

FEASIBILITY STUDY OF THE USE OF IRON ORE TAILINGS AS PIGMENT IN POLYPROPYLENE RESINS

ESTUDO DA VIABILIDADE DA UTILIZAÇÃO DE REJEITO DE MINÉRIO DE FERRO COMO PIGMENTO EM RESINAS DE POLIPROPILENO

ESTUDIO DE VIABILIDAD DEL USO DE RESIDUOS DE MINERAL DE HIERRO COMO PIGMENTO EN RESINAS DE POLIPROPILENO

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ABSTRACT

The Brazilian mining industry plays a significant role in the country's economy. However, it also generates large volumes of waste during the ore beneficiation process, resulting in severe environmental impacts. Recent researches have explored the potential of iron ore tailings as a raw material in the manufacturing of construction materials, aiming to reduce environmental impact. The distinctive coloring acquired by the produced materials can contribute to their application in certain segments where aesthetic factors are relevant. This study aimed to evaluate the feasibility of iron ore tailings, derived from the dam breach in Mariana-MG, as a pigment for polypropylene, widely used as a polymer matrix for composites, especially wood-plastic composites. Samples with different polypropylene and iron ore tailing contents, ranging from 1 to 10 % by mass, were developed using thermokinetic mixing, milling, extrusion, pelletization and hot compression processes. The produced specimens were analyzed for their appearance and evaluated for their mechanical performance. The results showed that the tailings presented high pigmentation power when incorporated into polypropylene, without compromising its mechanical properties and even providing a gain in flexural strength. However, the proportion between the amount of residual material used and the total volume of waste discarded in nature is very low, indicating limited potential for material reuse. This situation can be modified by using the tailings in the production of wood-plastic composites, where, in addition to acting as pigment, they can also act as reinforcing filer. This approach would allow the utilization of a considerable larger portion of the residual material.

KEYWORDS

Iron ore tailings, Pigmentation, Civil Engineering, Polymeric matrix.

RESUMO

A indústria mineradora brasileira desempenha um papel significativo na economia do país, no entanto, ela também gera grandes volumes de rejeitos durante o processo de beneficiamento dos minérios, resultando em impactos ambientais severos. Pesquisas recentes têm explorado o potencial do rejeito de minério de ferro como matéria-prima na fabricação de materiais de construção, com o objetivo de promover a redução do impacto ambiental. A coloração característica adquirida pelos materiais produzidos pode contribuir na sua aplicação em alguns



segmentos, onde o fator estético é relevante. Neste estudo, buscou-se avaliar a aplicabilidade do rejeito de minério de ferro, proveniente do rompimento de barragem ocorrido em Mariana-MG, como pigmento do polipropileno, amplamente utilizado como matriz polimérica de compósitos, em especial de madeiras plásticas. Foram desenvolvidas amostras com diferentes teores de polipropileno e rejeito de minério de ferro, este variando de 1 a 10 % em massa, a partir dos processos de mistura termocinética, moagem, extrusão, peletização e compressão a quente. Os corpos de prova produzidos foram analisados quanto à sua aparência, e avaliados quanto ao seu desempenho mecânico. Os resultados obtidos demonstram que o rejeito possui alto poder de pigmentação quando incorporado ao polipropileno, além de não comprometer sua performance quanto às propriedades mecânicas, proporcionando inclusive ganho de resistência à flexão. No entanto, a proporção entre a quantidade de material residual utilizada e o volume total de rejeito descartado na natureza é muito baixa, sugerindo limitado potencial de reaproveitamento do material. Essa situação pode ser modificada ao empregar o rejeito na fabricação de madeira plástica, onde além de desempenhar a função de pigmento, ele pode também atuar como carga de reforço. Essa abordagem permitiria a utilização de uma parcela consideravelmente maior do material residual.

PALAVRAS-CHAVE

Rejeitos de minério de ferro, Pigmentação, Engenharia Civil, Matriz polimérica.

