

Characterization of thermoplastic sintering properties: an exploratory study to evaluate its feasibility as an effective manufacturing method

Caracterização das propriedades de sinterização de termoplásticos: um estudo exploratório para avaliar sua viabilidade como um método eficaz de fabricação

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Resumo

Métodos de fabricação envolvem técnicas para produzir diversas peças e produtos, evoluindo com as mudanças tecnológicas, econômicas e sociais. A metalurgia do pó, uma metodologia notável, utiliza a sinterização de partículas para criar peças sólidas complexas, precisas e eficientes, superando métodos tradicionais baseados em remoção de material. Embora amplamente aplicada a materiais metálicos e cerâmicos, a exploração da sinterização em materiais poliméricos é limitada, exigindo estudos adicionais para avaliar a viabilidade. Se comprovada viável, essa técnica poderia revolucionar a reciclagem de materiais termoplásticos, oferecendo benefícios ambientais e promovendo uma economia circular sustentável. Este estudo concentra-se na caracterização das propriedades de sinterização do Poliamida-6 (Nylon) e do Ácido Polilático (PLA), compreendendo seu impacto nas propriedades finais do material. Posteriormente, testes destrutivos, como o teste de flexão de três pontos conforme ASTM D790, serão realizados em amostras para determinar propriedades mecânicas. O objetivo é validar a sinterização como um método eficaz para a fabricação de estruturas termoplásticas, analisando propriedades mecânicas e estruturais. Além disso, o estudo visa identificar limitações, desafios e oportunidades para a melhoria do método, avançando nas aplicações industriais e na qualidade do processo.

Palavras-chave: Métodos de Fabricação. Termoplástico. Sinterização. Nylon. PLA.

Abstract

Manufacturing methods involve techniques to produce diverse parts and products, evolving with technological, economic, and societal changes. Powder metallurgy, a notable methodology, utilizes

particle sintering to create complex, precise, and efficient solid parts, surpassing traditional removal-based methods. While extensively applied to metallic and ceramic materials, the exploration of sintering in polymeric materials is limited, necessitating further studies to assess feasibility. If proven viable, this technique could revolutionize thermoplastic material recycling, offering environmental benefits and fostering a sustainable circular economy. This study focuses on characterizing the sintering properties of Polyamide-6 (Nylon) and Polylactic Acid (PLA), understanding their impact on final material properties. Subsequently, destructive tests, like the three-point bending test per ASTM D790, will be conducted on specimens to determine mechanical properties. The aim is to validate sintering as an effective method for thermoplastic structure manufacturing, analyzing mechanical and structural properties. Additionally, the study aims to identify limitations, challenges, and opportunities for method improvement, advancing industrial applications and process quality.

Keywords: Manufacturing Methods. Thermoplastic. Sintering. Nylon. PLA.

1. Introduction

Sintering is a manufacturing methodology that involves the production of parts through the compaction of powdered materials followed by a thermal treatment, aiming to enhance the final piece by relieving stresses, adjusting the material's microstructure, and improving mechanical properties. This combination of compaction and thermal treatment offers the possibility to manufacture parts with complex geometries and customized properties, making it a manufacturing technique with potential applicability in high-value sectors, such as the automotive, aerospace, and biomedical industries.

Over the past two decades, sintering has emerged as a promising technology in the manufacturing of various materials, such as metals, ceramics, and their composites (Boulman et al., 2022). However, the application of sintering technique to polymeric materials remains relatively unexplored and requires further studies to assess its feasibility and effectiveness. This technique has the potential to be applied in the recycling of thermoplastic materials, which could provide significant environmental benefits by reducing the impact of improper disposal of these materials (Furtado et al., 2016).

This study aims to characterize the sintering properties of the thermoplastic materials Polyamide-6 (Nylon) and Polylactic Acid (PLA). Additionally, it seeks to validate powder metallurgy as an effective manufacturing technique for producing thermoplastic structures, aiming to identify potential limitations, challenges, and opportunities for improvement in this manufacturing method. A comprehensive understanding of the sintering properties of these thermoplastic materials and an assessment of the feasibility of powder metallurgy as a manufacturing method will provide a better insight into the capabilities and limitations of this technique.

