application of the quaternary mixture design approach, an underutilized tool, to determine the optimal proportions of the fundamental components of the developed construction material; and

(e) the study includes equations that model the performance of the proposed paints based on their basic components, predicting the behavior of the sustainable material in terms of viscosity, hiding power, and abrasion resistance. These equations also enable an optimization of these properties and the refinement of the paint formulation.

Material and methods

Materials

The pigments consisted of waste generated from granite and marble processing, obtained through the diamond wire cutting process and subjected to a pre-treatment. In line with the aim of sustainability, water was chosen as the solvent due to its abundance and environmentally friendly nature compared to conventional organic solvents. As for the binder, polyvinyl acetate resin (PVA) was selected for its low cost, water solubility, and easy availability in the market. Notably, the inclusion of additives commonly used in the paint industry was intentionally omitted due to their challenging procurement and high cost.

Methodology

Pre-treatment of granite and marble wastes

After being collected, the granite and marble wastes were separately submitted to a pre-treatment. These wastes were dried outdoors and sieved through an 8-mesh sieve (2.38 mm opening) to remove larger particles and homogenize the material. The pigments were prepared sequentially, based on the method developed by Cardoso *et al.* (2016), which consists of the disaggregation and dispersion of the particles in an aqueous medium using a Cowles disk coupled to a mechanical stirrer at 1500 rpm. Subsequently, the wastes in an aqueous medium were passed through an 400-mesh sieve (0.038 mm opening).

Characterization of granite and marble wastes

The wastes were characterized after being submitted to the pre-treatment. The following parameters were evaluated for physical characterization of the wastes: granulometric distribution curve, using a Bettersize 2000 laser particle size analyzer in the range of 0.02 μm to 2000 μm; specific mass, using a Le Chatelier's volumetric flask, according to NBR 16605 (ABNT, 2017); specific surface area by the BET method; and organic matter content by burning at 440 ºC, based on NBR 13600 (ABNT, 2022). Scanning electron microscope (SEM) images were obtained with a Leo 1430VP equipment for morphological characterization.

For chemical characterization, the materials were subjected to pH measurements according to the EMBRAPA (2017) and X-ray Fluorescence (XRF) using the Shimadzu Micro-EDX-1300 spectrometer, applying a high voltage (50 keV and 50 μA) and mapping 1200 points. For mineralogical characterization and identification of crystalline structures, X-ray Diffraction (XRD) was performed in a D8 Discover diffractometer (Bruker), using CuKα radiation (1.5418 Å) and 2θ ranging from 3° to 70°.

Experimental program

This research was based on an experimental design of mixtures with four components (quaternary), which allows for studying and optimizing the response variables as a function of four independent variables. The independent variables of the mixture design were the granite waste (GW) and marble waste (MW) pigments, ranging from 0 to 55% by mass; resin, ranging from 10 to 25% by mass, considering the volatile and non-volatile parts; and water, ranging from 35 to 45% by mass. These variation ranges were defined based on the studies developed by Lopes *et al.* (2019) and Tressmann *et al.* (2020). It should be noted that the variation range of water content was defined to ensure that the paint maintains adequate viscosity for proper application. This is aimed at preventing issues such as paint running (resulting from low viscosity) and the formation of an excessively thick paint film (caused by high viscosity) (Lopes *et al.*, 2019). In addition, the relationship between the mass of resin and pigment is compatible with the values adopted in PVA latex paints found on the market, which can vary between 10 and 43% (Silva; Uemoto, 2023). Table 1 and Figure 1 show the 35 formulations defined in the present experimental program, using the Minitab® 17 statistical analysis software, after selecting a design in extreme vertices of degree three, augmented by axial and central points.

Production and determination of paint performance characteristics and properties

To produce the paints, appropriate contents of granite and/or marble waste pigments were mixed with water and PVA resin according to the components' mass proportions defined in the statistical design. The mixing process was carried out with a Cowles disc coupled to a mechanical stirrer at a speed of 400 rpm.

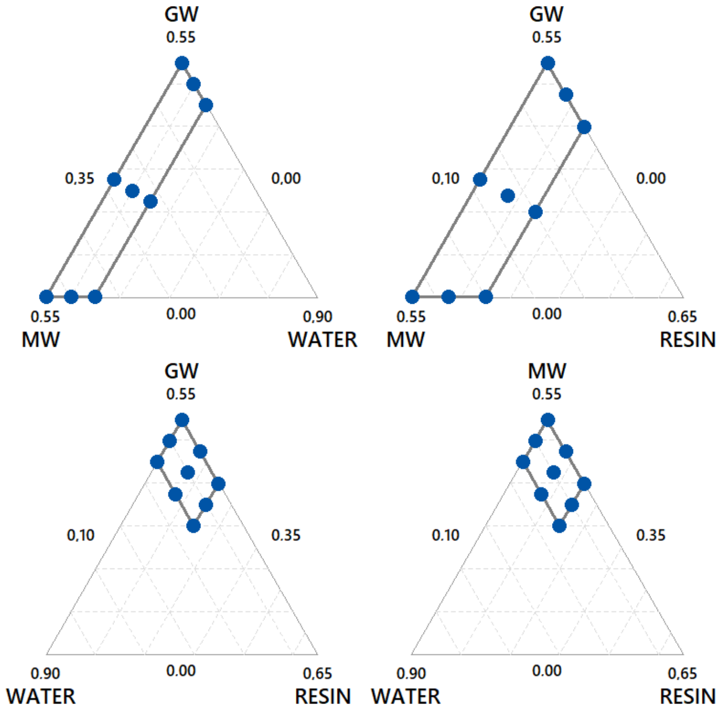
The paint samples were tested for viscosity, solids content, hiding power of the dry paint, and resistance to wet abrasion without abrasive paste. The kinematic viscosity was measured from the flow time of the fluid, in seconds, using a Ford cup viscometer with orifice No. 4, according to NBR 5849 (ABNT, 2015). The apparent and plastic viscosities were determined using a Couette rotational viscometers (Fann Model 35 viscometers), based on ISO 10414-1 (ISO, 2008). The solids content was calculated as the ratio of the oven-dried paint mass and the initial paint mass, according to D 3723-05 (ASTM, 2017). The hiding power of the dry paint was calculated as the maximum applied area (m²) per unit volume of paint (L), so that the coverage has a contrast ratio of 98.5%, based on D 2805-11 (ASTM, 2018) and NBR 14942 (ABNT, 2019a). The resistance to wet abrasion without abrasive paste was given by the number of cycles a paint film can resist until wear at least 80% of the area traversed by the brush, as recommended by D 4060-19 (ASTM, 2019) and NBR 15078 (ABNT, 2006).