RESUMEN

La industria minera brasileña desempeña un papel significativo en la economía del país. Sin embargo, también genera grandes volúmenes de residuos durante el proceso de beneficio del mineral, lo que resulta en graves impactos ambientales. Investigaciones recientes han explorado el potencial de los residuos de mineral de hierro como materia prima en la fabricación de materiales de construcción, con el objetivo de reducir el impacto ambiental. La coloración distintiva adquirida por los materiales producidos puede contribuir a su aplicación en ciertos segmentos donde los factores estéticos son relevantes. Este estudio tuvo como objetivo evaluar la viabilidad de los residuos de mineral de hierro, derivados del colapso de la represa en Mariana-MG, como pigmento para el polipropileno, ampliamente utilizado como matriz polimérica para compuestos, especialmente compuestos de madera y plástico. Se desarrollaron muestras con diferentes contenidos de polipropileno y residuos de mineral de hierro, que varían del 1 al 10% en masa, utilizando procesos de mezcla termocinética, molienda, extrusión, peletización y compresión en caliente. Los especímenes producidos se analizaron en cuanto a su apariencia y se evaluaron en cuanto a su rendimiento mecánico. Los resultados mostraron que los residuos presentaron un alto poder de pigmentación cuando se incorporaron al polipropileno, sin comprometer sus propiedades mecánicas e incluso proporcionando una ganancia en la resistencia a la flexión. Sin embargo, la proporción entre la cantidad de material residual utilizado y el volumen total de residuos descartados en la naturaleza es muy baja, lo que indica un potencial limitado para la reutilización del material. Esta situación puede modificarse mediante el uso de los residuos en la producción de compuestos de madera y plástico, donde, además de actuar como pigmento, también pueden actuar como material de refuerzo. Este enfoque permitiría la utilización de una porción considerablemente mayor del material residual.

PALABRAS CLAVE

Residuos de mineral de hierro, Pigmentación, Ingeniería Civil, Matriz polimérica.

1. INTRODUCTION

The Brazilian mining industry holds a strategic position in the country's economy, notably due to its significant contribution to iron ore production, representing approximately 1.4% of Brazil's Gross Domestic Product (GDP), according to data from IBRAM (2019). Although it has a crucial role in economic development, this industrial activity is also a source of considerable environmental impacts, particularly due to the generation of large volumes of waste during the ore beneficiation process. The growing global demand for iron ore has led to the exploitation of lower grade deposits, which has resulted in an increase in waste generation, requiring increasingly larger tailings dams for containment (CARNEIRO, 2020). According to the National Mining Agency (ANM, 2023), iron ore production reached approximately 430 billion tons in 2022, and the Institute for Technological Research (IPT, 2016) estimates that each ton of processed ore results in 0.4 tons of tailings, indicating the production of around 172 million tons of iron ore tailings (IOT) that year alone.

The challenges related to mining and tailings management became particularly evident after the disaster with the IOT dam in Mariana-MG, in November 2015, which resulted in severe environmental and social impacts (COURA, 2018). Approximately 40 million cubic meters of tailings were released, affecting 663 kilometers of watercourses and 1,469 hectares of vegetation (CARNEIRO, 2020), in addition to directly impacting about 2 thousand people, including casualties, injuries, illness, homeless, displaced and missing, as indicated by the Mariana Task Force report (2016).

Recently, researches have focused on the reuse of the IOT from the Mariana disaster as a raw material in the construction industry, aiming to transform this environmental liability into a resource. Studies indicate that the tailings can be incorporated into various construction materials, such as ceramic tiles (RUY, 2022), asphaltic mixtures (MORAES, 2022) and ceramic blocks (MENDES, 2019). In addition to enhancing certain properties, the characteristic coloring acquired by the materials when incorporating the tailings can favor their application in specific segments where aesthetic appeal is a significant differentiator.

Particularly, Moraes (2022) explored the use of IOT as a substitute for stone powder in asphalt mixtures, observing significant improvements in reflectance and reduction of surface temperature, which can mitigate the urban heat island phenomenon. Nascimento (2019)

evaluated the use of IOT as pigment in concretes, noting not only an aesthetic alteration with the acquisition of a reddish color but also improvements in mechanical strength and durability of the material.

