Characterization Of Temperature-Pressure Induced Morphology in High Molecular Weight Poly (Ethylene Oxide)

By

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B.S., Rensselaer Polytechnic Institute, 2018

Thesis

Submitted in partial fulfillment of the requirements for the
Degree of Master of Science in Biomedical Engineering at Brown University

PROVIDENCE, RHODE ISLAND

MAY 2020

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This thesis by Sheila Velagapudi is accepted in its present form by the Department of Biomedical Engineering as satisfying the thesis requirements for the degree of Master of Science.

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CAPSTONE DESIGN PROJECT

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I would like to thank my fellow lab mates as well. Cameron Baptista taught me so much throughout my master's degree and inspired me to be excited about my science and think innovatively. He gave me the opportunity to ask questions any time and is an essential person to the success of my thesis. I want to thank Derek Rott for being a welcoming member of the lab from the very first day and being incredibly supportive and collaborative throughout his time here. I want to thank Roni Azagury for showing me how being passionate for your work will lead to the best work and science possible. I want to thank Stacia for all her guidance throughout this process as well. Lastly, I want to thank the rest of the lab members including Travis Nyguen, Kosta Milavanovic, Rosa Kim, Shiffoni Sukhlal, Austin Lessin, and Megan Fife for giving me the most rewarding experience in this lab.

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Introduction

Poly (ethylene oxide) is a thermoplastic polymer shown to have a variety of uses in food, cell phones, apparel, medical devices, energy storages, and a continuing growing list of other uses. This poly has shown some limitations in certain uses based on its unsatisfactory thermal and mechanical properties. Previous work has attempted to overcome these limitations by creating copolymers or chemical modification.[1] However, the properties of poly (ethylene oxide) and effect of treatment to it has not been thoroughly studied to fully understand the behavior of the polymer.

Prior work on poly-L-lactic acid and polycaprolactone has shown that thermally treating polymers at various temperatures and pressures can create polymers with new morphology and unique melting properties that could be advantageous in certain applications. Previous work has shown that processing samples above and below the melting temperature resulted in different crystal packing structures and mesophase orientation. A baseline pressure of at least 5,000lbs was found to be necessary to induce these changes.[2] This work successful proved that a new phase could be induced in these polymers with potential benefits.

Based on these findings, this investigation is devoted to determining if poly (ethylene oxide) has the capability to induce new phases such as a mesophase and birefringence through thermal and mechanical treatment.

Polymer Morphology

Polymers are long chains of made of monomers. There can be crystalline, semi-crystalline, or amorphous. There can be a range of degree of crystallinity in polymers, which will affect the bulk properties of the polymer. The polymer chemical structure will determine the degree of crystallinity and glass transition temperature. These properties are determined by DSC and are dependent on the heating rate of the experiment, and the process to which the sample was exposed (temperature and pressure). A sample heated above its melting point and then cooled quickly will result in a lower degree of order as compared to a sample that was cooled slowly.

The properties engineered in a specific polymer depends on the final application. The most important property that determine the polymer properties are its chemical structure and orientation of polymer chains. Isotropic materials have no orientation in any direction, while anisotropic materials change with direction. Solid crystalline materials are anisotropic. These materials can show high strength and are often spun into high modulus fibers. This orientation of a polymer can be altered experimentally to show certain properties. This structure is determined during heating and cooling processes. This phenomenon will be investigated in this work to determine how treatment can induce specific morphologies in specific polymers for their intended use. A tightly packed and highly ordered polymer chain structure will be brittle, have high mechanical strength, and have a higher melting temperature. The temperature is determined by the chemical structure.[4]

Heat can be applied in multiple directions for multiple amounts of time, causing the polymer chains to reorder themselves when melted and cooled at different temperatures with respect to their glass transition temperature (T_g) and melting point (T_m). The glass transition temperature refers to the

temperature at which 30-50 carbon chains start to move in a material. At this point, the polymer transitions from a rigid state to a more flexible state, changing its internal structure and mechanical properties. The melting point refers to the temperature at which the crystal structure melt. Polymers melt over time by weakening these intermolecular forces that maintain structure.

Poly (ethylene oxide) Morphology

This investigation is focused on Poly (ethylene oxide) (PEO) with a high molecular weight of 300kDa. The structure of this thermoplastic is shown to the right in Figure 1. The general structure of this polymer is a chain with a repeat unit of O-CH2-CH2-. This polymer has a melting temperature of approximately 62-64°C and a glass transition temperature of -65°C.[5]

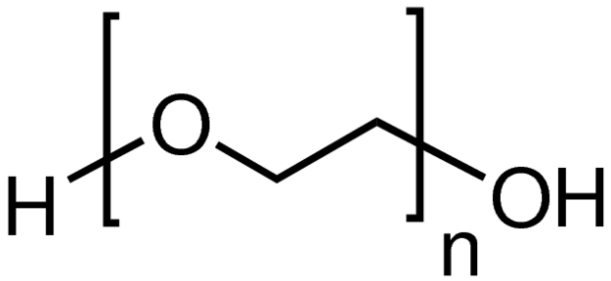


Figure 1. Structure of Poly (ethylene oxide)

Thermoplastics, such as poly (ethylene oxide) are usually a semi-crystalline polymer. The polymer is often not uniform all around, especially when treated. PEO is shown to not be affected by electrical conductivity which led to extensive research for its use in battery manufacturing. There are multiple properties of PEO that make them advantageous to use in electronics including: easy manufacturability, highly stability, compatible with other common materials in electronics, and relatively inexpensive to purchase. The disadvantage of PEO, which has potential to prevent its use in some electronics, is the high crystallinity of the polymer which creates an environment of low ionic conductivity.[6]

Based on the current use of poly (ethylene oxide) and its potential, this investigation is inspired by previous work to delve into the polymer itself to determine how its structure and properties change based on processing conditions.

Prior Work in the lab on polymer characterization

The ability to alter polymers is a phenomenon that has been investigated in the lab. In previous work, it was found that applying pressure at different temperatures induced a new mesophase in Poly-$_L$-Lactic Acid.

To study the effect, PLA sheets were purchased and treated hydraulic press. The sheets were subjected to multiple pressures between 2-20,000lbs for multiple times between 5-15 minutes. Samples were analyzed using Fourier Transform Infrared Spectroscopy, Differential Scanning Calorimetry, X-ray Diffraction, and polarized microscopy. The results of this investigation showed that the morphology of a polymer can be affected by temperature- and pressure- specific treatments and produce a mesogenic phase in the polymer. This work showed how to systematically measure the differences in the polymer after each treatment to investigate the effect of pressure and temperature[2].

The following investigation presented has taken inspiration from this previous work and attempts to see whether similar treatments can potentially be used to understand the effect of these conditions on polyethylene oxide.

Inducing Mesomorphic Orientation in Polymers

The mesogenic state in a polymer refers to an orientation between a full crystalline and amorphous polymer. This phase is thought to show properties of both phases (liquid-crystalline structures) which is advantageous for certain uses. This phase can be characterized by many methods one of them is polarized light, or Birefringence. Birefringence refers to the orientation of the polymer chains. An amorphous phase will show no birefringence, while crystalline polymers will show specific diffraction of the crystalline structure of the polymer[2]. A polymer with a mesophase structure will show the colorful structure present in a sample as shown in Figure 2 below. This phenomenon generally occurs in anisotropic samples because the orientation of polymer chains in those samples is in multiple directions. The different colors represent the amount this polymer is refracting light when placed between polarized filters.

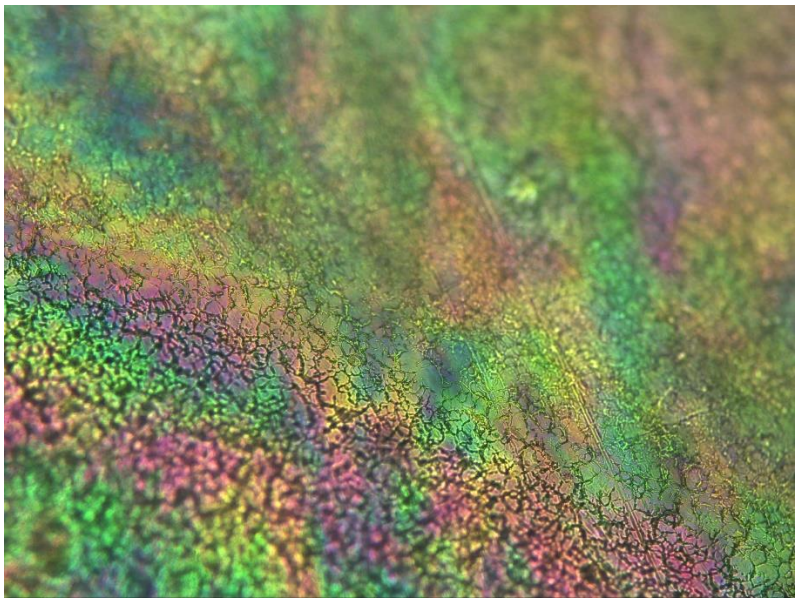


Figure 2. Birefringence of PEO sample treated with 20,000lbs, 62°C, 1 hour

MOTIVATION

Polymers have an immense use in various fields including biomedical engineering. The mesophase of polymers has proven to show even more beneficial properties. Determining the effect of heat and pressure on polymers helps understanding the final morphology of devices that are formed by melt extrusion where pressure is often applied. Poly (ethylene oxide) specifically is an inexpensive, water-soluble thermoplastic that has shown great use in many biomedical applications. This polymer has been investigated in its original state or as an addition to other polymers to develop new characteristics in the polymer. However, there is limited research into the ability to morph poly (ethylene oxide) and develop new structures within the polymer.

In our lab, determining if mesophases can be induced in a polymer can be incredibly useful in understanding their mechanical properties, degradation profiles, and performance in drug delivery systems. The results could be applied to any type of polymers used outside of the field of biomaterials.

Characterization Methods

Hydraulic Press

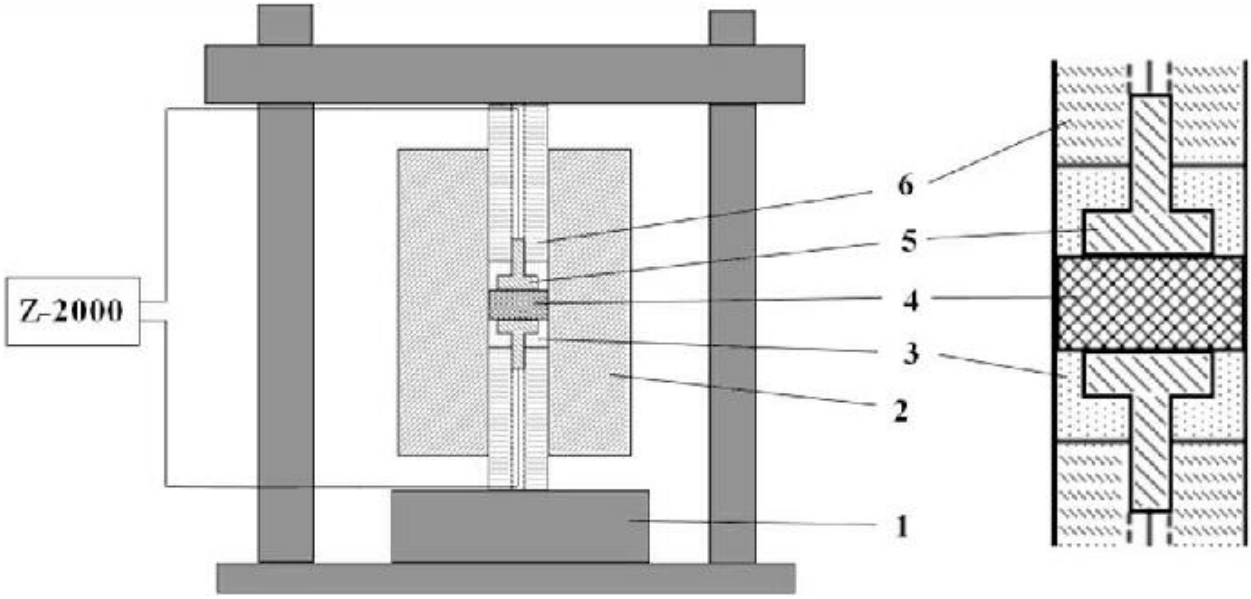


Figure 3. Schematic of Hydraulic Press

The hydraulic press applies pressure to a material on both ends. As shown in Figure 3, a sample is placed on a sheet between the two plates in the hydraulic press. When the test begins, two plates heated to a specific temperature apply a specified pressure to a material for a specific amount of time. This system can be used to heat a polymer past its melting temperature and the application of pressure can creates a new morphology after the pressure is released and the polymer cools down. The press can be used to cool a polymer as well before or during the test.

Optical Polarized Microscopy

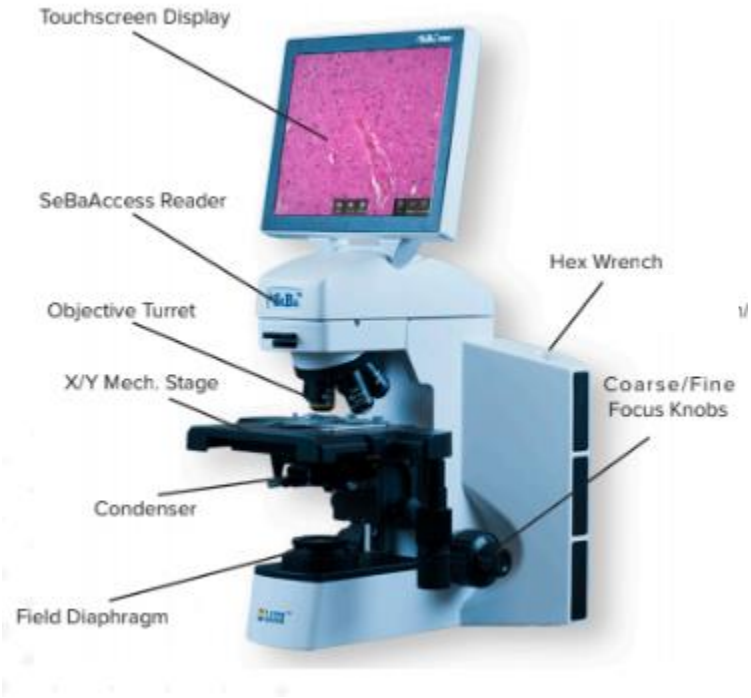


Figure 4. Schematic of SebaPro5 used for optical microscopy

The SebaPro 5 microscope (Figure 4) displays a high contrast image of either brightfield, darkfield, simple polarization, or phase contrast. In this investigation, the optical polarization technique was utilized to show birefringence in a polymer as it enhances the contrast of the image. The setup of optical polarized microscopy utilizes a polarize filter between the light source and another polarizer, an analyzer, which is positioned perpendicular to the sample and the polarizer. The image is produced based on the polymer sample's reaction to the plane polarized light. Different chains of the polymer will react to the light differently, forming the image that shows the birefringence of a polymer, indicated by bright fibril patterns. Before and after being pressed, samples were examined under a microscope to determine if and where there was birefringence. Sample photos were taken at the edge and middle section of the samples. If the sample was amorphous, darker monotone sections were seen under the microscope.

Crystalline regions show bright, rainbow colors as seen in Figure 2. Crystalline regions also show spherulites with the bright fibrils outward facing and straight, indicating a radial spherulite as shown below in Figure 5. Figure 5 shows an example of spherulites in poly (ethylene oxide). This morphology is seen in crystalline regions of a polymer. The fibrils are facing outwards resulting in a radial spherulite[7].

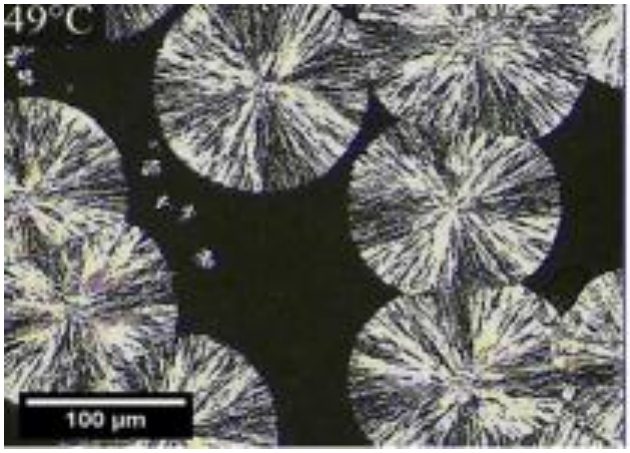


Figure 5. Radial spherulite of poly (ethylene oxide) crystallized on a glass slide at 49°C

Differential Scanning Calorimetry

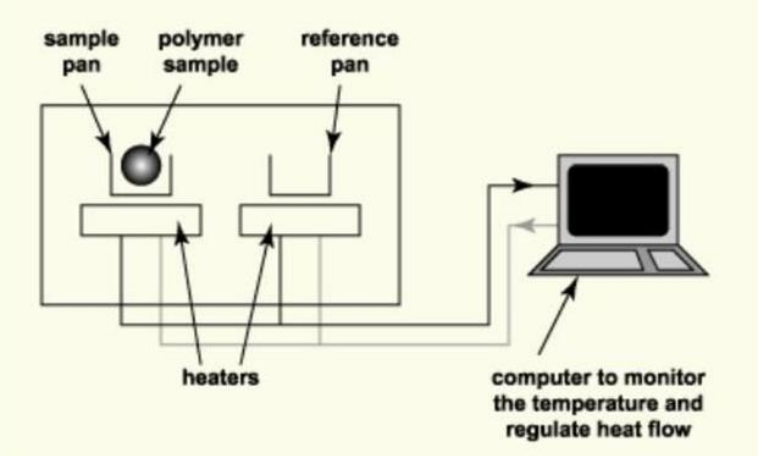


Figure 6. Schematic of Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) (Figure 6) is a thermoanalytical tool used to measure the heat required to increase the temperature of a sample to a specific temperature. The measurements acquired include the specific heat capacity and heat of fusion (melting enthalpy). The tool is commonly used to measure changes in enthalpy of polymers. These measurements can be used to determine the melting temperature, glass transition temperature, and crystallinity of a polymer. The sample preparation for this DSC includes slicing a small sample of the material of interest and placing it carefully into a sample pan which is then placed in the sample compartment of the DSC. The DSC will heat the sample and compare it to the sample in the reference compartment to determine the differential heat flows of each sample.[8]

A method was developed to determine the enthalpy of change for each sample in this investigation. The rate at which the sample is heated and the temperature to which the sample is heated to can be controlled. A typical result of a DSC test is shown below:

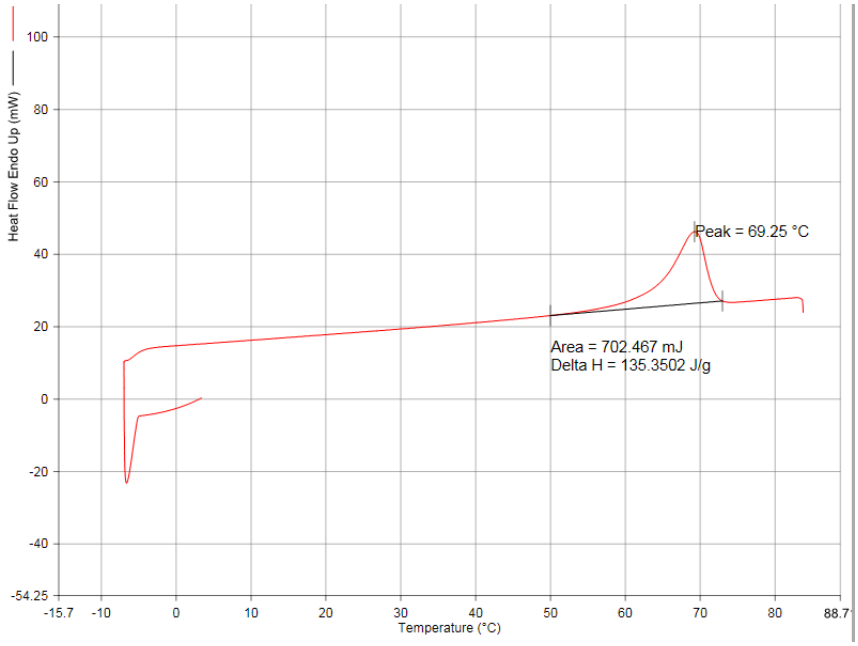


Figure 7. Sample Result of Differential Scanning Calorimetry – 1st heat of Sample pressed at room temperature and heated at 10°C/min

The first heat of fusion, sample shown in Figure 7, will demonstrate the new thermal properties of the material after processing. The material will then be cooled, and the second heat of fusion will determine the effect of the previous cooling step on the material properties. This can be useful in determining relaxation properties of the material. The rates of heating and cooling can be very fast or slow. The start and end temperatures will generally be below the glass transition temperature and above the melting point of the material, respectively. Various rates were used to heat and cool the samples in this investigation. An unaltered sample of poly (ethylene oxide) was used as a control comparison for each temperature or time-point measured. The more crystalline structure will usually show a higher glass transition temperature and capacity of heat.

X-ray Diffraction

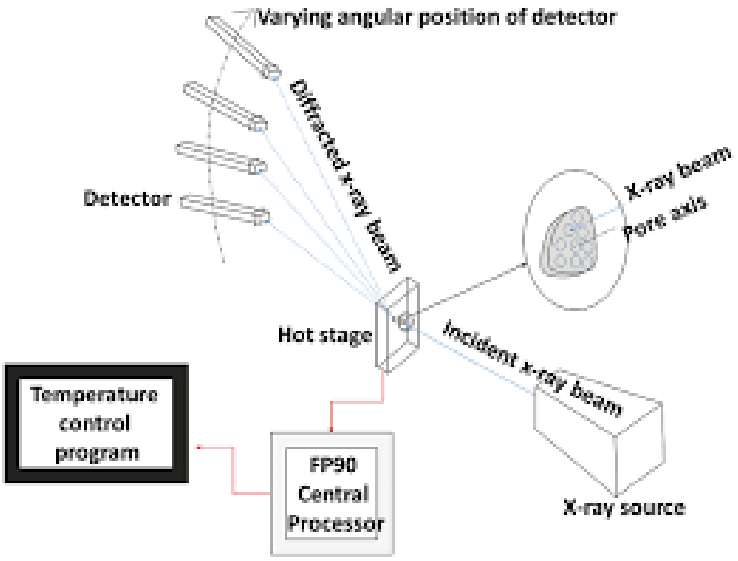


Figure 8. Schematic of X-Ray Diffraction

X-ray Diffraction (XRD) is used to determine molecular structure and crystallinity of a material. This technique works by placing a sample on a plate and using x-ray scattering from two laser beams to diffract off the material and either constructively or destructively interfere with one another (Figure 8). These rays are analyzed to determine the intensity and angle of diffraction and the structure of a material. The angle of diffraction can be used to determine interplanar atomic spacing, which can be used to determine the lattice structure of a material. These results will also show the percentage of crystallinity in a sample. Amorphous materials show a black space while more crystalline materials show narrow, high intensity peak.[9] This machine is used to analyze the crystal structure of a material. A small sample of polyethylene oxide was placed in the machine and five measurements between 15°C and 80°C were taken to measure the crystalline properties of the material. A sample of polyethylene was analyzed as well.

Instron

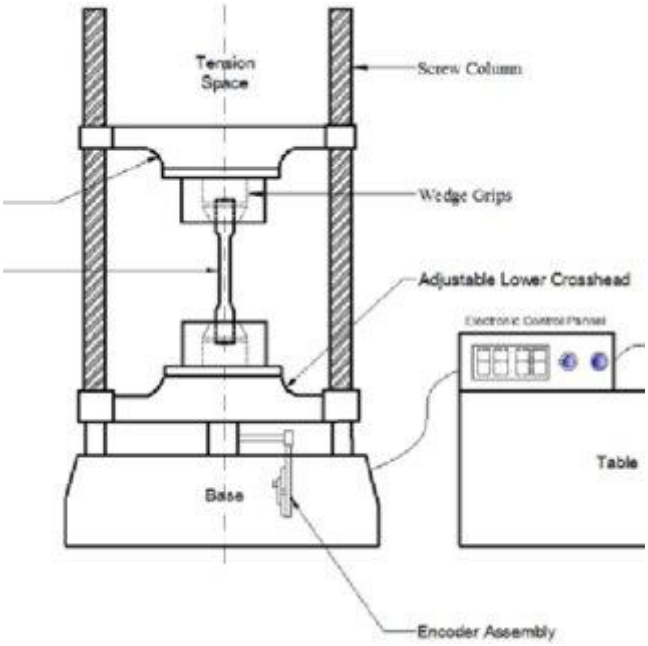


Figure 9. Schematic of Instron 3350

Instron machines (Figure 9) are used to measure mechanical properties of various materials. A tensile test can be created to measure the force needed to deform a specimen laterally at a rate decided in the test until the specimen fails and breaks apart. The results of this test can be used to determine the strength of the material. A compression test can be completed to measure the amount of force needed to deform a specimen under uniaxial load. A material is generally made into a dogbone shaped material to perform these tests based on ASTM standards.[10] A general tensile test on this machine begins with a dogbone shaped sample being placed between grips. The sample is adjusted in the grips to a starting point. The test is started on the computer and the tensile stress and strain is measured on a graph. An example of the results of a tensile test is shown below:

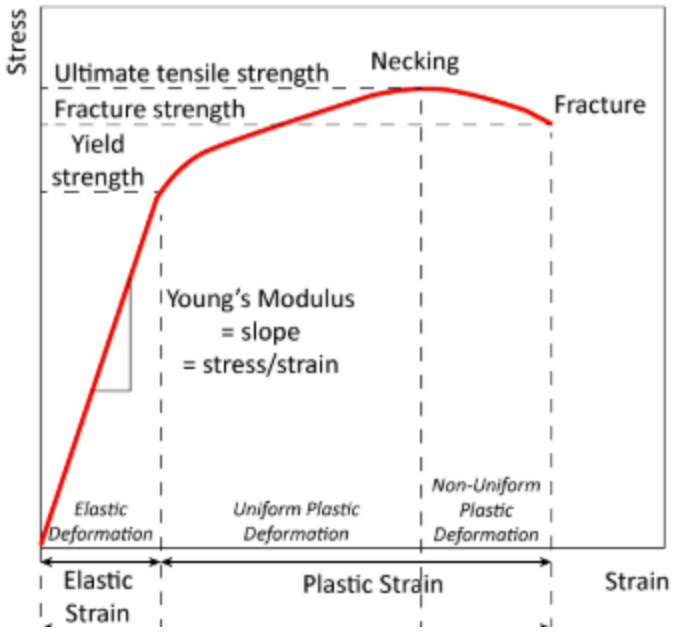


Figure 10. Results and analysis of sample tensile test in Instron

Overtime, the force required in the tension test can be measured for different polymers to determine the strength of polymers treated under various conditions. A sample result is shown in Figure 10. The tensile stress is then calculated by dividing the load applied to the sample by its cross-sectional area. The cross-sectional area of the polymer varies based on processing conditions. The ultimate tensile strength is calculated by determining the maximum load the sample could withstand before it fractured. The tensile strain is calculated dividing the change in length of the sample by its original length at the beginning of the test. This parameter relates to the elasticity of the sample. Elasticity at the maximum load and at break will describe how amorphous or crystalline the sample was at that time.

Specific Aims

This study examines the effect of temperature and pressure on polymers to on the ability to new morphology in poly (ethylene oxide). There is preliminary data that show the effect of thermal treatment on PLA and PCL, which was used has the basis for designing the experiments to study Poly (ethylene oxide), which has its own set of properties. This investigational study also examines the effect of temperature and pressure on poly (ethylene oxide) on the mechanical strength of the polymer. The strength of the polymer is important for its potential uses and therefore its need to degrade at certain rates.

Specific Aim 1: Characterize the effect of processing temperature (RT(25-30)°C, 55°C, and 62°C) on the morphology and strength of poly (ethylene oxide) samples.

Specific Aim 2: Evaluate the effect of processing time (30 seconds, 15 minutes and 60 minutes) on morphology and strength of poly (ethylene oxide) samples

The following characterization methods were used: differential scanning calorimetry (DSC), polarized microscopy, X-ray diffraction (XRD), and the Instron.

Materials and Methods:

Materials

High molecular weight poly (ethylene oxide) of M_w 300,000 was purchased from Sigma Aldrich. Samples of polyethylene were processed as well as a comparison to study the difference processing conditions can have on crystallinity in a similar polymer as well as study the effect of oxygen in the backbone of poly (ethylene oxide). Polyethylene is known to have thicker lamellae than polyethylene, so it is hypothesized to be more resistant to the same processing conditions.

Methods:

Hot Plate

High molecular weight poly (ethylene oxide) of M_w 300,000 was used. 150mg of powder was placed on a glass slide and placed on a hot plate at 80°C for 15 minutes to form a solid. Samples were taken off the hot plate and placed in bags until further processing.

Hydraulic Press - Processing of PEO

Samples were all pressed at a pressure of 20,000lbs. They were pressed at temperatures of RT (25-30°C), 55°C, and 62°C for 30 seconds, 15 minutes and 60 minutes. Samples were also pressed at 52°C and 57°C for the initial XRD scans.

Optical Polarized Microscopy

Polarized microscopy was completed on all samples before and after processing. Samples were placed on a glass slide between polarized light on the microscope at 40x magnification. Images of the center and the edge of the sample were taken to obtain a complete morphology analysis before and after processing.

Differential Scanning Calorimetry

The Perkin Elmer 7 DSC was used to for thermal analysis of the poly (ethylene samples). Samples were cut unprocessed or processed into small squares with a weight between 3-5mg. The sample weight on average was 4mg.

The StepScan parameters created in the DSC always followed this base pattern:

1. Mount sample at room temperature
2. Set temperature to 0°C or -10°C
3. Hold at 0°C or -10°C for 5 minutes
4. Start Test
5. Hold at 0°C or -10°C for 1 minute
6. Heat sample to 70°C, 75°C, or 80°C at 10°C/min
7. Hold at 70°C, 75°C, or 80°C for 1 minute
8. Cool sample to 0°C or -10°C X°C at 1°C, 10°C, or 100°C /min
9. Hold at 0°C or -10°C for 1 minute
10. Heat sample to 70°C, 75°C, or 80°C at 10°C/min

The sample was placed into the DSC at room temperature. The DSC was then set to a starting temperature of 0°C for the first 8 samples. The starting temperature was adjusted to -10°C to account for the glass transition temperature of the sample for all other samples. The DSC was set to and reached this starting temperature before the test was started. The final temperatures the samples were heated to were 70°C, 75°C, or 80°C. The X and Y values were variable based on the effect being studied in the investigation. The X value started at 70°C because it was above the melting point, but it seemed to be slightly low to fully melt the polymer well, so a higher temperature was chosen at 80°C, but that was deemed too high so a temperature of 75°C was chosen in the end. A study was complete to determine the difference in heat of fusion values if heated to 70°C, 75°C, or 80°C, and the differences were insignificant because the polymer was almost completely melted by 70°C. The rate of heating was always

maintained at 10°C/min. The rates of cooling tested varied to determine the effect of cooling rate on crystallinity in poly (ethylene oxide). These cooling rate values were chosen to be extreme to see the greatest effect. The cooling rates chosen were 1°C/min, 10°C/min, 100°C.

XRD and Hot Stage XRD

X-Ray Diffraction and Hot Stag X-ray Diffraction was performed using the Bruker D-8 Advance X-Ray Diffraction system. The DaVinci software was used to set parameters of 40kV and 40mA for the Cu X-Ray tube. Poly (ethylene oxide) samples were thermally treated at RT (25-30°C), 52°C, 57°C, 62°C in the hydraulic press for 15 minutes. A small square piece of each sample was cut using a box knife and placed on the XRD stage.

The Hot Stage XRD were completed on a similar set of samples. The samples used in the Hot Stage XRD were thermally treated at RT (25-30°C), 55°C, and 62°C for 15 minutes under 20,000lbs pressure. During the test, the sample was heated to various temperatures ranging from room temperature (25°C) to above the melting point to either 75°C or 85°C depending on the sample. The samples were heated to 25°C for the first scans. The samples were maintained at this temperature for 5 minutes prior to the beginning of the test. The sample in place was then heated to 45°C, 55°C, 65°C, and 75°C and left for 5 minutes before each test to allow the polymer to fully sit at that temperature. Scans were taken at each of these temperatures. In the both the XRD and Hot Stage XRD experiments, sample scans were run at 2θ from 10°-90° and scans were taken every 30 seconds. The scans were analyzed and adjusted using Diffract-Eva software.

Instron

Figure 11. Silicon Mold used to create dogbone Instron samples

The Instron 3350 was used to perform tensile tests on poly (ethylene oxide). According to ASTM standard, dogbone shaped samples were required, so a silicon mold (Figure 11) was made to create samples. Silicon molds were made using a silicon mix and a dogbone template. The template was left in the silicon mold and left to dry for 24 hours. The follow mold was created to the right and used to make every sample tested in the Instron. To ensure the amount of time the sample was melted in the mold did not affect the results, each day of testing, 700mg of PEO was spread evenly into the two dogbone molds and placed in the oven at 70°C for a 2 hours. This amount of powder was chosen because it mimicked the thickness of the samples used for all other tests. Once the samples were fully melted, the mold was taken out of the oven and left on the bench for 30 minutes to properly cool. Samples were carefully taken out of the mold and ready for testing. Control samples were taken out of the mold and a tensile test was run on each sample at 5mm/min until the sample broke. Samples to be processed were placed in the hydraulic press. The hydraulic press was set to a specific temperature and 20,000lbs pressure beforehand. The dogbone samples were placed on a metal slide and put on the hydraulic press plate and pressed for 15 minutes at RT (25-30)°C, 55°C, and 62°C. Once these samples were processed in the hydraulic press, a tensile test was run on each sample at 3-5mm/min until the sample broke. Rate had to be slightly adjusted for brittle samples processed at higher temperatures. Unprocessed samples had a thickness of 3mm, while samples processed at RT (25-30)°C, 55°C, and 62°C had a thickness of 2.5mm, 1.7mm, and 0.8mm respectively.

Results/Discussion:

Optical Microscopy

The purpose of this section is to discuss the comparison between unprocessed poly (ethylene oxide) polymer samples and processed samples. The processed samples can show the effect of pressure, heat, and treatment time on the birefringence and formation of spherulites of the poly (ethylene oxide) samples. Spherulites are an aggregation of spherical crystallites that form a larger structure[14].