Table 1 - **Proportions of paints designed in the Minitab® 17 statistical analysis software**

Id.	GW	MW	Water	Resin	Id.	GW	MW	Water	Resin
GW55.0%	0.55	0	0.35	0.1	GW10.6%+MW38.1%	0.106	0.381	0.375	0.138
GW50.0%	0.5	0	0.4	0.1	GW10.6%+MW33.1%	0.106	0.331	0.425	0.138
GW47.5%	0.475	0	0.35	0.175	GW10.6%+MW30.6%	0.106	0.306	0.375	0.213
GW45.0%	0.45	0	0.45	0.1	GW10.6%+MW25.6%	0.106	0.256	0.425	0.213
GW42.5%	0.425	0	0.4	0.175	GW15.0%+MW15.0%	0.15	0.15	0.45	0.25
GW40.0%	0.4	0	0.35	0.25	GW17.5%+MW17.5%	0.175	0.175	0.4	0.25
GW37.5%	0.375	0	0.45	0.175	GW18.8%+MW18.8%	0.188	0.188	0.45	0.175
GW35.0%	0.35	0	0.4	0.25	GW20.0%+MW20.0%	0.2	0.2	0.35	0.25
GW30.0%	0.3	0	0.45	0.25	GW21.3%+MW21.3%	0.213	0.213	0.4	0.175
MW55.0%	0	0.55	0.35	0.1	GW22.5%+MW22.5%	0.225	0.225	0.45	0.1
MW50.0%	0	0.5	0.4	0.1	GW23.8%+MW23.8%	0.238	0.238	0.35	0.175
MW47.5%	0	0.475	0.35	0.175	GW25.0%+MW25.0%	0.25	0.25	0.4	0.1
MW45.0%	0	0.45	0.45	0.1	GW25.6%+MW10.6%	0.256	0.106	0.425	0.213
MW42.5%	0	0.425	0.4	0.175	GW27.5%+MW27.5%	0.275	0.275	0.35	0.1
MW40.0%	0	0.4	0.35	0.25	GW30.6%+MW10.6%	0.306	0.106	0.375	0.213
MW37.5%	0	0.375	0.45	0.175	GW33.1%+MW10.6%	0.331	0.106	0.425	0.138
MW35.0%	0	0.35	0.4	0.25	GW38.1%+MW10.6%	0.381	0.106	0.375	0.138
MW30.0%	0	0.3	0.45	0.25	-	-	-	-	-

Note: Id.: sample identification; GW: granite waste; and MW: marble waste.

Figure 1 - **Experimental design of quaternary mixture defined in Minitab® 17**



All these characterization and performance tests were also carried out on eight economic category latex paints available on the Brazilian market. Thus, additional comparisons were made between the properties of paints based on granite and marble waste and commercial paints.

Statistical analysis

Based on the paint characterization and performance results, polynomial models (regression equations) were generated, considering the significance level of the terms (p-value < 0.05). The parameters used to optimize the performance of the paints were hiding power (target value of 4 m²/l) and abrasion resistance (target value of 100 cycles) for the economic latex paint category, according to NBR 15079-1 (ABNT, 2019b).

The mixture proportions associated with the best performance were determined using the statistical desirability function and the modeling of response surfaces. The individual desirability function (*di*), which varies from 0 to 1, was calculated according to Equation 1, when the goal was to maximize the results. In this equation, y is the response, L is the minimum value (considered as zero), T is the target value (assumed 4 m²/L for hiding power and 100 cycles for abrasion resistance), and s is the weight (assuming 1, either for hiding power and abrasion resistance).

$$d_i = \begin{cases} 0 \; se \; y < L \\ \left(\frac{y-L}{T-L}\right)^s \; se \; L \leq y \leq T \\ 1 \; se \; y > T \end{cases} \qquad \text{Eq. 1}$$

In addition, the global desirability (D), which also varies from 0 to 1, was calculated as the geometric mean of individual desirabilities (*di*), according to Equation 2, in which m is the number of responses studied in the optimization process. Zero means an unacceptable value, and 1 (one) is related to the most desirable value. All of these statistical analyzes were performed using the Minitab® 17 software.

$$D = \sqrt[m]{d_1 . d_2 \ldots d_m} \qquad \text{Eq. 2}$$

Microscopicanalyzes

Some granite and marble waste paints and commercial paints were selected for microscopic analysis of paint films. Microscopic images of the paints were taken using the Leo 1430VP scanning electron microscope. In this situation, SEM stubs were painted with three coats of paint.

In addition, the roughness of the paint films was determined using a 3D optical profilometer, model Contour GTK, adopting the "Ra" parameter. In this case, the substrates were glass slides painted with three coats of paint. The roughness was determined in the central part of the film. These measurements were performed in three different regions of the sample, and the average value was then calculated.

Results and discussion

Characterization of pigments

Figure 2 shows the particle size distribution curves of the pigments, and Table 2 shows the results of the physical characterization, organic matter content and pH of these materials.

The wastes had an average diameter lower than 10 µm. As per the values of D10 and D90, the range of variation in the size of particles from granite and marble waste is between 1µm and 40 µm. According to Diebold *et al.* (2022), the particle sizes of mineral fillers used in paint formulations generally cover the range of 0.01 to 45 µm, although most are between 0.5 and 10 µm. In paint formulation, particle size influences coverage, whiteness, gloss and adhesion (Qureshi, 2021).

The specific surface area of the granite waste was greater than that of the marble waste. A larger specific surface area is related to the smaller particle size (Karlsson *et al.*, 2019). As Rooney and Meldrum (2020) exemplified, the ivory black pigment contains grains with an approximate diameter of 10 µm, presenting an approximate specific surface area of 0.3 m²/g. In comparison, the white titanium pigment has grains of approximately 0.5 µm in diameter and an approximate specific surface area of 3 m²/g. This is one of the factors that directly influence the interaction between pigments and their surroundings (Sarkodie *et al.*, 2019). As particle size decreases, an increase in specific surface area is expected, resulting in greater resin consumption to coat the pigments (Lopes *et al*., 2019; Touazi *et al*., 2020).

The organic matter content of the wastes is lower than 1%, which can be considered insignificant. When this content is high, it can cause damage to paints exposed to solar radiation and, in particular, to its UV components, in addition to the possible attack of fungi and bacteria (Rossi; Simeoni; Quaranta, 2021; Tressmann *et al*., 2020).