The attainment of reliable results in this study will drive significant advancements in the field of thermoplastic sintering, opening new possibilities for the utilization of this technique in the manufacturing of complex and functional thermoplastic components.

2. Methods

2.1 Characterization

Characterization of raw materials: The behavior of powders during compaction and sintering operations depends on the individual characteristics of the particles, such as their morphology, dimensions, and size distribution. The powders used in this study were analyzed concerning the size of their particles (granulometric analysis) and compacted using two types of matrices: a cylindrical one for studying sintering properties and another with dimensions suitable for flexural testing following ASTM D790 standard.

Physical characterization of green compacts:

Green compact relative density: For the characterization of their physical properties, the green compact relative density method was employed on the cylindrical matrix samples. The green compact relative density (ρ_r) achieved by the test specimens after compaction is determined by the ratio of the real specific mass of the test specimen to the theoretical specific mass. The green compact relative density was calculated using the following expression, equation 1:

$$\rho_r = \frac{\rho_{\text{real}}}{\rho_t} \times 100, \quad (1)$$

Where ρ_r is the green compact relative density, [g/ cm³], ρ_{real} is the real specific mass of the green compact, [g/ cm³], and ρ_t is the theoretical specific mass of the material under study, [g/ cm³].

The pressure used in the compaction process is crucial for determining the green compact relative density; hence, the choice of an ideal tonnage is necessary. The compactions were carried out using a Hydraulic Press 15T Riosul Tools for applying the required pressure.

Sintering of samples: The green compacts, after uniaxial compaction, were subjected to a heat treatment, where the sintering process took place. Several tests were conducted on cylindrical matrix specimens, varying the sintering temperature and exposure time parameters. The objective of these tests was to select a range of temperature and exposure times that resulted in a sample with satisfactory appearance and strength, using the characteristics of the injected PLA as a parameter. The sintering process was performed in a conventional muffle furnace.

Densification: Densification (D) is a parameter used to quantify the change in specific mass of a compact after the sintering process. It can be calculated using equation 2:

$$D = \frac{\rho_s - \rho_v}{\rho_t - \rho_v} \times 100, \quad (2)$$

Where D is the densification, [%], ρ_s is the specific mass of the sintered sample, [g/ cm³], ρ_v is the specific mass of the green compact, [g/ cm³], and ρ_t is the theoretical specific mass of the material being worked [g/ cm³].

The value of densification represents a relative variation in the specific mass of a material, resulting from changes in its dimensions due to the sintering process. Positive values of (D) indicate a contraction of the material in its original dimensions, while negative values of (D) indicate expansion or dilation of the material.

Relative density of the sintered sample: For a better understanding of the density variations, the relative density of the sintered specimens was calculated in relation to the theoretical density of the material. The relative density is calculated using equation 3.

$$\rho_{rs} = \frac{\rho_v}{\rho_t} \times 100, \quad (3)$$

Where ρ_{rs} is the relative density of the sintered sample, [%], ρ_v is the specific mass of the green compact, [g/ cm³], and ρ_t is the theoretical specific mass of the material being worked [g/ cm³].

Porosity: Porosity was calculated based on the theoretical specific mass of PLA of 1,252 g/cm³ (Farah, Shady, et al., 2016) and the density of the sintered sample. Porosity is obtained using the equation 4.

$$P = 1 - \frac{\rho_s}{\rho_t} \times 100, \quad (4)$$

Where P is the porosity of the sample, [%], ρ_s is the density of the sintered material, [g/cm^3], and ρ_t is the theoretical density of the material under study [g/cm^3].

Mechanical characterization of sintered compacts:

Three-point bending test: To analyze the strength of the sintered samples, a test was conducted according to the ASTM D790 standard. Six specimens were subjected to a destructive three-point bending test. Based on the data obtained, it was possible to compare the strength of the sintered samples with the data from the injected PLA as a reference. Figure 01 illustrates the test configuration.



