Melting Efficiency for Various Polylactide Resins in a Co-rotating Intermeshing Twin Screw Extruder

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Abstract:

There are many grades of polylactide resins marketed by NatureWorks LLC under the Ingeo™ trade name. For commercial and technical reasons, Ingeo may be supplied as a neat resin or with the addition of external lubricant. The applied lubricant improves pellet flow through conveying systems, silos, and dryers with minimal influence on physical properties. Several studies have been performed comparing the differences in melting behavior, power load, and melt temperature in single screw extrusion, but to date no study has characterized the same parameters in twin screw extrusion.

Leistritz Extrusion and NatureWorks LLC have conducted experiments that examine the effect of externally applied lubricants on the melting performance in a twin screw extruder with multiple screw configurations and differing operating conditions (i.e. rotational screw speed), denoting the location of the onset of melting, power consumption, and melt temperature for lubricated and unlubricated high molecular weight, low melt flow rate (MFR), formulations.

Introduction:

NatureWorks LLC and Leistritz Extrusion have collaborated to conduct experiments examining the effects of externally applied ethylene-bis-stearamide (EBS) at levels less than two percent by weight on the melting performance of Ingeo polylactide (PLA) resins in a Leistritz 27-mm ZSE MAXX twin screw extruder. A naturally advanced materials company, NatureWorks offers a family of commercially available performance materials derived from locally abundant and sustainable natural resources.[1] Some of NatureWorks Ingeo resins are provided with a surface lubricant to reduce the friction and prevent sticking in conveying systems, silos, and dryers.

Twin screw extruders (TSEs) such as the Leistritz 27-mm ZSE MAXX, shown in figure 1, are a preferred manufacturing method for compounding bioplastics, including PLA. TSEs utilize modular barrels and segmented screws assembled on splined shafts. TSE motors transmit power into the gearbox/shafts and rotating screws which impart shear energy into the materials being processed. The modularity of TSEs allows for a wide range and refinement of many processing applications.

Figure 1- Leistritz ZSE 27 MAXX twin screw extruder used for processing.

Optimal processing of PLA in a twin screw extruder requires properly addressing the heat and shear sensitivity of the neat PLA, as well as its torque requirements. Poor processing practices such as high pressure, high melt temperature, excessive moisture content, and increased residence time can result in hydrolytic degradation and reduced mechanical properties.[3] Use of a surface lubricant provides many benefits in conveying and transportation, but also has implications on the melting performance of PLA in a twin screw extruder. Explicitly engineering a TSE to account for the differences in processing can increase the efficiency of melting, improve material properties, improve throughput, and reduce tool wear. A few key factors that influence the melting performance of PLA in a twin screw extruder include:

1.) Level of surface lubricant used
2.) Barrel temperature set points
3.) Length of the melting region
4.) Pellet to pellet and pellet to metal friction
5.) Screw rotation speed
6.) Screw element and pellet geometry

The factors that can be controlled through the design of a twin screw extruder are the temperature set points, length, and design of the melting region, screw speed, and screw element geometry. The influence of these various parameters in a TSE process can be observed in polylactide in the form of melt temperature, overall torque, energy input, onset of melting, and molecular weight degradation. These values can be measured in line or through analytical studies on the final product. Analysis of the specific energy is particularly useful when evaluating the mixing performance of a TSE and looking for drastic differences in processing. The specific energy can be calculated through combination of equations 1 and 2 below.

$$kW\ (applied) = \frac{0.97\ (gearbox\ efficiency) * kW(motor\ rating) * \%\ torque * screw\ RPM\ running}{Screws\ Max\ RPM}$$

Eq. 1

$$Specific\ Energy = \frac{kW\ (applied)}{kg/hr}$$

Eq. 2

For the purpose of this study, a twin screw extruder can be viewed as various regions with unique tasks. As shown in the image below, these process sections include the melting region, the mixing and conveying region, and finally a vent and discharge region. In the melting region, the pellets must absorb heat from the barrels and energy of the screw. This will allow the pellets to soften and begin to form a viscous melt. As the polymer reaches the first set of mixing blocks, they should be soft enough to mechanically deform without cracking or crunching. Audible crunching present in the process is indicative of higher levels of mechanical energy, which lead to higher motor loads and in essence, wasted energy. The formation of a viscous melt in the melting region will also help mixing, which is essential for a homogenous polymer melt. After melting, additional conveying and mixing will occur and materials with viscosity mismatch such as fillers and liquids may be added through the use of a side stuffer or liquid pump. The latter region includes a melt seal, conveying and venting, and pump/discharge. After the polymer leaves the discharge region of the screws, downstream processes such as pelletization, sheet extrusion, melt pumps, etc. are performed.