Figure 2 - **Particle size distribution curves of granite waste and marble waste pigments**

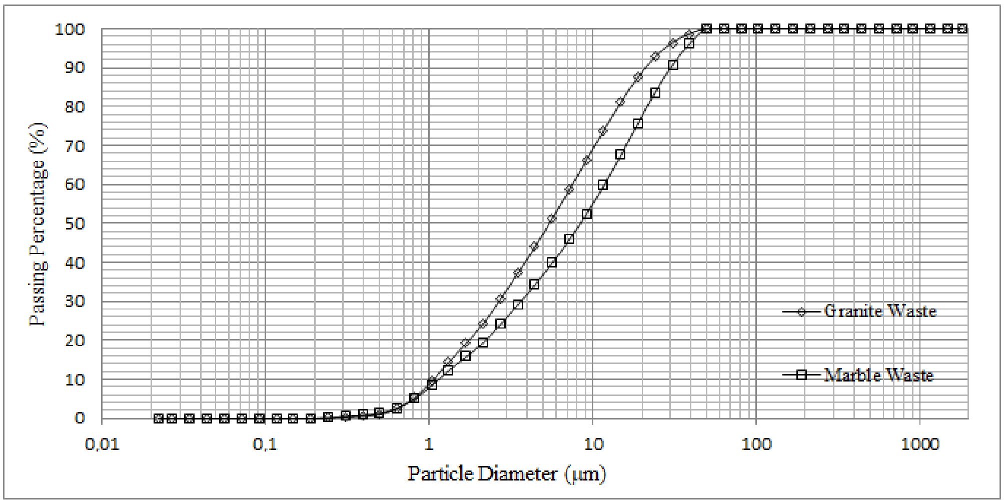


Table 2 - **Physical and chemical characterization of the pigments**

Pigment	Specific mass (g/cm³)	Specific surface area (m²/g)	Diameter (µm)			Organic matter content (%)	pH
			D10	D50	D90		
Granite waste	2.60	6.11	1.20	6.31	26.09	0.55	8.89
Marble waste	2.82	4.42	1.30	9.99	37.85	0.20	9.52

Note: D10: indicates the maximum diameter exhibited by 10% of the particles; D50: corresponds to the maximum diameter exhibited by 50% of the particles; D90: corresponds to the maximum diameter exhibited by 90% of the particles.

Granite and marble waste had an alkaline pH (pH > 7), as in the case of several inert pigments used in paint production, such as calcium carbonate and hydrated alumina. According to Fadhil, Nashaan and Hlihl (2020), the pH of the pigment significantly affects the performance properties of the paints, and the recommended pH is between 8 and 9. In the production of paints, an alkaline pH can mitigate the proliferation of microorganisms and enhance adhesion to the cementitious substrate, which also exhibits an alkaline pH, consequently influencing the ultimate durability of the paint film (Fazenda, 2009; Sumra; Pyam; Zainah, 2020). Nevertheless, research by Suma, Jacob and Joseph (2009) and Oliveira, Silva and Guerrini (2011) reveals that under alkaline conditions, polyvinyl acetate resin (PVA) can exhibit diminished hydrolytic stability, resulting in a decline in the reactivity index of the binder. According to Yamak (2013), the optimal pH range for polyvinyl acetate emulsions lies between 4.5 and 5.5.

The results of X-ray fluorescence (XRF) analyses are presented in Tables 3 and 4. The granite waste (Table 3) is mainly composed of SiO_2 and Al_2O_3, while the marble waste (Table 4) is mainly formed of CaO and MgO.

The results of mineralogical characterization based on X-ray diffraction (XRD) of the pigments are shown in Figure 3. The granite waste (Figure 3a) presented high-intensity peaks of quartz (SiO_2) and albite ($NaAlSi_3O_8$) and low-intensity signatures of muscovite ($KAl_2Si_3AlO_{10}(OH,F)_2$) and microcline ($KAlSi_3O_8$), while the marble waste (Figure 3b) presents strong peaks of calcite ($CaCO_3$) and dolomite ($MgCO_3$) and low-intensity peaks of quartz and muscovite. Several previous studies have already used these compoundsin the formulation of paints (Ahmed; Mohamed; Abd El-Gawad, 2019; Al-Kayiem *et al*., 2021; Komar *et al*., 2020; Krishana Mohan; Bhanuprakash; Mukherjee, 2022; Le *et al*., 2021).

Figures 4a and 4b show the morphological aspects of the granite waste and marble waste particles, respectively. The shape of the particles affects their packing conditions and, consequently, the hiding power of the paint film. Granite and marble wastes presented varied diameters, being composed of particles with irregular morphology and angular edges. This morphology is due to the ornamental stone processing process (Singh; Nagar; Agrawal, 2016). In addition to the shape of the particles, the SEM images show that the marble waste has larger particles compared to the granite waste. This variation in particle dimensions poses a challenge when comparing these residues. Instead of using a grinding treatment to standardize the grain size of the wastes, the current study exclusively concentrated on applying particles dispersion and sieving methods, as described in the "Pre-treatment of granite and marble wastes" section.

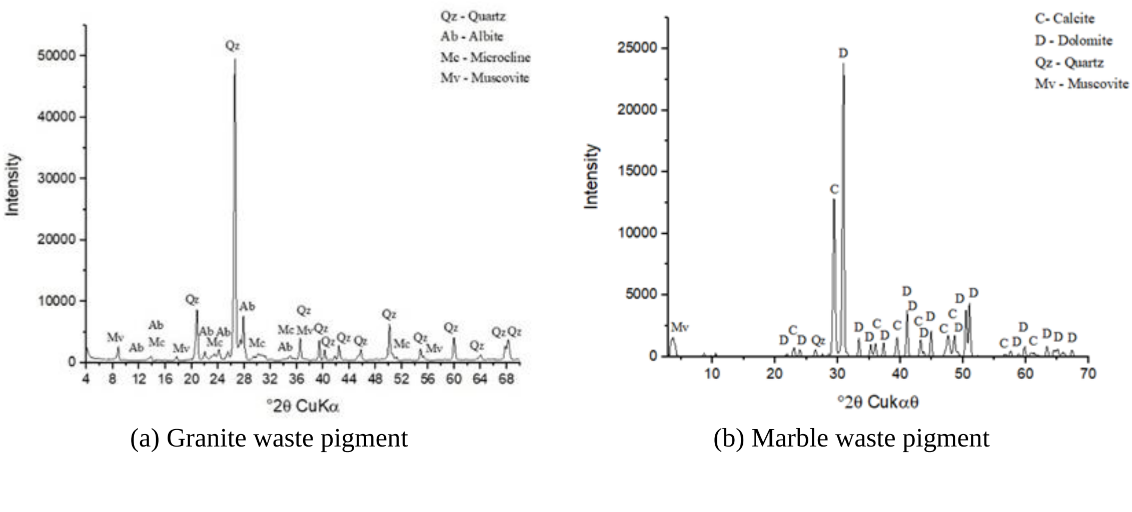
Table 3 - **Results of XRF analysis of granite waste pigments**