Unprocessed samples:

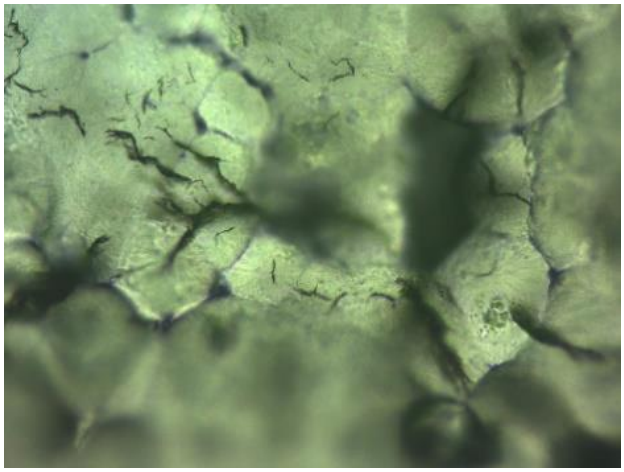


Figure 12. Middle Section of PEO Sample (40x magnification)

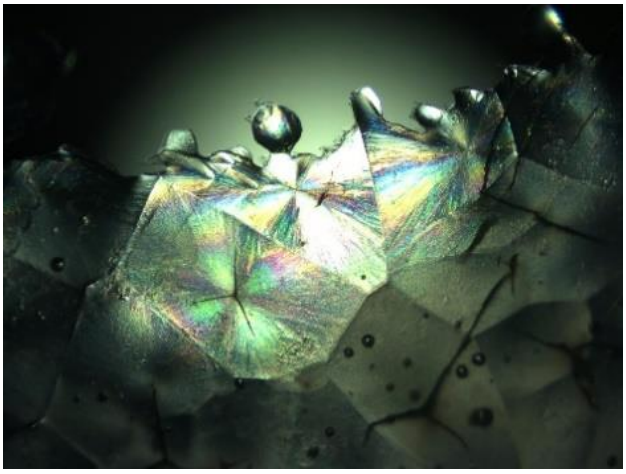


Figure 13. Edge Section of PEO Sample (100x)

The images in Figures 12 and 13 show original unprocessed polymer under 40x magnification. Figure 12 represents the middle region of an unprocessed sample and Figure 13 represents the edge region of an unprocessed sample. Samples investigated at the middle and edge of a sample tend to show different birefringence in unprocessed and processed circumstances. In Figure 12, an amorphous section of the polymer will be seen as space on the polymer. There is no birefringence seen in the middle section of this sample. This may be a direct result of the thickness of the sample.

Figure 13 also shows radial spherulites in the polymer as shown by the colorful, straight fibrils moving outward within the spherulites. As spherulites grow and interact with each other, they begin to grow into the empty space around them as seen in Figure 13. Spherulite formation was especially seen in samples processed at longer time points and at higher temperatures. The phenomenon is also observed in literature, indicating that the longer a polymer is maintained at a certain temperature, the larger the diameter of the spherulites formed as seen in the samples in Figure 2.[7]

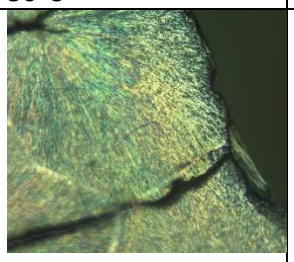
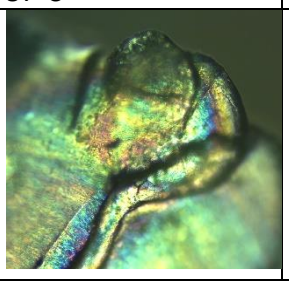
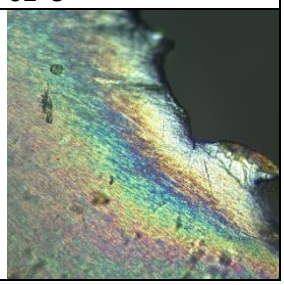
	30°C	50°C	57°C	62°C
15 min				

Table 1. Edge Samples - Processed in Hydraulic Press for 15 minutes at various temperatures (100x magnification)

Table 1 show microscopic images of the edge region of PEO samples processed at four different temperatures at 100x magnification. The images show a general trend of how the birefringence increased as the treatment temperature increased. Samples treated at 62°C consistently showed the most birefringence. This result is thought to be due to the polymer is fully melting before being removed from heat and pressure and then being cooled slowly to room temperature. When the polymer was melted above its melting point, the polymer would flow around the plate and become very thin. The samples processed at 30°C and 50°C show very large spherulites similar to the one seen by controls, yet more birefringence..

Edge Samples

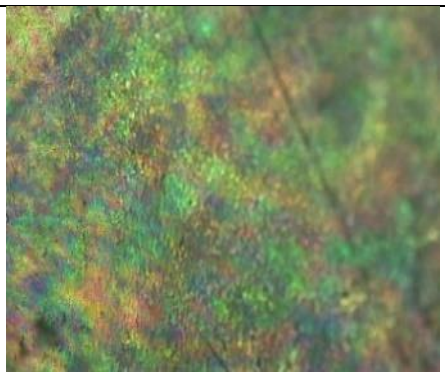
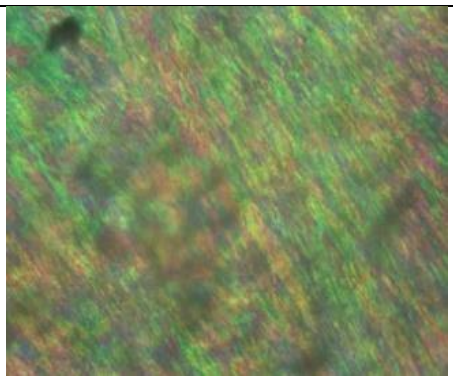
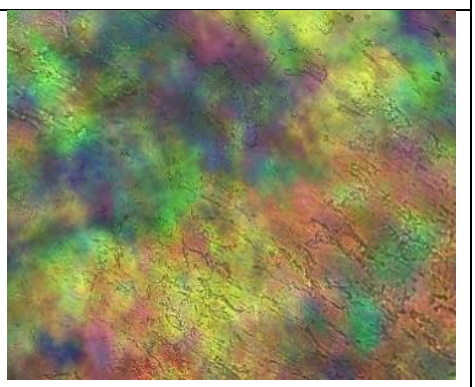
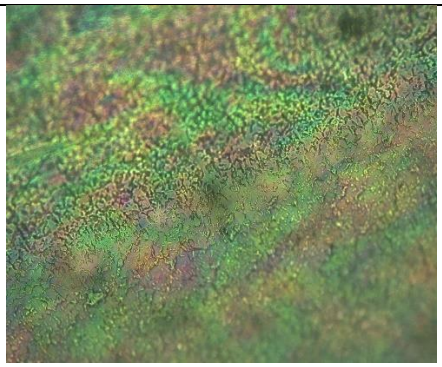
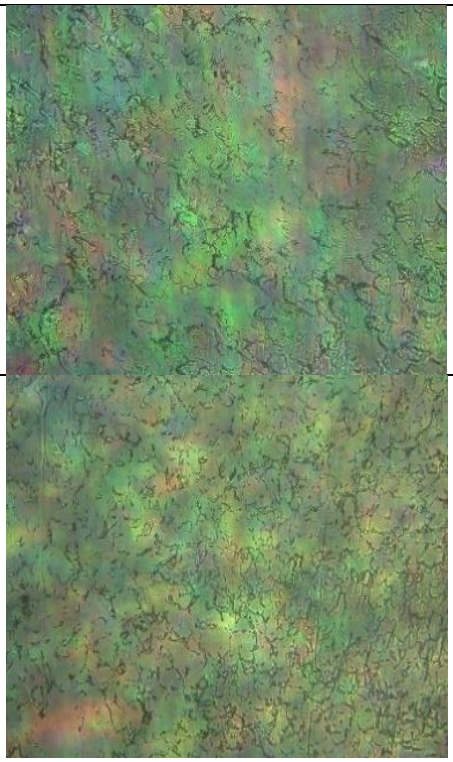
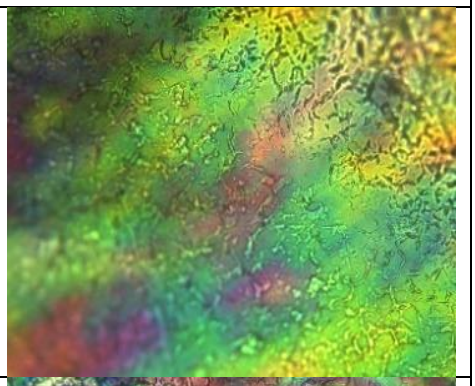
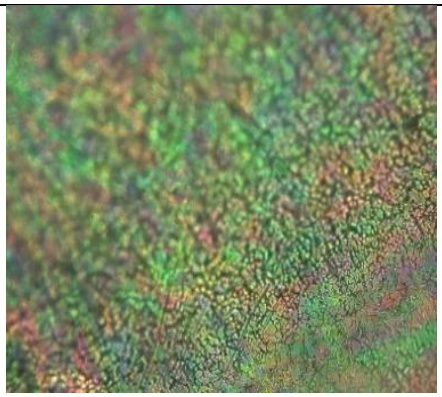
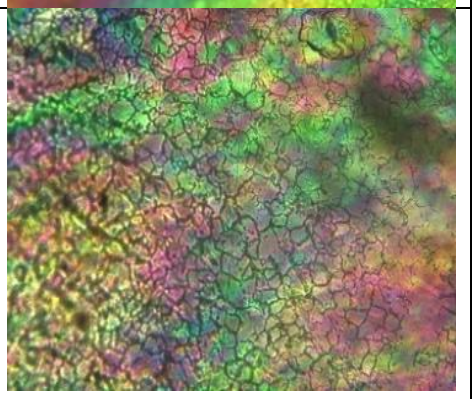
	RT (25-30)°C	55°C	62°C
30s			
15min			
1hr			

Table 2. Edge Samples - Processed in Hydraulic Press for 15 minutes at various temperatures and time points

(40x magnification)

Middle Samples

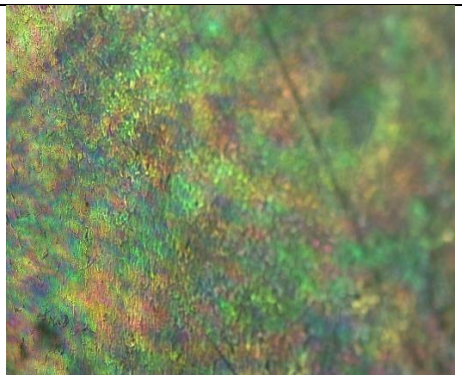
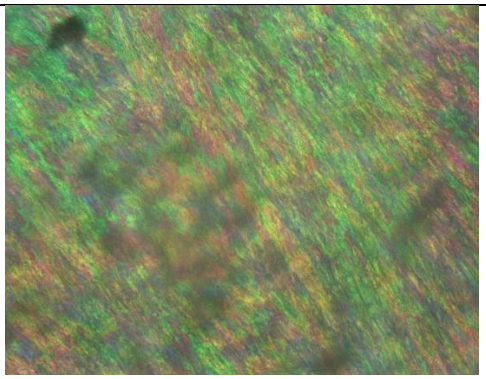
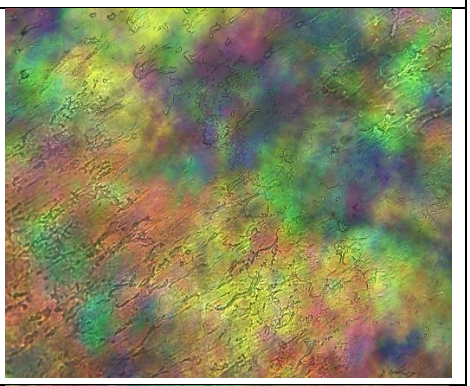
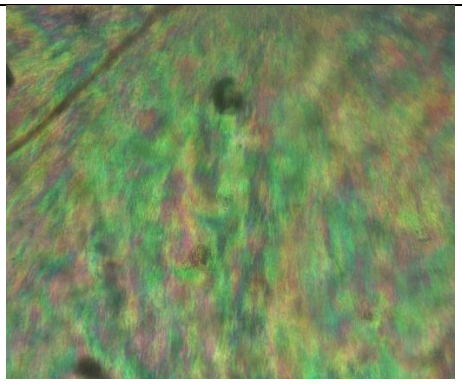
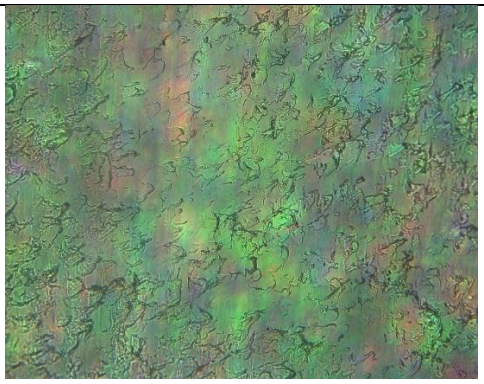
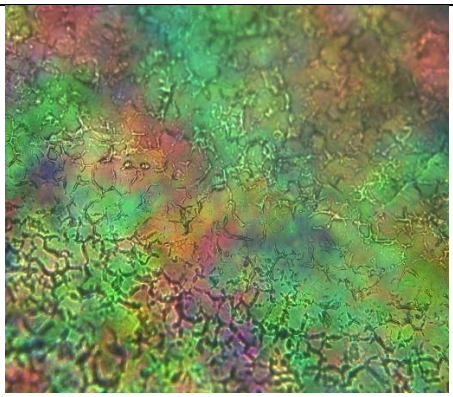
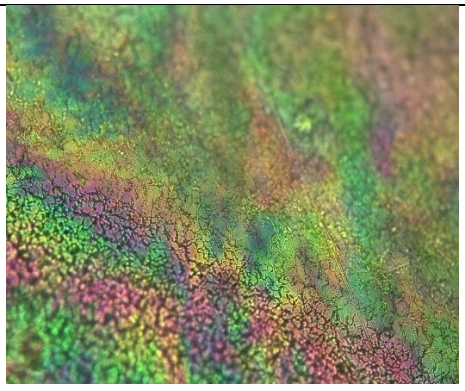
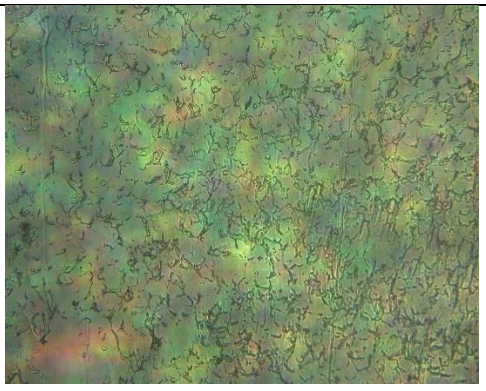
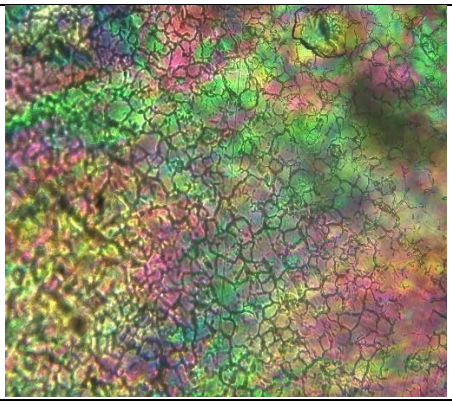
	RT (25-30)°C	55°C	62°C
30s			
15min			
1hr			

Table 3. Middle Samples - Processed in Hydraulic Press for 15 minutes at various temperatures and time points

(40x magnification)

Table 2 and Table 3 represent 40x magnification images of the edge and middle regions. Each sample processed at a different temperature and for a different amount of time. This analysis was conducted to determine if processing temperature or time of processing had an effect on birefringence and spherulite formation in the sample. As shown in Table 2, all samples processed at 62°C showed the most anisotropic birefringence as evidenced by the bright colors oriented in various directions. The samples processed for 15 minutes showed more anisotropic birefringence and spherulites than the samples processed for 30s. The analysis of these images led to the conclusion that the amount of anisotropic birefringence that can be induced dependent on the time and temperature of processing.[14] The processing temperature seems to have the greatest effect on inducing birefringence, thereby increasing the degree of orientation in the sample when processed for a substantial amount of time (at least 15 minutes) as evidenced in Table 2. There did not seem to be a stark difference in birefringence between the samples processed at 15 and 60 minutes.

Table 3 shows the birefringence from the middle region of a poly (ethylene oxide) sample treated at the same conditions as samples in Table 2. These images were taken from the middle of the samples to determine if there was similar birefringence throughout the sample by comparing edge and middle sections of an identically treated polymer. Based on the images in Table 3, the most anisotropic birefringence seen is in samples processed at 62°C. The samples processed at 62°C showed the most birefringence and showed that 15 minutes was enough processing time to reach a high amount of birefringence. Samples processed for an hour did not show a substantial difference in birefringence. Generally, the time of processing did not have a large effect on birefringence even when the temperature was kept constant. However, the temperature of processing did have a large effect on birefringence.

Therefore, the conclusions that can be made based on the optical microscopy images are that processing poly (ethylene oxide) right below its melting temperature for 15 minutes, is generally enough time to let it fully change to its final state at that temperature, can induce anisotropic birefringence and form spherulites, and therefore higher degree of crystallinity in the polymer. Overall, the temperature of processing had the largest effect on birefringence and orientation as well. These findings indicate that pressure and temperature may increase orientation and DSC can help quantify the results.

Differential Scanning Calorimetry

Unprocessed Samples

The following are the conditions of the DSC test run on the control (unprocessed) samples:

1. Mount sample at room temperature
2. Set temperature to 0°C
3. Hold at 0°C for 5 minutes
4. Start Test
5. Hold at 0°C for 1 minute
6. Heat sample to 70°C at 10°C/min
7. Hold at 70°C for 1 minute
8. Cool sample to 0°C at 10°C/min
9. Hold at 0°C for 1 minute
10. Heat sample to 70°C at 10°C/min

DSC Results – 1st heat	DSC Results – 2nd heat
92.7240 J/g (peak at 70.98°C)	85.1273 J/g (peak at 69.03°C)
106.5080 J/g (peak at 68.44°C)	107.7068 J/g (peak at 67.59°C)
108.3537 J/g (peak at 67.97°C)	116.2674 J/g (peak at 67.95°C)

Table 4. DSC Results of Unprocessed Samples

These three unprocessed samples in Table 4 serve as controls to compare to all the data shown below based on processing parameters. The 1st and 2nd heat do not have significantly different values. The 1st heat value of the 1st sampled does vary somewhat compared to the other 1st heat values. This is most likely due to the difference in homogeneity of the sample. The 2nd heat values vary slightly from the 1st heat values, indicating the DSC test may influence the crystallinity of the polymer. This data also indicates there is some variability in results.

Processed Samples

To begin the investigation into the effect of processing time and temperature, multiple processing times and temperatures were applied to samples of poly (ethylene oxide) to determine their effect on the heat of fusion values and degree of crystallinity in DSC tests. The following data sets in Tables 5-10 show the results of the DSC tests on these processed samples.

Thermal analysis of effect of processing time on samples processed at various temperatures

The following are the conditions of the DSC test run on the processed samples:

Process conditions

1. Mount sample at room temperature
2. Set temperature to -10°C
3. Hold at -10°C for 5 minutes
4. Start Test
5. Hold at -10°C for 1 minute
6. Heat sample to 75°C at 10°C/min
7. Hold at 75°C for 1 minute
8. Cool sample to -10°C at 10°C/min
9. Hold at -10°C for 1 minute
10. Heat sample to 75°C at 10°C/min

Results of DSC test:

Temperature °C Pressed	Time	DSC Results – 1st heat	DSC Results – 2nd heat
RT (25-30)	30s	127.2886 J/g	127.0410 J/g
RT (25-30)	15min	126.8983 J/g	125.4656 J/g
RT (25-30)	60min	122.0888 J/g	118.7509 J/g

Table 5. DSC Results of Samples Processed at RT (25-30)°C for various time points

Temperature °C Pressed	Time	DSC Results – 1st heat	DSC Results – 2nd heat
55	30s	141.0969 J/g	130.6836 J/g
55	15min	142.9969 J/g	127.9931 J/g
55	60min	126.2957 J/g	111.6135 J/g

Table 6. DSC Results of Samples Processed at 55°C for various time points

Temperature °C Pressed	Time	DSC Results – 1st heat	DSC Results – 2nd heat
62	30s	150.8021 J/g	130.5737 J/g
62	15min	149.8143 J/g	127.3179 J/g
62	60min	138.1823 J/g	114.4877 J/g

Table 7. DSC Results of Samples Processed at 62°C for various time points

In Table 5, samples were processed at room temperature for 30 seconds, 15 minutes, and 60 minutes. It is seen that the heat of fusion in the 1st heat and 2nd heat is very similar, but as the time of processing increases, the 2nd heat lowers slightly. In Table 6, the samples are processed at 55°C for 30 seconds, 15 minutes, and 60 minutes. The samples processed at 30 seconds and 15 minutes had much higher heat of fusion values for the 1st heat than the room temperature samples in Table 5, which indicates samples processed at 55°C had a higher degree of crystallinity. The room temperature and 55°C samples processed for 60 minutes both showed very similar heat of fusion values, indicating similar crystallinities.[9] The samples processed at 55°C showed much larger differences in the 1st and 2nd heat of fusion values, indication processing conditions in the DSC decreased the crystallinity in these samples much more than it did in the samples processed at room temperature. In table 7, the samples were

processed at 62°C for 30 seconds, 15 minutes, and 60 minutes. The 1st heat of fusion values are higher than samples processed at room temperature or 55°C, indicating higher degree of crystallinity as well. Samples processed at 62°C also show a fairly large decrease in heat of fusion values for the second heat, indicating the processing conditions of the DSC decreased the degree of crystallinity in the samples more than any other group.

Thermal analysis of effect of processing temperature of samples processed for various times

The following are the conditions of the DSC test run on the processed samples:

Process conditions

1. Mount sample at room temperature
2. Set temperature to -10°C
3. Hold at -10°C for 5 minutes
4. Start Test
5. Hold at -10°C for 1 minute
6. Heat sample to 75°C at 10°C/min
7. Hold at 75°C for 1 minute
8. Cool sample to -10°C at 10°C/min
9. Hold at -10°C for 1 minute
10. Heat sample to 75°C at 10°C/min

Temperature °C Pressed	Time	DSC Results – 1st heat	DSC Results – 2nd heat
30	30s	127.2886 J/g	127.0410 J/g
55	30s	141.0969 J/g	130.6836 J/g
62	30s	150.8021 J/g	130.5737 J/g

Table 8. DSC Results of Samples Processed at various temperatures for 30 seconds

Temperature °C Pressed	Time	DSC Results – 1st heat	DSC Results – 2nd heat
30	15min	126.8983 J/g	125.4656 J/g
55	15min	120.2809 J/g	111.6540 J/g
62	15min	149.8143 J/g	127.3179 J/g

Table 9. DSC Results of Samples Processed at various temperatures for 15 minutes

Temperature °C Pressed	Time	DSC Results – 1st heat	DSC Results – 2nd heat
30	60min	122.0888 J/g	118.7509 J/g
55	60min	126.2957 J/g	111.6135 J/g
62	60min	138.1823 J/g	114.4877 J/g

Table 10. DSC Results of Samples Processed at various temperatures for 60 minutes

Tables 8-10 above depict the effect of processing temperature when the samples are processed for different time (30 seconds, 15 minutes, or 60 minutes) The samples processed a short amount of time (30 sec), depict increased crystallinity as the process temperature increases, which is shown by the increasing heat of fusion values in Table 8. The same trend is generally found for samples processed at 15 minutes (Table 9) and 60 minutes (Table 10). Based on this data, another general trend found is that for the same period of processing time, the crystallinities increased as the processing temperature increases. This phenomenon is shown by the increase of the heat of enthalpy values as process temperature increases. The time of processing does not seem to affect the 2nd heat of fusion values as much as the temperature of processing does as shown by the varied 2nd heat of fusion results in Tables 8-10 which correlate with temperature much more than time of processing. (Should we take out 2ndheat)

Thermal analysis of effect of different cooling rates in DSC test

The purpose of this analysis was to determine how the rate of cooling affected the degree crystallinity of the sample. The investigation was supposed to determine if it took a varying amount of energy to heat the sample to the same final temperature if the sample heated at one rate (10°C) and then was cooled very slowly (1°C/min), normal rate (10°C/min), or very quickly (100°C/min). The samples were heated to a value between 70°C-80°C, but differences in heat of fusion values were not shown to vary significantly between identical samples heated to any of these temperatures since they are all above the melting temperature.

The following are the conditions of the DSC test run on the processed samples:

Process conditions

1. Mount sample at room temperature
2. Set temperature to -10°C
3. Hold at -10°C for 5 minutes
4. Start Test
5. Hold at -10°C for 1 minute
6. Heat sample to (70-80)°C at 10°C/min
7. Hold at (70-80)°C for 1 minute
8. Cool sample to -10°C at 1°C, 10°C, or 100°C /min
9. Hold at -10°C for 1 minute
10. Heat sample to (70-80)°C at 10°C/min

Temperature °C Pressed	Heating rate	Cooling rate	DSC Results – 1st heat	DSC Results – 2nd heat
30	10°C	1°C	118.0366 J/g	140.9872 J/g
30	10°C	10°C	128.9473 J/g	125.5562 J/g
30	10°C	100°C	133.5276 J/g	123.8196 J/g
55	10°C	1°C	127.7293 J/g	139.1452 J/g

55	10°C	10°C	130.6723 J/g	119.6513 J/g
55	10°C	100°C	78.8995 J/g	69.6067 J/g
62	10°C	1°C	145.7385 J/g	138.0457 J/g
62	10°C	10°C	135.3502 J/g	133.7821 J/g
62	10°C	100°C	151.0036 J/g	124.4533 J/g

Table 11. DSC results of samples cooled at various rates

The 1st heat of fusion value in Table 11 represents the degree of crystallinity of the polymer after processing in the hydraulic press. The DSC test was programmed to heat this sample at a rate of 10°C/min and give this 1st heat of fusion value. The sample was then cooled at these various rates of 1°C/min, 10°C/min, and 100°C/min. The samples were held at -10°C and then heated for a second time at 10°C/min. This heating step gives the 2nd heat of fusion value and shows the effect of the cooling process on the sample.

Overall, this data showed that the cooling rate does influence the heat of fusion values and therefore affects the degree of crystallinity of the samples. The samples processed at RT (25-30)°C show a much larger difference in heat of fusion values between the 1st and 2nd when cooled very slowly (1°C/min) vs the normal rate (10°C/min) or at the much quicker rate (100°C/min). This large difference in 1st and 2nd heat of fusion indicates that as this polymer was cooled slowly over time, the degree of crystallinity increased drastically compared to the degree of crystallinity of original processed polymer. The next sample processed at RT (25-30)°C was cooled at 10°C/min and did not show a significant difference, just slightly lower values between the 1st and 2nd heat of fusion, indicating the degree of crystallinity did not change much based on this cooling process. The next sample processed at RT (25-

30)°C was cooled at 100°C/min and showed a decrease in heat of fusion, indicating this very quick cooling rate decreased the degree of crystallinity of the polymer.

The samples processed at 55°C showed differences in heat of fusion patterns when cooled at 1°C/min, 10°C/min, 100°C/min. Samples cooled at 1°C/min showed a higher heat of fusion value in the 2nd heat, indicating a higher degree of crystallinity after processing. Samples cooled at 10°C/min showed a lower heat of fusion value in the 2nd heat, indicating a lower degree of crystallinity processing. Samples cooled at 100°C/min showed a lower heat of fusion value in the 2nd heat, indicating a lower degree of crystallinity. The samples processed at 55°C show a similar trend to the samples processed at RT (25-30)°C, but show less of a difference in the sample processed at 1°C/min and a larger difference in the samples cooled at 10°C/min and 100°C/min.

The samples processed at 62°C showed differences in heat of fusion patterns when cooled at 1°C/min, 10°C/min, 100°C/min as well. Samples cooled at 1°C/min showed a lower heat of fusion value in the 2nd heat, indicating a lower degree of crystallinity after processing. Samples cooled at 10°C/min showed a similar heat of fusion value in the 2nd heat, indicating a similar degree of crystallinity processing. Samples cooled at 100°C/min showed a significantly lower heat of fusion value in the 2nd heat, indicating a significantly lower degree of crystallinity. The samples processed at 62°C sometimes show a similar trend to the samples processed at RT (25-30)°C or 55°C.

All these processed samples showed some similarity in how cooling rate affected their crystallinity. The most significant differences in 1st and 2nd heat of fusion were in the RT samples cooled at 1°C/min and 62°C samples cooled at 100°C/min. In addition, both the RT and 55°C samples showed an increase in crystallinity when cooled slowly. The 62°C samples were not as affected by a slow cooling

rate (1°/min). Both the RT and 55°C samples were slightly affected by the fast cooling rate (100°C/min), showing slightly less crystallinity after cooling. The 62°C samples showed a similar trend, showing less crystallinity, but the 62°C showed a much lower degree of crystallinity based on this cooling rate. All of the samples showed the least difference in heat of fusion rates when cooled at the normal rate (10°C/min). Therefore, it can be concluded that very fast and very slow cooling rates have the greatest effect on the degree of crystallinity, and it varies based on the original processing conditions of the poly (ethylene oxide) samples.

Thermal analysis of difference between heat of fusion between edge or middle pieces of sample in DSC test

The purpose of this experiment was to determine if there were differences in the degree of crystallinity in different areas of the sample. This experiment could elucidate how the different regions of a polymer are affected by processing and if it is possible to create a homogenous sample in terms of crystallinity.

The following are the conditions of the DSC test run on the processed samples:

1. Mount sample at room temperature
2. Set temperature to 0°C
3. Hold at -10°C for 5 minutes
4. Start Test
5. Hold at -10°C for 1 minute
6. Heat sample to 75°C at 10°C/min
7. Hold at 75°C for 1 minute
8. Cool sample to -10°C at 10°C/min
9. Hold at -10°C for 1 minute
10. Heat sample to 75°C at 10°C/min

Temperature °C Pressed	Edge or Middle	Time	DSC Results – 1st heat
Unprocessed	Edge	N/A	124.9978 J/g
Unprocessed	Middle	N/A	106.5080 J/g
RT (25-30)	Edge	15min	130. 4174 J/g
RT (25-30)	Middle	15min	133.7974 J/g
55	Edge	15min	142.9969 J/g
55	Middle	15min	143.8491 J/g
62	Edge	15min	149.8143 J/g
62	Middle	15min	148.0988 J/g

Table 12. DSC results comparing 1st heat of edge and middle of each sample processed at various temperatures

As shown in Table 12, unprocessed (no applied pressure or heat) poly (ethylene oxide) samples showed clearly different results between the edge and middle of the polymer samples. The middle samples had a lower degree of crystallinity than the edge of the sample. The polymers processed at various temperatures for 15 minutes each showed similar results middle and edge of the polymer. This finding indicates that the pressure and heat applied created a more uniform sample, which resulted in similar heat of fusion values in the DSC analysis. The ability to create this uniform structure is beneficial for ensuring consistent properties throughout the entire film during manufacturing.

Thermal analysis of Polyethylene

This experiment was conducted to determine how DSC processing would affect another polymer similar to poly (ethylene oxide). Poly (ethylene oxide) is a modification of poly (ethylene), so this experiment would elucidate the effect the oxide bond in poly (ethylene oxide) has on the degree of crystallinity of this polymer.

The following are the conditions of the DSC test run on the processed samples:

1. Mount sample at room temperature
2. Set temperature to 0°C
3. Hold at -10°C for 5 minutes
4. Start Test
5. Hold at -10°C for 1 minute
6. Heat sample to 145°C at 10°C/min
7. Hold at 145°C for 1 minute
8. Cool sample to -10°C at 10°C/min
9. Hold at -10°C for 1 minute
10. Heat sample to 145°C at 10°C/min

Melting Point of Polyethylene is 115°C-135°C. Temperature above melting point was chosen in DSC test.

Temperature °C Pressed	Time	DSC Results – 1st heat	DSC Results – 2nd heat
Untreated	15min	203.6385 J/g	202.0732 J/g
30	15min	209.0675 J/g	208.5626 J/g
50	15min	191.6483 J/g	191.3508 J/g
60	15min	222.2421 J/g	221.0752 J/g
75	15min	202.2520 J/g	198.6449 J/g

Table 13. DSC results of polyethylene processed at various temperatures for 15 minutes

As shown in Table 13, the temperature applied to polyethylene samples while in the hydraulic press seemed to show no trend or ability to change heat of fusion values. Polyethylene had higher heat of fusion values compared to poly (ethylene oxide), indicating higher degree of crystallinity, which may be the cause of its resistance to change structure at the same temperatures that poly (ethylene oxide) did.

Overall, the DSC data in Tables 1-13 indicate that processing time and temperature can play a large role in inducing new morphologies and crystallinities in poly (ethylene oxide). It was also determined that the cooling rate of the sample could also induce new morphology. These properties of poly (ethylene oxide) show that the polymer structure can be malleable based on processing conditions.

X-ray Diffraction

Polyethylene

The purpose of this section was to determine the crystal structure of polyethylene, as it was chosen to be a low molecular weight polymer comparison. Polyethylene is known to have thicker lamellae that poly (ethylene oxide) so it is less likely to changed structure at the same processing conditions applied to poly (ethylene oxide). The polyethylene samples were processed in the hydraulic press in similar conditions to those that applied to poly (ethylene oxide). The polyethylene samples were processed for 15 minutes under 20,000lbs of pressure at varying temperatures.

Control 30°C 50°C 60°C

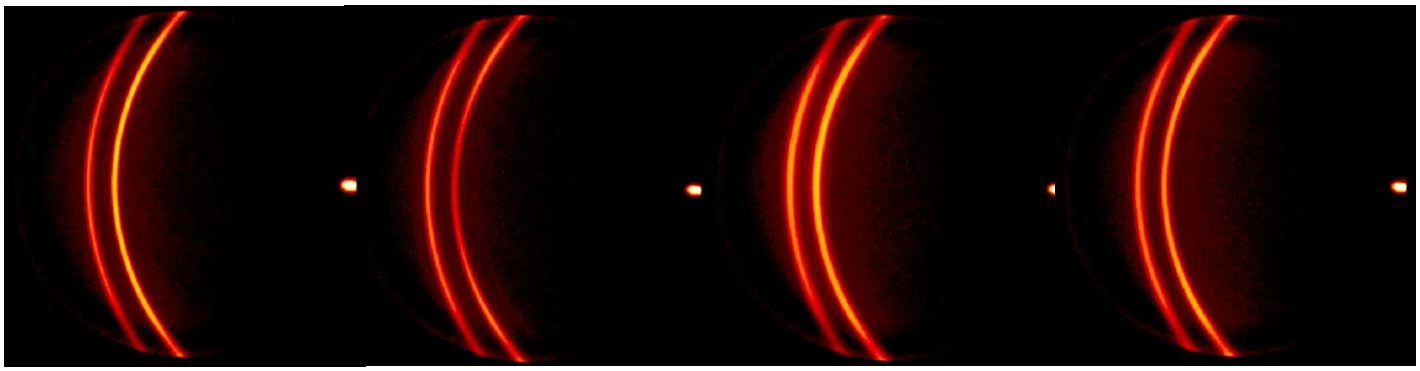


Figure 14. XRD scans of polyethylene samples unprocessed or processed at 30°C, 50°C, or 60°C.