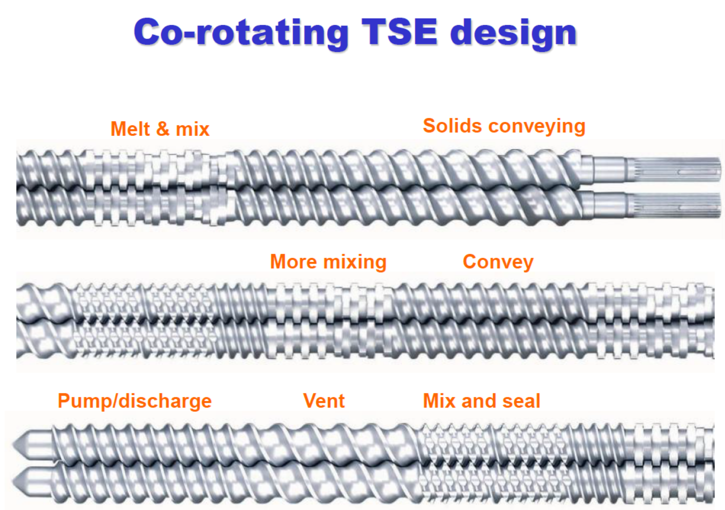


Figure 2- Co-rotating screws in a twin screw extruder split into various separate regions. These will be referred to as the melting, mixing/conveying, and pumping/discharge regions.[2]

Methods and Equipment:

Twin screw extrusion (TSE) experiments processing NatureWorks LLC Ingeo™ Biopolymer 4032D were performed using the three screw designs in figure 3 below. Neat Ingeo 4032D pellets as well as pellets with medium and high levels of externally applied surface lubricant were processed on a Leistritz ZSE 27 MAXX extruder with 28.3 mm diameter screws, 5.7 mm flight depth, 1.66 OD/ID, torque rating of 304 NM for both screws, and 1200 max rpm.