Pigment	SiO_2	Al_2O_3	Na_2O	K_2O	CaO	SO_3	Fe_2O_3	P_2O_5	Others
Granite waste	80.53	8.20	6.25	2.79	0.98	0.54	0.32	0.24	0.47

Table 4 - **Results of XRF analysis of marble waste pigments**

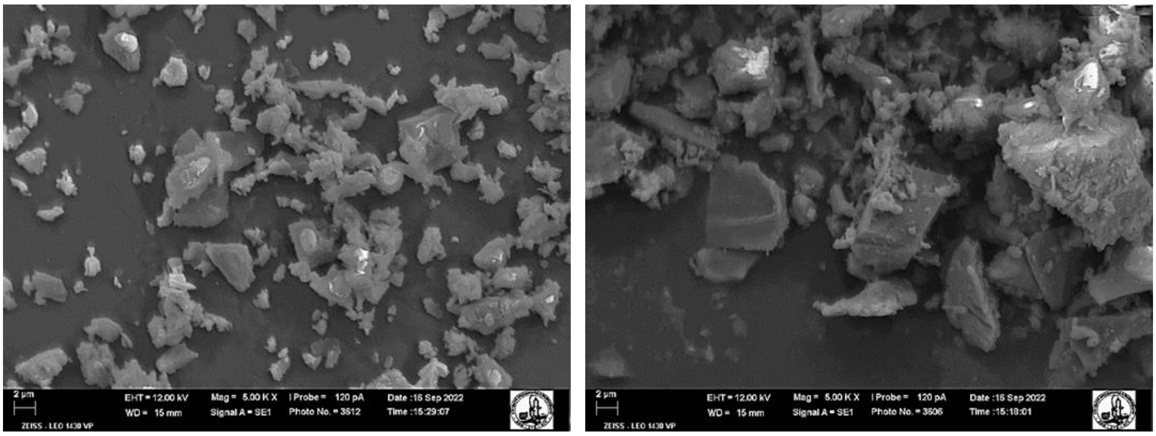
Pigment	CaO	MgO	SiO_2	K_2O	SO_3	Others
Marble waste	76.59	16.68	4.91	1.02	0.77	0.03

Figure 3 - **Result of the XRD analysis**



(a) Granite waste pigment (b) Marble waste pigment

Figure 4 - **SEM of the particles**



(a) Granite waste pigment (b) Marble waste pigment (Mag.= 500 X)

Table 5 - **Characteristics and properties of the paints**

Sample	SM (g/l)	SC (%)	pH	KV (s)	AV (cP)	PV (cP)	AR (cycles)	HP (m²/l)	D
GW55.0%	1336.70	59.00	7.30	19.00	84.00	77.00	40.00	14.20	0.63
GW50.0%	1468.20	55.50	7.40	17.00	65.50	60.00	49.00	10.50	0.70
GW47.5%	1353.30	56.10	7.40	19.00	70.50	69.00	115.00	4.60	1.00
GW45.0%	1419.90	50.50	7.40	14.40	28.50	35.00	64.00	6.20	0.80
GW42.5%	1183.90	50.80	7.40	16.50	41.00	42.00	69.50	4.30	0.83
GW40.0%	1243.00	52.20	7.30	20.70	68.50	60.00	143.00	3.30	0.91
GW37.5%	1335.00	46.20	7.30	14.10	25.00	28.00	107.50	3.60	0.95
GW35.0%	1318.60	47.00	7.30	17.20	43.00	39.00	127.00	3.20	0.89
GW30.0%	1270.40	42.00	7.20	14.30	24.50	22.00	160.00	2.10	0.72
MW55.0%	1353.90	60.50	7.60	17.10	52.00	52.00	23.00	7.30	0.48
MW50.0%	1343.80	56.20	7.60	14.00	42.50	38.00	28.00	6.50	0.53
MW47.5%	1591.40	57.10	7.50	17.50	60.50	48.00	80.00	5.50	0.89
MW45.0%	1374.70	50.70	7.50	12.80	31.50	20.00	40.00	5.40	0.63
MW42.5%	1439.40	52.10	7.50	15.80	50.50	31.00	94.00	3.60	0.92
MW40.0%	1393.60	53.40	7.50	17.00	38.50	42.00	97.50	1.00	0.49
MW37.5%	1340.00	47.60	7.50	14.60	22.50	17.00	62.50	3.30	0.72
MW35.0%	1321.20	47.70	7.40	14.80	23.50	20.00	115.00	1.50	0.61
MW30.0%	1175.10	43.60	7.30	14.60	26.00	19.00	115.00	0.90	0.47
GW10.6%+MW38.1%	1203.10	55.30	7.60	16.70	46.00	42.00	72.00	4.60	0.85
GW10.6%+MW33.1%	1274.10	49.30	7.60	13.60	26.00	26.00	85.00	4.70	0.92
GW10.6%+MW30.6%	1220.70	51.60	7.50	16.60	34.50	36.00	132.00	3.30	0.91
GW10.6%+MW25.6%	1336.00	47.60	7.50	14.00	22.00	19.00	140.00	2.20	0.74
GW15.0%+MW15.0%	1272.20	42.00	7.40	14.10	20.50	18.00	170.00	1.50	0.61
GW17.5%+MW17.5%	1239.40	46.80	7.40	16.20	33.00	29.00	160.00	2.50	0.79
GW18.8%+MW18.8%	1256.90	45.80	7.40	13.30	17.50	16.00	115.00	3.90	0.99
GW20.0%+MW20.0%	1242.40	51.10	7.40	17.50	50.50	45.00	148.00	1.90	0.69
GW21.3%+MW21.3%	1169.60	50.80	7.50	16.90	35.00	38.00	112.00	3.00	0.87
GW22.5%+MW22.5%	1368.70	50.60	7.50	14.80	29.00	29.00	39.00	6.90	0.62
GW23.8%+MW23.8%	1360.90	55.60	7.60	17.60	55.00	56.00	110.00	4.30	1.00
GW25.0%+MW25.0%	1232.30	56.00	7.60	14.40	49.50	52.00	43.50	6.10	0.66
GW25.6%+MW10.6%	1309.60	45.50	7.40	14.30	22.50	25.00	142.50	2.80	0.84
GW27.5%+MW27.5%	1318.60	59.40	7.60	17.70	66.50	67.00	52.50	6.00	0.72
GW30.6%+MW10.6%	1315.90	50.60	7.40	18.40	44.50	42.00	143.00	2.80	0.84
GW33.1%+MW10.6%	1309.40	51.00	7.50	14.90	27.00	35.00	68.00	6.70	0.82
GW38.1%+MW10.6%	1346.40	54.60	7.50	18.80	51.00	53.00	65.00	5.40	0.81

Note: SC: Solids Content; SM: Specific Mass; KV: Kinematic Viscosity (represented by the flow time of the fluid in the viscometer Ford cup No. 4); AV: Apparent Viscosity; PV: Plastic Viscosity; AR: Abrasion Resistance; HP: Hiding Power; D: Desirability. The lines highlighted in gray represent the paints produced only with granite waste, only with marble waste, and with both granite waste and marble waste that presented the best performance (higher desirability values).