Figure 1 - Three-point bending test

3. Results and Discussion

3.1 Granulometric analysis of materials

Through data provided by the suppliers of the raw materials, the particle size distribution of the materials used can be seen in Table 01.

Table 1 – Granulometric of Raw Materials.

Material	Granulometry [mesh]
Nylon	7 to 100
PLA	100

Upon analyzing Table 01, it is evident that there is an irregularity in the particle size of Nylon. This irregularity has a negative effect on the material's compaction process, as the lack of uniformity in particle size during uniaxial compaction can lead to inadequate density and undesired heterogeneity in internal stresses, as depicted in Figure 02. These factors can directly impact the sintering stage during the material's thermal process.

To ensure the integrity and reliability of the results, a decision was made to exclude the investigation of Nylon in this study due to its inconsistent and inadequate data. This allowed the focus to be directed solely on materials exhibiting more favorable characteristics for the compaction and sintering processes.

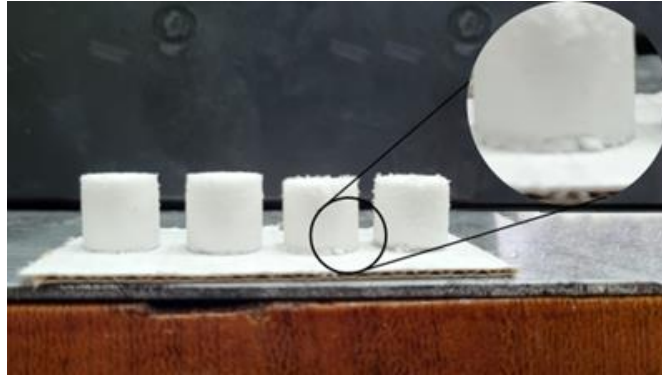


Figure 2 - Heterogeneity observed in the Nylon sample

3.2 Sinterization Properties

Compaction pressure: After selecting PLA as the main material of study for this work, its samples were subjected to different compaction pressures as shown in Table 02.

Table 2 - Table of Relative Density

Pressures used / Mpa	Real specific mass in green state (preal) / g/cm ³	Real specific mass in green state (preal) / g/cm ³	Relative density (ρ_r) / %, g/cm ³
43.4		0.887	70.85
86.8		0.91	72.68
104.1		0.984	78.59
121.5	1.252 ⁽¹⁾	0.984	78.59
138.9		0.983	78.51
156.2		0.996	79.55
173.6		0.982	78.43

Note: (1) Specific mass of PLA in solid state (Farah, Shady, et al., 2016).

By subjecting the material to different pressures, it is possible to analyze the degree of green compact compatibility, as shown in Figure 03.

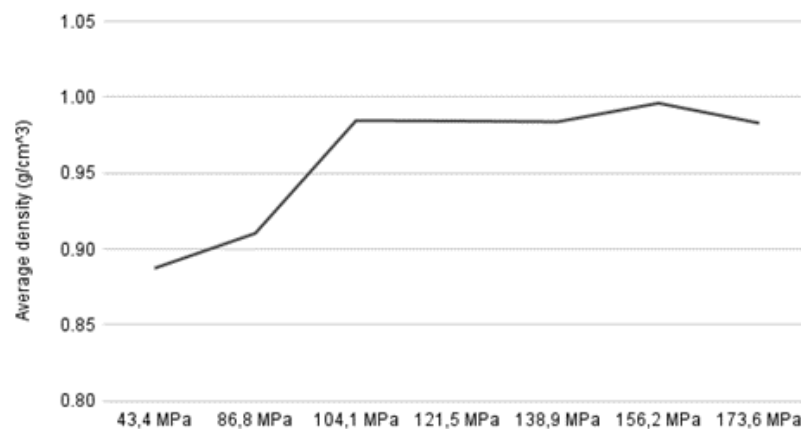


Figure 3 - Green compact compressibility curve of PLA

With the aim of preserving the physical integrity of the compaction matrix, the decision was made to select the minimum compaction pressure required to achieve a satisfactory relative density.

Based on the green compaction curve, it was observed that after applying a compaction pressure of 138,9 MPa, the difference in average density between subsequent pressures is low. Therefore, it would make sense to choose 138,9 MPa as the compaction pressure to be used in the study.