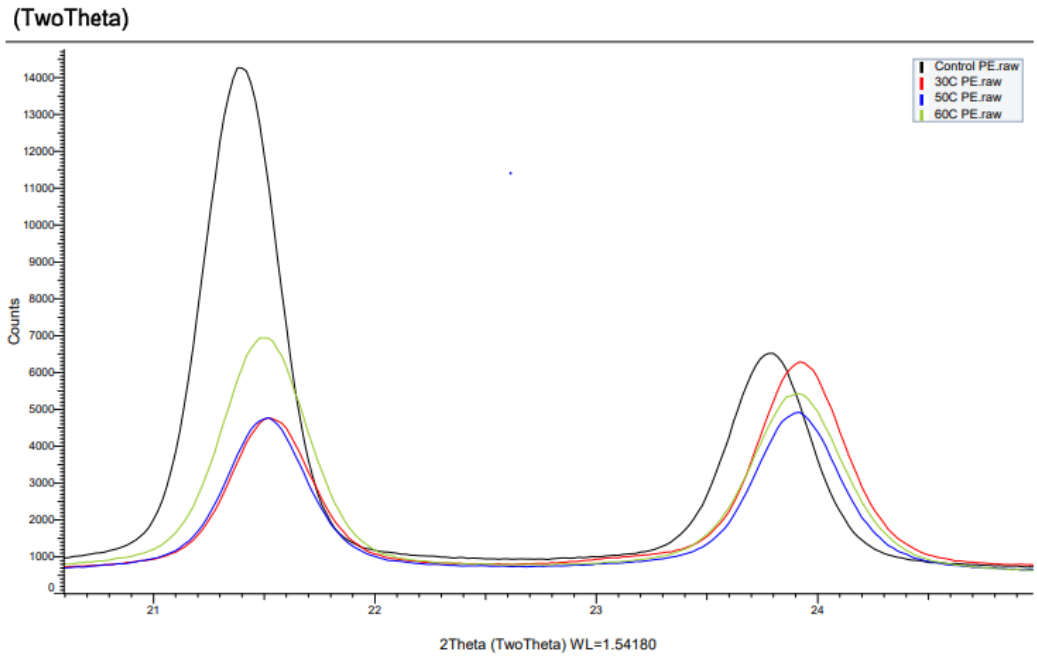


Figure 15. XRD histogram of polyethylene samples unprocessed or processed at 30°C, 50°C, or 60°C.

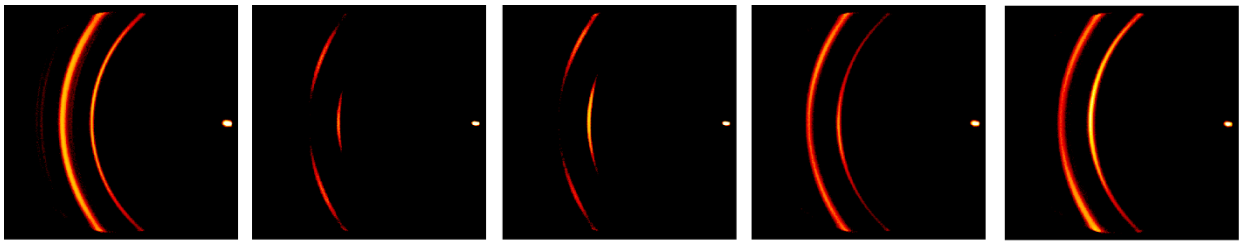
Shown in Figure 14, the unprocessed sample of the polyethylene XRD scans show clear rings in the XRD scans indicating a semi-crystalline polymer. The sample processed at 30°C show a slightly diminishing ring. The samples processed at 50°C and 60°C show a higher intensity and slightly broader ring. These samples also show a slight amorphous halo, similar to the XRD profile of the unprocessed polymer.

The XRD spectra of the polyethylene scans are shown in Figure 15. The height of the peak has been shown to correlate to the degree of crystallinity of a sample. In Figure 15, the first large peak is starkly higher in intensity and height than the processed polymers, indicating the unprocessed polymer had very high degree of crystallinity[12]. The scan labeled 60°C shows a slightly high intensity scan and peak in the histogram, indicating a higher degree of crystallinity in this polymer compared to the other processed samples, but still less than unprocessed polyethylene. Processing the polymer at any

temperature under the 20,000lbs for 15 minutes seems to be enough to create a change in morphology, creating a more amorphous polymer sample. Temperature does not seem to play a large factor at all in terms of the degree of crystallinity.

PEO

The samples in Figures 16 and Figure 17 below show the XRD scans and histogram of poly (ethylene oxide) samples that were unprocessed or processed in the hydraulic press under 20,000lbs, at temperatures of RT (25°C), 52°C, 57°C, or 62°C, for 15 minutes. In this analysis, Figures 16 and 17 will be used to study the structure and crystallization of poly (ethylene oxide) after processing at various conditions.



PEO Unaltered **25°C** **52°C** **57°C** **62°C**

Figure 16. XRD scans of processed PEO at various temperatures

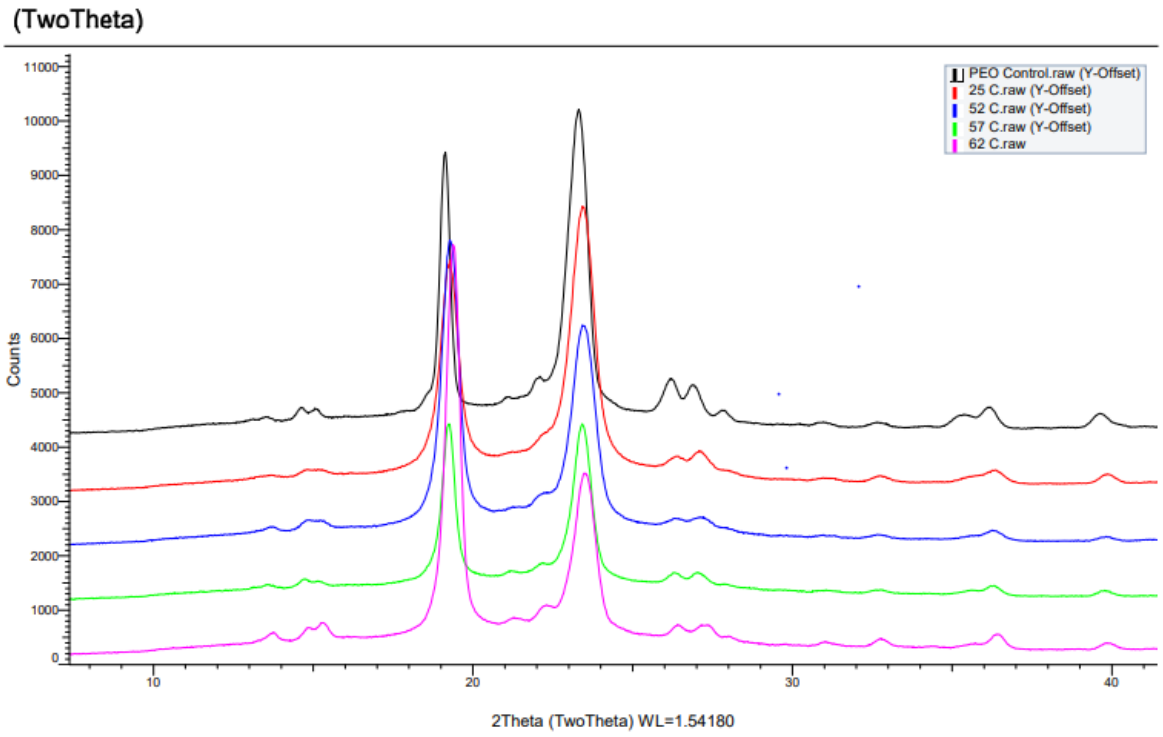


Figure 17. XRD histogram of processed PEO at various temperatures

In Figure 16, the 2D image of the unaltered PEO (far left image) sample shows two thick low intensity bands. This is a typical reflection of an alpha crystal structure. The 2D images labeled 25°C, 52°C, 57°C, 62°C represent a sample processed in the hydraulic press at these temperatures, respectively before being analyzed in the XRD. The first two of these scans, 25°C and 52°C samples show high intensity reflections at the equator dominantly, indicating partial lamellae orientation and a heterogenous slip at these temperatures.[12] The next two samples labeled 57°C and 62°C show broader intensity at the equator of the bands than the previous samples, which is indicative of smaller crystal size or increasing structural order in the sample. This broadening at the equator of the samples can indicate a crystal-like mesogenic phase, especially in the 62°C sample. This conclusion suggests that processing the polymer at higher temperatures (closer to the melting point) can result in disordering of the original crystal

conformation into a new one. This also indicates that the sample processed at 62°C has lamellar fragmentation and a high degree of crystallinity when processed at these higher temperatures.

Based on literature, pure PEO will show high intensity diffraction peaks at 19.36° and 23.72° because of the order of polyether side chains and strong intermolecular forces connecting chains in poly (ethylene oxide).[14] These high intensity, narrow peaks indicate a semi-crystalline polymer. These peaks are seen in the spectra of Figure 17 for all the samples for the unprocessed and processed samples. The peaks correlating to each poly (ethylene oxide) sample become incrementally less intense and shorter in height as the sample processing temperature increases towards the melting temperature. Many other peaks at the higher 2θ region are also less intense. This decrease in intensity indicates a decrease in the degree of crystallinity in the backbone of the PEO.[15] The peaks also slightly broadened as the processing temperature increased, indicating a possible mesogenic phase in the polymer.[13] The peak of the 62°C sample was high in intensity and had a broader peak further suggesting the presence of the crystal-like mesophase also seen in the XRD scans of the 62°C sample.

HOT STAGE XRD RESULTS – Poly (ethylene oxide)

The images in Table 14 represent the Hot Stage XRD scans of poly (ethylene oxide) samples that were either unprocessed or processed in the hydraulic press under 20,000lbs, at temperatures of RT (25-30°C), 55°C, or 62°C, for 15 minutes. Samples were cut into small squares and placed on the slide on the stage of the XRD. The scans were taken at temperatures of 25°C, 45°C, 55°C, 65°, and 75°C. The sample intensity was adjusted in the Bruk Eva Diffract Software to see the degree of crystallinity in the sample.

XRD Temperature

Processing Temperature

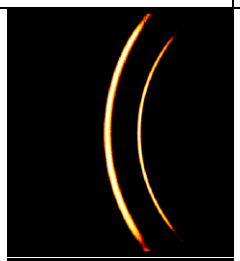
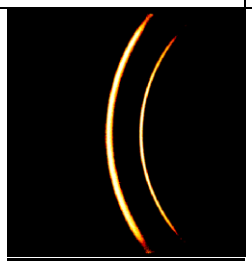
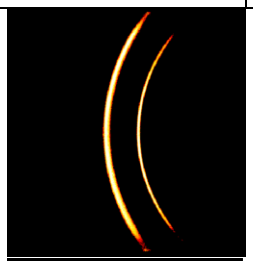
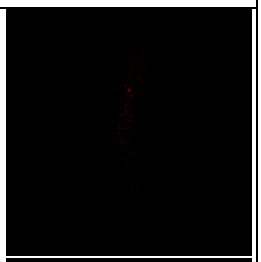
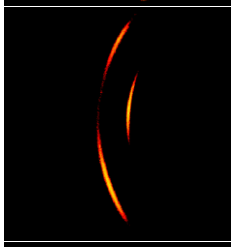
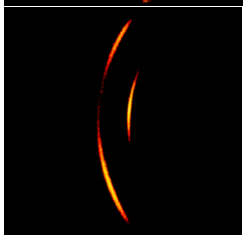
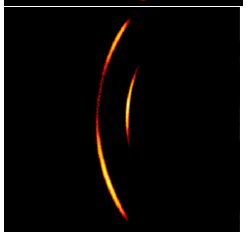
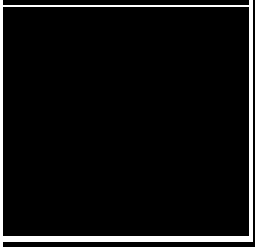
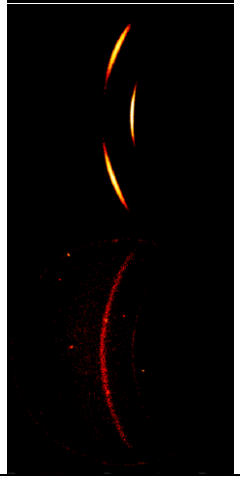
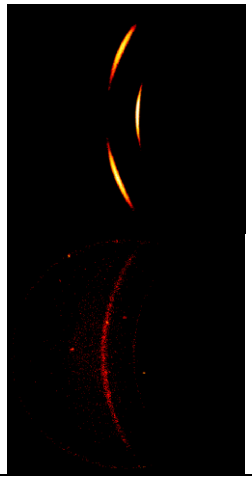
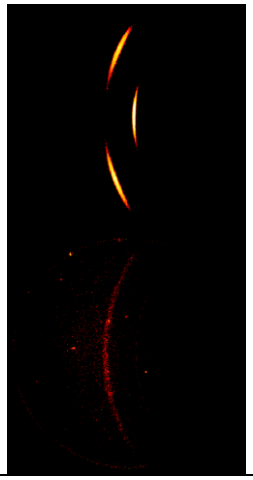
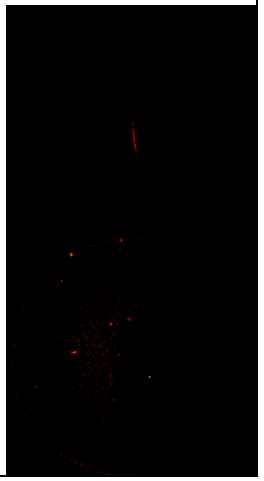
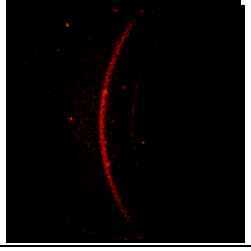
Hot Stage XRD	25°C	45°C	55°C	65°C	75°C
Unprocessed					
RT – (25-30)°C					
55°C					
62°C					

Table 14. Hot Stage XRD Results of Poly (ethylene oxide)

The images in the unprocessed row (Top row of Table 14) show normal high intensity bands that indicate a semi-crystalline polymer. The scans in the RT row (2nd row of Table 14) show much higher intensity peaks at the equator and sharp rings that indicate a high degree of crystallinity and partial lamellae orientation. The scans in the 55°C row (3rd row of Table 14) show higher intensity peaks than both the previous samples and also show sharper rings compared the unprocessed samples. This indicates a very high degree of crystallinity. The scans in 62°C row (4th row of Table 14) are dimmer, potentially because of the thickness of the sample. However, they also show a highly crystalline structure

based on their sharp rings. It is shown in the 25°C column that as the samples are treated closer to the melting point, they show more intense and shark peaks, indicating high degrees of crystallinity. All the samples showed similar XRD scans as the sample was melted to 75°C. The unprocessed and room temperature processed samples showed a fully amorphous structure at 75°C, while the 55°C and 62°C samples displayed some intensity even above the melting point. The 62°C also showed a broader band at all temperatures while melting, indicating a possible crystal-like mesogenic phase.[13] Overall, this data shows the structure of the samples are stable throughout the melting process regardless of processing temperature. The scans also depict the possibility of a mesogenic phase of the samples processed at 62°C, maintained throughout the melting process in the XRD.

INSTRON RESULTS (Pilot Study)

Poly (ethylene oxide) powder was placed into silicon mold. The mold was put into the oven at 80°C for 30 minutes. Samples were then left to dry for 1 hour in the mold (Figure 18). Some samples were kept as unprocessed control samples. The other samples were pressed at a pressure of 20,000 for 15 minutes at varying temperatures. Samples were pressed at room temperature (25°C -30°C), 55°C, and 62°C. An unprocessed sample was used as reference. Measurements were taken for all the samples before and after testing to take into the account that the pressure and temperature from the hydraulic press could alter the shape of the samples. The pressing condition did change the shape of the dogbone samples when the samples were pressed at 55°C and 62°C. Samples to recut as close to the dogbone shaped as possible using the mold and place in the sample holders to perform the tensile test (Figure 19).

Figures 31 and 32 show the processing of melting the samples to create the dogbone shape and how a sample processed at 55°C looked when fracturing during the tensile test in the Instron. A sample tensile stress curve of a control sample is shown in Figure 20 below. As a tensile force was applied to the samples, a curve similar to Figure 20 would be produced, which indicated how the specimen deformed over time before fracture.

Figure 18. Samples drying after oven into dogbone shapes

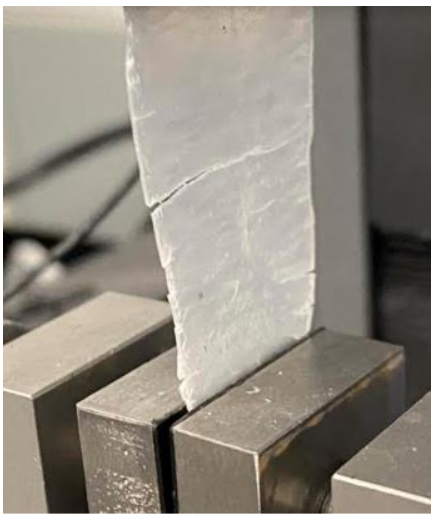


Figure 19. Sample processed at 55°C breaking after tensile test in Instron

Sample result:

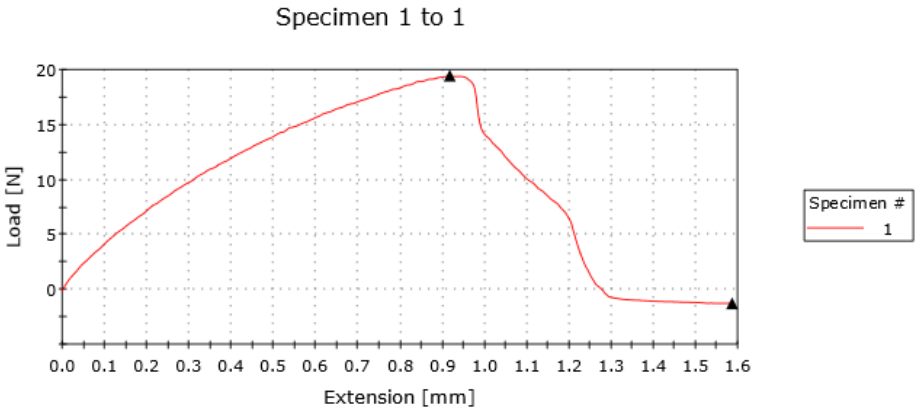


Figure 20. Instron results of unprocessed samples

There was some variation in the maximum average load of some of the samples as they slipped through the grips or as they stretched. Some of the control samples stretched slightly more than others when they had a lower degree of crystallinity or little lamellar orientation, allowing for more ductility instead of fracturing quickly. Sometimes these polymers stretched a small amount before they started slipping from the grips, especially in the control samples. It is possible the polymer structure became

more crystalline because of the stretching during the slipping. That could be a reason that in following tensile tests, some of the samples fractured slightly quicker. There were many attempts to prevent slipping in the Instron starting with the control samples. Eventually, after using some tape, the control samples began to slip less. This tape was not used on the rest of the samples as they were slipping less often. The samples processed at RT, 55°C, and 62°C did not have as much of an issue with slipping. These samples would more often slip one or two times, but then would fracture. These samples were also more brittle and thereby less rubbery than the unprocessed or control samples, and therefore less slippery in the grips. To try to determine the maximum strength the polymer had at any point, the sample with the highest maximum load was chosen for comparison between the polymers.[16]

The three parameters of focus in this section of the investigation are the tensile stress, and the tensile strain, and ductility of the sample. These values are shown in Table 15 bellow. The tensile stress and tensile strain were calculated and normalized by the dimensions of each sample for comparison. The ductility of the samples was analyzed quantitatively based on the values in Table 15 and qualitatively by the graphs. Ductility refers to a material's ability to withstand tensile force by undergoing deformation until failure. In contrast, a brittle sample in this context would fracture quickly upon load with little deformation or strain. The following equation was used to calculate the tensile stress of the samples at maximum load:

$$Tensile\ Stress\ (KPa) = \frac{Maximum\ Load\ (N)}{Width\ (mm)\ x\ Thickess\ (mm)} x1000$$

The following equation was used to calculate the tensile strain of the sample at maximum load:

$$Tensile\ Strain\ (\%) = \frac{Change\ in\ Length\ (mm)}{Total\ Length\ (mm)}$$

Sample Description	Dimensions (LengthxWidthxThickness)(mm)	Maximum Load (N)	Tensile Stress (KPa)	Tensile Strain (%)
Control 1	20x10x3	20.22	674	≤4.5
Control 2	20x10x3	19.45	648	≤4.5
Room Temperature 1	23x11x2.5	19.11	695	≤4.3
Room Temperature 2	23x11x2.5	21.79	792	≤4.3
55°C Sample 1	30x20x1.7	51.60	1518	≤1.7
62°C Sample 1	40x30x0.8	51.87	2161	≤1.3

Table 15. Instron results of control and processed poly (ethylene oxide) samples

Table 15 above provides calculated values of the tensile stress and tensile strain based on the maximum load and cross-sectional area of each sample of poly (ethylene oxide) tested. The dimensions of each sample are shown in the second column of Table 15. The dimensions of the samples processed at room temperature do not vary significantly from the dimensions of the control samples. The thickness of the samples processed at 55°C and 62°C was significantly lower than the control or room temperature processed samples. All samples took two attempts to fracture, and the maximum load and corresponding values are shown in Table 15. The maximum load in the third column of Figure 15 refer to the maximum force the sample could withstand during the tensile test.

All the control samples and room temperature processed samples had similar tensile stress as shown in the column labeled tensile stress at maximum load in Table 15. The room temperature processed samples were put under 20,000lbs for 15 minutes, and this pressure alone did not seem to create much difference from the control samples in terms of visible modifications, in the amount of force the sample could withstand, or in the tensile stress or strain at maximum load, but the samples were very slippery in the Instron machine, so this may have had an effect on these values for the room temperature processed samples.

The thickness of the samples processed at 55°C and 62°C was significantly lower than the thickness of room temperature and control samples, and these samples were able to withstand very

high maximum loads. The sample processed at 55°C showed a much higher tensile stress (1518Kpa) and much lower tensile strain (~1.7) to that of the samples processed at room temperature (750Kpa, 4.3) or control samples (~660Kpa, ~4.5), indicating the 55°C samples comparatively showed more brittle behavior under load. This behavior could be due to high crystallinity in the 55°C processed samples and possible lamellar orientation in the tensile direction. The samples processed at 62°C similarly showed very high tensile stress (2161Kpa) and very low tensile strain (~1.3) similar to the 55°C processed samples. The samples processed at 62°C actually showed a significantly higher tensile stress than even 55°C, indicating the samples processed at 62°C may have also had an even higher degree of crystallinity and more lamellar orientation in the tensile direction. The behavior of samples processed at 62°C were not very elastic and very brittle, causing a quick fracture with no necking under tensile load.

The estimated young's modulus values were calculated as well. The following equation was used to calculate the Young's Modulus of the sample at maximum load:

$$Young's\ Modulus\ (Kpa) = \frac{Tensile\ Stress\ (Kpa)}{Tensile\ Strain\ (\%)}$$

This property varies based on the tensile stress and strain values calculated, which may have been impacted by slipping. Generally, the trend seem in the estimated young's modulus values is that the young's modulus of the control and room temperature processed samples were similar at about 100-200KPa, indicating a more less stiff and less crystalline polymer compared to the other processed samples. The young's modulus increased dramatically when processed at 55°C (~900) and 62°C (~1650), indicating a much stiffer and highly crystalline material at these conditions. Therefore, the heating temperature did have a dramatic effect on the stiffness and ductility of poly (ethylene oxide).

Summary of Results

This investigation began by studying unprocessed high molecular weight poly (ethylene oxide) under polarized light, a qualitative method. This qualitative analysis indicated that there was spherulite formation and small amounts of birefringence and semi-crystallinity in poly (ethylene oxide). Upon processing this polymer in a hydraulic press at RT (25-30)°C, 55°C and 62°C at various time points, further polarized microscopy indicated that pressure and temperature could induce more spherulite formation. This data also showed that processing poly (ethylene oxide) at higher temperatures, close to its melting point would induce the most birefringence and high degree of crystallinity. To study this poly (ethylene oxide) quantitatively, unprocessed and processed poly (ethylene oxide) were run in the DSC. This DSC product the heat of fusion required to melt the polymer before and after processing conditions. Overall, it was indirectly shown that processing poly (ethylene oxide) at higher temperatures, close to the melting point increased the degree of crystallinity. These high temperature processing conditions caused the heat of fusion to increase and therefore indicated the degree of crystallinity increased as well. This data supports the observations found through polarized microscopy. The XRD shows qualitative and quantitative data. The XRD 2D scans of the unprocessed and processed poly (ethylene oxide) results indicated that the unprocessed poly (ethylene oxide) had some degree of crystallinity, and processed poly (ethylene oxide) had a higher degree of crystallinity and a crystal-like mesogenic phase induced. The Instron testing showed that increasing the processing temperature of the polymer increased the mechanical stress and Young's Modulus at maximum load, and decreased the mechanical strain, indicating a brittle and stiff polymer can be induced with highly crystalline structure at high processing temperatures. Overall, similar behavior is seen in all the qualitative and quantitative analysis of all the tests.

Potential Applications of Poly (ethylene oxide)

As mentioned earlier, there are numerous applications for poly (ethylene oxide). There is already preliminary as a controlled drug delivery as PEO is biocompatible and malleable as discussed in this investigation. This drug delivery can include use as a plasticizer in the film-coating of food supplements in tablet or capsule form as well. Poly (ethylene oxide) can be used as a scaffold. PEO is also known for being an asset in electrospinning of biopolymer solutions.[1]

In the electronic sphere, PEO is very highly thought to be a useful tool in batteries as discussed earlier. The processing methods in this investigation were in part determined based on the notion that ionic conductivity in poly (ethylene oxide) can be increased by creating a mesogenic phase in the polymer. This mesogenic phase should provide the polymer with the higher conductivity and lower rigidity needed to be successful in the battery.[17] PEO can also be used in other electronics such as frames, circuit boards, and screens if it can be processed into use in those industries. This polymer can also be used in packaging of materials including electronics or food because of its high strength and durability.

Another industry that utilizes polymers is the apparel, especially the footwear industry. Polymers are used in many shoes, especially sneakers to aid in performance and comfort. It can also be used as an elastic band in sports bras or pants with an elastic waist. There is such a diverse range of applications that poly (ethylene oxide) can contribute to if properly understood.

Conclusion

Poly (ethylene oxide) has shown incredible potential in so many applications. This investigation covered a preliminary look into the capabilities of poly (ethylene oxide). Poly (ethylene oxide) has shown to respond very differently to processing at various temperatures and at short time points of 15 minutes. This short processing alone shows birefringence and a mesogenic phase in certain samples which can indicate a tunable mesogenic polymer with brand new properties. This investigation also shows a pattern of evidence for different conditions. The samples processed at 55°C and 62°C have consistently shown to form a highly crystalline structure with lamellar orientation. Further research can be done to study poly (ethylene oxide) processed at many temperatures to continue determining if the new properties are advantageous for certain applications. It is recommended to study this polymer under various pressure amounts and perform compression tests to understand the material behavior under applied stress. It is important to study this polymer and the new properties that can be induced in it. Overall, this polymer proved to show interesting properties when subjected to thermal and mechanical treatment, but much further research needs to be done to fully understand the possibilities of poly (ethylene oxide).

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Appendix 1 – Differential Scanning Calorimetry

Differential Scanning Calorimetry – Poly (ethylene oxide)
Sample 1 - Process conditions – Unprocessed – Edge

1. Hold heat at 0°C
2. Heat sample to 70°C at 10°C/min
3. Hold at 70°C for 1 minute
4. Cool sample to 0°C at 10°C/min
5. Hold at 0°C for 1 minute
6. Heat sample to 70°C at 10°C/min

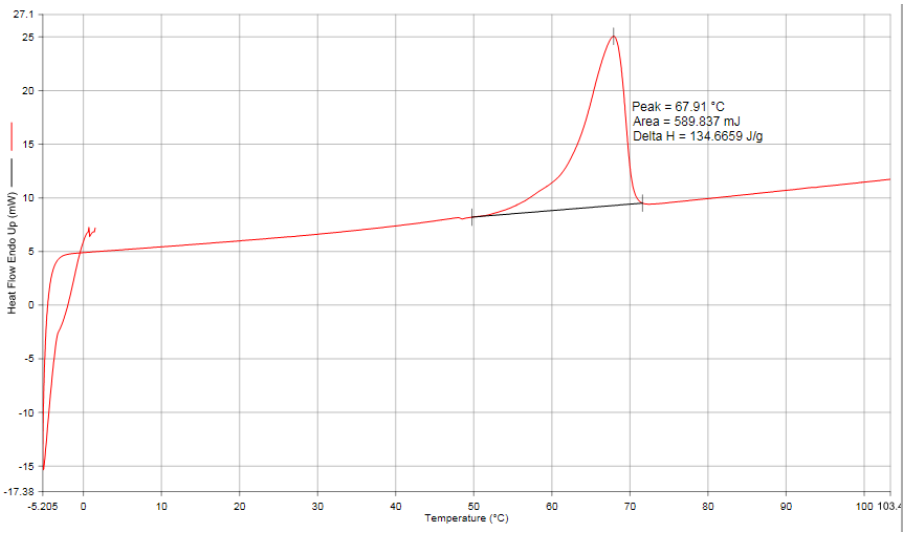


Figure 1 (a). 1st heat

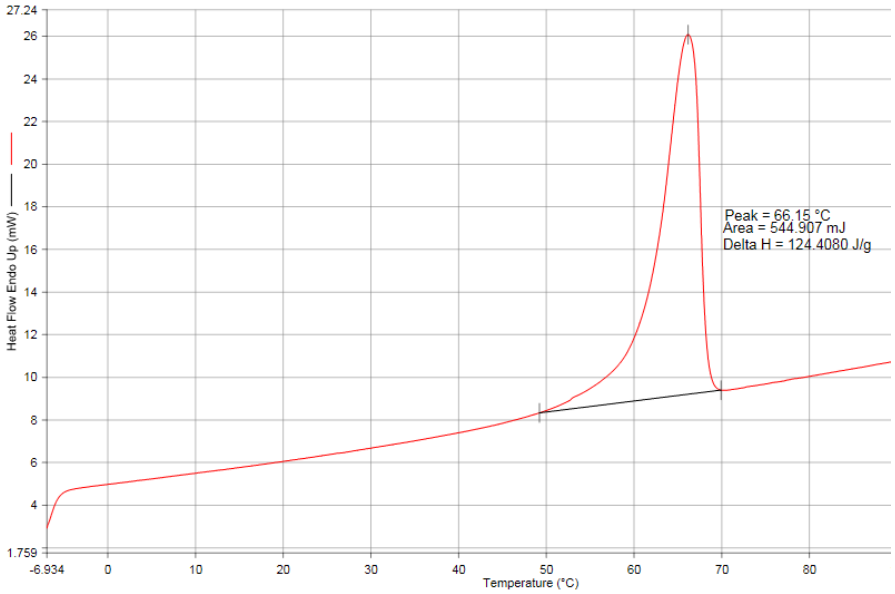


Figure 1 (b). 2nd heat

Differential Scanning Calorimetry
Sample 2 - Process conditions – Unprocessed – Edge

1. Hold heat at 0°C
2. Heat sample to 100°C at 10°C/min
3. Hold at 100°C for 1 minute
4. Cool sample to 0°C at 10°C/min
5. Hold at 0°C for 1 minute
6. Heat sample to 100°C at 10°C/min

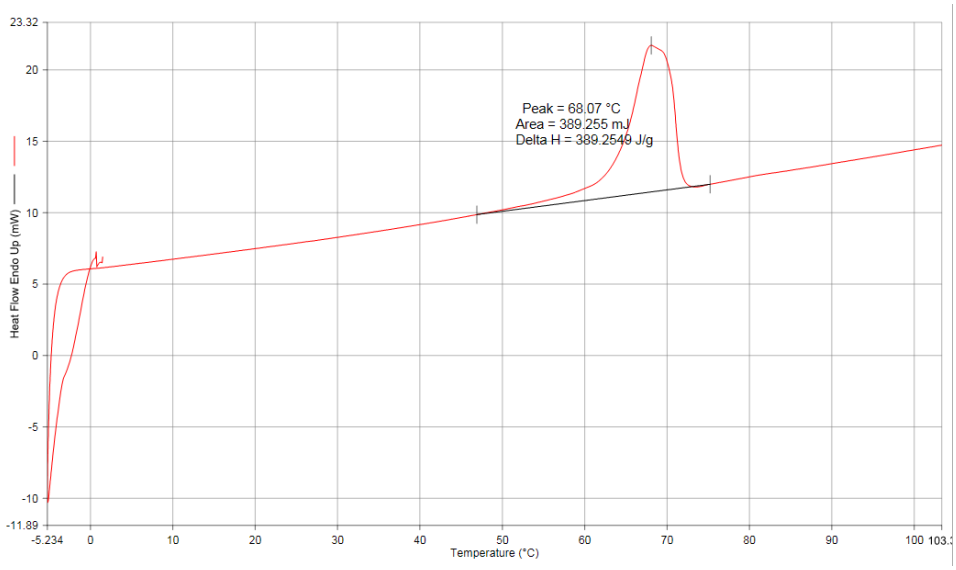


Figure 2(a). 1st heat

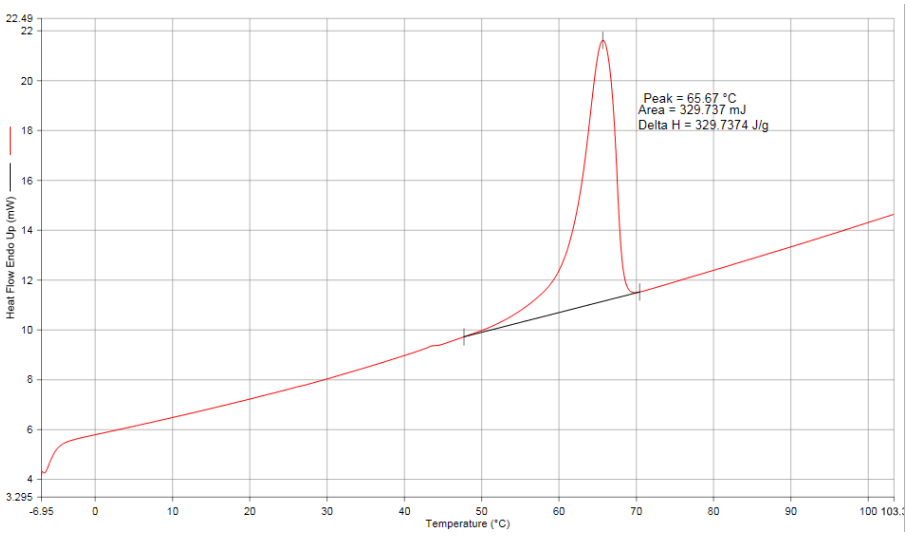


Figure 2(b). 2nd heat

Differential Scanning Calorimetry
Sample 3 - Process conditions – Unprocessed – Edge

1. Hold heat at 0°C
2. Heat sample to 75°C at 10°C/min
3. Hold at 75°C for 1 minute
4. Cool sample to 0°C at 10°C/min
5. Hold at 0°C for 1 minute
6. Heat sample to 75°C at 10°C/min