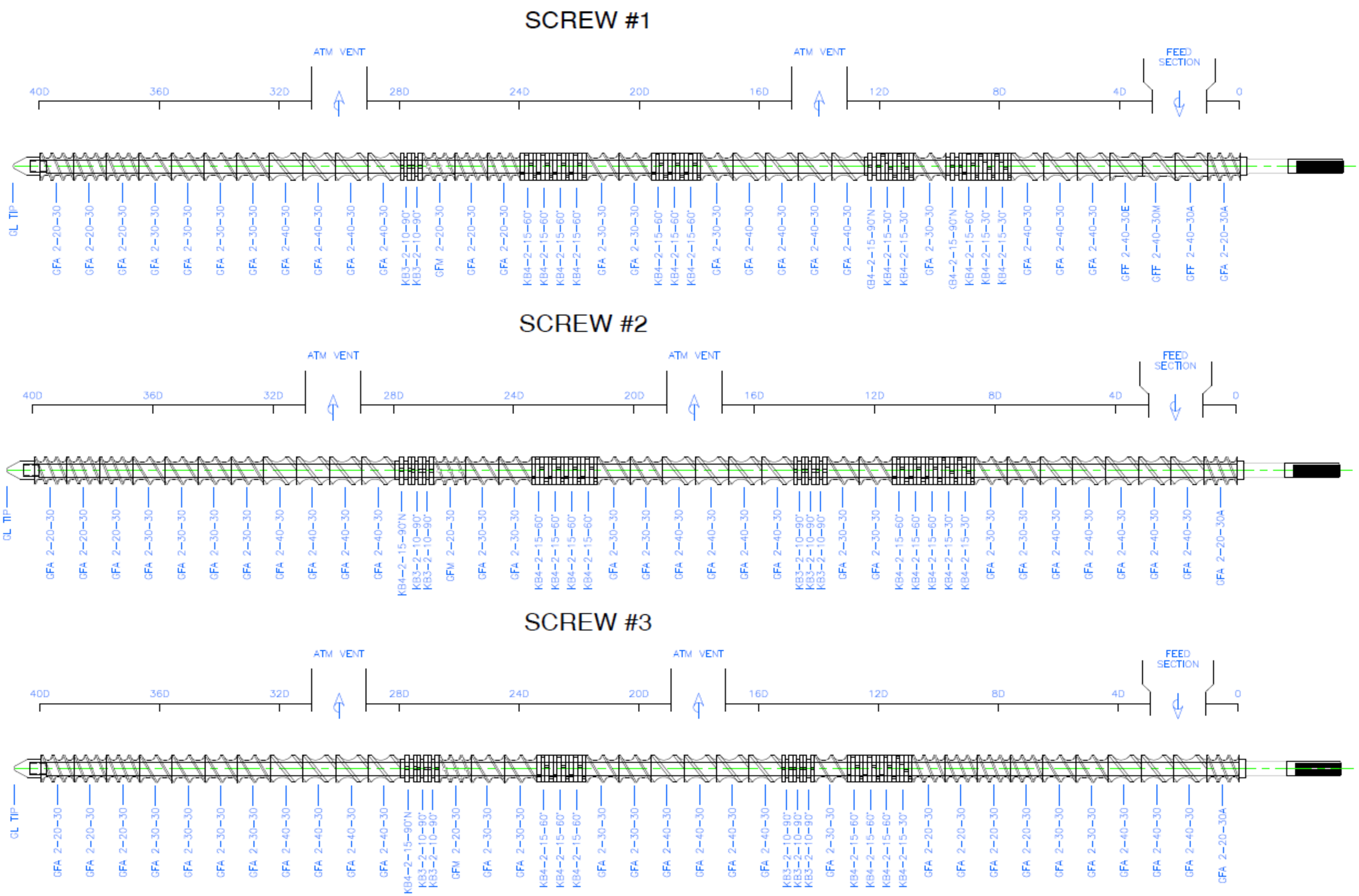


Figure 3- Three screw designs used over the course of this study.

Close attention was given to the melt temperature, melt pressure, specific energy, overall torque, residence time, and onset of melting. The parameters were measured and logged and the specific energy calculated using a state of the art twin screw extrusion control system. An image of the two melt temperature probes and two pressure transducers used in this study are shown in figure 4 below where the internal (deep) melt probe was fixed at 0.25” melt penetration.

