



# Effect of Spent Coffee Grounds on Crystallization & Properties of Injection-Molded PLA

## Introduction

Poly(lactic acid) (PLA) has seen increased usage as a promising bioplastic. Its usage has been limited by the slow crystallization rate during injection molding. This forces economically viable products to be molded into an amorphous phase of PLA, with reduced physical properties in comparison to crystalline PLA [1]. To achieve high crystallinity, post-processing techniques, such as tempering, are used but required additional production time and cost.

In comparison, a nucleating agent be utilized to provide sites that promote crystal growth throughout PLA samples. Components in spent coffee grounds (SCG) provide excellent sites for nucleation during PLA injection molding. With high concentration of dietary fibers, lipids and nitrogen-based compounds, SCG promote crystallization [2]. The worldwide coffee industry provides an ample source of SCG that plastic processors can work with to supply a biobased, food-safe nucleation agent.

## Materials and Processing Methods

Ingeo™ 3100HP was supplied by NatureWorks as the injection grade poly(lactic acid) (PLA), designed for crystallization processes. Spent Coffee Grounds (SCG) were collected from were collected from the on-campus café and size reduced through a 1mm mesh. All materials were dried prior to use. A PLA-SCG blend was compounded through a twin-screw extruder. With 80 wt% of PLA and 20 wt% SCG, the compounded blend was then pelletized for injection molding processes.

Table 1: Non-variable injection molding parameters

Injection Molding Parameters	Values
Nozzle Temperature (°C)	165.5
Mold Temperature (°C)	95.0
Pack/hold Time (s)	20
Cooling Time (s)	90

## Injection Molding

Utilizing a Toshiba EC85SXII, samples were injection molded into a dual cavity mold for D638 Type-1 specimens. 16 replicates were produced based on a run based on a 2x2 DOE with one center point as seen in Table 2. To investigate the effect of shear and pressure, injection velocity and packing/hold pressure were selected at factors. Non-variable factors, such as cooling time, mold temperature, and packing time, were consistent throughout all runs (Table 1).

Table 2: 2x2 DOE with one center point for injection molding of PLA-SCG

Run	Injection Velocity (in <sup>3</sup> /s)	Packing Pressure (ksi)
(+, +)	4.0	6.5
(+, -)	4.0	3.5
(-, +)	1.0	6.5
(-, -)	1.0	3.5
(0, 0)	2.5	5

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## Abstract

As poly(lactic acid) (PLA) becomes a promising bioplastic, its slow crystallization rate limits its use cases due to lower mechanical properties. Nucleating agents allow for sites stimulating PLA crystallization, however most commercially available limit PLA's biodegradability and food-safe use. In this research, spent coffee grounds (SCG) was investigated for its performance as a nucleating agent for injection molded PLA under different injection velocities and packing pressures. The degree of crystallinity, tensile behavior, flexural behavior of 80-20 PLA-SCG injection molded samples were characterized and analyzed via a 2x2 Design of Experiment (DOE).

## Differential Scanning Calorimetry (DSC)

Three 3-8mg samples were made from tensile gauge region for each DOE run. The samples were run through a heat-cool-heat procedure (10C°/min ramp) on a TA Discovery X3. The degree of crystallinity of the PLA-SCG samples was determined from the first heat cycle by using equation (1).

$$Cryx \% = \frac{\Delta H_m - (\Delta H_{cc} + \Delta H_{pt})}{Wf_{PLA} * \Delta H_{m100\%}} (1)$$

where,  $\Delta H_m$  is the melting enthalpy,  $\Delta H_{cc}$  is the cold crystallization enthalpy,  $\Delta H_{pt}$  is the phase transition enthalpy,  $\Delta H_{m100\%}$  is the degree of crystallinity of 100% crystallized PLA, which is 93.7J/g based on literature [3], and  $Wf_{PLA}$  is the weight fraction of PLA, which was kept constant at 80%.

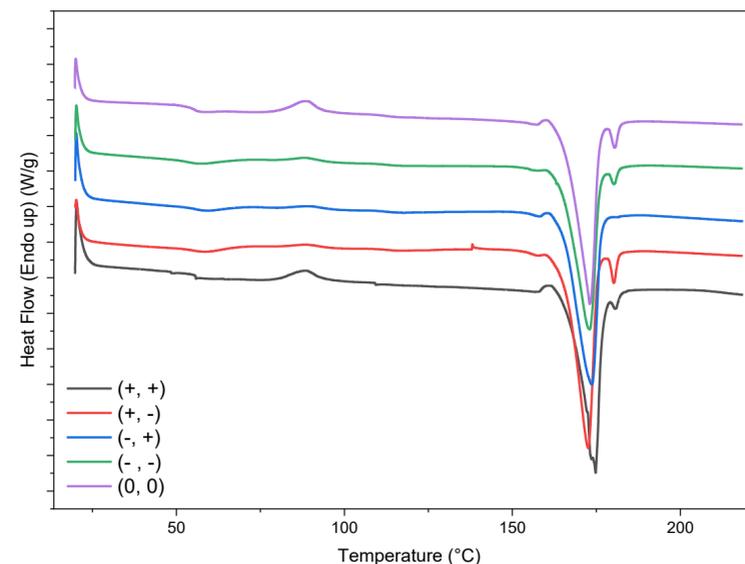


Figure 1: DSC curves of the PLA-SCG samples

From the DSC analysis, the PLA-SCG were highly crystalline with certain samples containing both  $\alpha'$  and  $\alpha$  phases of crystallization. As seen in Table 3, the average crystalline of all samples ranged from 48% to 58%. The ANOVA analysis of the crystallinity percentage showed only packing pressure was significant and limited crystallization in PLA-SCG.

Table 3: Degree of crystallinity, Young's modulus, and Flexural modulus of PLA-SCG samples

Run	Average Crystallinity (%)	Young's Modulus (MPa)	Flexural Modulus (MPa)
(+, +)	47.98 ± 5.02	1880.25 ± 58.39	3826.14 ± 130.32
(+, -)	57.76 ± 8.26	1875.50 ± 141.27	3751.67 ± 71.43
(-, +)	49.32 ± 2.72	1981.75 ± 71.86	4095.04 ± 54.00
(-, -)	55.49 ± 4.76	2116.75 ± 80.53	3664.27 ± 171.58
(0, 0)	52.28 ± 7.55	2028.25 ± 130.54	3776.64 ± 93.05

## Mechanical Testing (Tensile and Flexural)

Mechanical testing was conducted with a Shimadzu Autograph AGS-X Series load frame. The head speed for tensile tests was set to 5.5mm/min in accordance with ASTM D638. ASTM D790 defined the flexural head speed at 2.2mm/min. ANOVA analysis was conducted to determine parameters' effects on PLA-SCG modulus.

Based on mechanical testing, PLA-SCG samples were able to achieve comparable Young's and Flexural modulus to neat PLA. Young's Modulus ranged from 1880 to 2116 MPa but was negatively affected by injection velocity increasing. In comparison, flexural modulus was negatively affected by packing pressure increases but achieved a range of 2750 to 4095 MPa.

## Conclusion

Spent Coffee Grounds provide a promising solution to produce highly crystalline PLA products, upwards up of 60% crystalline. By eliminating post-processing steps, the cost and cycle of manufacturing is significantly reduced for industries. In addition, Adding SCG in neat PLA for injection molding can significantly reduce the crystallization time required during injection molding. Additional research to optimize SCG concentration will provide insight into the nucleation effect of SCG for PLA.

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## References

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