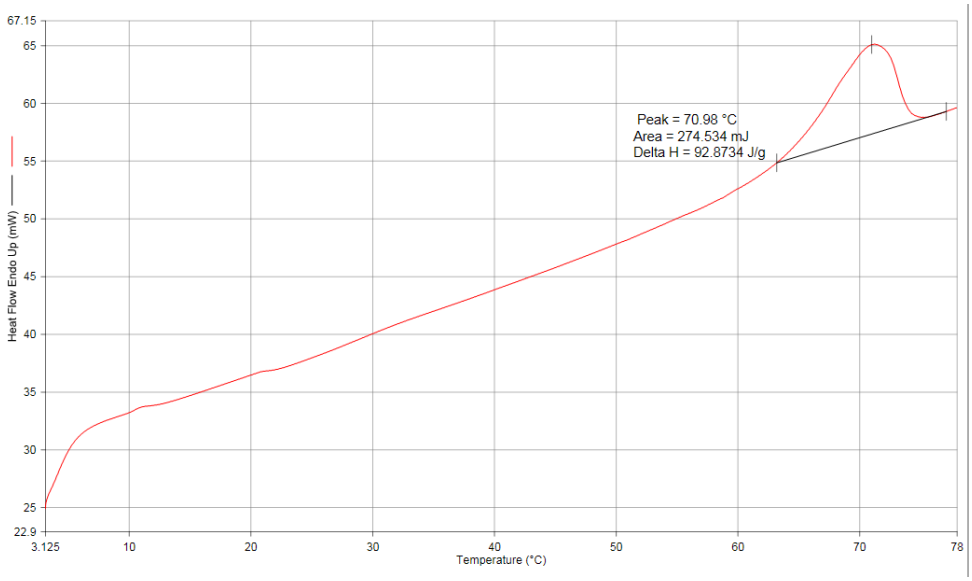


Figure 3(a). 1st heat

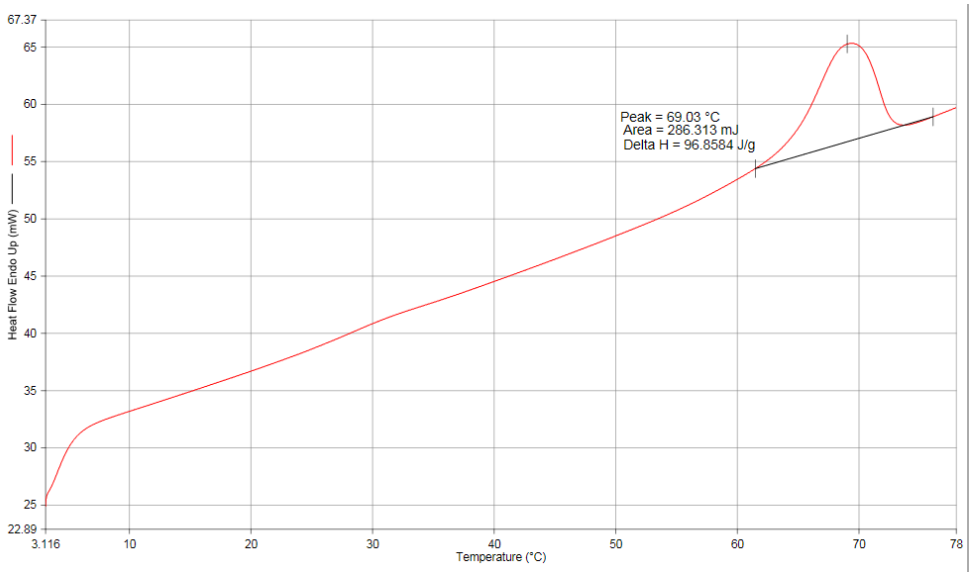


Figure 3(b). 2nd heat

Differential Scanning Calorimetry
Sample 4 - Process conditions – Unprocessed – Edge

1. Hold heat at 0°C
2. Heat sample to 70°C at 10°C/min
3. Hold at 70°C for 1 minute
4. Cool sample to 0°C at 10°C/min
5. Hold at 0°C for 1 minute
6. Heat sample to 70°C at 10°C/min

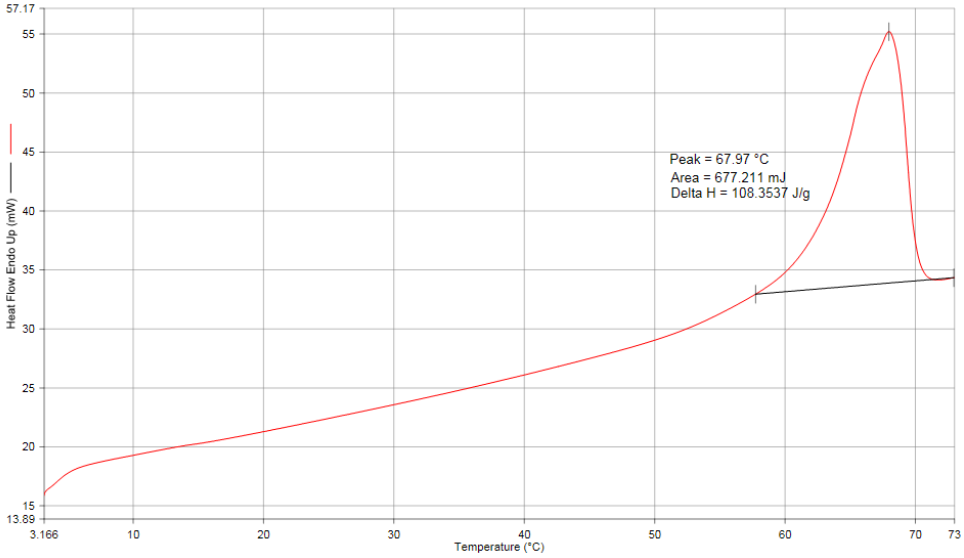


Figure 4(a). 1st heat

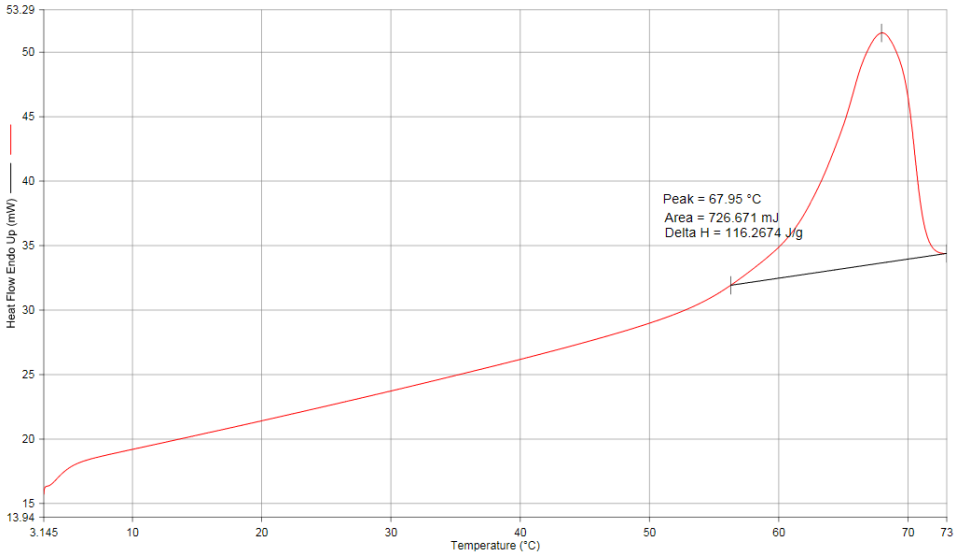


Figure 4(b). 2nd heat

Differential Scanning Calorimetry
Sample 5 - Process conditions - 55°C – 15 minutes – Edge

1. Hold heat at 0°C
2. Heat sample to 70°C at 10°C/min
3. Hold at 70°C for 1 minute
4. Cool sample to 0°C at 50°C/min
5. Hold at 0°C for 1 minute
6. Heat sample to 70°C at 10°C/min

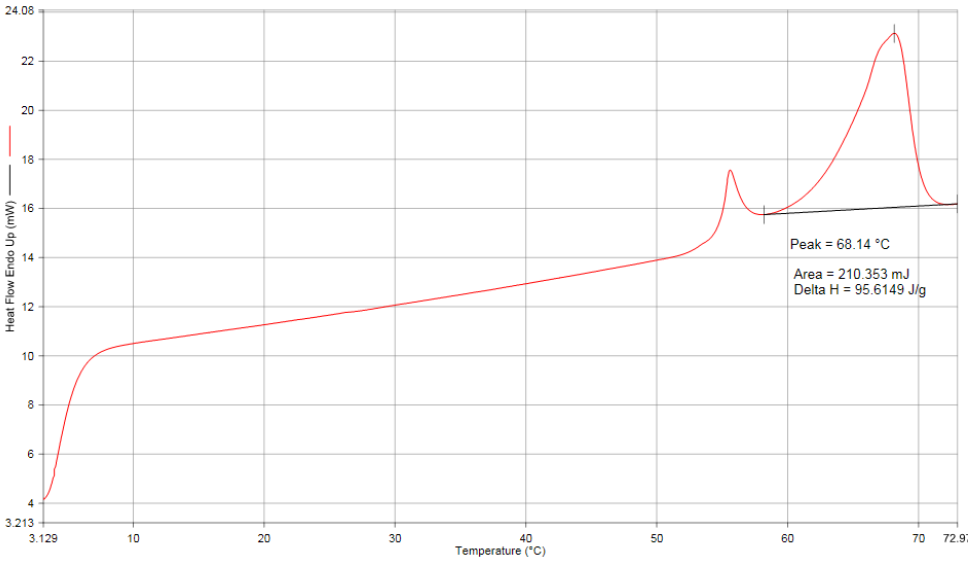


Figure 5(a). 1st heat

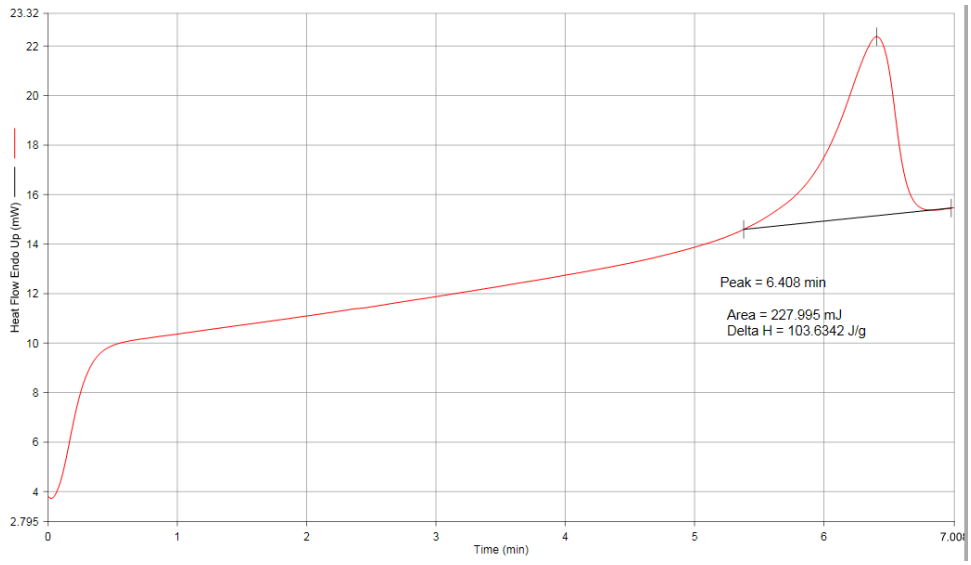


Figure 5(b). 2nd heat

Differential Scanning Calorimetry
Sample 6 - Process conditions - 55°C – 15 minutes – Edge

1. Hold heat at 0°C
2. Heat sample to 75°C at 10°C/min
3. Hold at 75°C for 1 minute
4. Cool sample to 0°C at 10°C/min
5. Hold at 0°C for 1 minute
6. Heat sample to 75°C at 10°C/min

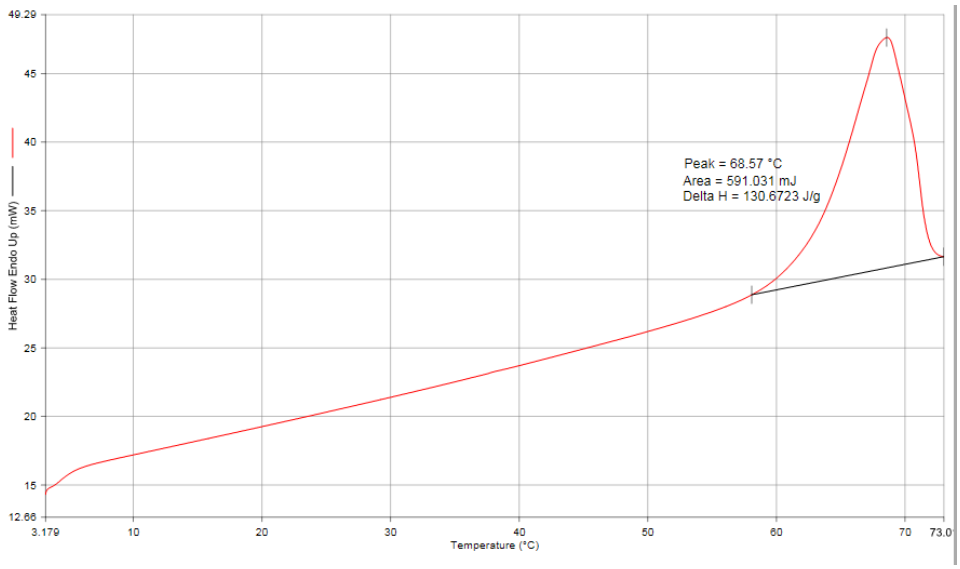


Figure 6(a). 1st heat

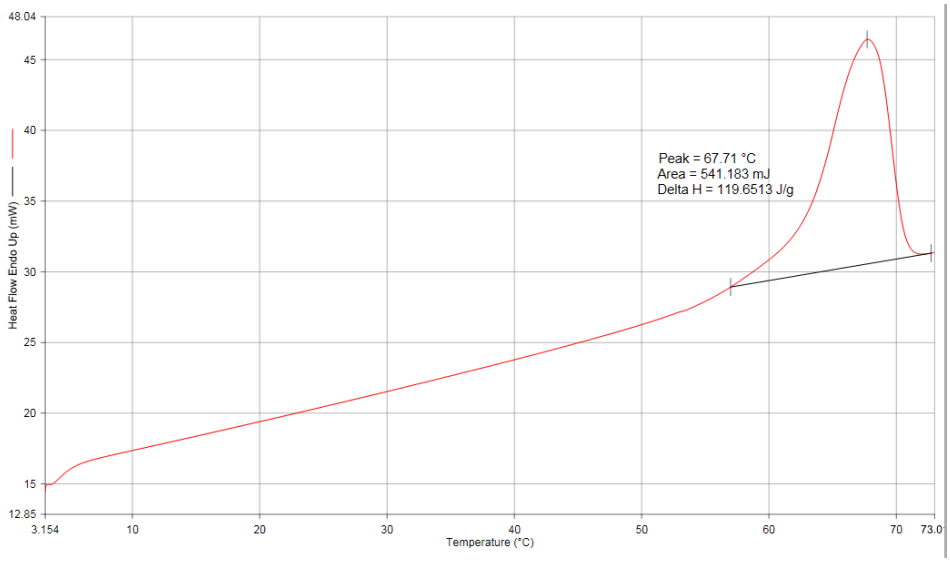


Figure 6(b). 2nd heat

Differential Scanning Calorimetry
Sample 7 - Process conditions - 62°C – 15 minutes – Edge

1. Hold heat at 0°C
2. Heat sample to 70°C at 1°C/min
3. Hold at 70°C for 1 minute
4. Cool sample to 0°C at 10°C/min
5. Hold at 0°C for 1 minute
6. Heat sample to 70°C at 10°C/min

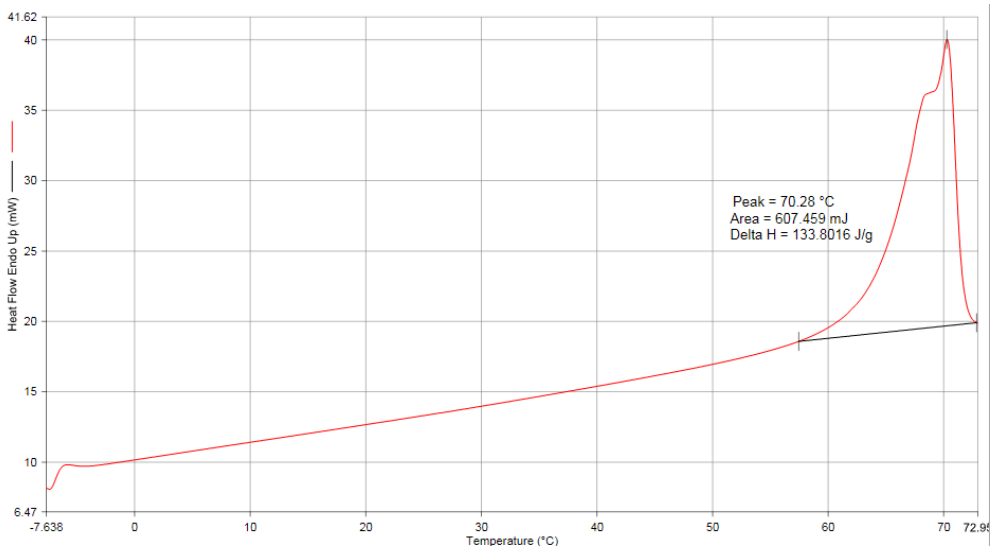


Figure 7(a). 1st heat

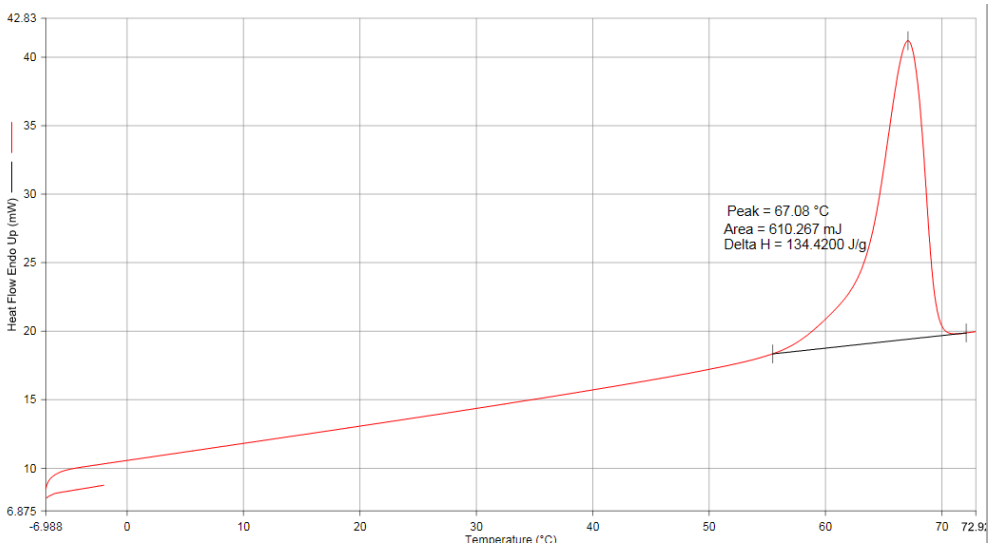


Figure 7(b). 2nd heat

Differential Scanning Calorimetry
Sample 7 - Process conditions - 62°C – 15 minutes – Middle

1. Hold heat at 0°C
2. Heat sample to 70°C at 1°C/min
3. Hold at 70°C for 1 minute
4. Cool sample to 0°C at 10°C/min
5. Hold at 0°C for 1 minute
6. Heat sample to 70°C at 10°C/min

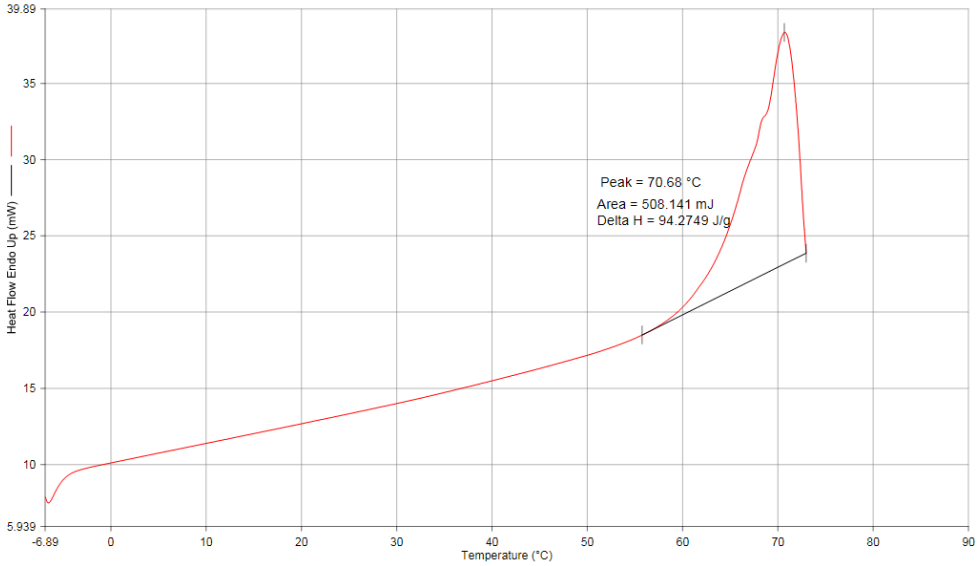


Figure 7(c). 1st heat

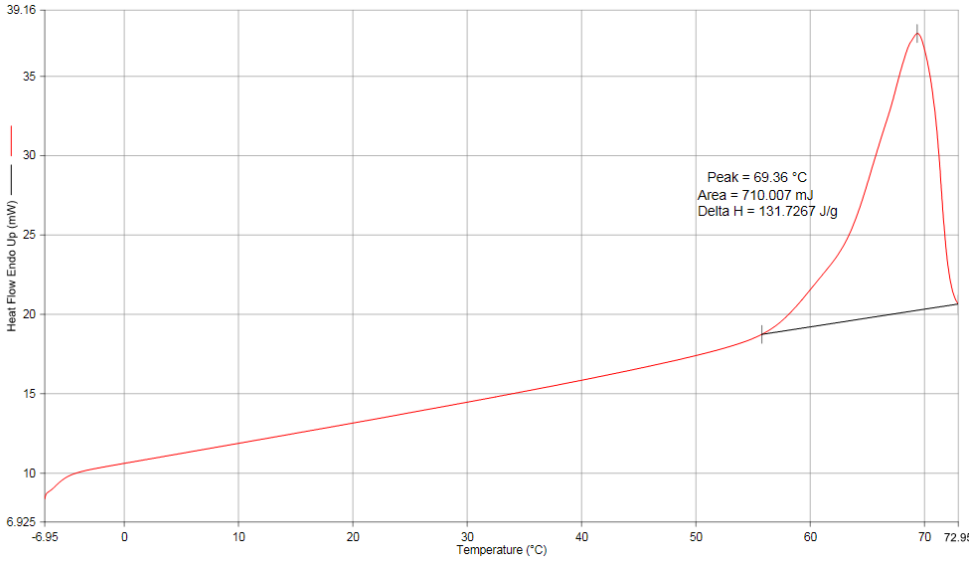


Figure 7(d). 2nd heat

Differential Scanning Calorimetry
Sample 8 - Process conditions - 55°C – 15 minutes – Edge

1. Hold heat at 0°C
2. Heat sample to 70°C at 1°C/min
3. Hold at 70°C for 1 minute
4. Cool sample to 0°C at 10°C/min
5. Hold at 0°C for 1 minute
6. Heat sample to 70°C at 10°C/min

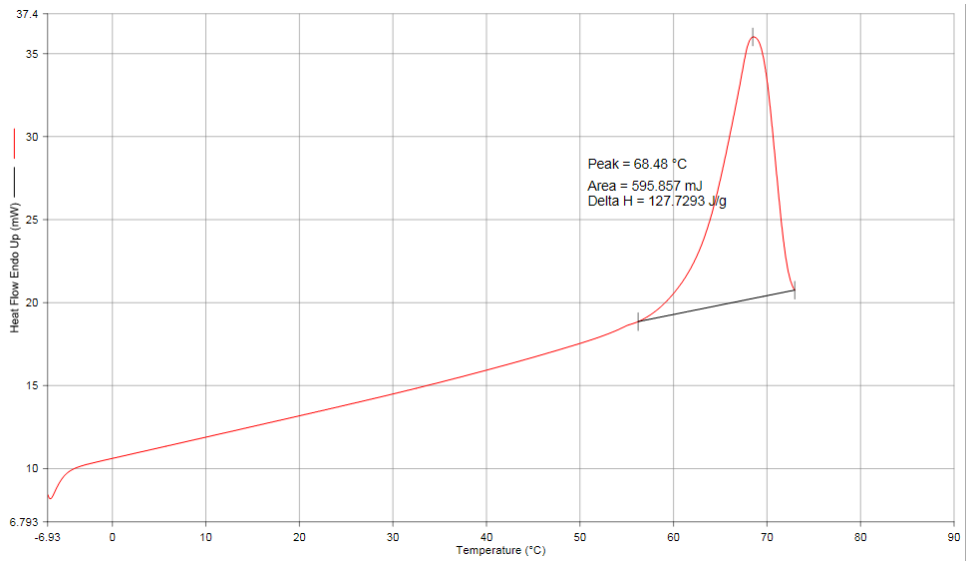


Figure 8(a). 1st heat

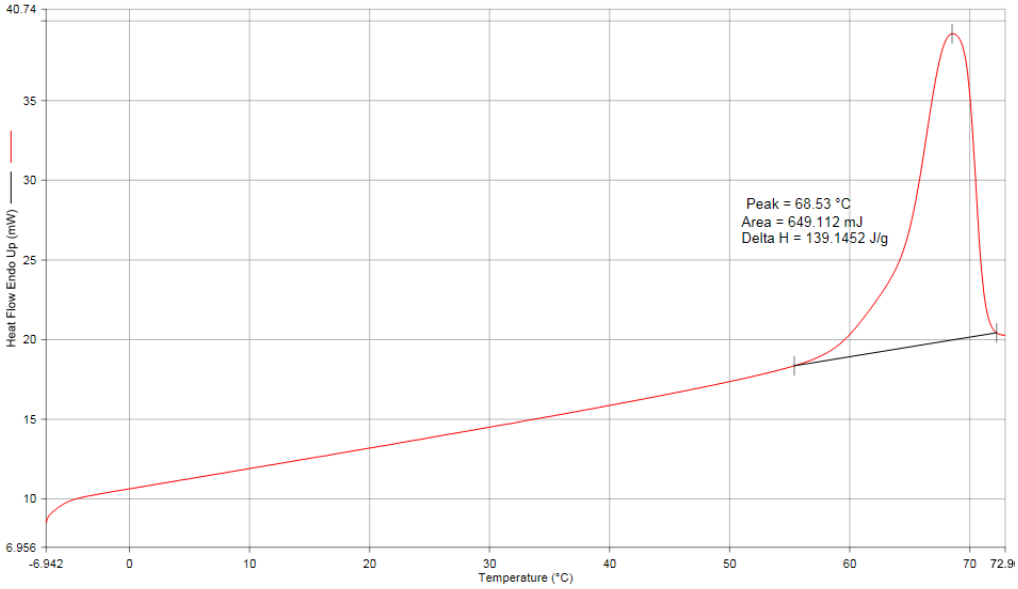


Figure 8(b). 2nd heat

Differential Scanning Calorimetry
Sample 9 - Process conditions - 55°C – 15 minutes – Edge

1. Hold heat at 0°C
2. Heat sample to 70°C at 100°C/min
3. Hold at 70°C for 1 minute
4. Cool sample to 0°C at 10°C/min
5. Hold at 0°C for 1 minute
6. Heat sample to 100°C at 10°C/min

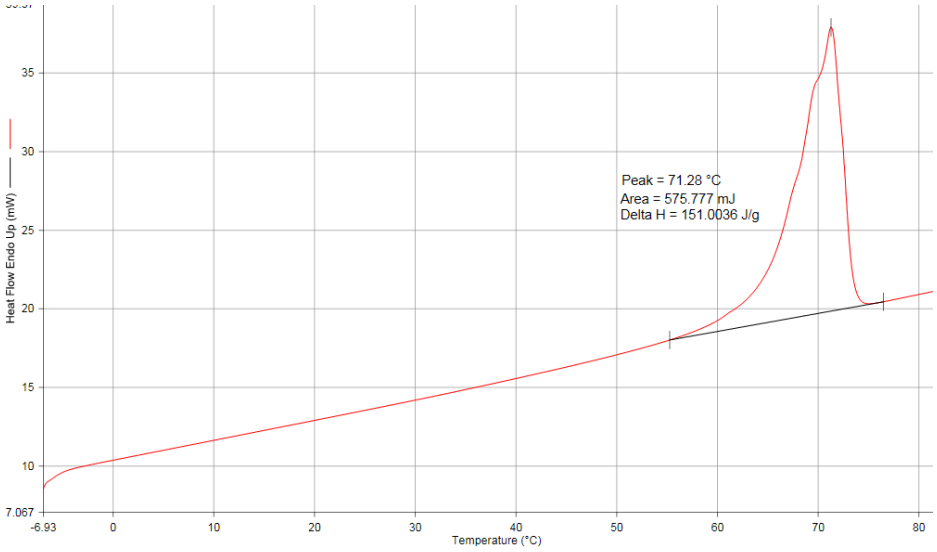


Figure 9(a). 1st heat

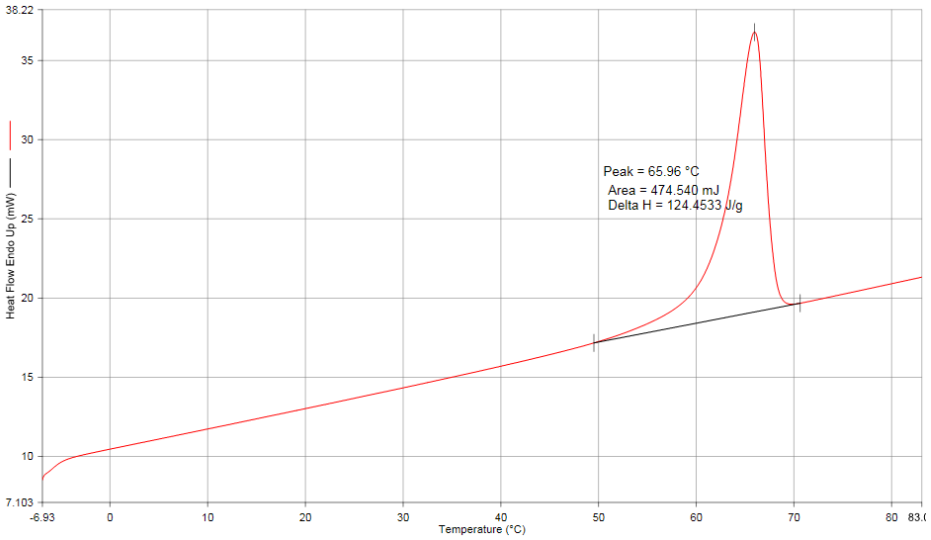


Figure 9(b). 2nd heat

Differential Scanning Calorimetry
Sample 9 - Process conditions - 55°C – 15 minutes – Middle

1. Hold heat at 0°C
2. Heat sample to 70°C at 100°C/min
3. Hold at 70°C for 1 minute
4. Cool sample to 0°C at 10°C/min
5. Hold at 0°C for 1 minute
6. Heat sample to 70°C at 100°C/min

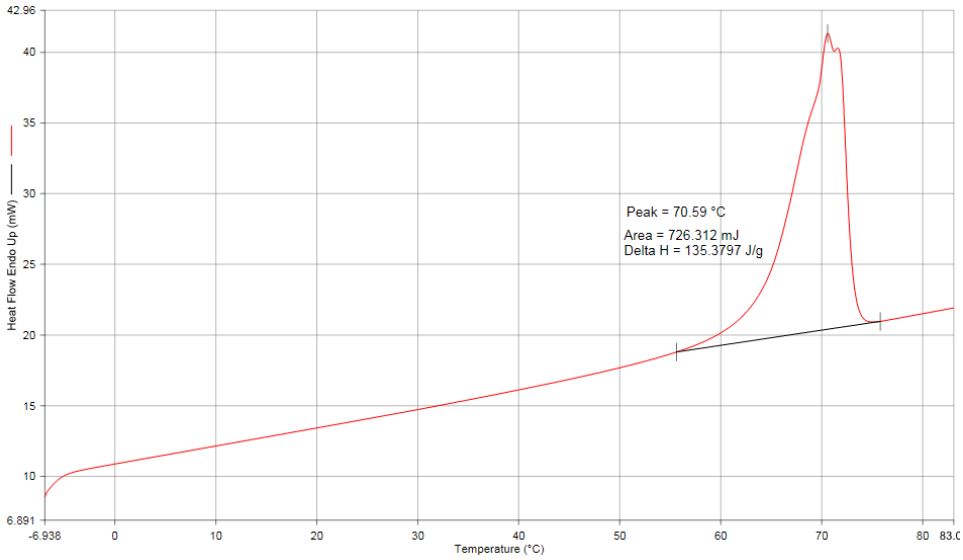


Figure 9(c). 1st heat

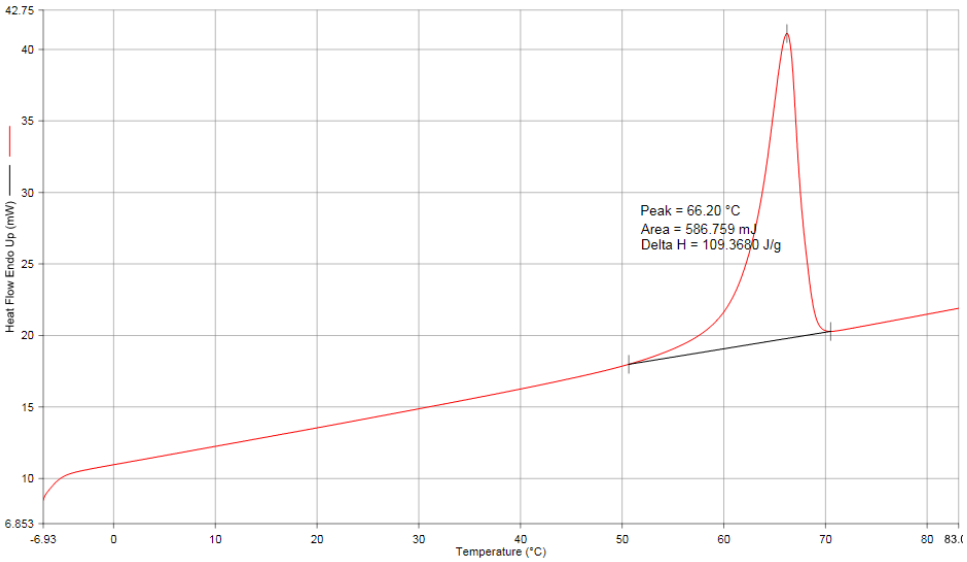


Figure 9(d). 2nd heat

Differential Scanning Calorimetry
Sample 9 - Process conditions - 55°C – 15 minutes – Edge