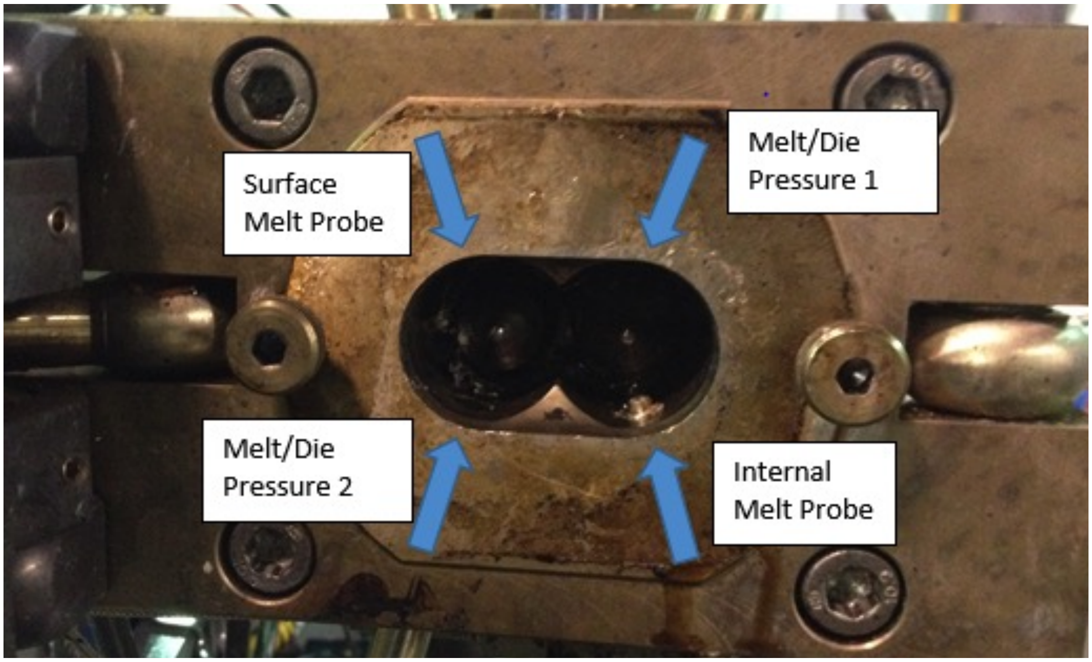


Figure 4- Locations of the two melt pressure transducers, the surface (shallow) melt probe, and internal (deep) melt probe at the end of the 27-mm TSE used.

The onset of melting of PLA was determined by removing the vent at zone 3, which immediately follows the kneading blocks present in zone 2. An image of the melt extrudate freely flowing out of the extruder can be observed below. After removal from the extruder, the collected polymer was immediately quenched in room temperature water to preserve the melt image.

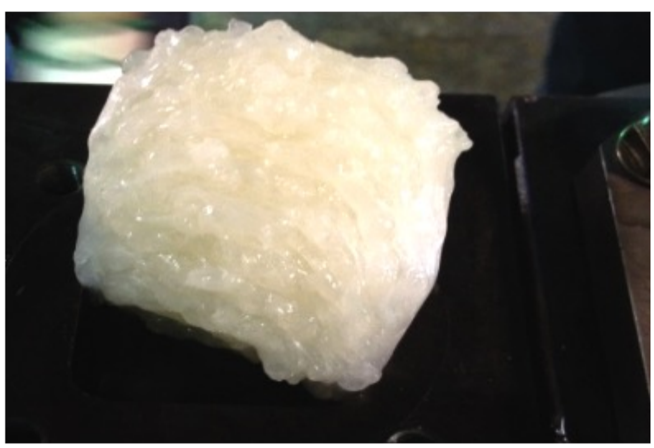


Figure 5- Melt extrudate collected from highly lubricated PLA at zone 3 vent for screw design 1.

Results and Discussion:

Figure 6- Melting region of screw design 1 using GFF screw elements and kneading blocks in zone 2.

Examining the Lubricant- Screw Designs 1 & 2:

Screw design 1 is typical of a screw optimized for melting and mixing of unlubricated polylactide. The melting region, as shown in the image above, consists of three GFF2-40-30 elements, three GFA2-40-30 elements, and four kneading blocks varying from 30° to 90° forward twist between each division. This is a relatively short melting zone for PLA that is advantageous when processing unlubricated polymer due to the high friction and shear energy absorbed by the polymer. The addition of the surface lubricant EBS altered the pellet-to-pellet and pellet-to-metal friction experienced in the extruder. The reduction of friction from surface lubricant in this design reduced the shear energy and frictional heat applied to pellets. As a result, the melting efficiency of the PLA pellets was reduced and the polymer was too solid as it approached the first set of kneading blocks. This was observed in the form of torque spikes and audible pellet crunching. Photos of the melt extrudate for various levels of surface lubricant can be found in figure 7. On the far left, neat Ingeo™ 4032D shows almost complete, uniform melting while the highly lubricated PLA on the right shows a high portion of unmelted pellets. The physical cracking of the pellets is not desired in a twin screw extrusion process as it is an indication of inefficient melting, excessive tool wear, lower throughput, torque overload, and increased probability of catastrophic events such as broken elements and shafts.