Another innovative application of IOT is in the wood-plastic composites, which are formed by a polymeric matrix and a reinforcement phase, both organic and inorganic (BRASKEM, 2016). In this application, IOT acts both as an inorganic reinforcing filler and as a pigment, a fundamental component for these composites. Santos *et al.* (2022) emphasize that the visual aspect of wood-plastic composites, which should resemble natural wood, is essential to valorize the material and promote its acceptance in the market as an alternative to tree species. The relevance of inorganic pigments, such as IOT, also lies in their thermal resistance, which is especially useful as many polymers require molding temperatures above 200 °C (JAFELICCI, 2019). Studies by Bressiani *et al.* (2021) and Coura (2018) show that the addition of IOT in polymeric matrices improves both the aesthetic characteristics and the mechanical and thermal properties of the composites.

In the composition of wood-plastic composites, thermoplastic polymers are often preferred, and amongst these, Polypropylene (PP) stands out due to its mechanical strength, lightweight, transparency, ease of recycling and high processing capability (MONSORES *et al.*, 2017). Thus, this study aims to explore the impact and feasibility of using IOT, derived from the tragic collapse of the Fundão dam in Mariana-MG, as a pigment agent in PP. This investigation seeks to expand the applicability of PP not only as a substrate for wood-plastic composites but also in other composites where aesthetic value is important. Furthermore, this study proposes to analyze how IOT affects the mechanical properties of PP, varying according to the amount of tailings incorporated into the composition.

2. METHODOLOGY

2.1 Preparation of testing specimen

In this study, the materials used were Iron Ore Tailings (IOT) and virgin Polypropylene (vPP). The IOT, collected after the dam's collapse in Mariana-MG, was acquired already processed by a mining company located in Congonhas-MG, dried and without agglomerations. The vPP, provided by Braskem, has a flow index of 3.5 g/10 min and density of 0.905 g/cm³ (BRASKEM, 2020).

For the production of the specimens, the vPP and IOT were thermokinetically homogenized, enhancing the dispersion of IOT and vPP. For this purpose, a laboratory homogenizer (model MH-100, MH Equipments) was used. Initially, the proportion of IOT ranged from 1 to 5%, aiming to highlight its function as a pigment instead of acting as a reinforcement filler. Moreover, samples were prepared with 10% of IOT to evaluate its influence on the mechanical properties. Additional samples with 2% of IOT incorporated with Industrial Black recycled Polypropylene (IBrPP) and Laboratory Blue recycled Polypropylene (LBrPP), from Nonwoven fabric (NW) from masks, were also produced to analyze the IOT's pigment capability to cover the recycled polymers' colors. The samples' composition is described in Table 1.

Sample	PP Mass (g)	IOT Mass (g)	PP (%m)	IOT (%m)
100vPP	180	0,00	100	0
99vPP1IOT	180	1.82	99	1
98vPP2IOT	180	3.67	98	2
97vPP3IOT	180	5.57	97	3
95vPP5IOT	180	9.47	95	5
90vPP10IOT	180	20.00	90	10
98IBrPP2IOT	180	3.67	98	2
98LBrPP2IOT	180	3.67	98	2

Table 1 – Composition of the samples used

As mentioned, the procedure involved a thermokinetic mixer, where the fusion and homogenization of the polymer with the IOT were obtained by the heat generated by friction and rotation. The resulting product was then cooled and ground in a knife mill (Marconi, model MA 580) to obtain smaller particles. These particles were subsequently extruded in a single-screw extruder (Thermo Scientific, HAAKE Polylab), set at 175 °C in three heating zones and a speed of 45 rpm. The extruded material, with an average diameter of 3.40 mm, was cut into pellets approximately 3.70 mm in length using a pelletizer (AX Gran, AXPlástico). Finally, the pellets were molded into plates of 150 mm x 100 mm x 1 mm, using a heated hydraulic press (SOLAB, model SL11) at 180 °C under a pressure of 25 MPa for 5 minutes. The plates, after pressing and cooling, were cut into test specimens with dimensions of 10 mm x 13 mm x 1 mm for subsequent analyses. The experimental procedures are outlined in the flowchart in Figure 1.