1. Hold heat at 0°C
2. Heat sample to 70°C at 1°C/min
3. Hold at 70°C for 1 minute
4. Cool sample to 0°C at 10°C/min
5. Hold at 0°C for 1 minute
6. Heat sample to 70°C at 1°C/min

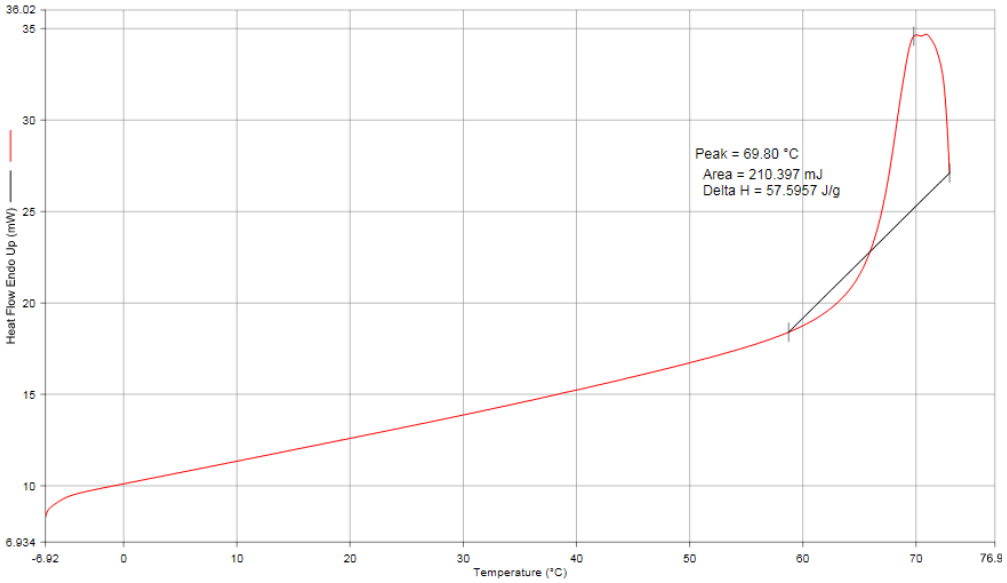


Figure 9(e). 1st heat

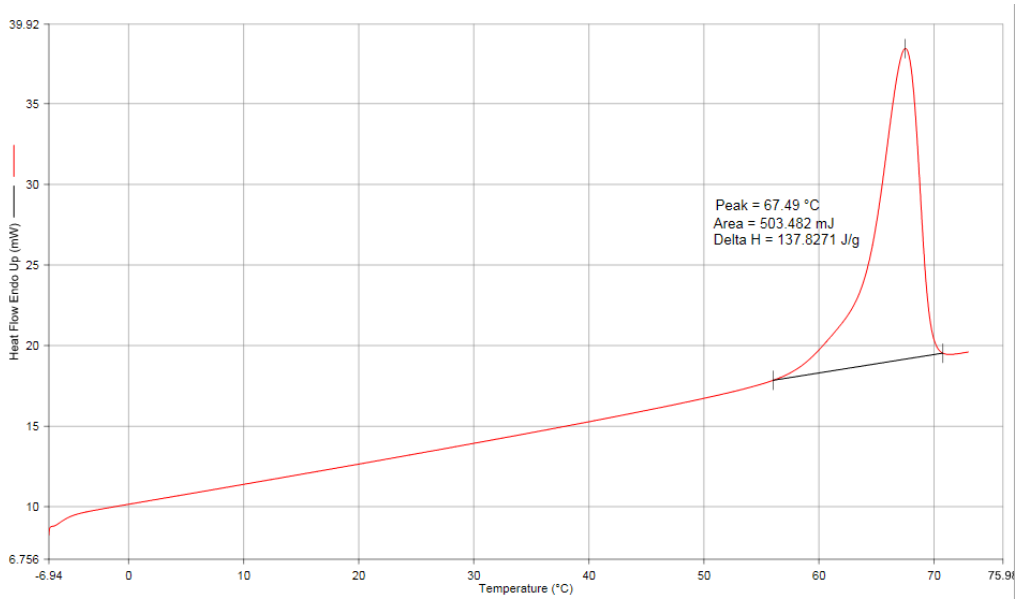


Figure 9(f). 2nd heat

Differential Scanning Calorimetry
Sample 9 - Process conditions - 55°C – 15 minutes – Middle

1. Hold heat at 0°C
2. Heat sample to 70°C at 1°C/min
3. Hold at 70°C for 1 minute
4. Cool sample to 0°C at 10°C/min
5. Hold at 0°C for 1 minute
6. Heat sample to 70°C at 1°C/min

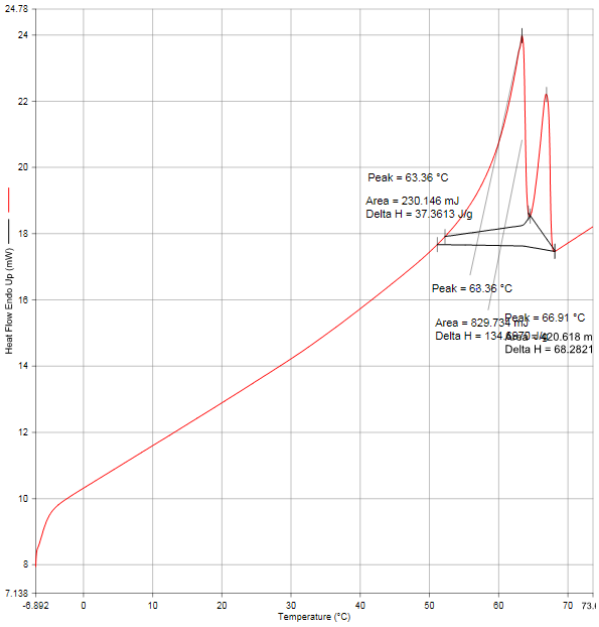


Figure 9(g). 1st heat

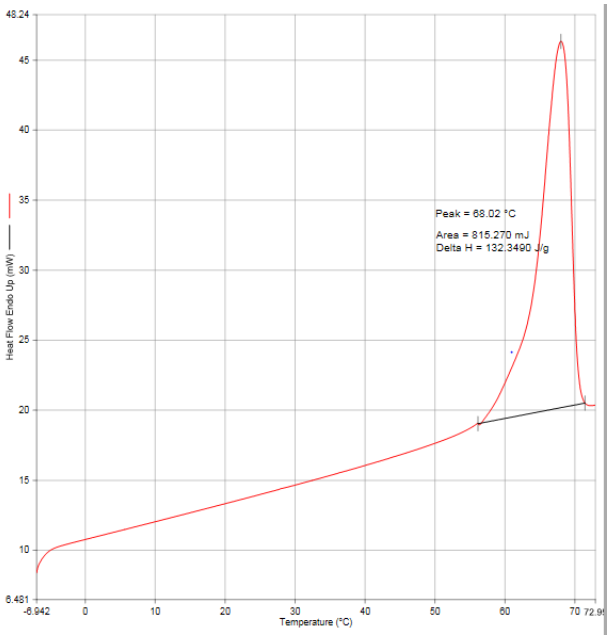


Figure 9(h). 2nd heat

Differential Scanning Calorimetry
Sample 10 - Process conditions - 62°C – 15 minutes – Edge

1. Hold heat at -10°C
2. Heat sample to 80°C at 10°C/min
3. Hold at 80°C for 1 minute
4. Cool sample to -10°C at 100°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 80°C at 10°C/min

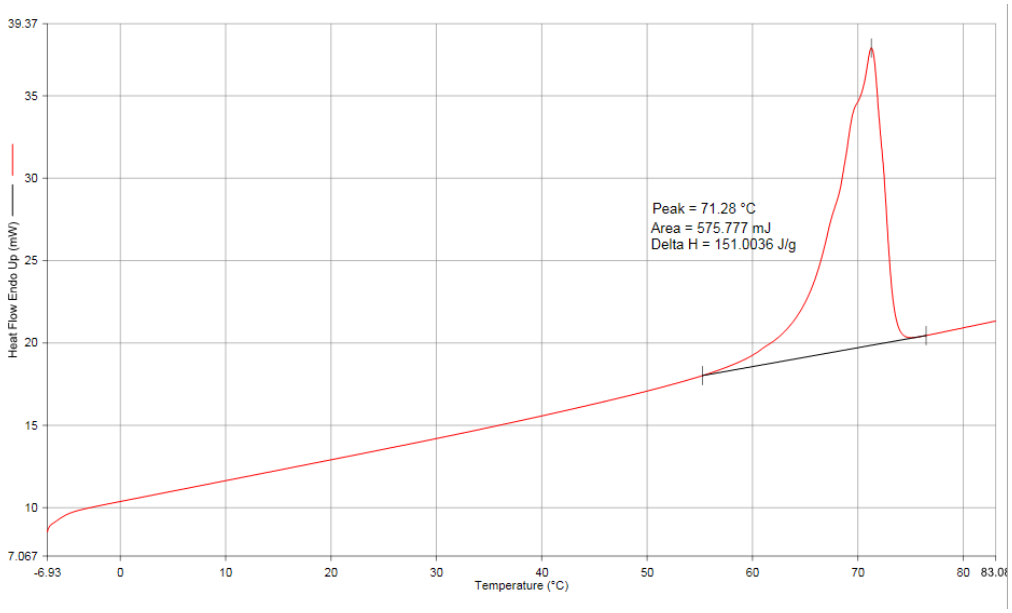


Figure 10(a). 1st heat

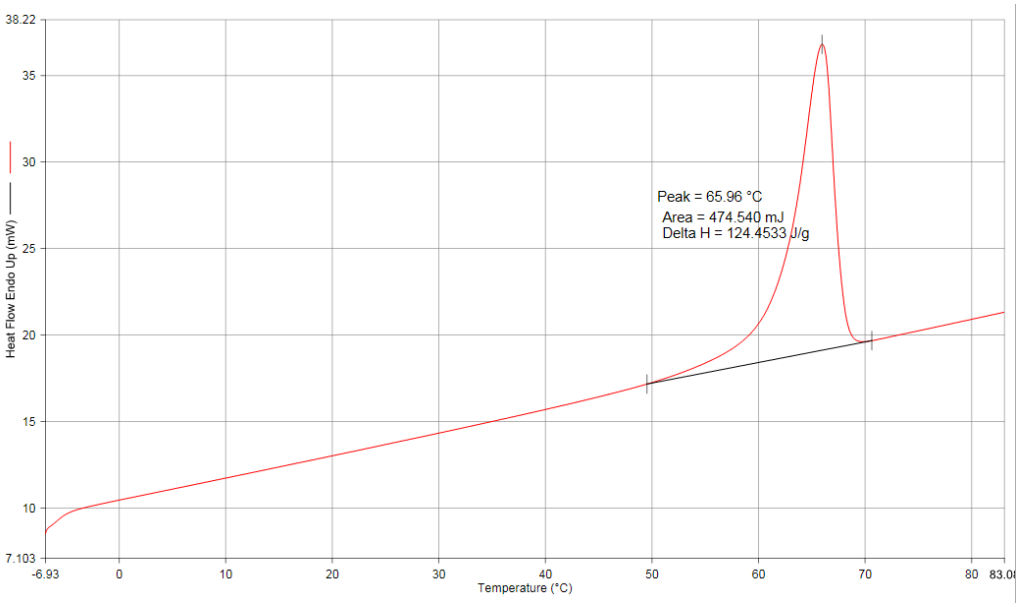


Figure 10(b). 2nd heat

Differential Scanning Calorimetry
Sample 10 - Process conditions - 62°C – 15 minutes – Middle

1. Hold heat at -10°C
2. Heat sample to 80°C at 10°C/min
3. Hold at 80°C for 1 minute
4. Cool sample to -10°C at 100°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 80°C at 10°C/min

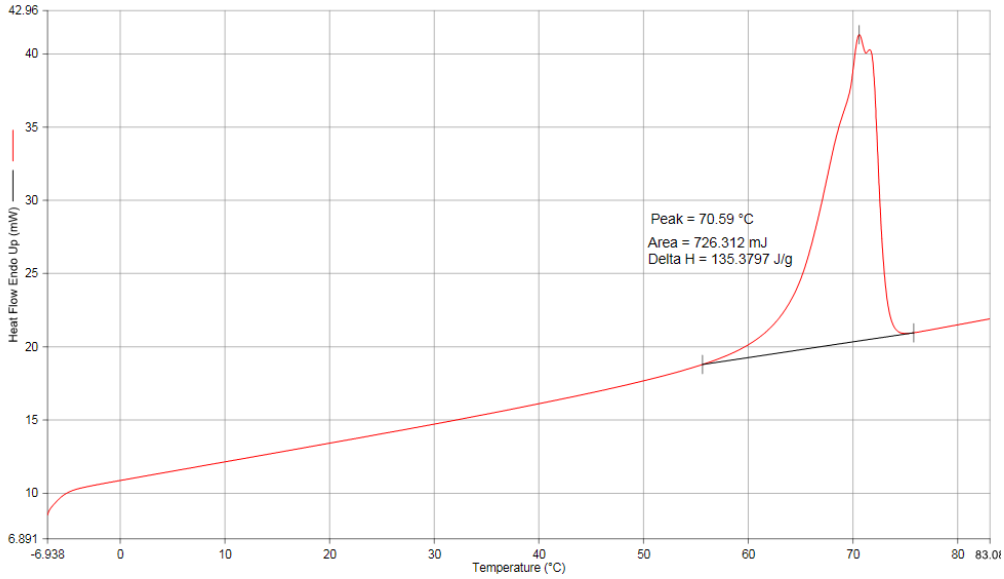


Figure 10(c). 1st heat

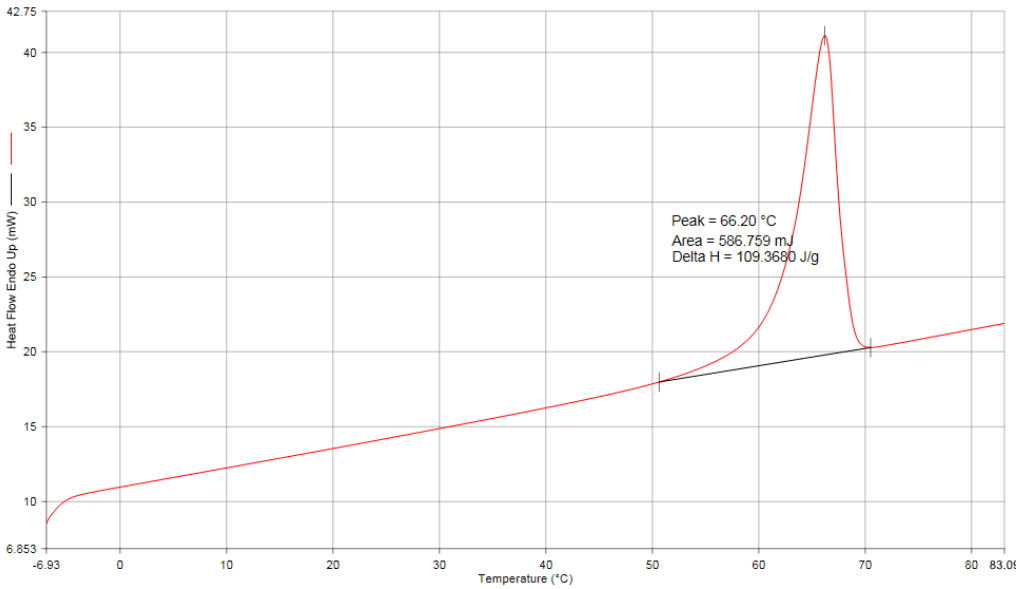


Figure 10(d). 2nd heat

Differential Scanning Calorimetry
Sample 11 - Process conditions - 62°C – 15 minutes – Edge

1. Hold heat at -10°C
2. Heat sample to 80°C at 10°C/min
3. Hold at 80°C for 1 minute
4. Cool sample to -10°C at 1°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 80°C at 10°C/min

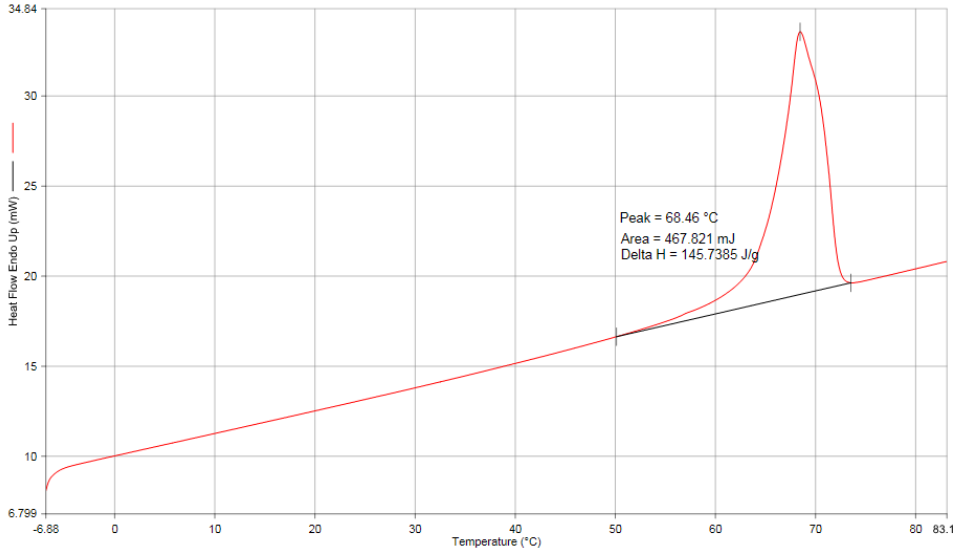


Figure 11(a). 1st heat

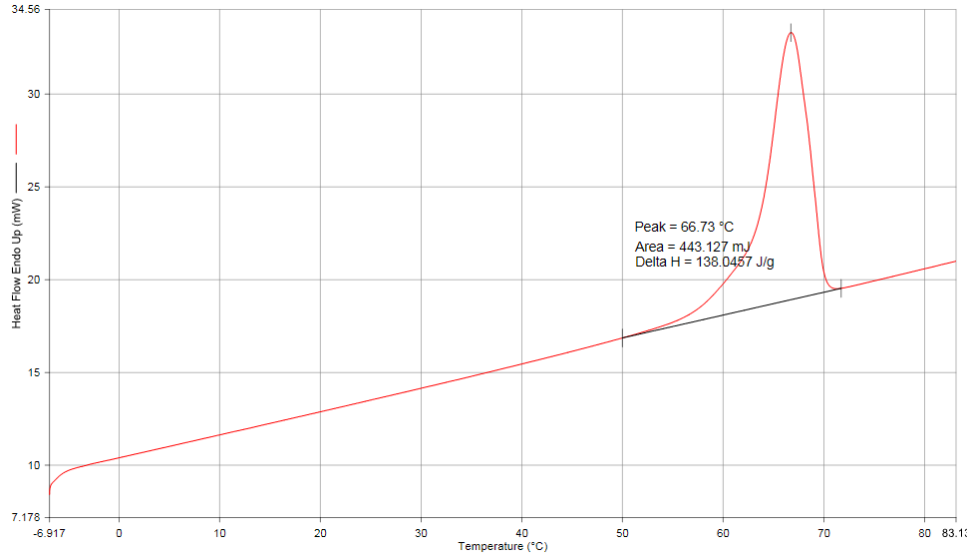


Figure 11(b). 2nd heat

Differential Scanning Calorimetry
Sample 11 - Process conditions - 62°C – 15 minutes – Middle

1. Hold heat at -10°C
2. Heat sample to 80°C at 10°C/min
3. Hold at 80°C for 1 minute
4. Cool sample to -10°C at 1°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 80°C at 10°C/min

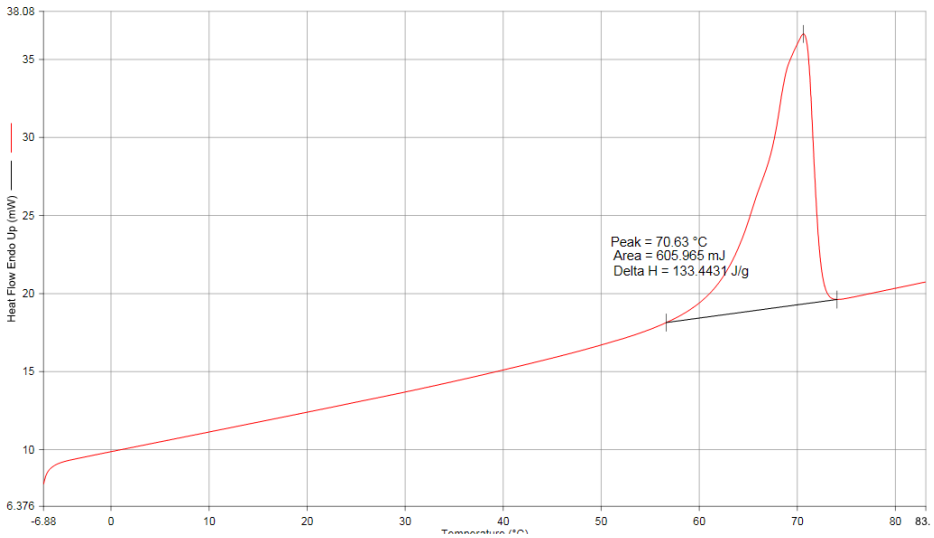


Figure 17(c). 1st heat

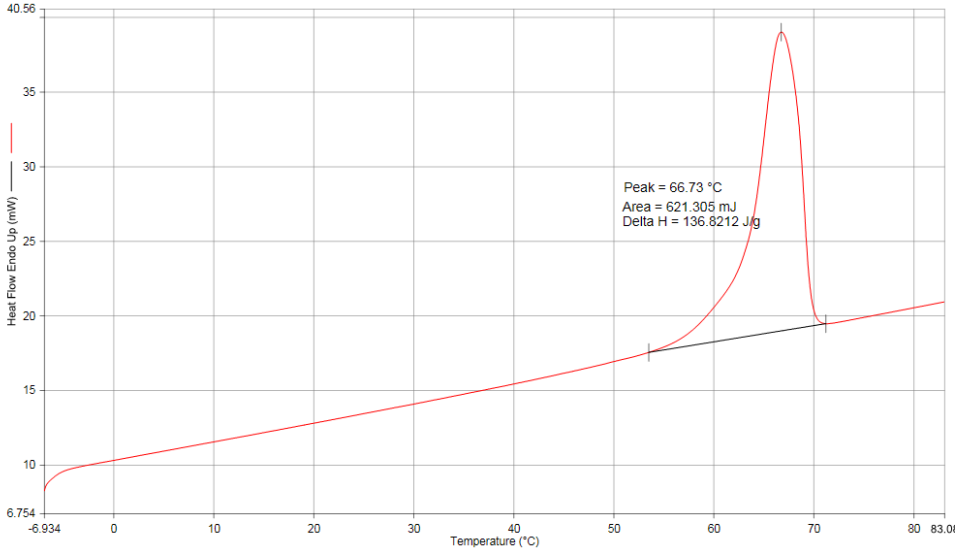


Figure 17(d). 2nd heat

Differential Scanning Calorimetry
Sample 12 - Process conditions - 62°C – 15 minutes – Edge

1. Hold heat at -10°C
2. Heat sample to 80°C at 10°C/min
3. Hold at 80°C for 1 minute
4. Cool sample to -10°C at 100°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 80°C at 10°C/min

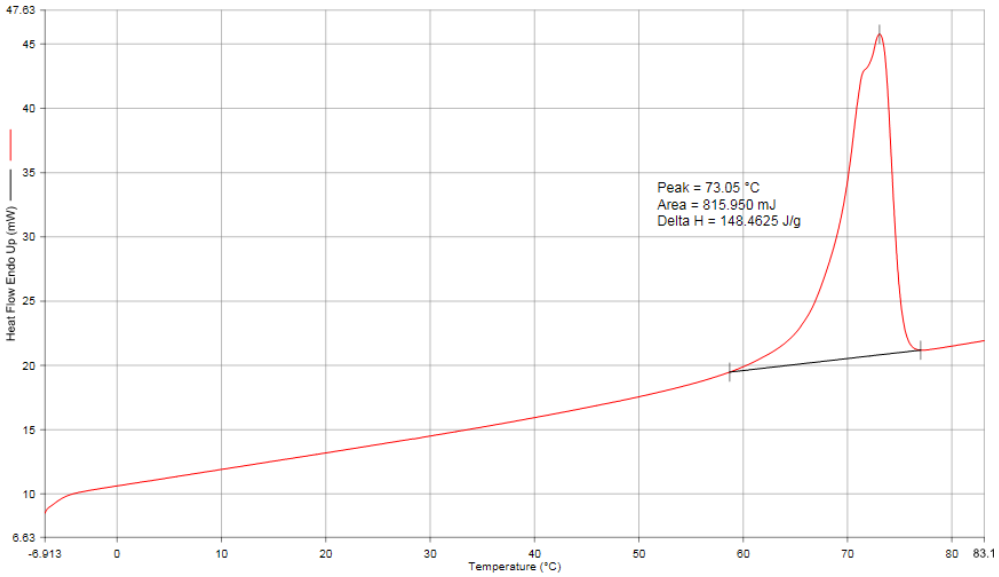


Figure 12(a). 1st heat

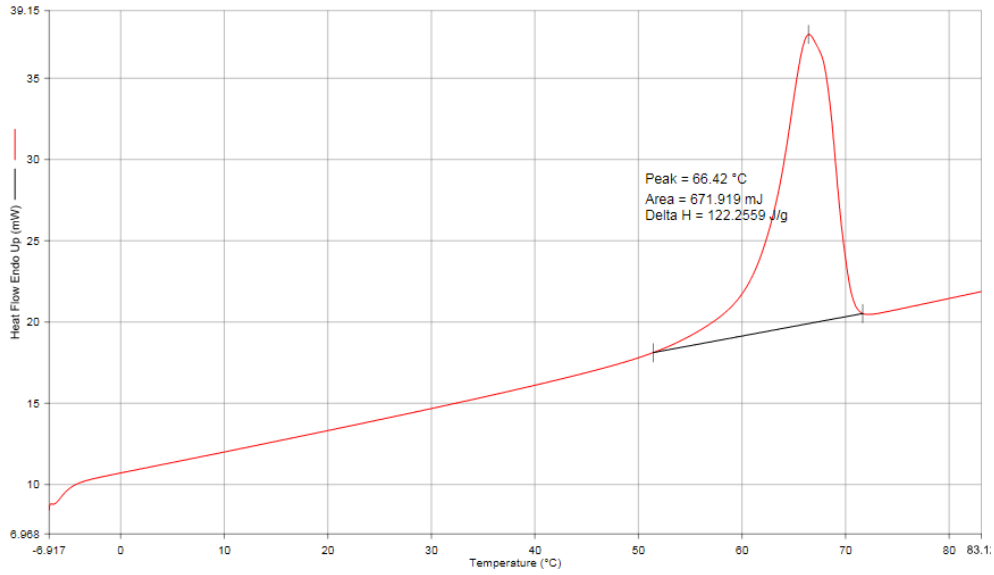


Figure 12(b). 2nd heat

Differential Scanning Calorimetry
Sample 12 - Process conditions - 62°C – 15 minutes – Middle

1. Hold heat at -10°C
2. Heat sample to 80°C at 10°C/min
3. Hold at 80°C for 1 minute
4. Cool sample to -10°C at 100°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 80°C at 10°C/min

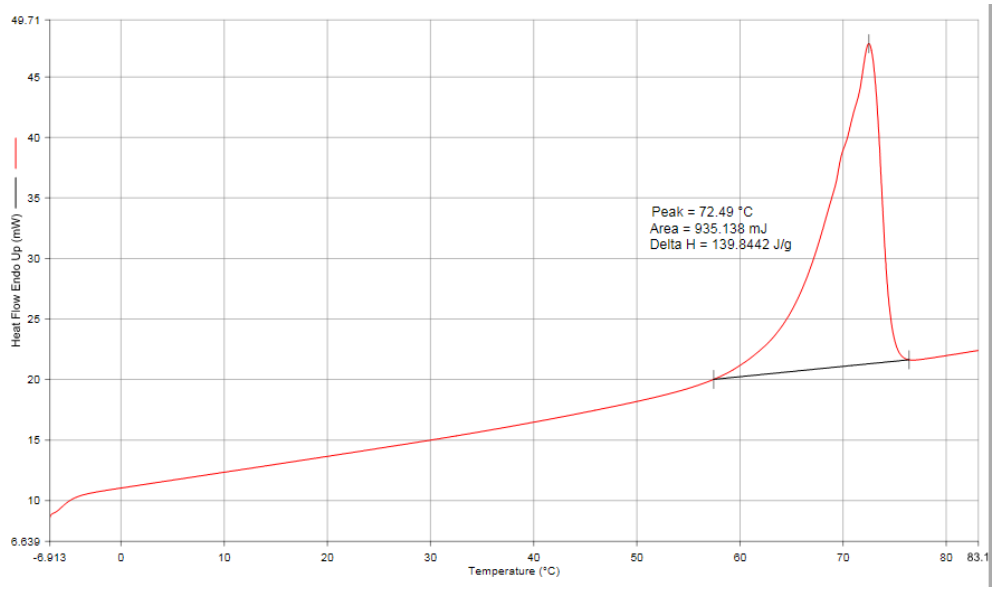


Figure 12(c). 1st heat

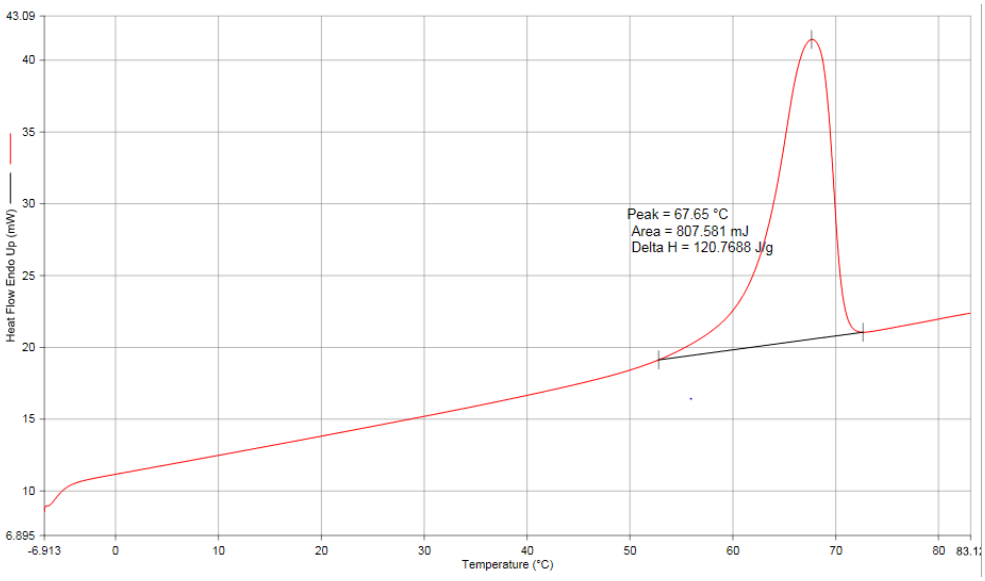


Figure 12(c). 2nd heat

Differential Scanning Calorimetry
Sample 13 - Process conditions - 62°C – 15 minutes – Edge

1. Hold heat at -10°C
2. Heat sample to 80°C at 10°C/min
3. Hold at 80°C for 1 minute
4. Cool sample to -10°C at 1°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 80°C at 10°C/min

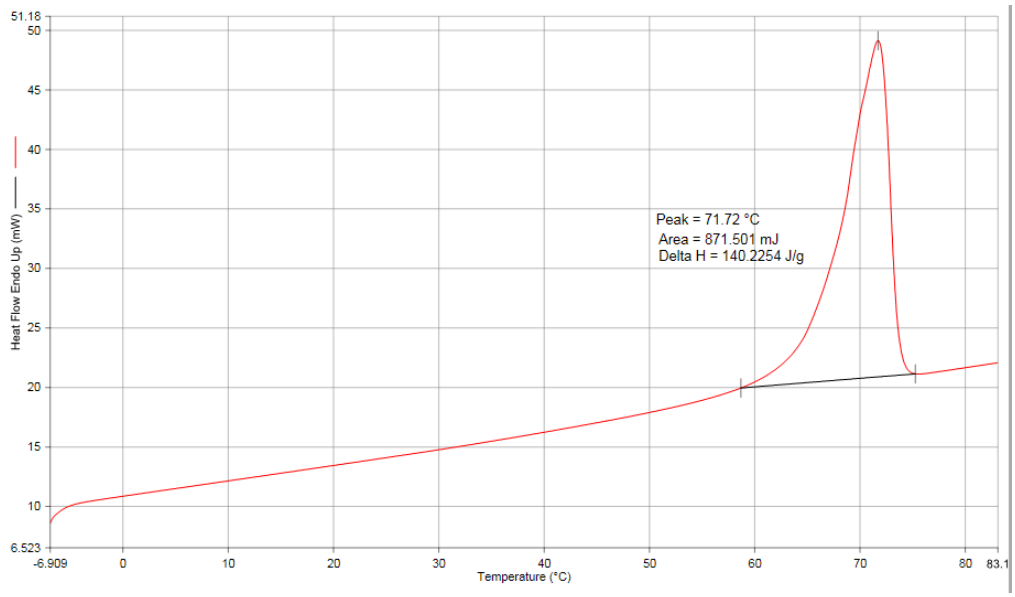


Figure 13(a). 1st heat

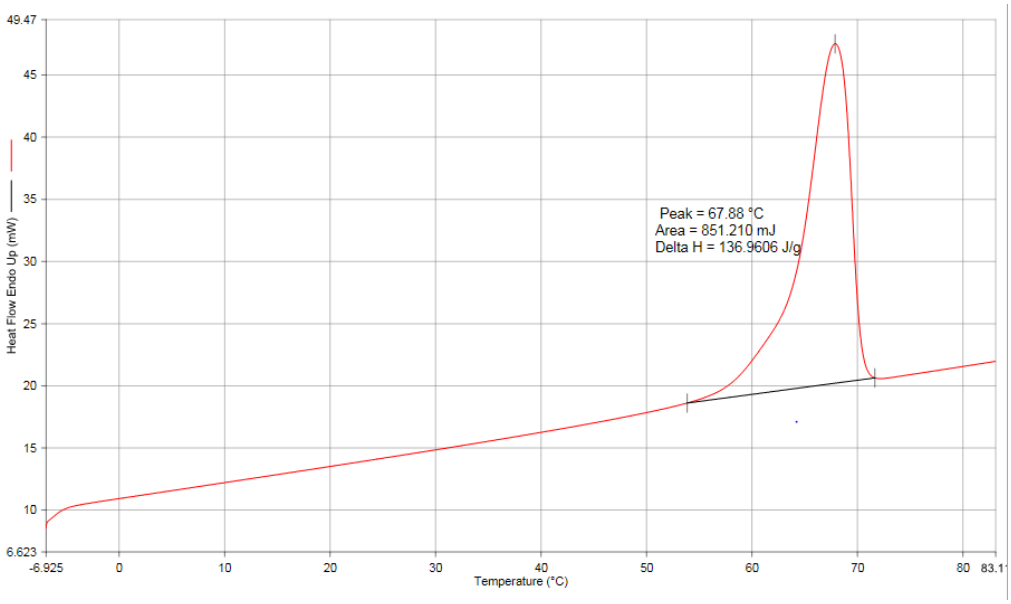


Figure 13(b). 2nd heat

Differential Scanning Calorimetry
Sample 13 - Process conditions - 62°C – 15 minutes – Middle