Characterization and performance of paints

Table 5 presents the results of the different properties of the paints: specific mass (SM), solids content (SC), pH, kinematic viscosity (KV), apparent viscosity (AV), plastic viscosity (PV), abrasion resistance (AR), hiding power (HP), in addition to the desirability (D) results obtained from the statistical analysis.

Table 6 presents the polynomial models (regression equations) that describe the characteristics and performance properties of the paints. These models explain how each component affects the final behavior of the paint; whether there are any significant interactions between the components; whether the contribution of each component is positive or negative; and whether the behavior is linear or parabolic. In addition, the coefficient of determination (R^2) was presented. This coefficient indicates the fitness level of a generalized statistical model to the observed values of a random variable. Since all R^2 values are greater than 80%, it is possible to conclude that the developed models explain the data variance well.

Table 7 presents the characteristics and properties of commercial paints used for comparisons with the granite and marble waste paints. It is noteworthy that the kinematic viscosity was not determined. As commercial paints have low fluidity, possibly due to the presence of thickening additives, it was not possible to measure their viscosity using the Ford cup viscosimeter No. 4.

The specific mass of the paints produced in this research ranged from 1169.6 g/L to 1591.4 g/L, compatible with the variation range found in commercial paints (1232.1 - 1530.3 g/L).

The solids content of the paints prepared in the present paper ranged from 42.0 to 60.5%. It is observed that commercial paints have a lower solids content, ranging from 32.8 to 54.3%. According to Lopes *et al.* (2019), a higher solids content provides greater opacity to paints. Furthermore, a higher solids content indicates greater waste consumption in the case of paints produced with granite and marble waste, i.e., the paint is more environmentally friendly. The regression equation generated for this parameter indicates that the granite and marble wastes have a similar contribution and that the water has an almost null contribution compared to the other parameters.

Table 6 - **Valid polynomial models for the paints series with granite and marble waste pigments**

Regression Equations	R^2
SC= 97.28a + 99.61b + 3.69c + 45.06d	98.20%
pH = 7.05a + 7.46b + 7.66c + 5.01d + 1.79ab + 5.63ad + 5.44bd	87.62%
KV = 34.66a + 30.58b – 10.33c + 35.85d	85.26%
AV = 214.4a + 186.8b - 159.6c + 110.9d	82.24%
PV = 270.5a + 227.7b – 194.5c + 286.5d - 571.4ad - 544.6bd	96.12%
AR= - 51.79a – 98.20b + 29.49c + 614.09d +386.48ab	88.24%
HP= 20.71a + 16.07b + 1.95c – 20.88d – 23.39ab	81.81%

Note: a - Granite waste; b - Marble waste; c - Water; and d - Resin.

Table 7 - **Characteristics and properties of commercial paints**

Commercial Paint	SM (g/l)	SC (%)	pH	KV (s)	AV (cP)	PV (cP)	AR (cycles)	HP (m²/l)
A	1520.20	41.60	9.90	-	67.50	25.00	95.00	7.50
B	1391.80	43.30	10.60	-	105.00	43.00	28.00	6.90
C	1311.20	38.00	10.00	-	55.00	28.00	52.00	4.80
D	1232.10	32.80	7.80	-	65.00	42.00	26.00	8.50
E	1530.30	54.30	9.30	-	95.50	40.00	38.00	10.10
F	1260.60	35.80	9.00	-	77.50	31.00	155.00	7.60
G	1416.00	44.60	9.90	-	84.00	16.00	264.00	10.60
H	1278.80	35.90	8.80	-	112.00	17.00	290.00	11.10

Note: SC: Solids Content; SM: Specific Mass; KV: Kinematic Viscosity; AV: Apparent Viscosity; PV: Plastic Viscosity; AR: Abrasion Resistance; and HP: Hiding Power. The lines highlighted in gray indicate the commercial paints that presented the best performances.

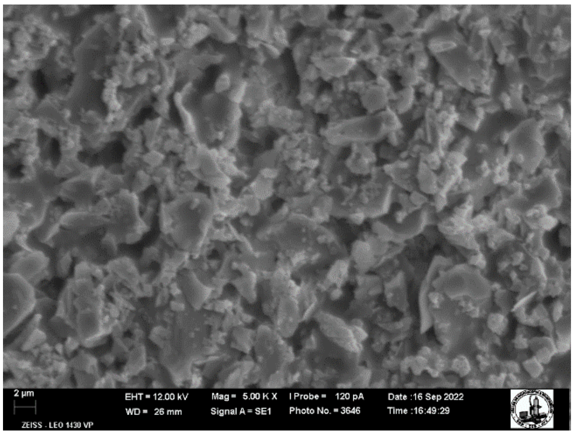
The pH of paints containing only granite waste ranged from 7.1 to 7.4. The paints produced only with marble waste had a pH in the range of 7.3 to 7.7. In addition, the paints produced with the two types of waste ranged from 7.4 to 7.7. These results are related to the pH values of the components used in the mixtures and their quantities: granite waste (pH = 8.89), marble waste (pH = 9.52), water (pH = 6.70), PVA resin (pH = 4.50). One can notice that the solution contains an excess of OH^- ions, indicating the occurrence of chemical reactions between the pigments and other paint components. According to the regression equation, significant interactions exist between the wastes and between the wastes and the resin, indicating a parabolic behavior of these components. Moreover, commercial paints have a higher pH, ranging from 7.8 to 10.6, due to the use of acrylic emulsion as resin, which has a pH in the range of 8.5 to 10.0. It is important to highlight that the main substrates used to apply real estate paints are cementitious materials with an alkaline pH. Therefore, if the paint has a lower pH, the substrate must receive a sealer or correction base to improve the adhesion of the paint, avoiding pathologies and increasing the durability of the finish (Fazenda, 2009).