Figure 1: Flowchart of raw materials and experimental procedures.

2.2 Mechanical Characterization

The mechanical characterization of the samples was performed following standardized procedures. For the tensile tests, six specimens of each composition, including the control sample (100% vPP) and the blends with IBrPP and LBrPP, were tested in a universal testing machine (Instron, model EMIC 23-20) at a speed of 50 mm/min, according to ASTM D638 (2014) standard. The results represent the average of the six tests performed. Flexural strength was determined using the same testing machine, operating at a speed of 30 mm/min in alignment with ASTM D790 (2007) standard. Four specimens of each composition were subjected to the test, and the results obtained are the arithmetic mean of these measurements.

3. RESULTS AND DISCUSSION

3.1 Sample Analysis

The visual analysis of the specimens, as illustrated in Figure 2, reveals the influence of the Iron Ore Tailings (IOT) on the coloration of the Polypropylene (PP), with variations proportional to the amount of IOT incorporated. The designation of the specimens reflects their specific compositions, for example, the sample 99vPP1IOT consists of 99% by mass of virgin Polypropylene (vPP) and 1% by mass of IOT. Control samples were prepared using only pure resin (100vPP), and comparative samples were prepared using Industrial Black recycled PP (98IBrPP2IOT) and Laboratory Blue recycled PP (98LBrPP2IOT).

It was observed that the IOT provided a distinctive coloration to the PP, demonstrating its effectiveness as a pigment even at low concentrations. In the specimens produced with vPP (transparent), the intensification of the

hue was notable with increasing IOT content, although the samples with 3, 5 and 10% IOT presented indistinctive colors when observed under the same ambient light. This observation is consistent with Chen et al. (2016), who highlighted the effectiveness of inorganic pigments in imparting color to light-toned polymers, maintaining thermal stability even at low concentrations.

Samples containing recycled PP exhibited distinctive behaviors regarding coloration. The 98IBrPP2IOT sample did not show significant color change due to its original dark hue. On the other hand, adding IOT to the LBrPP resulted in a greenish coloration, indicating that the polymer base decisively influences the final coloration of the specimens.



Figure 2: Samples' testing specimen.

The prevalence of a red-ochre hue in the samples indicates the presence of hematite (Fe_2O_3) in the tailings (BRANCO, 2015). This composition was confirmed by Coura (2018), who characterized a sample of IOT from the same origin through X-ray fluorescence analysis and found a predominance of 58.1% of iron oxide. The same hue was verified in the study conducted by Galvão et al. (2018), in which IOT was used as a base to reddish pigments in the production of sustainable paints.

When examining the resulting plates under direct light (Figure 3), a clearer differentiation between the samples was observed, highlighting the influence of the IOT content on coloration and opacity. The vPP, due to its translucent nature, when combined with IOT, a material of high opacity, especially when compared to organic pigments (TORRES, 2018), resulted in specimens with noticeable variations in transparency and color.



Figure 3: Samples' plates against light.

While the images of the specimens (Figure 2) initially suggest a seemingly homogeneous mixture of the samples, the analysis of the plates under direct light (Figure 3) reveals areas with varied transparency in samples containing up to 5% of IOT. This heterogeneity is less pronounced in the sample with only 1% of tailings. Such variation in coloration and opacity across the same plates suggests that the pelletized IOT grains exhibit a diversity in pigment intensity, resulting in a non-uniform color distribution. This phenomenon indicates a possible loss of homogeneity in the mixture, believed to have occurred during the extrusion process. During this processing phase, it is likely that the unintentional introduction of additional vPP, used in cleaning the extruder before and after extrusion, contributed to the observed variation, subtly modifying the proportion of the mixture. Notably, the sample containing 10% IOT stands out significantly for the increased opacity compared to the other samples made from vPP, reinforcing the influence of IOT concentration on the optical properties of the material.