1. Hold heat at -10°C
2. Heat sample to 80°C at 10°C/min
3. Hold at 80°C for 1 minute
4. Cool sample to -10°C at 1°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 80°C at 10°C/min

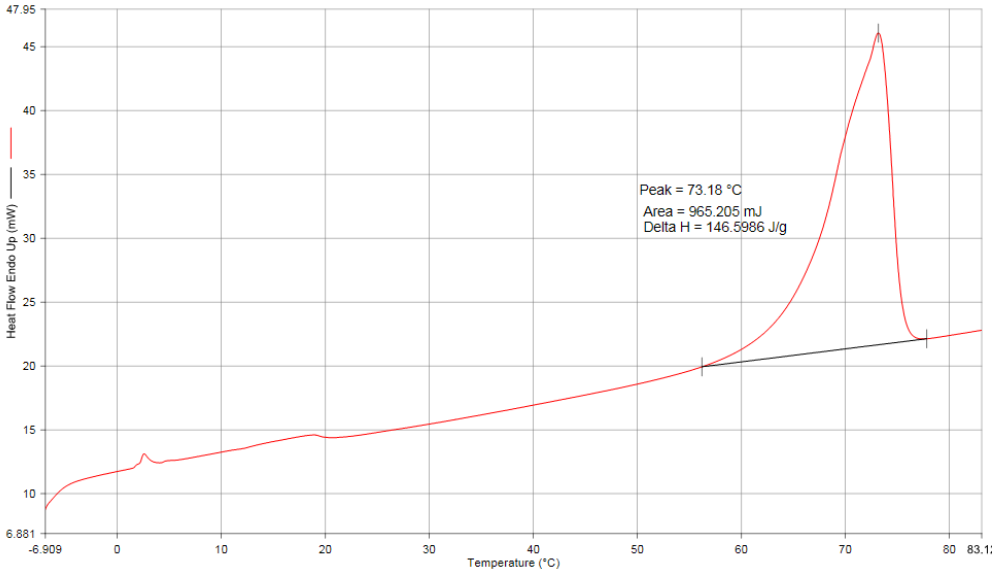


Figure 13(c). 1st heat

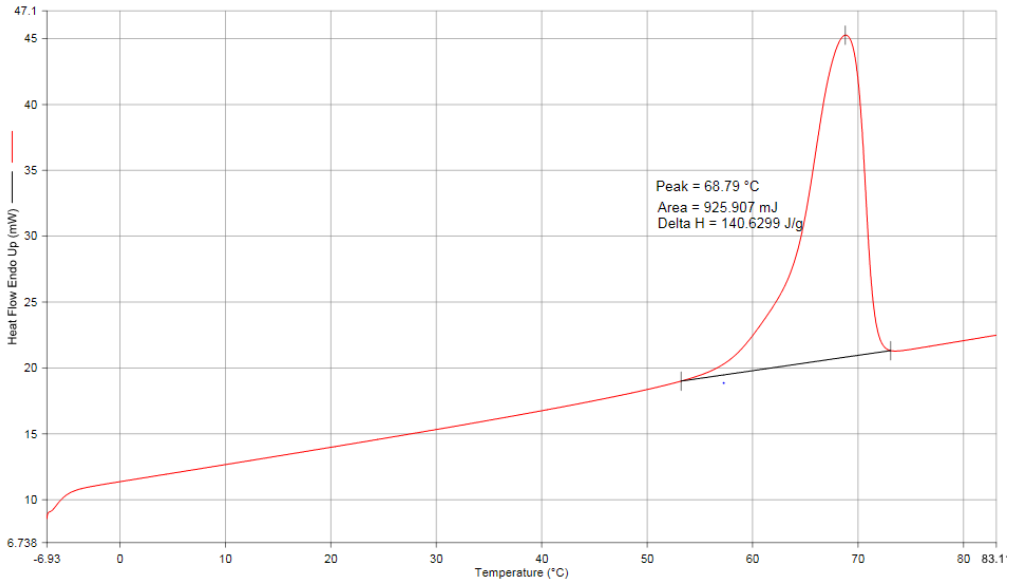


Figure 13(c). 2nd heat

Differential Scanning Calorimetry
Sample 14 - Process conditions – RT(25-30)°C – 15 minutes – Edge

7. Hold heat at -10°C
8. Heat sample to 70°C at 10°C/min
9. Hold at 70°C for 1 minute
10. Cool sample to -10°C at 100°C/min
11. Hold at -10°C for 1 minute
12. Heat sample to 70°C at 10°C/min

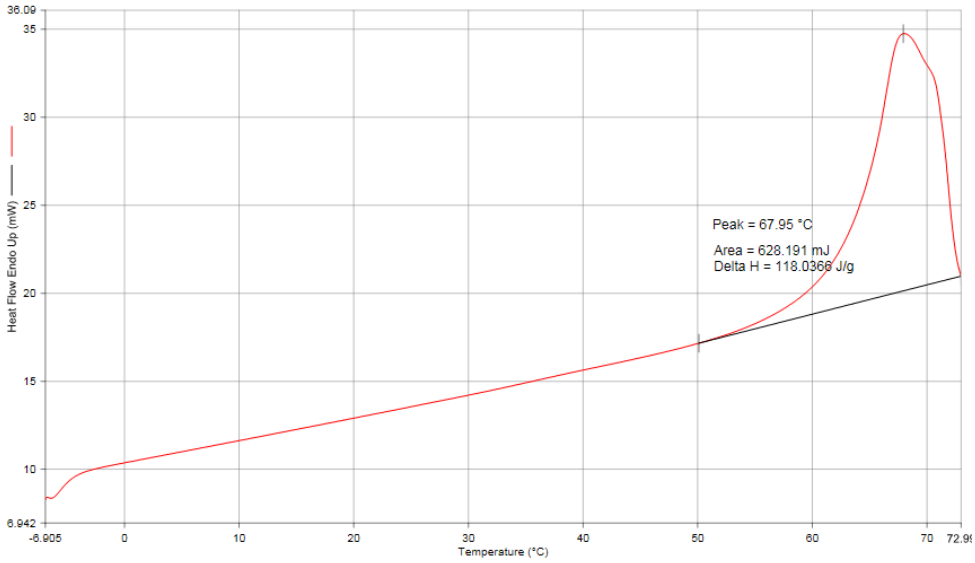


Figure 14(a). 1st heat

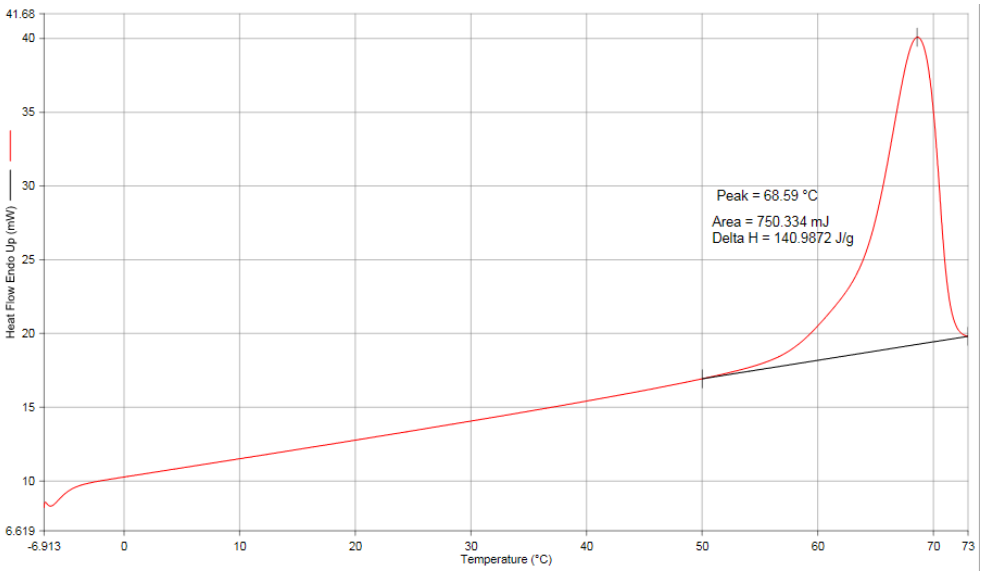


Figure 14(b). 2nd heat

Differential Scanning Calorimetry
Sample 14 - Process conditions – RT(25-30)°C – 15 minutes – Edge

7. Hold heat at -10°C
8. Heat sample to 70°C at 10°C/min
9. Hold at 80°C for 1 minute
10. Cool sample to -10°C at 1°C/min
11. Hold at -10°C for 1 minute
12. Heat sample to 70°C at 10°C/min

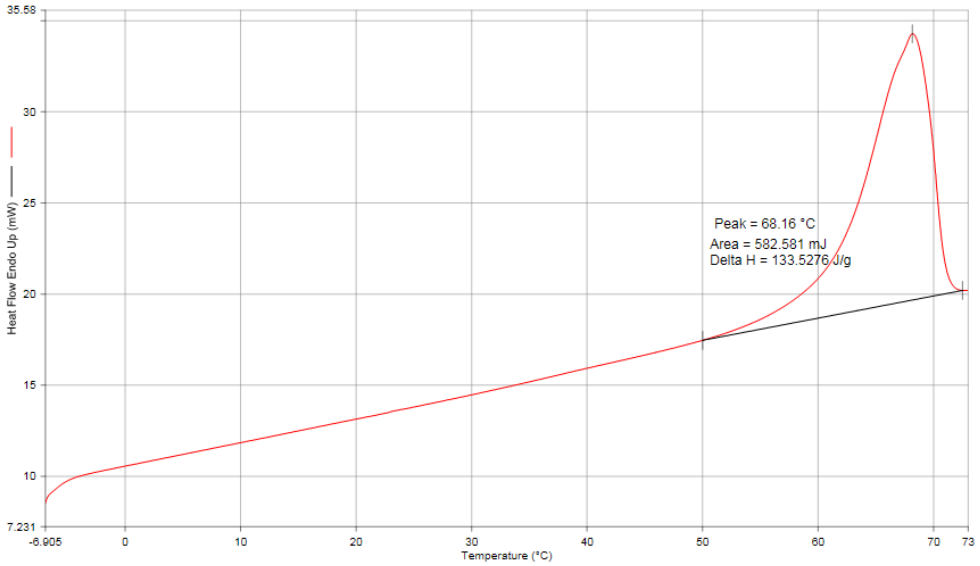


Figure 14(c). 1st heat

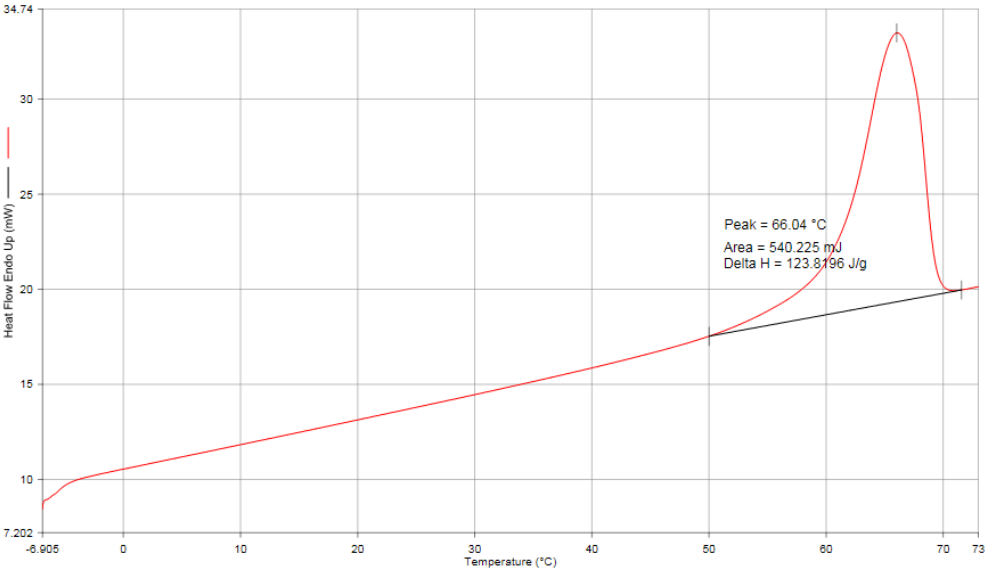


Figure 14(d). 2nd heat

Differential Scanning Calorimetry
Sample 15 - Process conditions – RT(25-30)°C – 15 minutes – Edge

13. Hold heat at -10°C
14. Heat sample to 80°C at 10°C/min
15. Hold at 80°C for 1 minute
16. Cool sample to -10°C at 10°C/min
17. Hold at -10°C for 1 minute
18. Heat sample to 80°C at 10°C/min

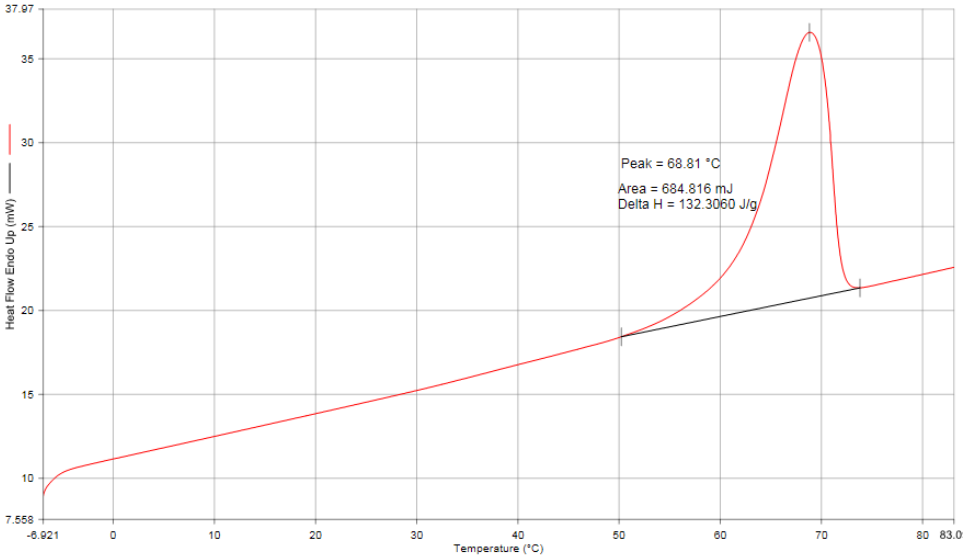


Figure 15(a). 1st heat

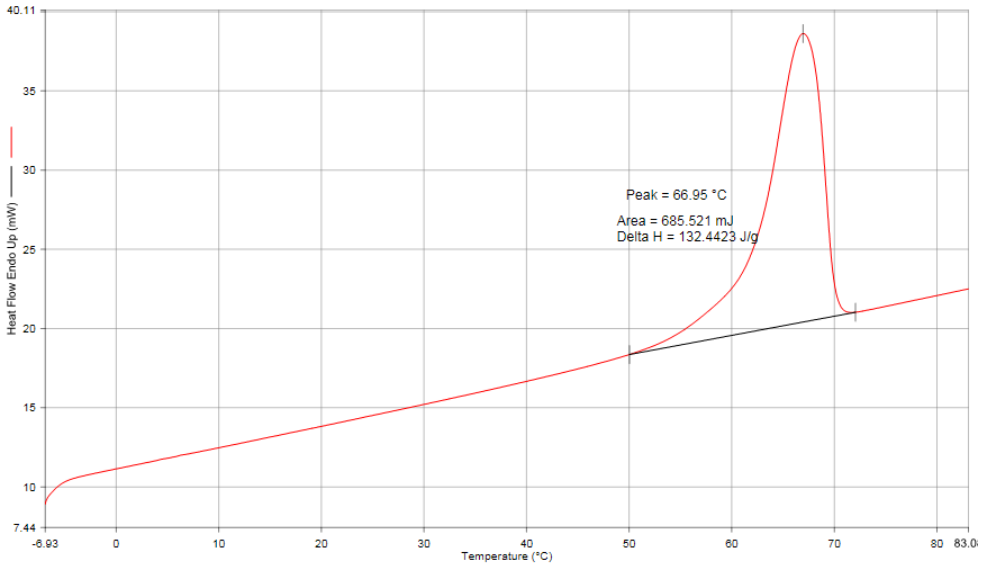


Figure 15(b). 2nd heat

Differential Scanning Calorimetry
Sample 16 - Process conditions – RT(25-30)°C – 30 seconds – Edge

1. Hold heat at -10°C
2. Heat sample to 75°C at 10°C/min
3. Hold at 75°C for 1 minute
4. Cool sample to -10°C at 10°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 75°C at 10°C/min

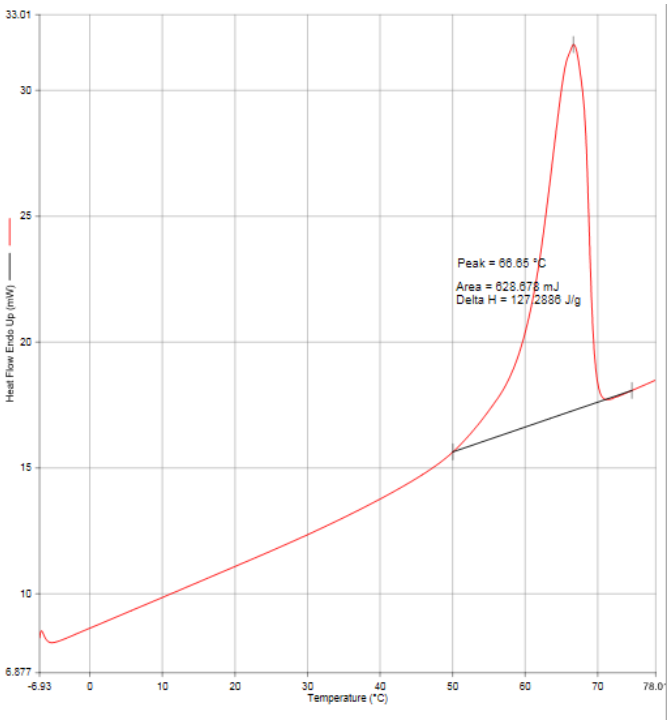


Figure 16(a). 1st heat

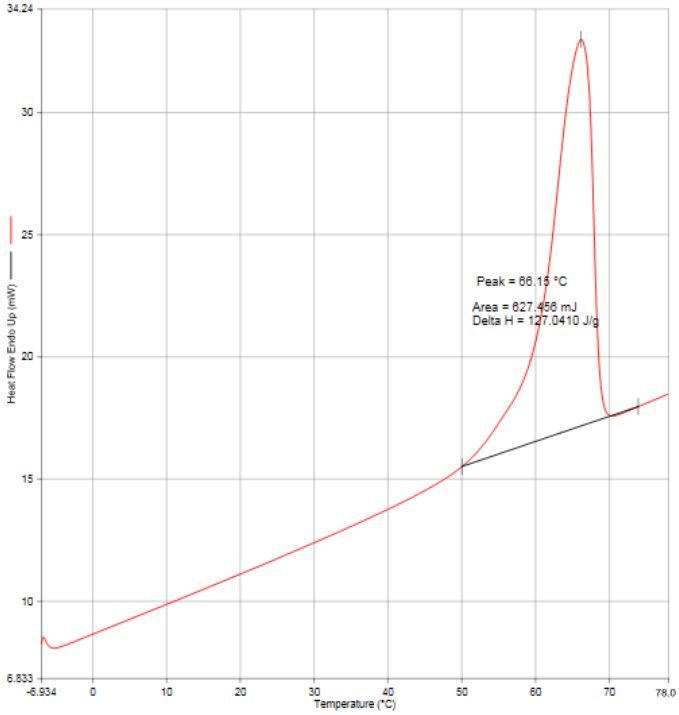


Figure 16(b). 2nd heat

Differential Scanning Calorimetry
Sample 16 - Process conditions – RT(25-30)°C – 30 seconds – Middle

1. Hold heat at -10°C
2. Heat sample to 75°C at 10°C/min
3. Hold at 75°C for 1 minute
4. Cool sample to -10°C at 10°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 75°C at 10°C/min

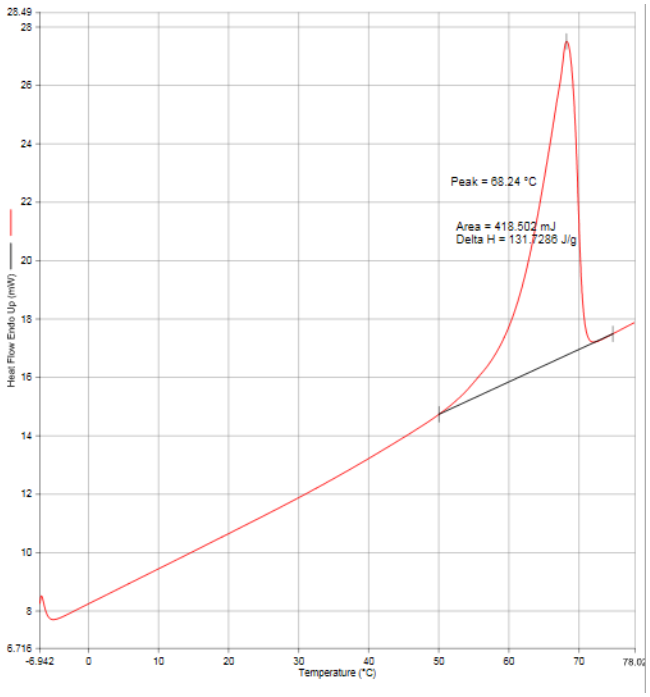


Figure 16(c). 1st heat

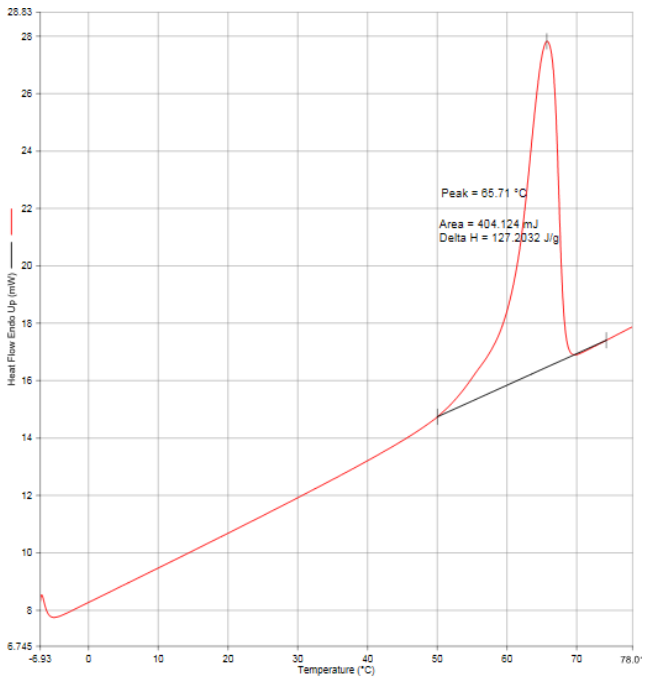


Figure 16(d). 2nd heat

Differential Scanning Calorimetry
Sample 17 - Process conditions – RT(25-30)°C – 15 minutes – Edge

1. Hold heat at -10°C
2. Heat sample to 75°C at 2°C/min
3. Hold at 75°C for 1 minute
4. Cool sample to -10°C at 2°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 75°C at 2°C/min

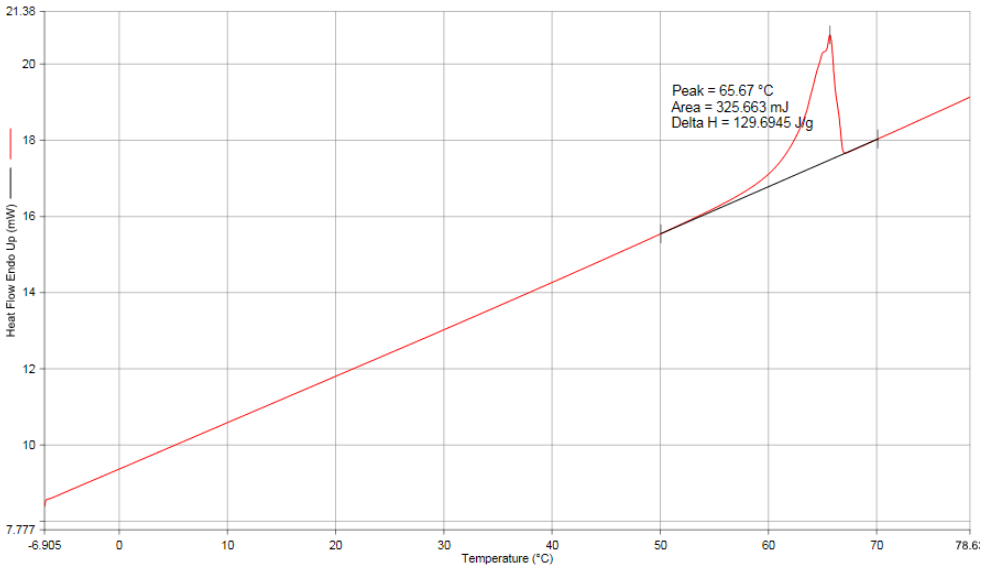


Figure 17(a). 1st heat

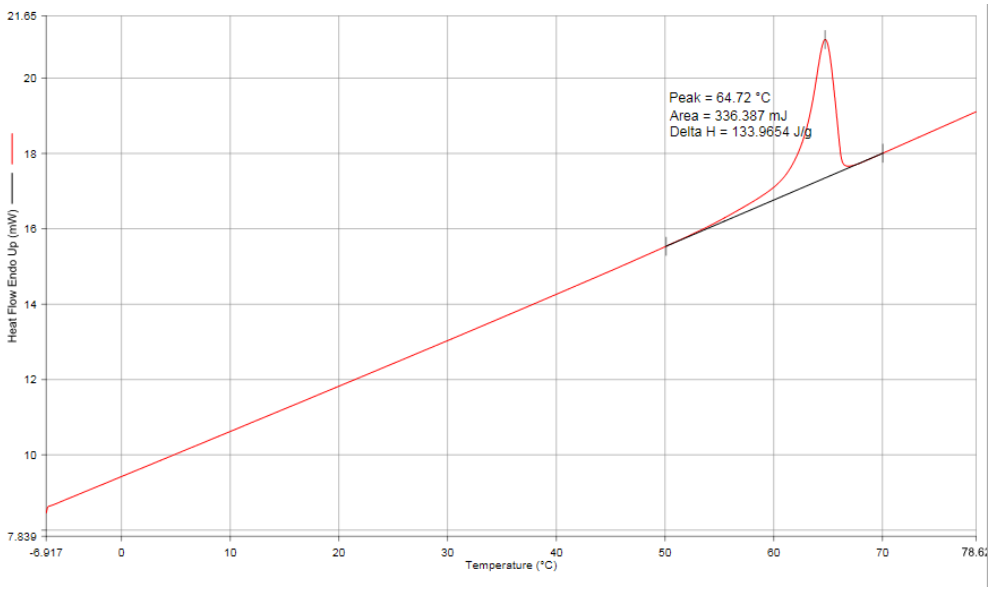


Figure 17(b). 2nd heat

Differential Scanning Calorimetry
Sample 17 - Process conditions – RT(25-30)°C – 15 minutes – Edge

1. Hold heat at -10°C
2. Heat sample to 75°C at 2°C/min
3. Hold at 75°C for 1 minute
4. Cool sample to -10°C at 10°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 75°C at 2°C/min

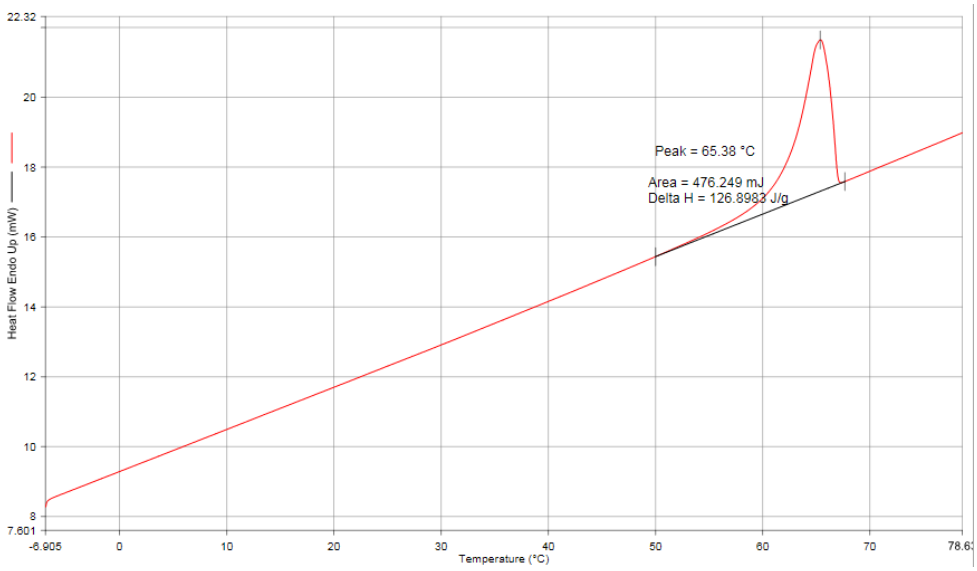


Figure 17(c). 1st heat

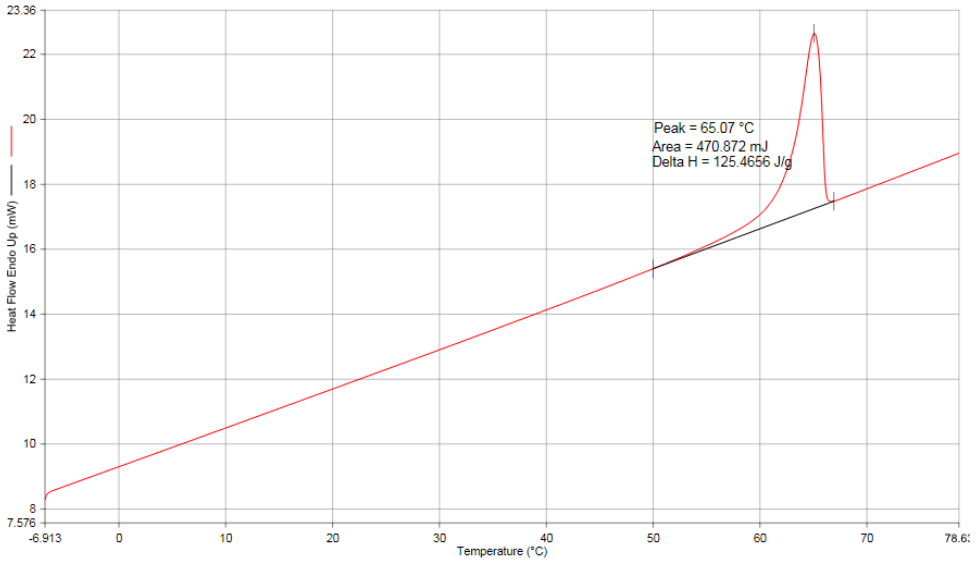


Figure 17(c). 2nd heat

Differential Scanning Calorimetry
Sample 17 - Process conditions – RT(25-30)°C – 15 minutes – Edge

1. Hold heat at -10°C
2. Heat sample to 75°C at 2°C/min
3. Hold at 75°C for 1 minute
4. Cool sample to -10°C at 100°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 75°C at 2°C/min

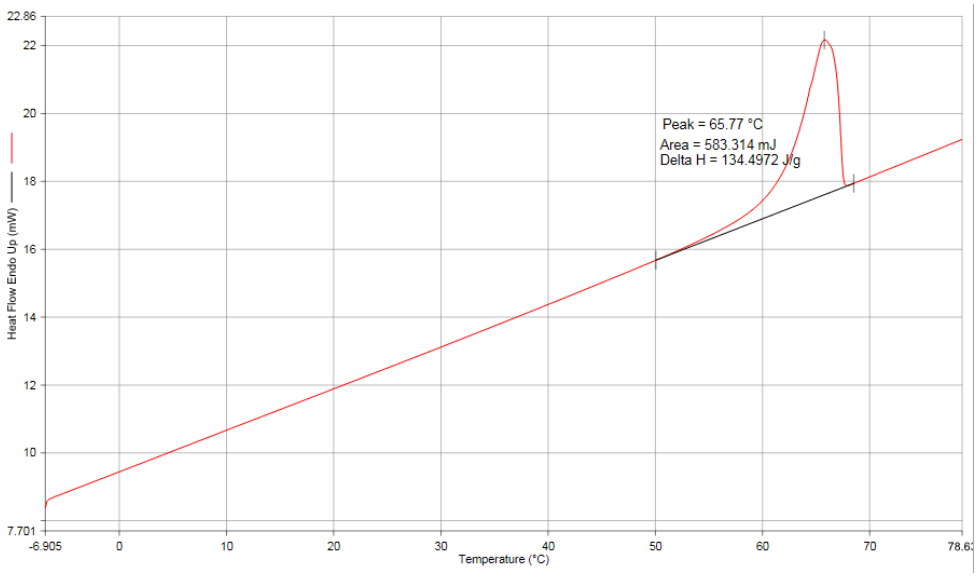


Figure 17(e). 1st heat

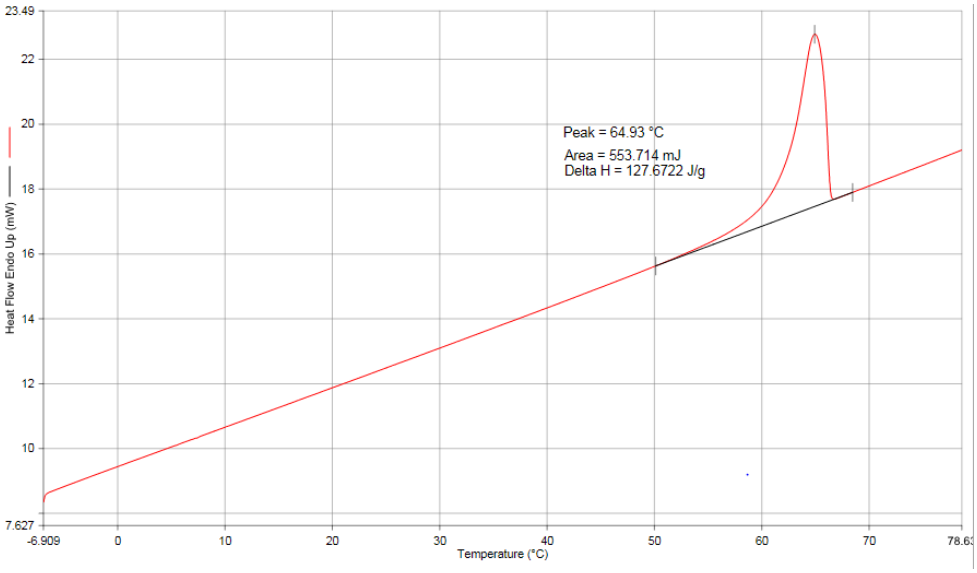


Figure 17(f). 2nd heat

Differential Scanning Calorimetry
Sample 18 - Process conditions – RT(25-30)°C – 1 hour – Edge