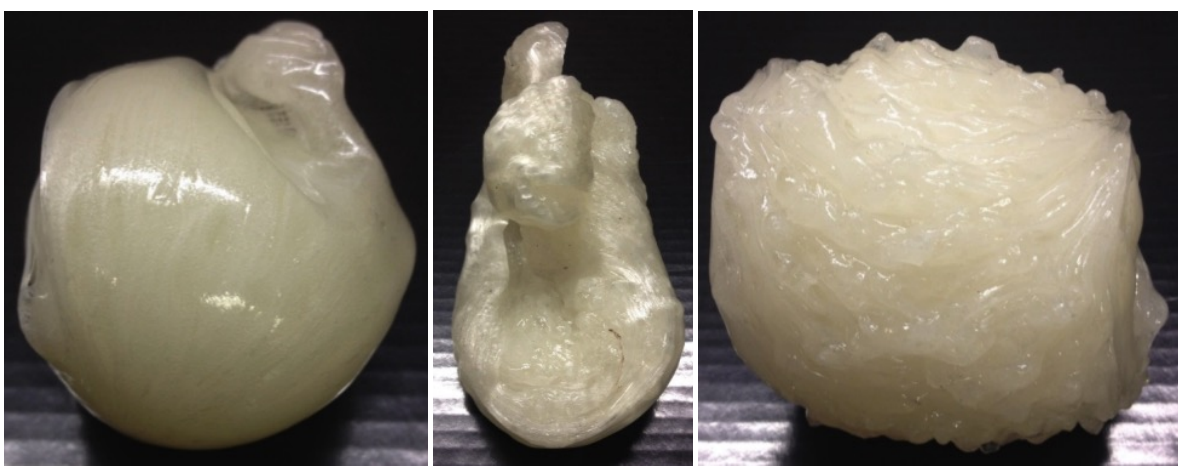
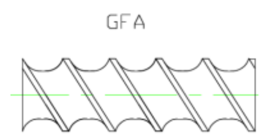


Figure 7- Images of melt extrudate removed from zone 3. From left to right: unlubricated Ingeo™ 4032D, medium level of surface lubricant, and high level of surface lubricant.

The learnings from screw design 1 were used to engineer a revised temperature profile and screw configuration, referred to here as screw design 2. In screw design 2, the melting region was extended by 30-mm to allow for increased heating and softening prior to the first set of kneading blocks. Also, the GFF screw elements used in the feed throat of design 1 were replaced with GFA elements of equivalent pitch and length. As shown in figure 8, GFF's are freely cut, non-self-wiping elements while GFAs are closely-meshing, self-wiping elements. The use of GFA elements reduced the free volume and increased the energy input into the polymer through additional pellet to screw surface contact, increased pellet to pellet friction, and additional contact with the heated barrel wall.[4] The temperature profile early in the screw was also increased from 210°C to temperatures as high as 222°C in the melting region. The increased temperature in the melting region caused the polymer to melt faster and reduced the overall load on the screws.



	Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6	Zone 7	Zone 8	Zone 9	Zone 10
Screw Design 1	210°C	210°C	210°C	210°C	210°C	210°C	210°C	210°C	210°C	210°C
Screw Design 2	220°C	222°C	222°C	212°C	212°C	212°C	202°C	202°C	202°C	210°C

Figure 8- Schematic of GFF and GFA elements used in screw designs 1 and 2, respectively. Also included are the temperature profiles used for processing at 35 kg/hr and 350 RPM.

A comparison of the specific energy input while operating at 350 RPM and 35 kg/hr with a medium level of surface lubricant in screw designs 1 and 2 is included in figure 9 below. It can be seen that an increase in temperature set points in the melting zone and extension of the melting zone reduced the load on the extruder. The audible crunching sounds that were experienced when processing lubricated PLA were eliminated using screw design 2. The temperature profile for screw design 2 also efficiently managed the final melt temperature. This provides additional benefits such as improved melt strength for downstream processes such as sheet extrusion, reduced degradation, and efficient mixing throughout the screw. One thing to note is the extension of the melting zone required the atmospheric vent to be moved one barrel downstream form zone 3 to zone 4. However, relocation of the mixing blocks and increased viscosity along the process length is still expected to provide quality dispersion of additives.