However, for the fabrication of the three-point flexural test specimen, a tonnage 10,35 times higher than the one used for the cylindrical sample would be required. The press used in this study has a maximum capacity of 15 tons, which would not be sufficient to produce the flexural specimen.

Therefore, due to infrastructure constraints, the compaction pressure was set at 86,8 MPa, as it allowed for compaction while preserving the integrity of the compaction matrix, hydraulic press, and machine operator.

Temperature and exposure time: After compacting the samples, they must undergo a heat treatment according to the procedures described in section 3.2.2.2. The objective of these tests is to select a range of temperature and exposure times that result in samples with satisfactory appearance and strength, considering the characteristics of the injected PLA as a reference parameter.

After compacting the samples, they must undergo a heat treatment according to the procedures described in section 3.2.2.2. The objective of these tests is to select a range of temperature and exposure times that result in samples with satisfactory appearance and strength, considering the characteristics of the injected PLA as a reference parameter.

The samples were subjected to a temperature range ranging from 150 to 200 °C, with 5-degree Celsius increments for each test. Additionally, different exposure times were applied during the heat treatment, ranging from 40 minutes to 2 hours, with 20-minute increments in each step. Table 03 presents a portion of the selected temperature range for the study, along with the response of the material sample regarding temperature and exposure time variations.

Table 3 - Table of Sinterization Analysis

Temperature Ranges / °C	Time Ranges / h				
	00:40	01:00	01:20	01:40	02:00
155	No sintering	No sintering	No sintering	No sintering	No sintering
165	No sintering	No sintering	Sintering	Oxidation	Oxidation
175	Partial sintering	Sintering	Degradation	Degradation	Oxidation
180	Partial sintering	Degradation	Degradation	Degradation	Degradation

It was observed that for temperatures below the range of 160 degrees Celsius, the material showed no reaction to the thermal treatment, regardless of the exposure time. Additionally, it was also observed that for temperatures above 180 degrees Celsius, material degradation occurred.

PLA is susceptible to thermal decomposition when exposed to high temperatures. The intense heat from the muffle furnace can result in PLA degradation, causing changes in mechanical properties due to the formation of chemical bonds in the polymer chains. Additionally, PLA exposure to oxygen in the environment during the sintering process can trigger oxidation reactions. This can lead to the formation of oxidized compounds and changes in material color. These characteristics and effects can be observed in Figure 04, which illustrates the impacts of temperature and oxidation on PLA during the sintering process.

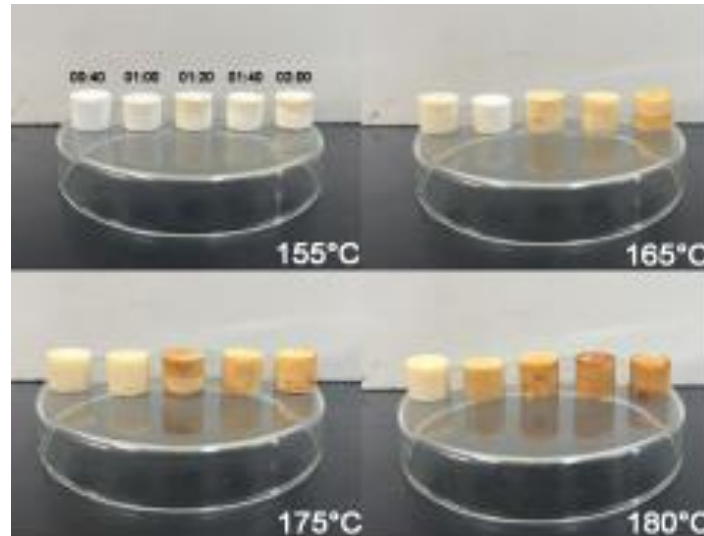


Figure 4 - Behavior of the samples within their study ranges

Based on visual analysis using the injected PLA as a reference, the temperature of 175°C and the exposure time of 1:00 hour were selected. These choices were based on the ability of this temperature range to promote material sintering while minimizing visually perceptible oxidation and degradation compared to the other analyzed samples.