In addition to coloration, the effectiveness of a pigment is highly affected by the average particle size and its granulometric distribution, as emphasized by Buxbaum and Pfaff (2005). In this context, IOT emerges as a raw material with promising potential. This is because, for the efficient applicability of a residue rich in iron oxide as a pigment, it is essential that the raw material possess a granulometric distribution concentrated in ranges of smaller particle sizes. This characteristic is essential, as it can directly affect the hue manifested by hematite, ranging from red to intense violet (TAVARES, 2012; MONTEDO et al., 2004). A granulometric analysis of the IOT conducted by Coura (2018) revealed that the particles of the materials used in the present work are

distributed in a range from 0.01 to 70 μm , with about 50% of the material having a diameter smaller than 8.16 μm . These values are comfortably within the limits considered ideal for pigments, which are between 0.01 and 10 μm , as described by Buxbaum and Pfaff (2005).

3.2 Mechanical resistance

Figures 4 and 5 show the Stress/Strain curves and the average values of the modulus of elasticity, respectively, obtained by the tensile strength tests.

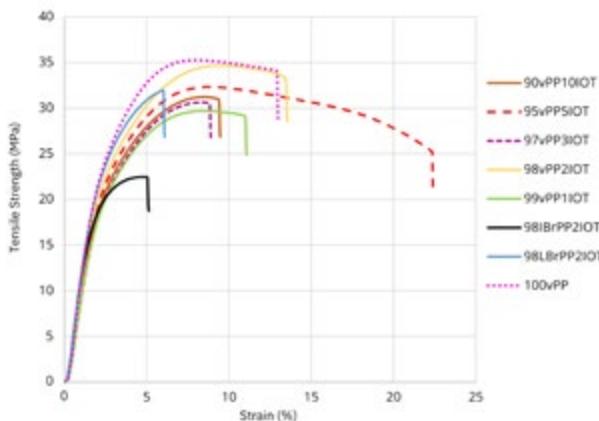


Figure 4: Tensile strength and strain of the samples.

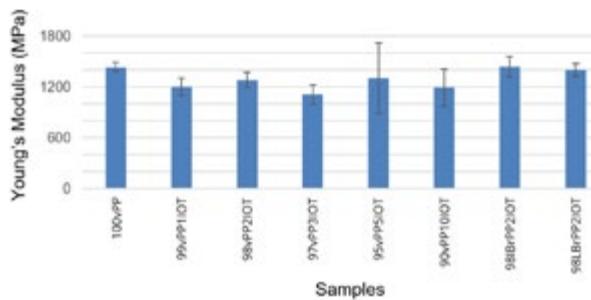


Figure 5 – Tensile modulus of elasticity of the samples.

In general, the addition of IOT resulted in a decrease in the tensile strength of the vPP. However, this behavior was not linear. For example, when compared to the control sample (100vPP), the addition of 1% IOT resulted in a 15.80% loss of strength, while the addition of 2% IOT presented a reduction of only 1.67%. Samples produced with a recycled PP matrix also showed inferior performance compared to the vPP, specially the 98IBrPP2IOT sample standing out for having the lowest tensile strength. However, all samples, except for the latter mentioned, achieved tensile strengths superior or

very close to the expected values for vPP, according to the literature: from 31 to 41.4 MPa (CALISTER; RETHWISCH, 2012), indicating that the incorporation of IOT at low levels does not significantly compromise the performance of PP in this property.

The analysis of the modulus of elasticity graphs reveals that the addition of tailings did not result in an increase in this property, unlike what was observed by Coura (2018), who found a gradual increase in the value of the modulus of elasticity in composites with 20% IOT. It is worth remembering that Coura (2018) produced composites with 20% IOT so that the tailings would act as reinforcement filler, unlike the present study in which the focus is the pigmentation of PP. With lower IOT contents, a variation in the modulus of elasticity was observed, with only the comparison sample 98IBrPP2IOT exceeding the average value achieved by the vPP. However, all the samples presented stiffness values within the expected range for PP, according to Calister and Rethwisch (2012): 1140 to 1550 MPa, indicating that the pigmentation of PP with IOT does not pose risks to its stiffness.

Figures 6 and 7 present the Stress/Strain curves and the average values of the modulus of elasticity, respectively, obtained by the flexural strength tests.