1. Hold heat at -10°C
2. Heat sample to 75°C at 10°C/min
3. Hold at 75°C for 1 minute
4. Cool sample to -10°C at 10°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 75°C at 10°C/min

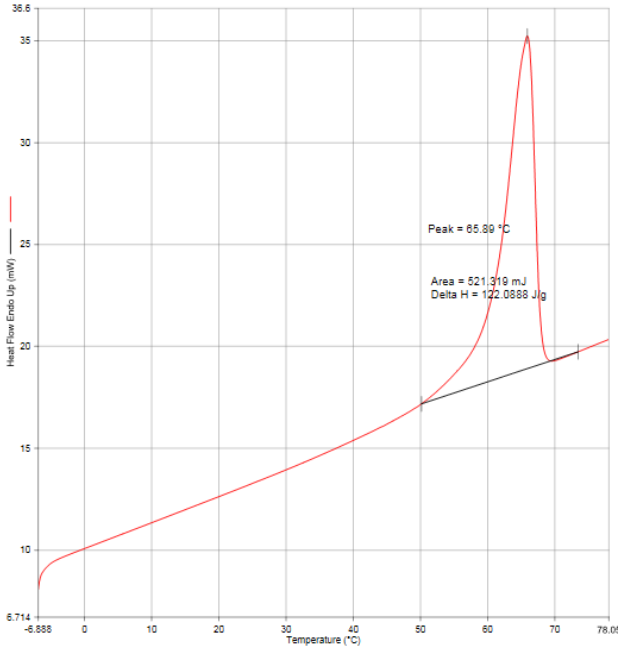


Figure 18(a). 1st heat

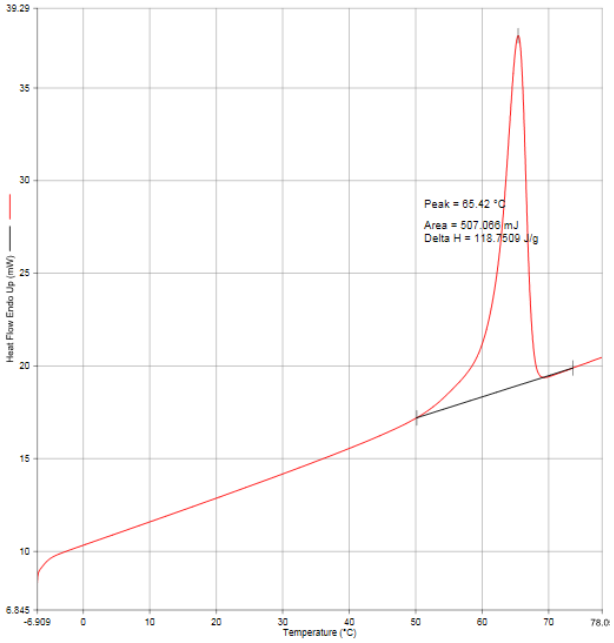


Figure 18(b). 2nd heat

Differential Scanning Calorimetry
Sample 18 - Process conditions – RT(25-30)°C – 1 hour – Middle

1. Hold heat at -10°C
2. Heat sample to 75°C at 10°C/min
3. Hold at 75°C for 1 minute
4. Cool sample to -10°C at 10°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 75°C at 10°C/min

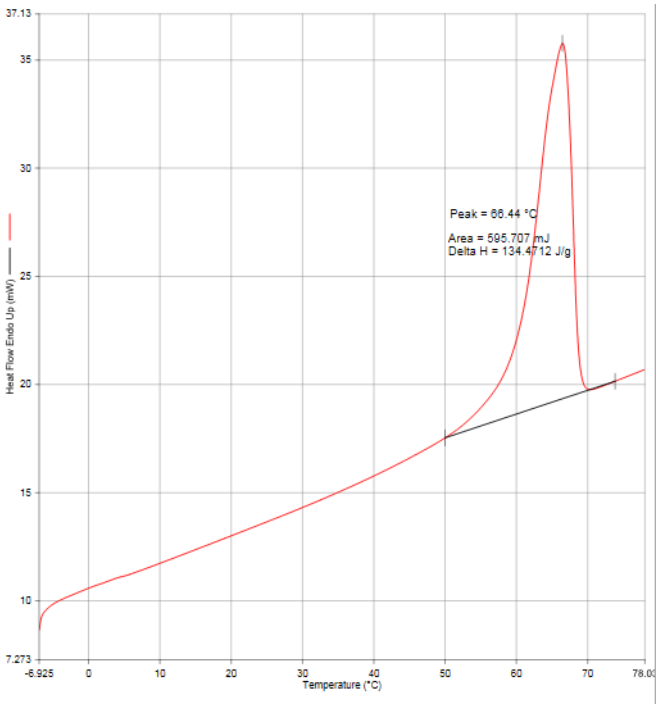


Figure 18(c). 1st heat

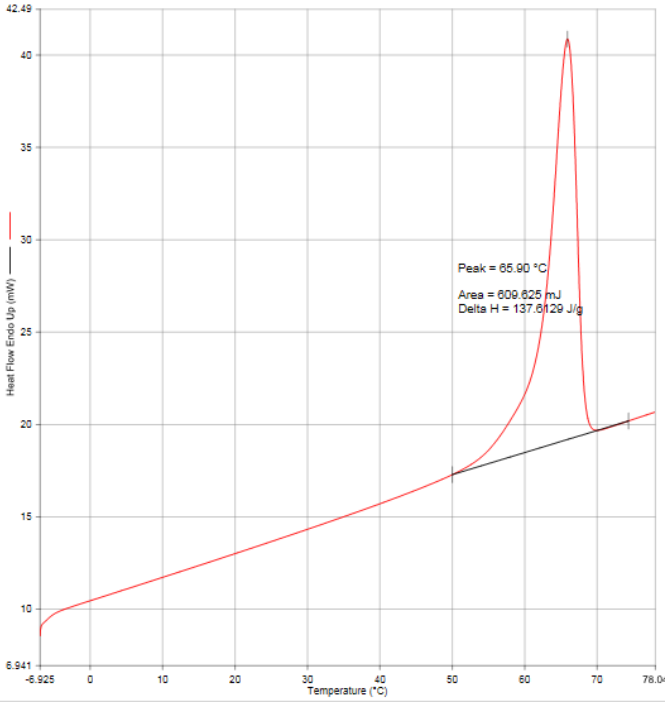


Figure 18(d). 2nd heat

Differential Scanning Calorimetry
Sample 19 - Process conditions – 55°C – 30 seconds – Edge

7. Hold heat at -10°C
8. Heat sample to 75°C at 10°C/min
9. Hold at 75°C for 1 minute
10. Cool sample to -10°C at 10°C/min
11. Hold at -10°C for 1 minute
12. Heat sample to 75°C at 10°C/min

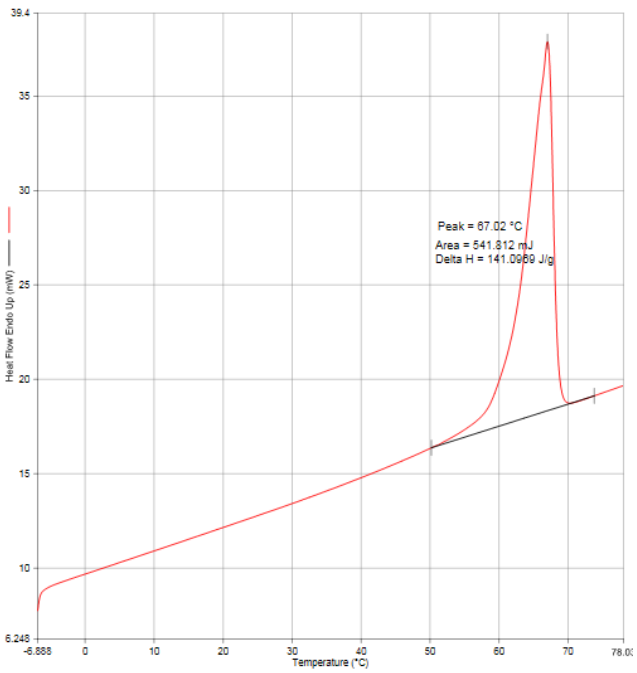


Figure 19(a). 1st heat

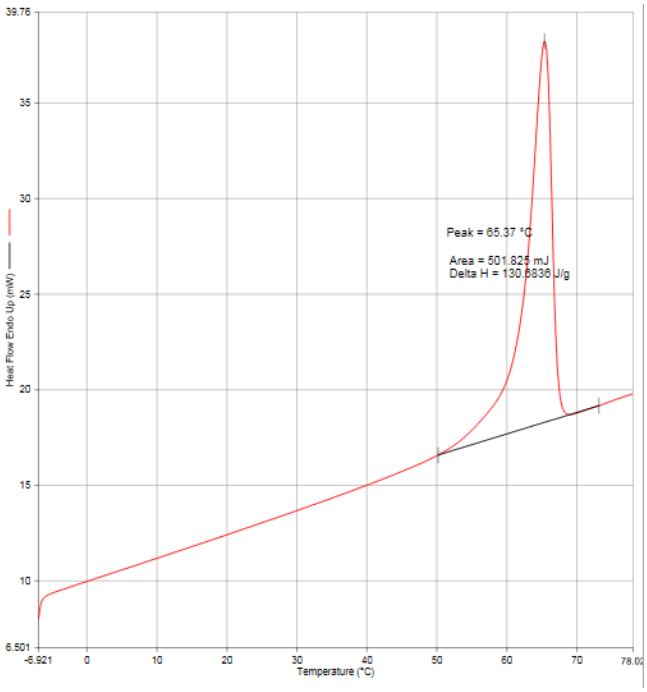


Figure. 19(b). 2nd heat

Differential Scanning Calorimetry
Sample 19 - Process conditions – 55°C – 30 seconds – Middle

7. Hold heat at -10°C
8. Heat sample to 75°C at 10°C/min
9. Hold at 75°C for 1 minute
10. Cool sample to -10°C at 10°C/min
11. Hold at -10°C for 1 minute
12. Heat sample to 75°C at 10°C/min

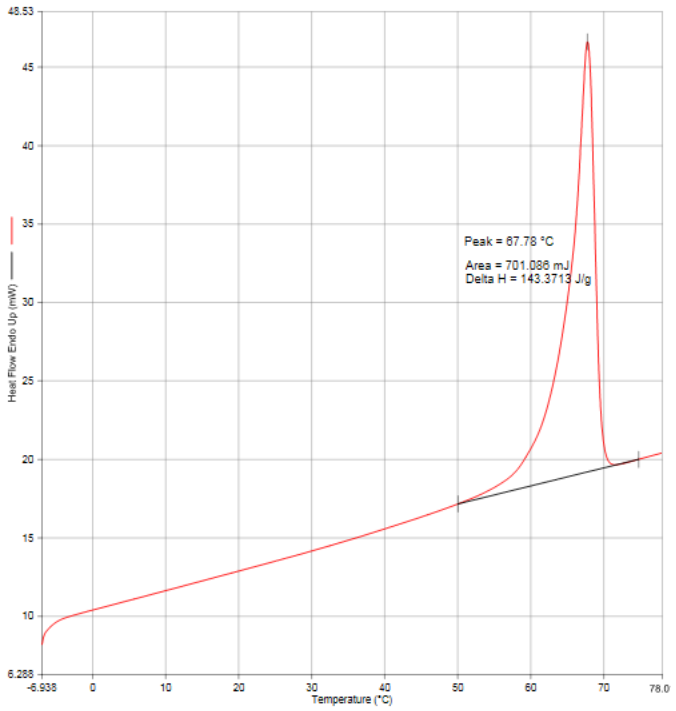


Figure 19(c). 1st heat

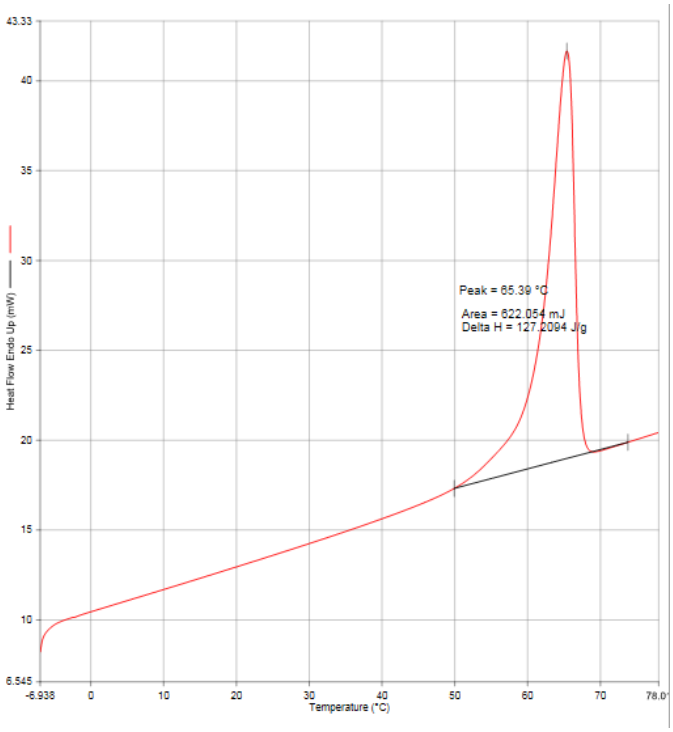


Figure 19(c). 2nd heat

Differential Scanning Calorimetry
Sample 20 - Process conditions – 55°C – 15 minutes– Edge

7. Hold heat at -10°C
8. Heat sample to 75°C at 10°C/min
9. Hold at 75°C for 1 minute
10. Cool sample to -10°C at 10°C/min
11. Hold at -10°C for 1 minute
12. Heat sample to 75°C at 10°C/min

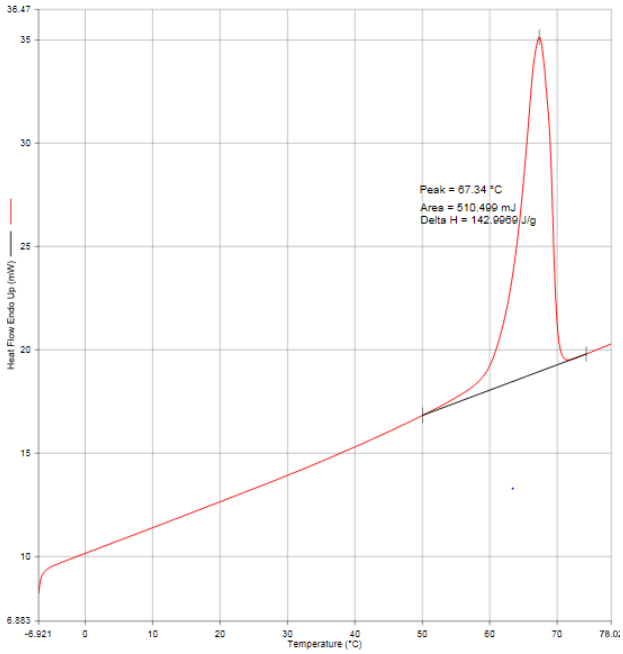


Figure 20(a). 1st heat

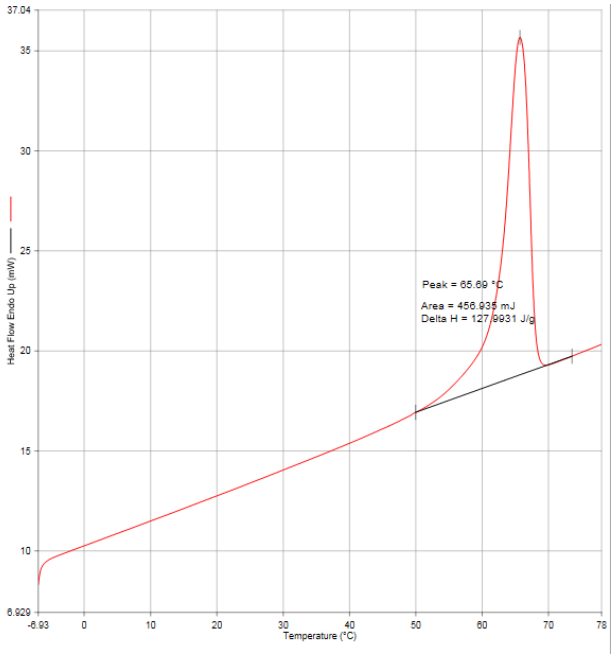


Figure 20(b). 2nd heat

Differential Scanning Calorimetry
Sample 20 - Process conditions – 55°C – 15 minutes – Middle

7. Hold heat at -10°C
8. Heat sample to 75°C at 10°C/min
9. Hold at 75°C for 1 minute
10. Cool sample to -10°C at 10°C/min
11. Hold at -10°C for 1 minute
12. Heat sample to 75°C at 10°C/min

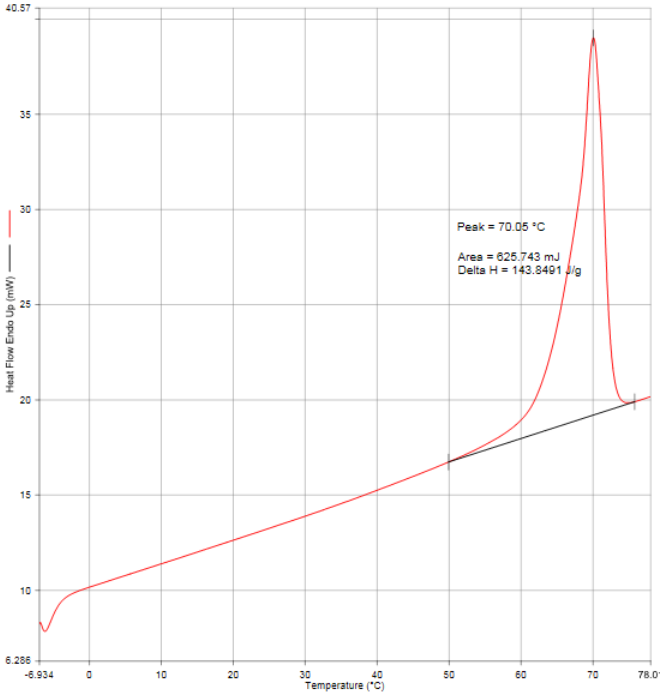


Figure 20(c). 1st heat

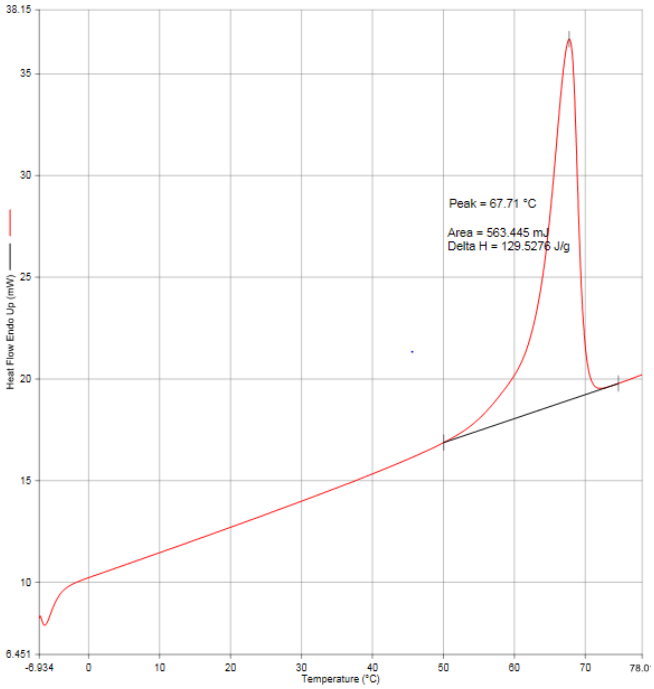


Figure 20(d). 2nd heat

Differential Scanning Calorimetry
Sample 21 - Process conditions – 55°C – 1 hour – Edge

13. Hold heat at -10°C
14. Heat sample to 75°C at 10°C/min
15. Hold at 75°C for 1 minute
16. Cool sample to -10°C at 10°C/min
17. Hold at -10°C for 1 minute
18. Heat sample to 75°C at 10°C/min

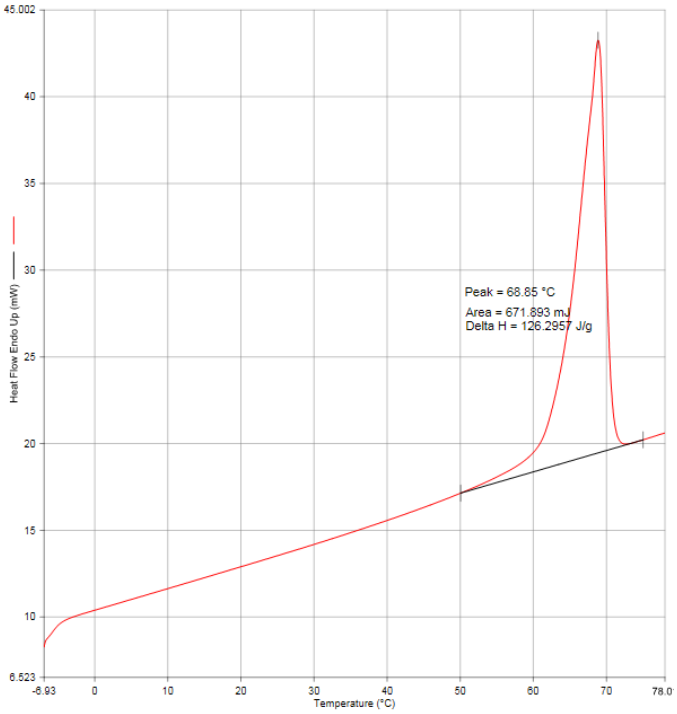


Figure 21(a). 1st heat

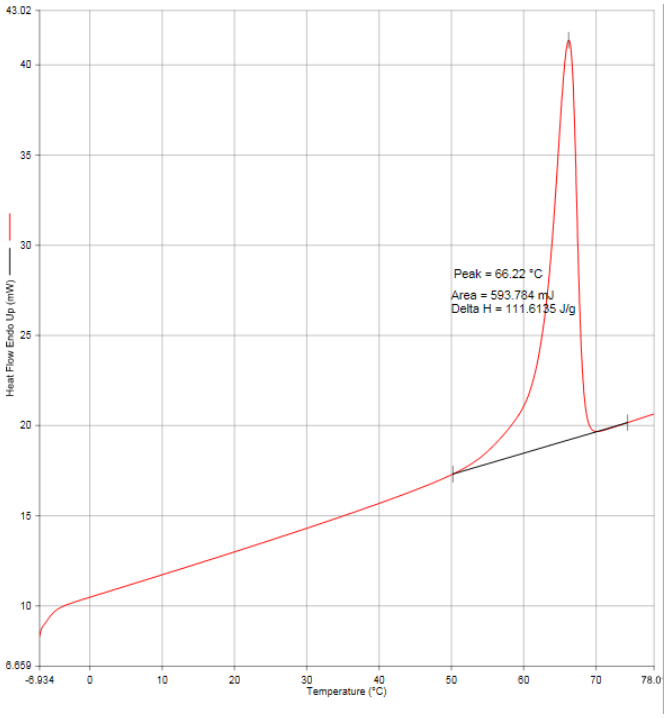


Figure 22(b). 2nd heat

Differential Scanning Calorimetry
Sample 21 - Process conditions – 55°C – 1 hour – Middle

13. Hold heat at -10°C
14. Heat sample to 75°C at 10°C/min
15. Hold at 75°C for 1 minute
16. Cool sample to -10°C at 10°C/min
17. Hold at -10°C for 1 minute
18. Heat sample to 75°C at 10°C/min

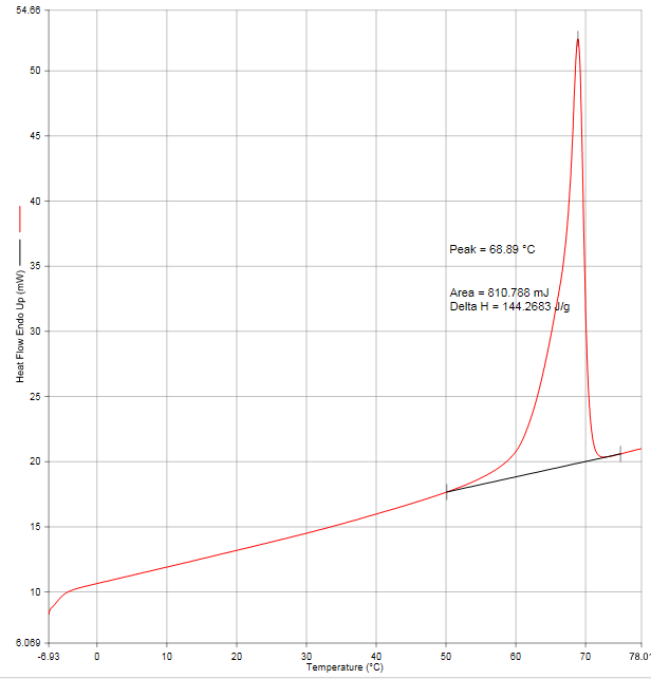


Figure 21(c). 1st heat

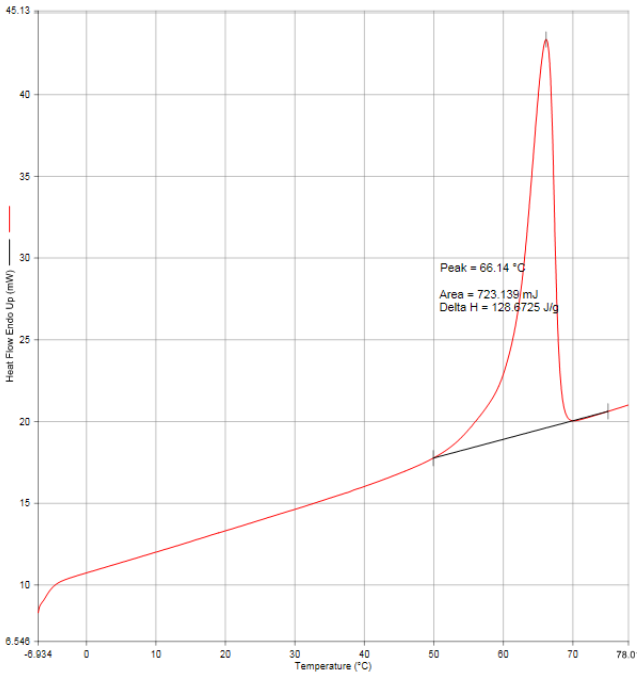


Figure 22(d). 2nd heat

Differential Scanning Calorimetry
Sample 22 - Process conditions – 62°C – 30 seconds– Edge

13. Hold heat at -10°C
14. Heat sample to 75°C at 10°C/min
15. Hold at 75°C for 1 minute
16. Cool sample to -10°C at 10°C/min
17. Hold at -10°C for 1 minute
18. Heat sample to 75°C at 10°C/min

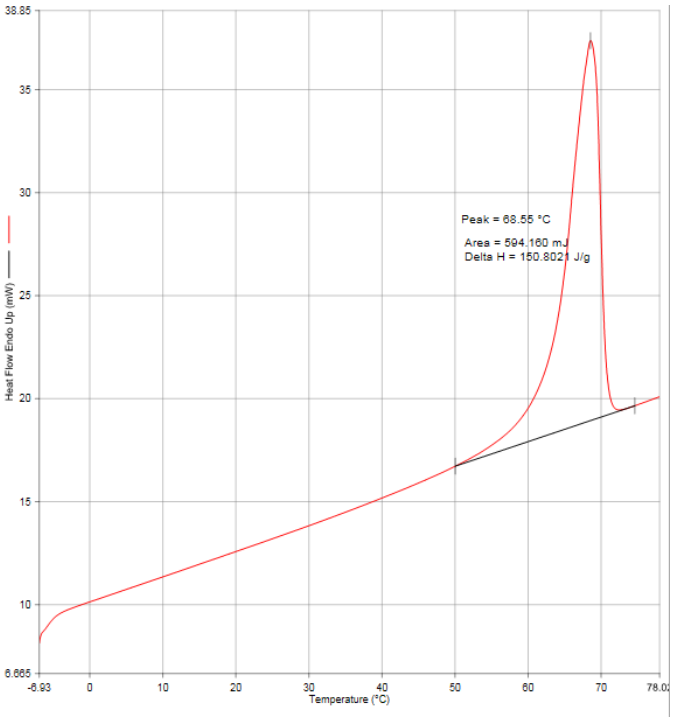


Figure 22(a). 1st heat

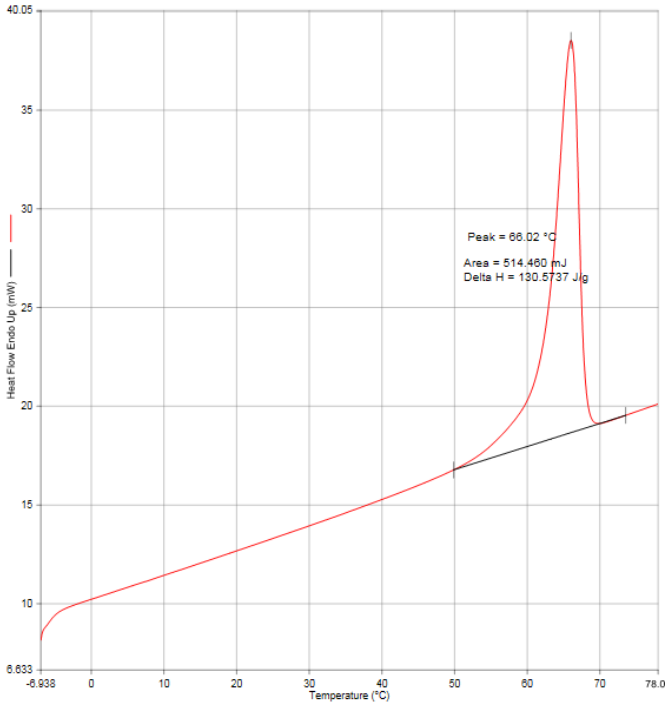


Figure 22b). 2nd heat

Differential Scanning Calorimetry
Sample 22 - Process conditions – 62°C – 30 seconds – Middle

13. Hold heat at -10°C
14. Heat sample to 75°C at 10°C/min
15. Hold at 75°C for 1 minute
16. Cool sample to -10°C at 10°C/min
17. Hold at -10°C for 1 minute
18. Heat sample to 75°C at 10°C/min

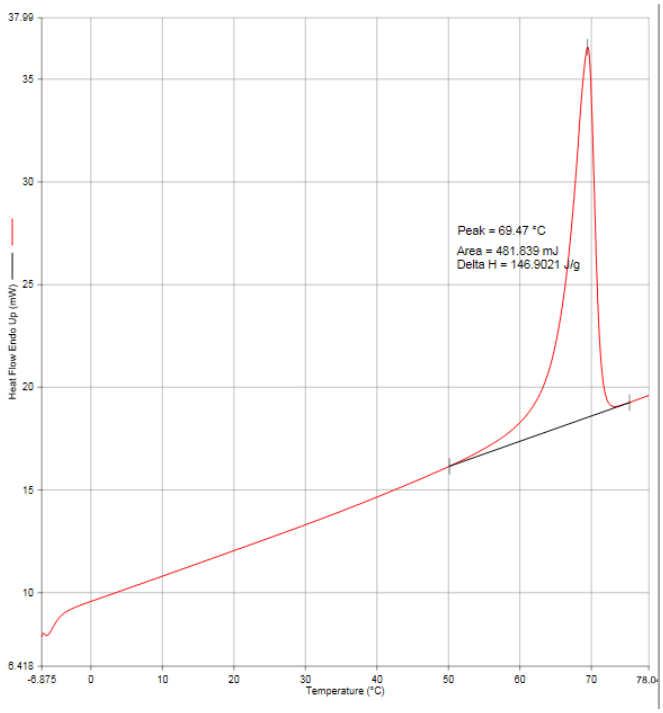


Figure 22(c). 1st heat

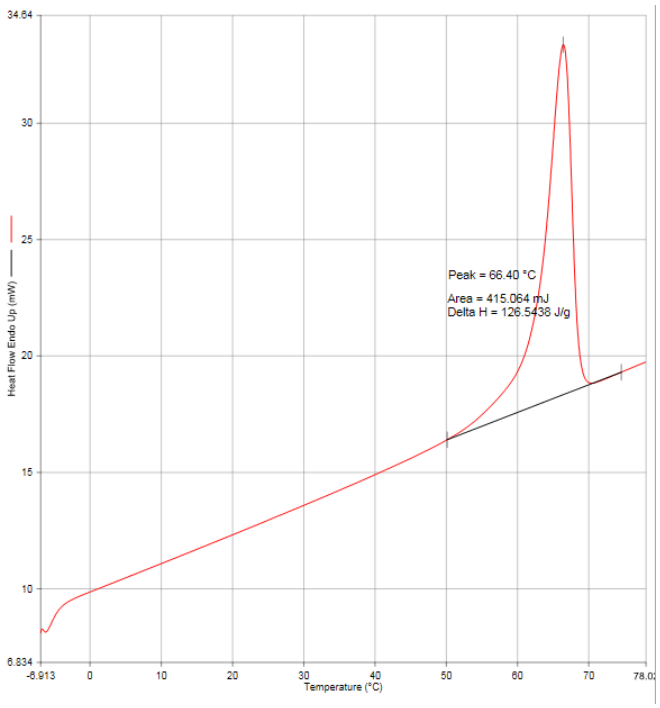


Figure 22(c). 2nd heat

Differential Scanning Calorimetry
Sample 23 - Process conditions – 62°C – 15 minutes – Edge

19. Hold heat at -10°C
20. Heat sample to 75°C at 10°C/min
21. Hold at 75°C for 1 minute
22. Cool sample to -10°C at 10°C/min
23. Hold at -10°C for 1 minute
24. Heat sample to 75°C at 10°C/min