Therefore, it can be concluded that to achieve successful uniaxial compaction of PLA, it is necessary to subject the material to a temperature of 175°C for a period of 1 hour.

3.3 Physical Properties of Sintered Parts

Densification, relative density, and porosity: The calculated physical properties for the sintered parts are presented in Table 04:

Table 4 – Physical Properties of the Samples

Physical Properties			
ρ_v	0.961	D	-0.4296
ρ_s	0.836	ρ_{rv}	76.7572
ρ_t	1.252	P	0.33227

It can be observed that there is a decrease in the relative specific mass from the green compact (ρ_v) to the sintered compact (ρ_s). It can be concluded that during the sintering process, significant expansion of the material occurs, which is supported by its negative densification (D).

Furthermore, it is noticeable that the relative specific mass of the green compact compared to the theoretical value is 76.75%. This value is attributed to the non-use of the appropriate compaction pressure of 138.9 MPa, which results in a porosity rate between the material grains, as evidenced by the porosity (P).

3.4 Mechanical Properties of Sintered Parts

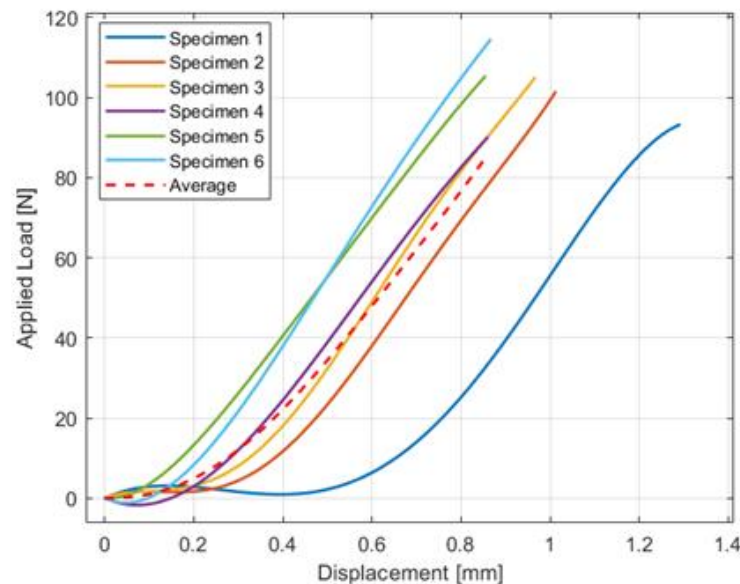
Three-point bending test: Through the three-point bending test, the following properties were obtained and are presented in Table 05:

Table 5 – Mechanical Properties – Three-Point Bending Test

Mechanical Properties	Sinterized Samples
Rupture Load / N	108.84
Maximum displacement / mm	0.9126
Ultimate tensile stress / MPa	5.945
Percentage of deformation / %	1.11
Elastic modulus / MPa	712.16

It is possible to observe that the sintered samples had an average elastic modulus of 712,16 MPa, representing only 20,34% of the theoretical elastic modulus of PLA of 3500 MPa (Farah, Shady, et al., 2016). This factor may be directly related to the non-utilization of the appropriate compaction pressure of 138,9 MPa.

However, it is possible to observe in Figure 06 a pattern in the rupture load of the specimens, showing a brittle failure close to 100N.

**Figure 5 - Rupture loads of sintered samples**

The consistent values of rupture stress demonstrate that the manufacturing process applied in the production of the test specimens is efficient, allowing for reliable results due to its predictable behavior and relatively uniform properties across different samples.

4. Conclusions

In conclusion, sintering through uniaxial compaction has proven to be a valid manufacturing methodology for thermoplastic materials, providing consistent results in three-point bending tests. However, it is a technique that requires improvements. Future studies should focus on identifying the appropriate compaction pressure to maximize mechanical properties and on mitigating material oxidation during the process. With further refinements, this approach has the potential to significantly enhance the manufacturing process of thermoplastic materials, driving advancements in various industrial applications.

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