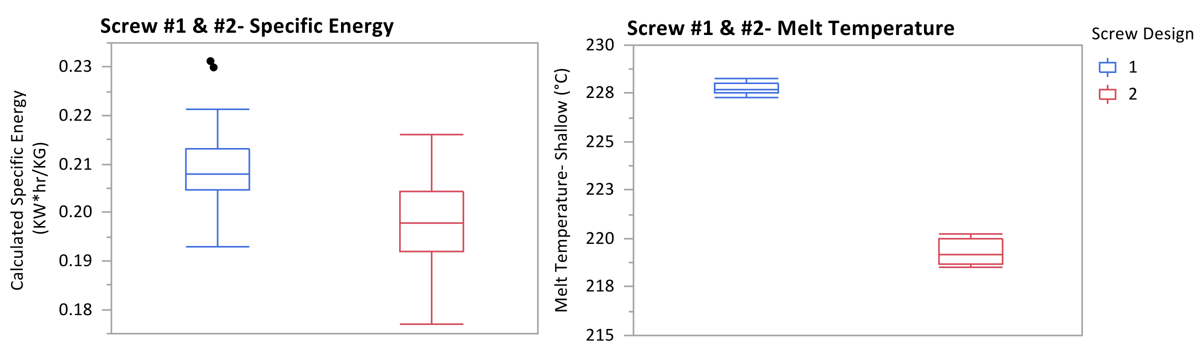


Figure 9- Specific energy and melt temperature while processing medium level of lubricant at 350 RPM and 35 kg/hr on a Leistritz 27-mm ZSE MAXX. Melt temperature measured with shallow, surface thermocouple.

Optimizing the Process- Screw Designs 2 & 3

Further optimization of screw design 2 was targeted with varied screw speeds, temperature set points, lubricant levels, and a modification that resulted in screw design 3. In screw design 3, the melting region was extended by an additional 60 mm beyond that of screw design 2. The conveying elements in the melting region of design 3 were altered to include two fewer 30 mm, 40° pitched elements and instead used four additional 30-mm, 20° pitched elements. The ability to extend the screw an additional 60 mm will depend on the process being used. In some cases, this may not be an option due to additional feeders and/or mixing necessary in the mixing and conveying portion of the screw. However, this design further improved the melting prior to mixing by increased pellet to metal surface area and increased residence time in the heated barrel prior to the first set of kneading blocks.

The temperature profiles and feed rates for screws 2 and 3 were altered beyond the work performed in screws 1 and 2. The increased temperature is designed to increase melting early in the screw, manage melt temperature throughout the process, decrease the torque on the screw shafts, and increase the overall throughput. The feed rates for all materials was increased to 45 kg/hr due to improved melting performance of the screws.

	Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6	Zone 7	Zone 8	Zone 9	Zone 10
Screw Design 2	220°C	240°C	230°C	230°C	220°C	210°C	210°C	210°C	210°C	210°C
Screw Design 3	220°C	240°C	230°C	230°C	220°C	210°C	210°C	210°C	210°C	210°C

Figure 10- Temperature profiles used for a comparison and optimization of screw designs 2 and 3.

The final melt temperature of Ingeo 4032D was measured at the barrel surface as well as 0.25” (6.4 mm) into the melt with the use of a variable melt probe. The recorded temperatures from this process at screw speeds varying from 325 to 600 RPM can be observed in figures 11 and 12. As expected, the internal melt temperature is significantly higher than the surface temperature in both screw designs due to reduced influence of heat transfer from the barrel. The differences in melt temperature of lubricated and unlubricated feeds can be explained by decreased melting performance. The reduction of frictional heat early in the screw results in a cooler melt throughout the extruder. One thing to note is the maximum melt temperature observed in screw design 2 was 243°C while the maximum temperature in design 3 was 240°C. The difference of 3°C may seem marginal, however, it may have a significant influence on degradation in processes where regrind is incorporated and the polymer may be exposed to these temperatures 5-10 times. It is recommended that each process be optimized with close attention paid to the molecular weight, melt temperature, torque load, and mechanical properties for any process.