In general, viscosity results from cohesion between fluid molecules, with kinematic (KV), apparent (AV) and plastic viscosity (PV) being measured. KV is a gravity-based measure; AV is measured at a single point, keeping shear constant; VP is a measure of the friction resulting from the collision between the particles. The results described by the regression equations revealed that water is the only component that contributes negatively to viscosity because the greater the amount of water, the lower the viscosity of the fluid. The highest viscosity values were observed for the paints with the addition of granite waste. The differences in viscosity caused by the type of pigment are due to their different morphological and physical characteristics, such as size, particle size distribution and specific surface area. Smaller particles with a higher specific surface area have a greater number of particle-particle interactions and an increase in flow resistance, leading to increased viscosity. This result was also observed by Wolosiak-Hnat *et al.* (2019) when comparing paints with additions of different pigments (TiO_2, ZnO, $BaSO_4$). It is important to emphasize that pigments have different chemical compositions. Therefore, another factor that can influence viscosity is the interaction of the resin with the pigments. In this sense, a significant negative interaction between the waste particles and the resin was observed only in the case of PV, indicating a parabolic behavior of these components. The results also proved that increasing the amount of PVA resin considerably increases the viscosity. In terms of comparisons with commercial paints, it is observed that the AV (17 to 84 cP) and PV (16 to 77 cP) of granite and marble waste paints presented ranges of variation in common with the AV (55 to 112 cP) and the PV (16 to 43 cP) of commercial paints. The differences in viscosity values may be attributed to the reduced size of the pigment particles used in commercial paints. It is important to emphasize that the viscosity of the paint affect the application of the product, the coverage, the visual aspect, the durability, and the resistance of the dry paint film. Therefore, the amount of solvent present in the paints was controlled (Bilgin *et al.*, 2022; Fazenda, 2009).

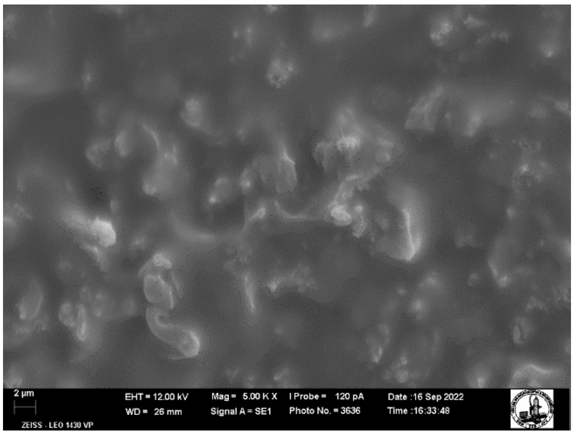
Abrasion resistance is especially affected by the amount of resin in the mixture. When formulating paints, it is necessary to add enough resin content to envelop the pigment particles and allow the paint to adhere properly to the desired surface (Sarkodie *et al*., 2019). With the results obtained evidenced that the resin variation range used in the experimental program is quite comprehensive. This allows for the production of paints with varying degrees of abrasion resistance, ranging from very low (indicative of low resin consumption) to higher levels (indicative of high resin consumption), which are compatible with the values required by NBR 15079-1 (ABNT, 2019b). Figure 5 presents electron microscope images of paints produced only with granite waste and paints produced only with marble waste. The paints with the highest amount of waste and those with the highest amount of resin were selected for these microscopic analyses. In Figures 5a and 5c, it is possible to observe a low resin content compared to the amount of pigments, resulting in low abrasion resistance. The smaller the amount of resin, the rougher the paint film, the greater the friction with the brush and the lower the abrasion resistance. In contrast, Figures 5b and 5d show an excess of resin associated with materials with high abrasion resistance. In Figure 5b, excess resin and a smoother surface are observed due to the smaller particle size of the granite residue, which guarantees fewer empty spaces between the grains and concentration of excess resin on the surface. In Figure 5d, there is also an excess of resin, but with a rougher surface due to the presence of larger particles and more spaces between them, which are being filled by the resin. Therefore, the excess resin is not entirely concentrated on the surface.

According to the regression equation, the wastes contribute negatively to abrasion resistance, and the marble waste exhibited the most harmful effects on this parameter. Figure 5 shows that the paint films with marble waste had larger particles, which provided greater roughness to the paint. On the other hand, granite waste had a finer granulometry and was predominantly formed by SiO_2, which is often used in the paint industry to improve abrasion and scratch resistance and provide surface superhydrophobic abilities (Al-Kattan *et al*., 2015; Nguyen; Nguyen; Nguyen, 2020). Therefore, paints produced with granite waste had a higher abrasion resistance.

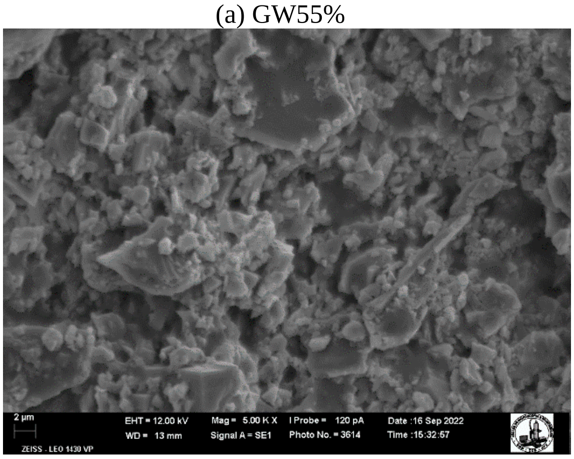
Figure 5 - **SEM of the paint films**



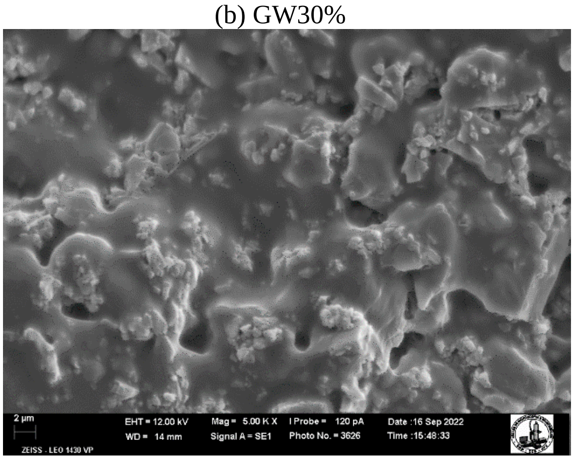
(a) GW55%



(b) GW30%



(c) MW55%



(d) MW30% (Mag.= 500 X)

The paints produced only with granite waste presented abrasion resistance ranging from 40 to 160 cycles. The abrasion resistance of marble waste paints ranged from 23 to 115 cycles. Additionally, paints produced with both granite and marble waste had abrasion resistance between 39 and 170 cycles. Commercial paints present an abrasion resistance ranging from 26 to 290 cycles. Therefore, it is possible to observe that all waste paints presented abrasion resistance similar to those of paints available in the market, except for the MW55.0% sample, which presented an abrasion resistance of 23 cycles. Low abrasion resistance causes reduced durability of coatings, rapid surface degradation due to contact with abrasive materials, and a decrease in the substrate's lifespan (Fazenda, 2009). Some commercial paints have better performance than the paints produced in this study. It happens because the paint industry generally works with pigments with particle sizes between 0.1 and 10 µm (Sarkodie *et al*., 2019), depending on the paint type and use. Moreover, acrylic resins and various additives are used in those materials, impacting production costs and the environment.