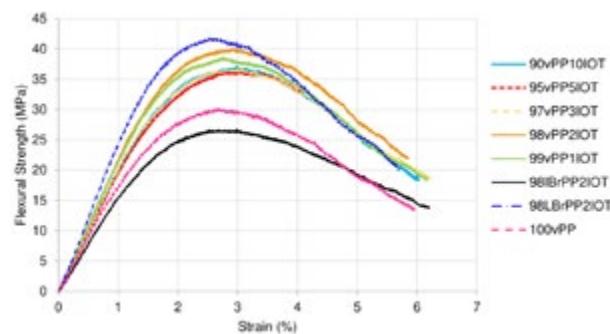


Figure 6 – Flexural strength and strain of the samples.

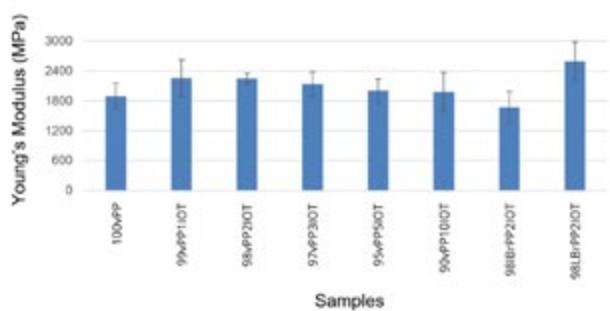


Figure 7 – Flexural modulus of elasticity of the samples.

In contrast to the tensile strength tests, an improvement in the performance of vPP was observed with the addition of IOT regarding flexural strength and stiffness. The sample with addition of 2% IOT even presented a 28% gain compared to vPP. The two comparison samples exhibited extreme performances, with 98IBrPP2IOT showing the worst strength while 98LBrPP2IOT achieved the highest. Although a progressive decrease in the stiffness of the specimens with the increase of IOT content was observed, all exceeded the modulus of elasticity of the vPP. The PP recycled from NW fabric also stood out regarding stiffness, possibly due to the matrix used. Thus, the addition of IOT did not compromise the behavior of vPP regarding flexion, enhancing its resistance, which is advantageous in applications such as decks in civil construction, one of the main applications of wood-plastic composites.

4. CONCLUSION

Based on the results obtained and the analysis performed, this study demonstrates that the Iron Ore Tailings (IOT) from the collapse of the Fundão dam in Mariana-MG can be effectively used as a pigment agent in the coloration of polymers, particularly Polypropylene (PP). The significant coloration achieved with minimal additions of IOT, and its proportional intensification with increasing concentrations of IOT in the samples, highlight its efficiency as a pigment. Despite small reductions in the tensile strength of virgin PP (vPP) due to the addition of IOT, the values remained close to those expected for vPP, as described in the literature. Regarding flexural properties, an increase up to 28% in material strength was observed with the addition of IOT.

It is noteworthy that the pigmentation is often achieved with low levels of pigment material, resulting in a low volume of IOT reused compared to the generated waste, raising questions about the environmental efficiency of this technique. However, the results of this study demonstrate the promising potential of mining waste as a highly efficient and economic viable option for the pigment industry. Moreover, they contribute to the valorization of industrial by-products and the reduction of the environmental impacts associated with improper disposal of these residues.

In addition to the results achieved, it is important to highlight that the shades obtained in this study evoke familiar tones, with an appearance capable of eliciting affective memories associated with natural wood. This

aspect makes IOT a promising option for coloring wood-plastic composites, providing an attractive and familiar aesthetic to the final products. For future research in this field, it is recommended to conduct a wider variety of tests, especially those aimed at evaluating the performance of IOT as a pigment in different scenarios. This includes darkness and accelerated weathering tests, aiming to optimize the efficiency of IOT as a pigment and explore its potential contribution to sustainability.

It is also critical to evaluate tests with wood-plastic composites, both with and without the presence of IOT, to understand how this would affect the production volume of the composite. Even though there are doubts about the potential reuse of IOT solely for coloring pure polymers, its use in the wood-plastic composite production offers a different perspective. In this case, the residual material does not only act as a pigment but also serve as reinforcement, allowing for the utilization of a substantially larger volume of waste.

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