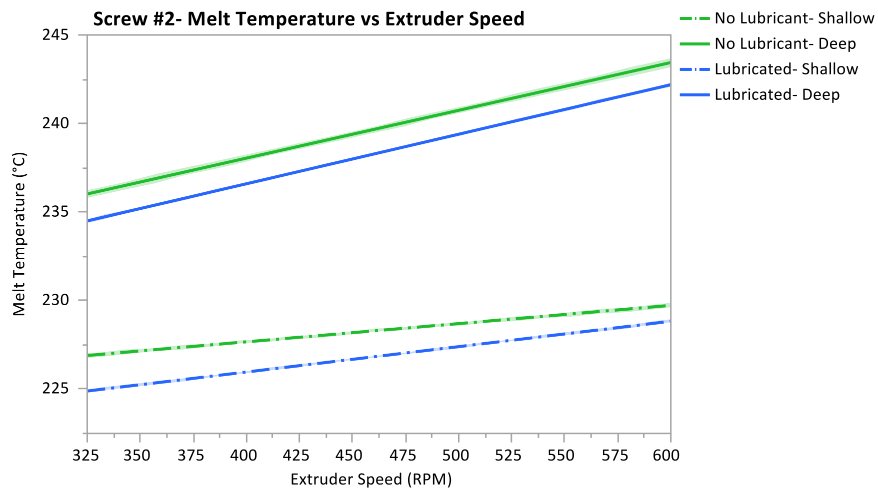


Figure 11- Melt temperature for shallow and deep thermocouple measured on lubricated and unlubricated PLA at the die prior to discharge in screw design 2.

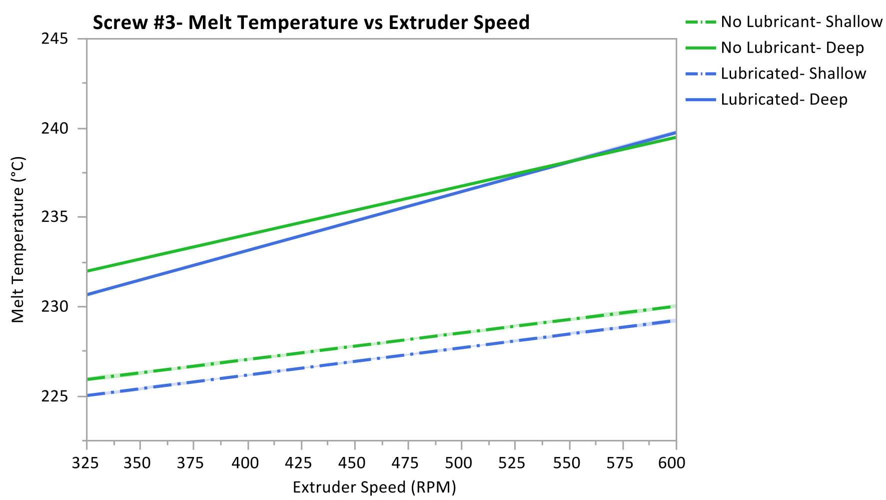


Figure 12- Melt temperature for shallow and deep thermocouple measured on lubricated and unlubricated PLA at the die prior to discharge in screw design 3.

In addition to monitoring the melt temperature, close attention was paid to the specific energy input in screw designs 2 and 3. In figure 13 it is clear that screw design 2 is adding more energy to the polymer melt. One would expect that design 2 is providing more efficient mixing while design 3 is a lower work screw. Each of these designs provides benefits for different applications. When compounding a performance enhancing material such as an impact modifier, screw design 2 may be preferred due to enhanced dispersive mixing. However, screw design 3 may be beneficial for compounding in a colorant powder or other highly hygroscopic material where low residence time, increased throughput, and low melt temperatures are desired.