Regarding the hiding power, the regression equations indicate that the higher the percentage of pigments, the greater the paint coverage. In addition, paints produced with granite waste showed better hiding power than paints with marble waste. Pigment particle size is a critical parameter that affects surface finishing. According to Sarkodie *et al.* (2019), the tendency for a pigment to have good hiding power is also related to its good light-absorbing or -scattering properties. Fine particles produce a uniform surface because the angle of incident light is close to the angle of reflected light.

The paints produced only with granite waste presented hiding power ranging from 2.10 to 14.20 m²/L. The hiding power of marble waste paints ranged from 0.90 to 7.30 m²/L. Additionally, paints containing both granite and marble wastes had hiding power ranging between 1.50 and 6.90 m²/L. Comparing these values with those verified in commercial paints (ranging from 4.80 to 11.10 m²/L), it is possible to observe that several paints produced in this study presented hiding power values lower than the minimum value verified in commercial paints. In fact, the paint industry generally works with fine pigments down to the nanometer scale, high refractive index, various additives and chemical treatments/reagents to produce modified pigments for satisfactory applications (Sarkodie *et al*., 2019). It is crucial to highlight that low hiding power leads to inadequate coverage, increased product consumption to cover the substrate (requiring more layers), and reduced product yield (Fazenda, 2009).

It is important to note that abrasion resistance and hiding power are antagonistic properties. In the regression equations, it is possible to observe that the components that contribute positively to the hiding power have an opposite effect on abrasion resistance. Thus, it is necessary to determine the contents of each material that would provide satisfactory performance for the paint.

Figure 6 shows the response surface generated for the statistical desirability function (D). The red line highlights the region studied in this work. In some regions, it is possible to obtain desirability values equal to 1, meaning that there are paint samples with a hiding power greater than 4 m²/L and abrasion resistance greater than 100 cycles. These paints perform satisfactorily according to the economical category NBR 15079-1 (ABNT, 2019b). The paints with the best performances are those composed of 0.41-0.47 GW, 0.35 water and 0.18-0.24 PVA resin or 0.21-0.28 GW; 0.17-0.27MW; 0.35 water, 0.17-0.20 PVA resin. So, paints produced only with marble waste had less satisfactory performances.

Furthermore, the paints with the best performance had the lowest water contents used in this experimental design. However, it does not mean that water impairs the performance of paints. According to the regression equations, water contributes positively to abrasion resistance and hiding power. However, other components promote more significant performance gains. In addition, the paints composed of granite and marble waste required less resin to perform satisfactorily. This behavior can be attributed to the lower specific surface area of the marble waste particles, which would require a smaller amount of resin to cover the grains.

Figures 7 and 8 show comparisons between experimental analyses of samples of the commercial paints with the best performances (F, G and H) and granite waste and/or marble waste paints with the best performances (GW47.5%, MW42.5% and GW23.8%+MW23.8%). The SEM images indicated that commercial paints have a greater number of small pigment particles. However, it is also possible to identify larger particles, probably the mineral fillers used in industrial paints. Furthermore, an excess of resin cannot be seen in SEM images of commercial paints. In contrast, Figures 7a, 7b, and 7c show some regions of high resin concentration, which may even indicate a non-uniform distribution of the binder in the paint film.

The profilometry images of the paint films illustrate that the smallest pigment particles promote less roughness (RG) to the film, which impacts the resistance to scratching and abrasion. According to Figure 8, the roughness of waste paints is more than 200% higher than the roughness of commercial paints.

Figure 6 - **Response surface generated for the statistical desirability function (D)**

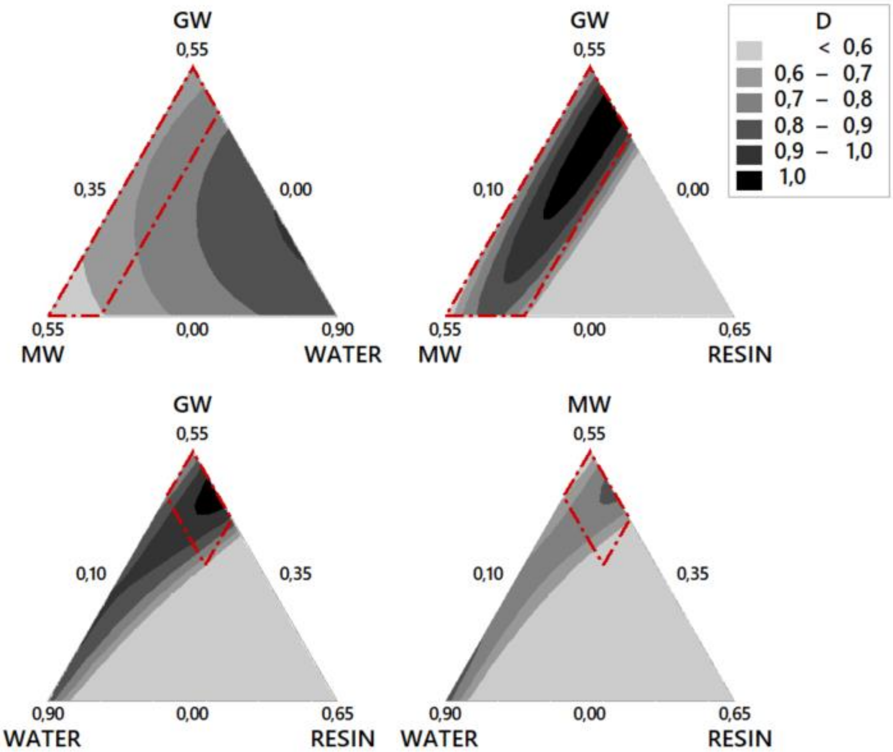
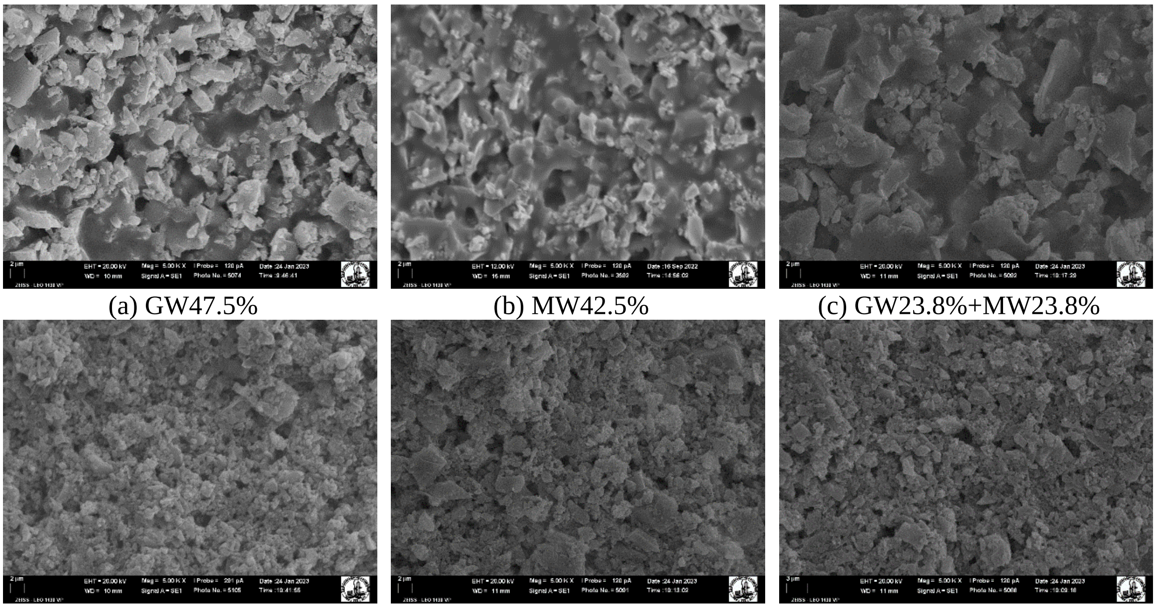


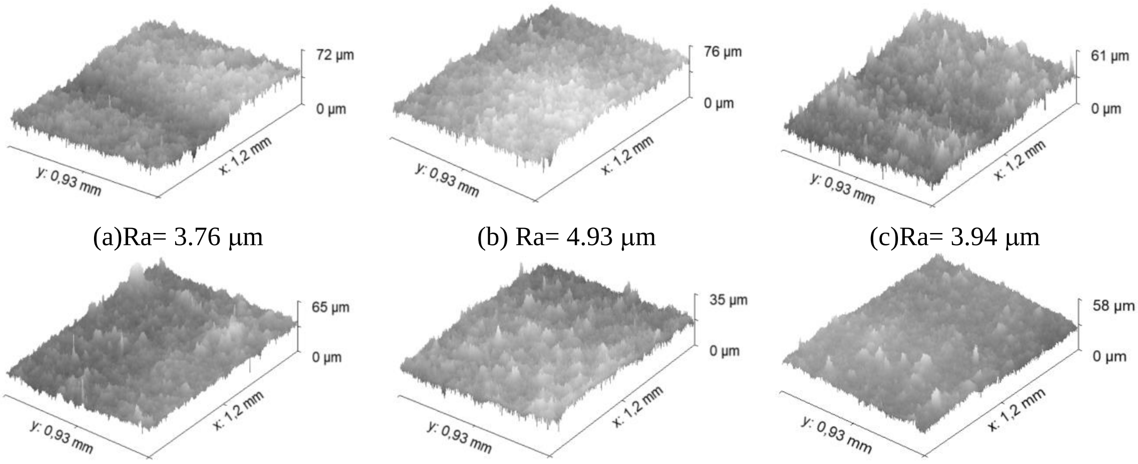
Figure 7 - **SEM of paint films**



(a) GW47.5% (b) MW42.5% (c) GW23.8%+MW23.8%

(d) F (e) G (f) H

Figure 8 - **Profilometry of paint films: (a) GW47.5%; (b) MW42.5%; (c) GW23.8%+MW23.8%; (d) F; (e) G; (f) H**



(a)Ra= 3.76 µm (b) Ra= 4.93 µm (c)Ra= 3.94 µm

(d) Ra= 2.69 µm (e) Ra= 2.38 µm (f) Ra= 1.77 µm

Note: Ra: Roughness.

Conclusion

This study aimed to optimize the composition of paints produced using waste materials from ornamental stone processing by employing a quaternary mixture design, with the goal of achieving performance compatible with technical standards and commercial paints. The following conclusions were drawn from the results of the experimental tests:

(a) the experimental design of mixtures composed of four components (granite waste, marble waste, water, and PVA resin) allowed for the development of polynomial models that described the characteristics and properties of the paints;

(b) although a special cubic model with fourth-order terms was utilized, most of the polynomial models exhibited linear behavior, indicating that the interactions between the components were not statistically significant;

(c) paints formulated with granite waste demonstrated superior hiding power and abrasion resistance compared to those produced with marble waste. This can be attributed to the smaller particle size and favorable chemical composition of granite waste for paint formulation;

(d) the hiding power was positively influenced by the type and amount of pigment in the mixture, while the resin content positively affected abrasion resistance;

(e) the optimal paint formulations consisted of 41-47% granite waste, 35% water, and 18-24% PVA resin, or alternatively, 21-28% granite waste, 17-27% marble waste, 35% water, and 17-20% PVA resin. These formulations met the minimum requirements for hiding power and abrasion resistance set by the technical standards for the economic paint category; and

(f) the optimal paints developed in this study exhibited similar values of solids content, hiding power, and abrasion resistance compared to commercial paints. However, it is important to note that the granite and marble waste paints did not contain additives, acrylic resin, or undergo complex pigment treatments. Consequently, the paints developed in this research offer environmentally friendly alternatives.

Overall, this study demonstrates the potential of utilizing waste materials from ornamental stone processing to produce paints with satisfactory performance and reduced environmental impact. The optimized formulations can contribute to the development of sustainable paint solutions that align with technical standards and offer economic advantages. It is important to emphasize that further complementary studies are needed to assess the durability of these paints. Additionally, efforts should be made to explore the use of additives to enhance the performance (abrasion resistance and hiding power) and durability of the developed product. Future research is also strongly recommended to investigate the effects of different types of waste grinding treatments (variations in grinding equipment, duration, speed, etc.) to further understand their impact on the performance of the sustainable paints. Cost-effectiveness assessments of paint production should also be conducted to express the economic viability of the product.

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