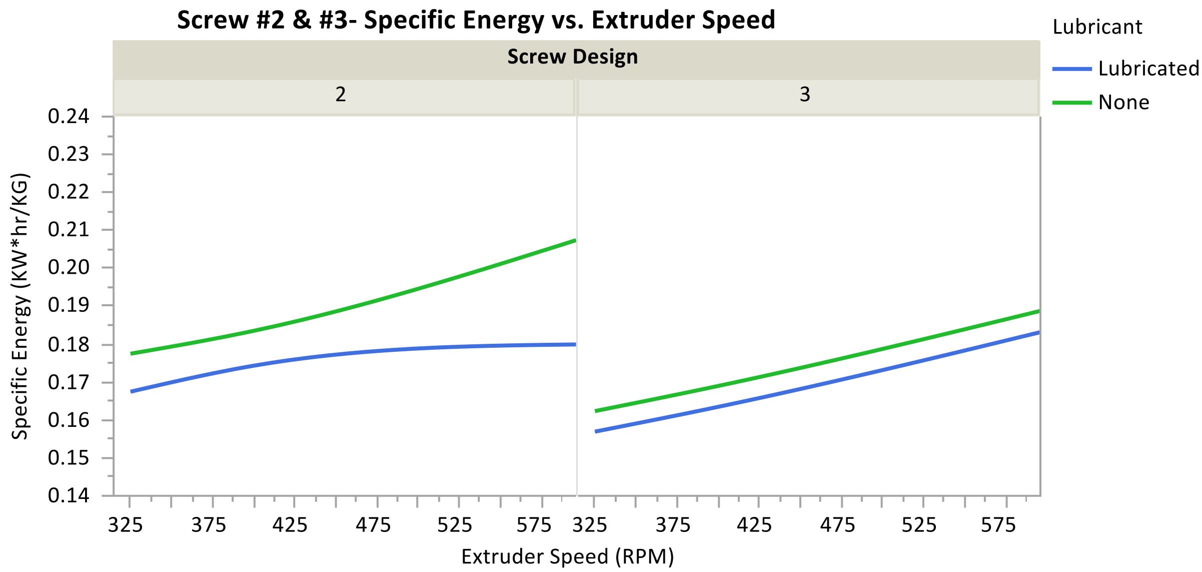


Figure 13- Specific energy vs extruder speed for designs 2 and 3 with lubricated and unlubricated PLA.

For each of the conditions applied to screw designs 2 and 3, the polymer extrudate was strand cut and analytical testing performed. It was concluded that each of the screw designs and process conditions resulted in greater than 92% molecular weight retention after a single pass, the surface lubricant level was maintained constant, the melt temperature did not exceed 245°C, pressure did not exceed 300 psig, and residence time was held maintained between 15 and 20 seconds. The surface lubricant used in the studies has been tested extensively and does not significantly influence the mechanical properties, rheology, or degradation of polylactide at the concentrations used. Screw designs 2 and 3 offer viable options for melting and processing lubricated PLA but should be further optimized based on desired throughput, mixing efficiency, and polymer mechanical properties.

Conclusions:

The processing of NatureWorks' Ingeo™ 4032D was observed with and without the application of EBS surface lubricant while processing with three different screw configurations in a Leistritz 27-mm ZSE MAXX. Key differences were noted in the melting behavior of lubricated PLA including delayed melting, reduced load on the extruder, and a reduction of melt temperature. In screw design 1, unlubricated PLA exhibited quality melting behavior while lubricated PLA caused torque spikes and unmelted pellets immediately following the first set of kneading blocks. In designs 2 and 3, optimized for processing lubricated PLA, quality product was observed under all processing conditions tested. This was determined as greater than 92% molecular weight retention, melt temperatures of less than 245°C, specific energy input of 0.15 to 0.21 kW-hr/kg, and sufficient melting/softening prior to further processing. For the general practitioner, this work suggests some practical guidelines for optimizing a twin screw compounding process when PLA makes up a major portion of the blend. If motor loads seem excessively high or there is audible cracking and grinding of pellets coming from the extruder, the process, and therefore product, would benefit from a change in the screw design. Increasing the heat transfer into the pellet bed prior to the first kneading section generally will lead to lower energy consumption, improved process stability and reduced melt temperature. This work performed by NatureWorks LLC and Leistritz Extrusion has identified multiple solutions to improve optimize the processing of lubricated Ingeo™ resin grades, such as extending the melting region of the screw, altering temperature profiles, improving the modes of heat transfer through increased surface area and frictional/shear heat.

References and Notes:

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3.) Garlotta, Donald. "A Literature Review of Poly(Lactic Acid)." *ResearchGate*. Journal of Polymers and the Environment, Vol. 9, No. 2, April 2001 (q 2002), n.d. Web. 09 May 2016.
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