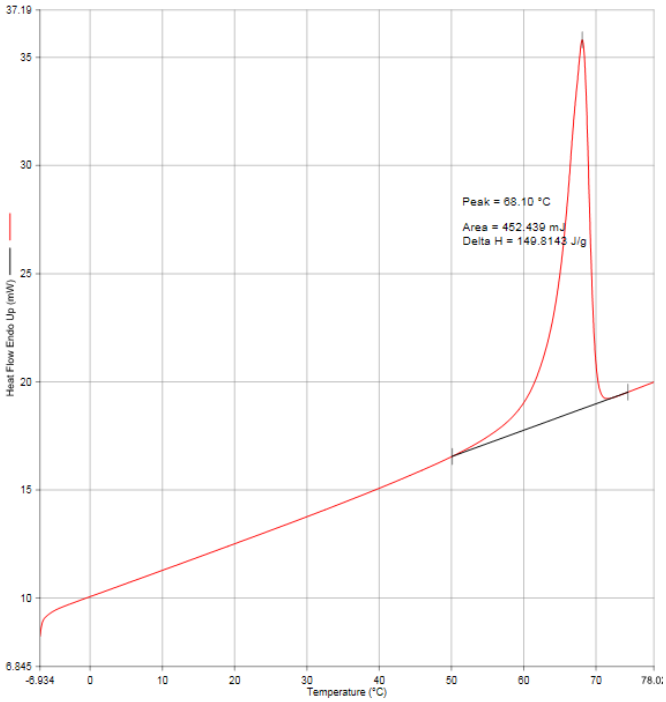


Figure 23(a). 1st heat

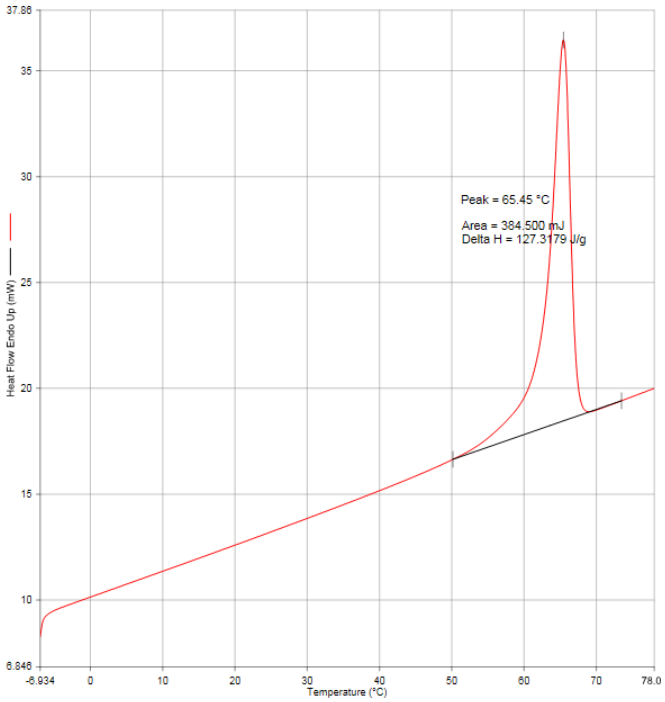


Figure 23(b). 2nd heat

Differential Scanning Calorimetry
Sample 23 - Process conditions – 62°C – 15 minutes – Middle

19. Hold heat at -10°C
20. Heat sample to 75°C at 10°C/min
21. Hold at 75°C for 1 minute
22. Cool sample to -10°C at 10°C/min
23. Hold at -10°C for 1 minute
24. Heat sample to 75°C at 10°C/min

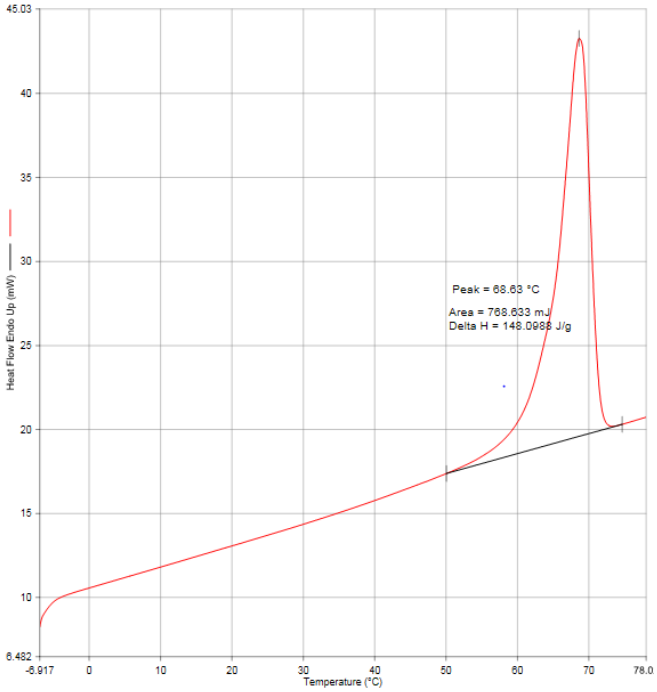


Figure 23(c). 1st heat

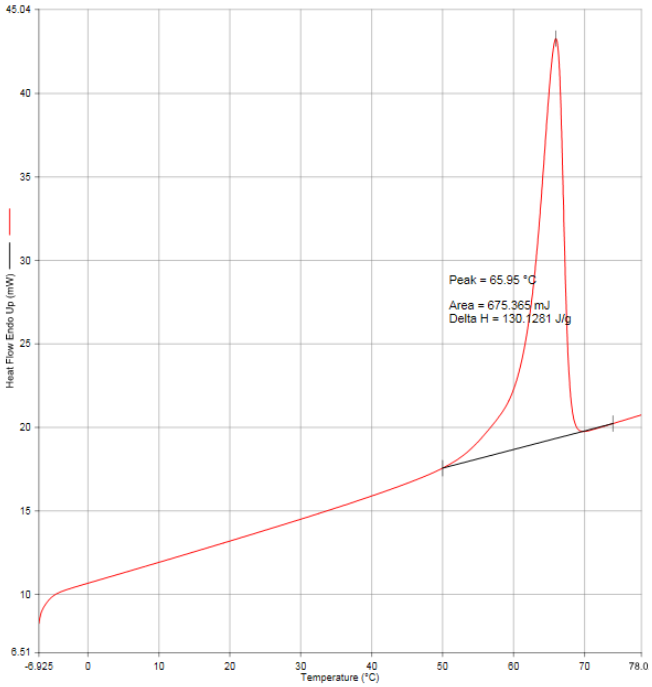


Figure 23(d). 2nd heat

Differential Scanning Calorimetry
Sample 24 - Process conditions – 62°C – 1 hour – Edge

19. Hold heat at -10°C
20. Heat sample to 75°C at 10°C/min
21. Hold at 75°C for 1 minute
22. Cool sample to -10°C at 10°C/min
23. Hold at -10°C for 1 minute
24. Heat sample to 75°C at 10°C/min

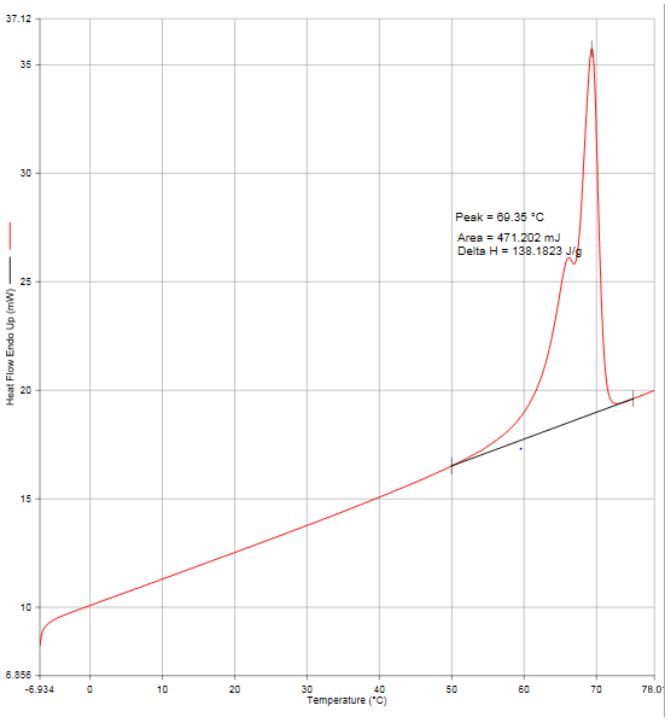


Figure 24(a). 1st heat

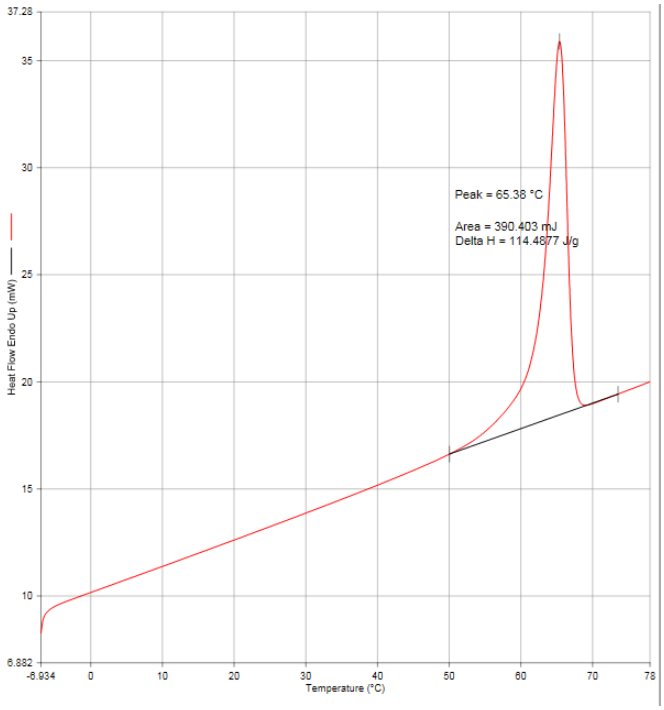


Figure 24(b). 2nd heat

Differential Scanning Calorimetry
Sample 24 - Process conditions – 62°C – 1 hour – Middle

19. Hold heat at -10°C
20. Heat sample to 75°C at 10°C/min
21. Hold at 75°C for 1 minute
22. Cool sample to -10°C at 10°C/min
23. Hold at -10°C for 1 minute
24. Heat sample to 75°C at 10°C/min

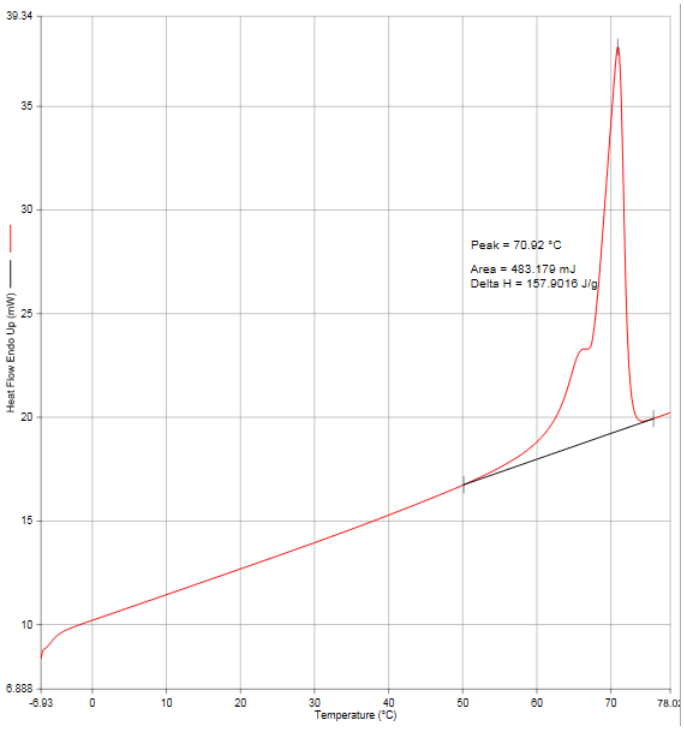


Figure 24(c). 1st heat

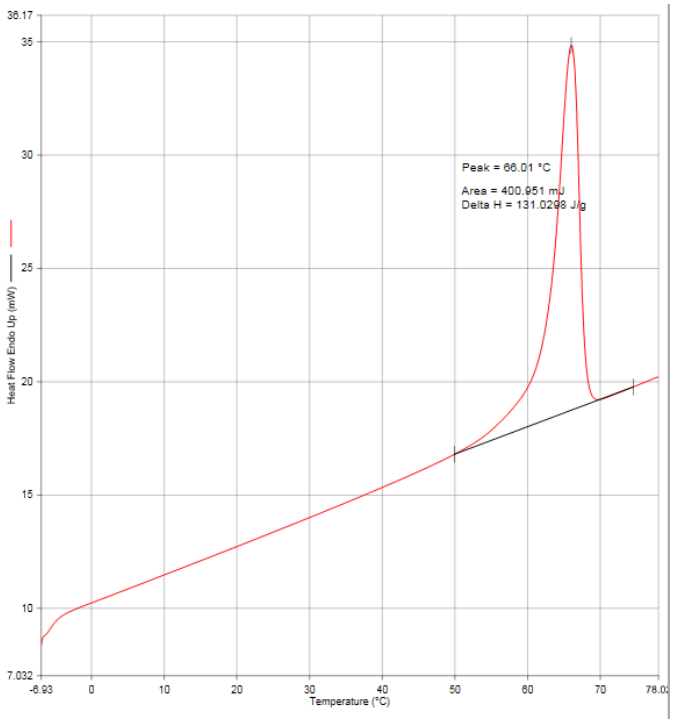


Figure 24(d). 2nd heat

Differential Scanning Calorimetry – Polyethylene
Sample 25 – Process conditions – Untreated

1. Hold heat at -10°C
2. Heat sample to 145°C at 10°C/min
3. Hold at 145°C for 1 minute
4. Cool sample to -10°C at 10°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 145°C at 10°C/min

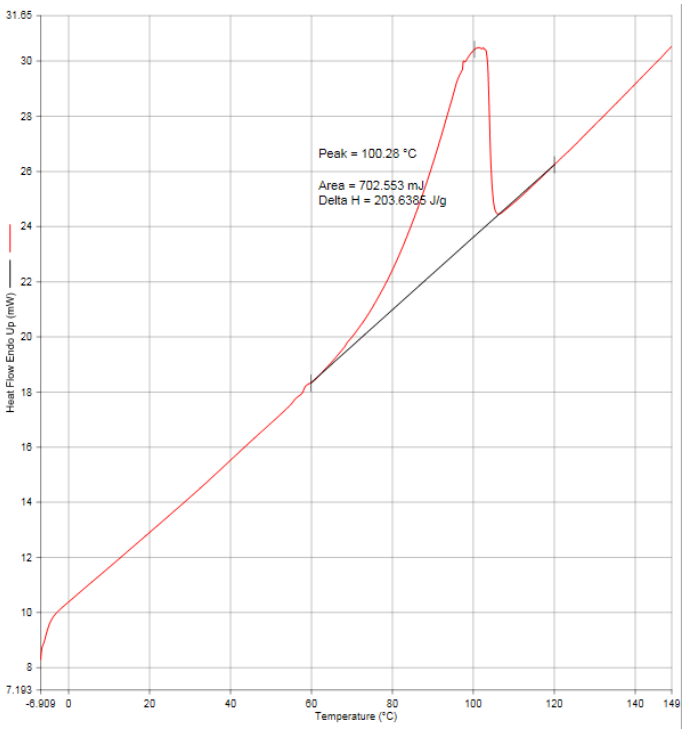


Figure 25(a). 1st heat

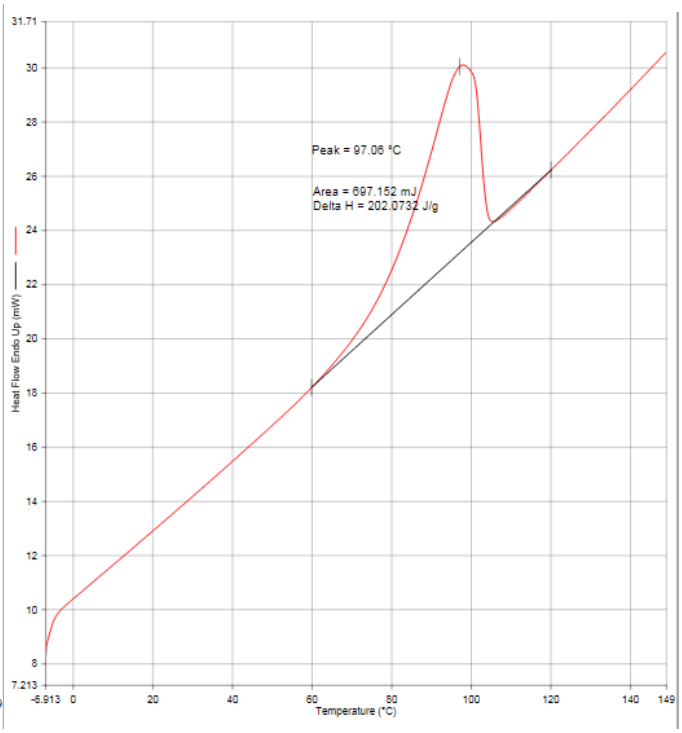


Figure 25(b). 2nd heat

Differential Scanning Calorimetry – Polyethylene
Sample 26 – Process conditions – RT(25-30)°C – 15 minutes

1. Hold heat at -10°C
2. Heat sample to 145°C at 10°C/min
3. Hold at 145°C for 1 minute
4. Cool sample to -10°C at 10°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 145°C at 10°C/min

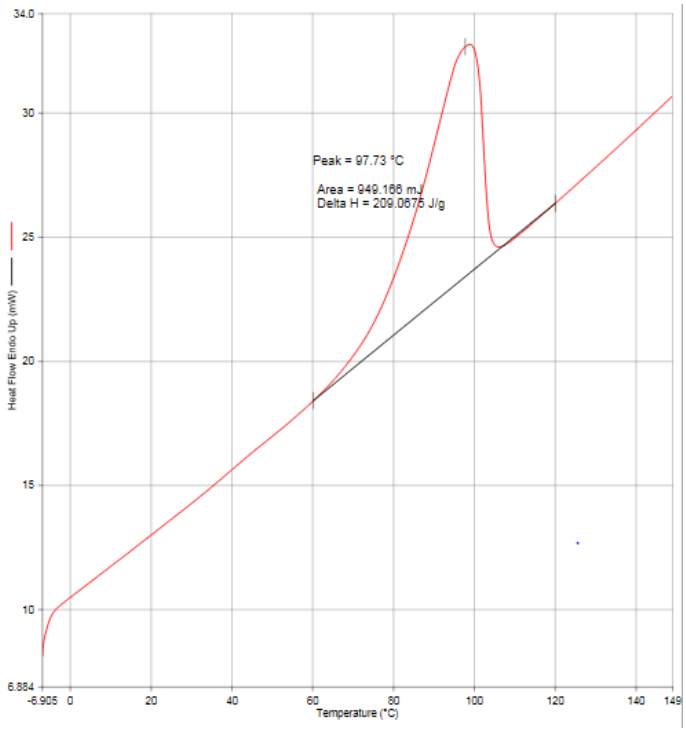


Figure 26(a). 1st heat

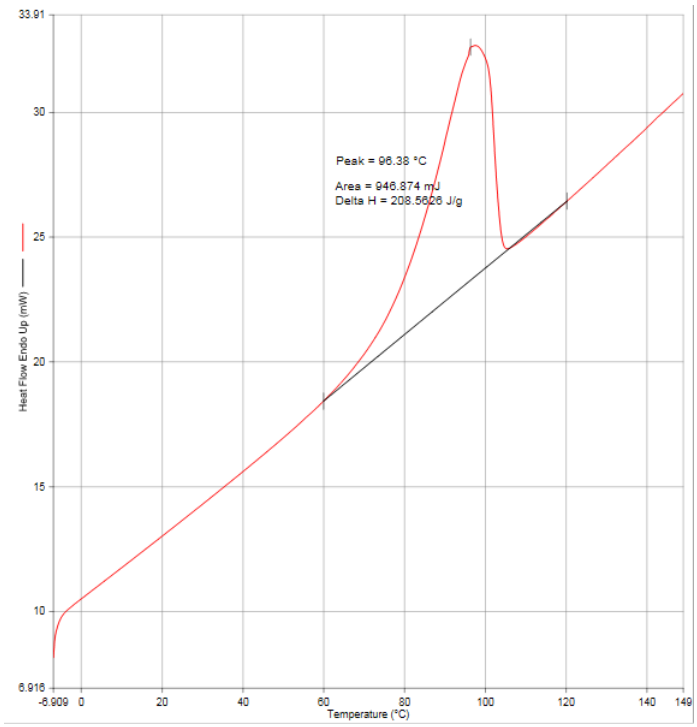


Figure 26(b). 2nd heat

Differential Scanning Calorimetry – Polyethylene
Sample 27 – Process conditions – 50°C – 15 minutes

1. Hold heat at -10°C
2. Heat sample to 145°C at 10°C/min
3. Hold at 145°C for 1 minute
4. Cool sample to -10°C at 10°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 145°C at 10°C/min

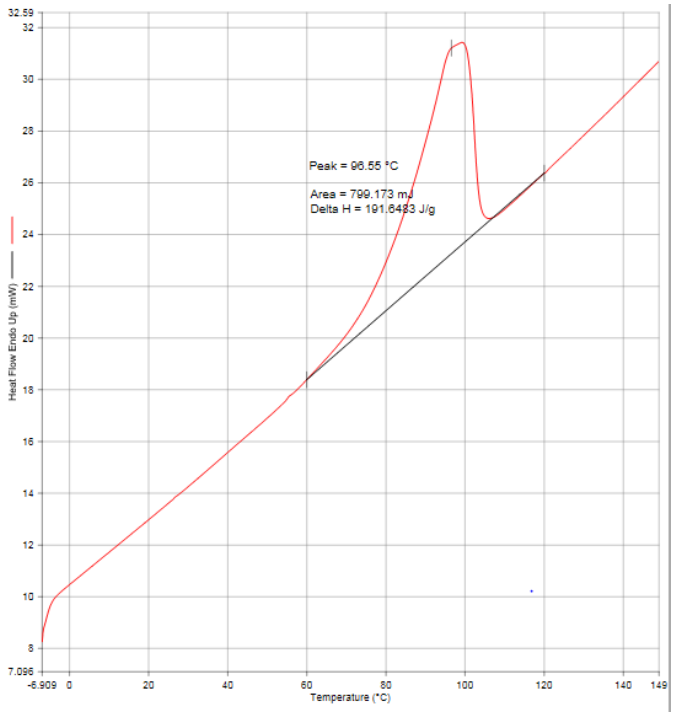


Figure 27(a). 1st heat

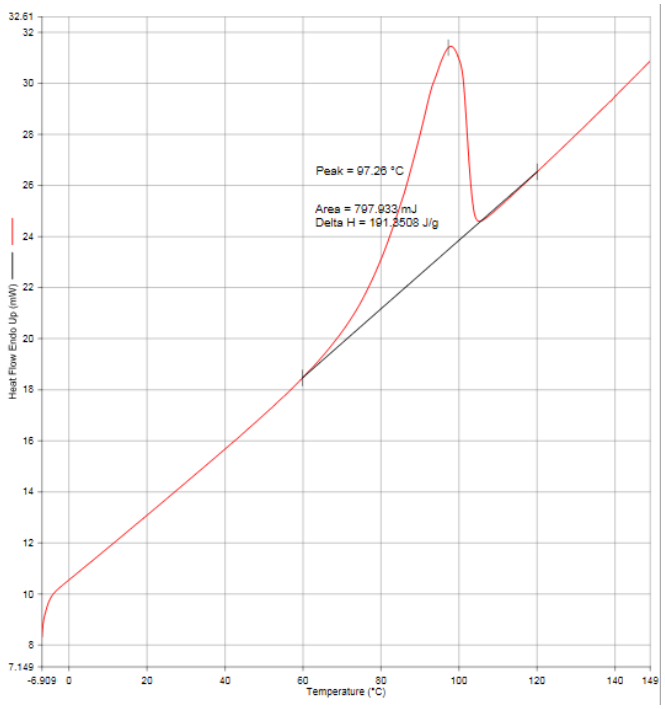


Figure 27(b). 2nd heat

Differential Scanning Calorimetry – Polyethylene
Sample 28 – Process conditions – 60°C – 15 minutes

1. Hold heat at -10°C
2. Heat sample to 145°C at 10°C/min
3. Hold at 145°C for 1 minute
4. Cool sample to -10°C at 10°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 145°C at 10°C/min

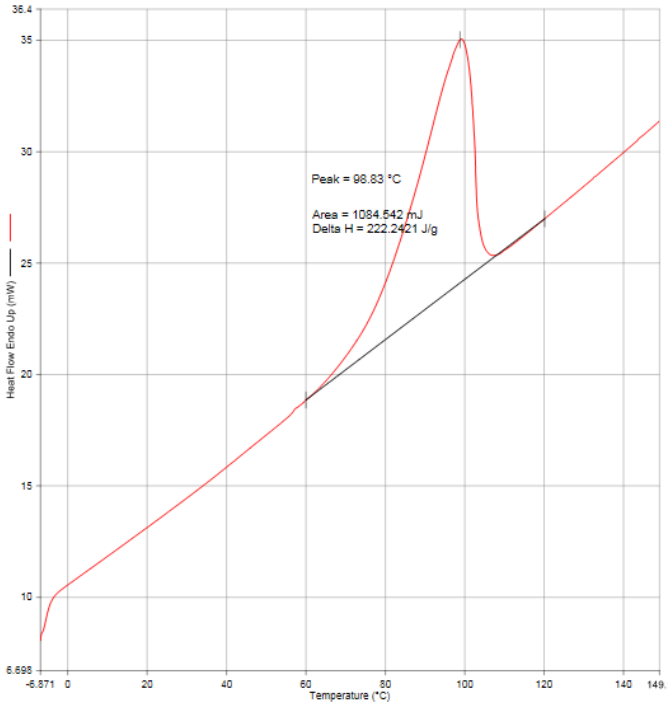


Figure 28(a). 1st heat

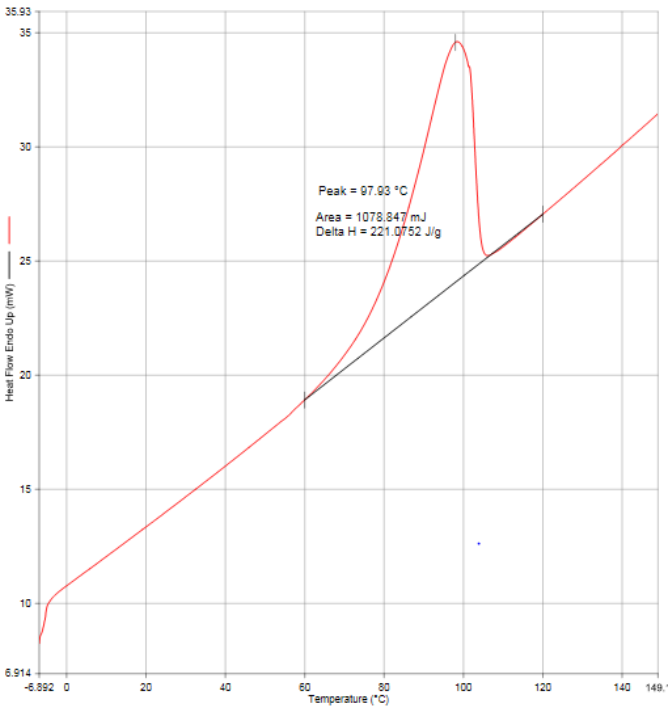


Figure 28(b). 2nd heat

Differential Scanning Calorimetry – Polyethylene
Sample 29 – Process conditions – 75°C – 15 minutes – Middle

1. Hold heat at -10°C
2. Heat sample to 145°C at 10°C/min
3. Hold at 145°C for 1 minute
4. Cool sample to -10°C at 10°C/min
5. Hold at -10°C for 1 minute
6. Heat sample to 145°C at 10°C/min

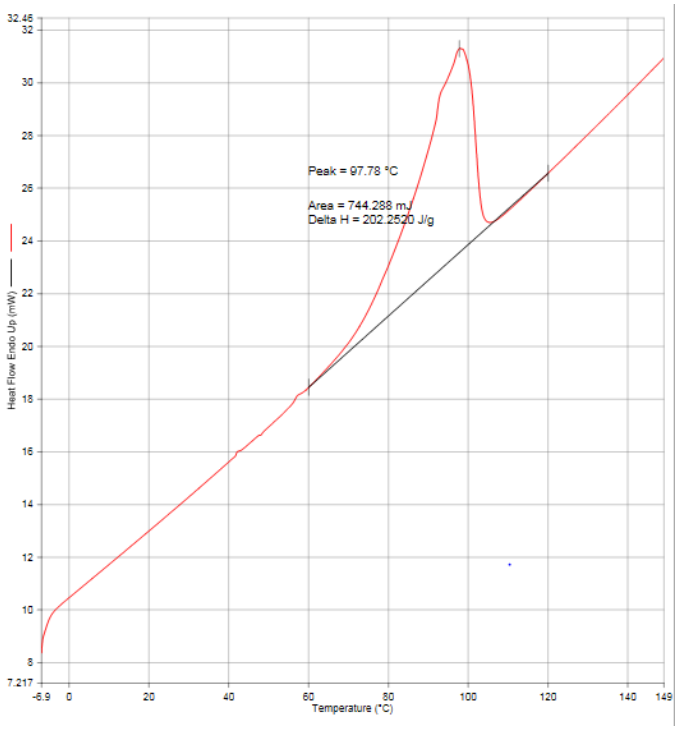


Figure 29(a). 1st heat

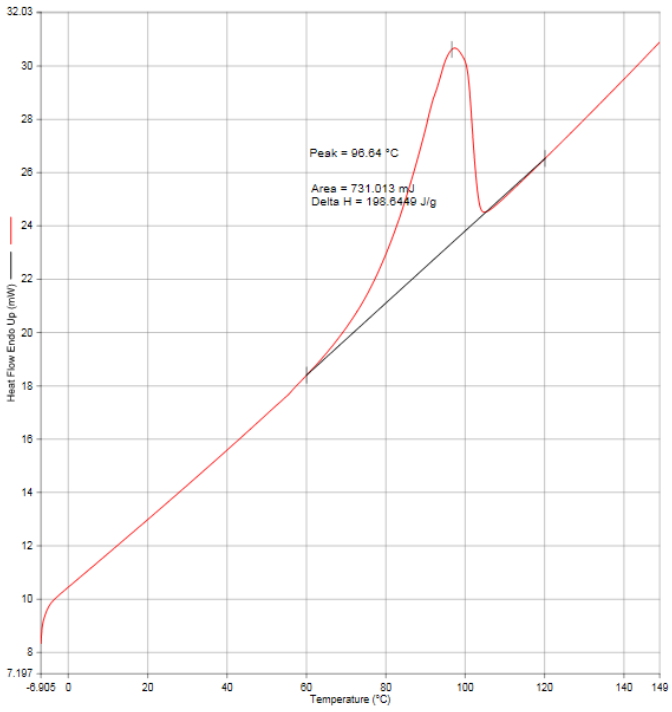
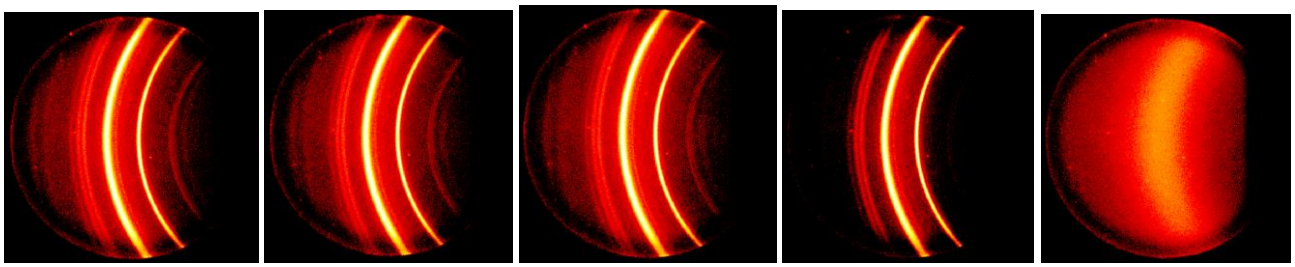


Figure 29(b). 2nd heat

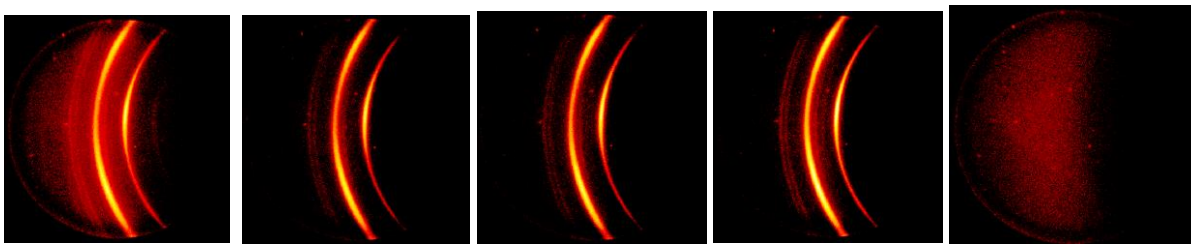
Appendix 2 – X-Ray Diffraction Scans

XRD Raw unaltered data



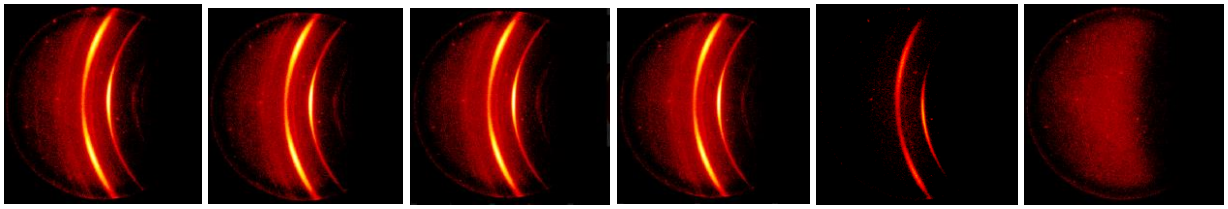
25°C **45°C** **55°C** **65°C** **75°C**

Figure 30. XRD results – Untreated



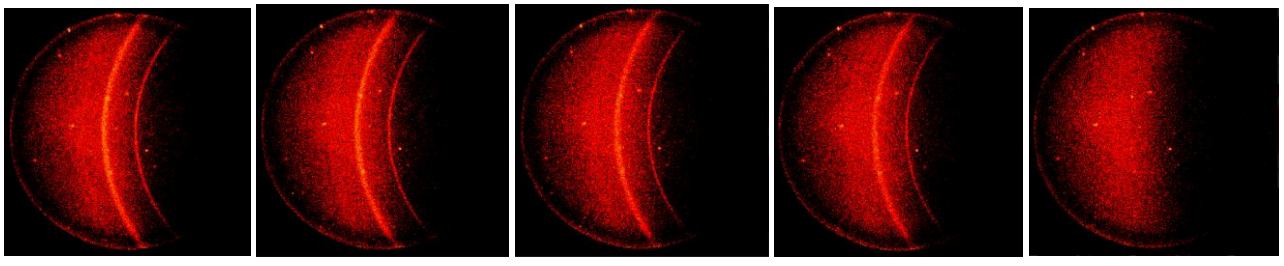
25°C **45°C** **55°C** **65°C** **75°C**

Figure 31. XRD results - 25°C – RT



25°C **45°C** **55°C** **65°C** **75°C** **85°C**

Figure 32. XRD results - 55°C



25°C **45°C** **55°C** **65°C** **75°C**

Figure 33. XRD results - 62°C

Appendix 3 – Instron Results

Instron – Poly (ethylene oxide)

Control Sample 5

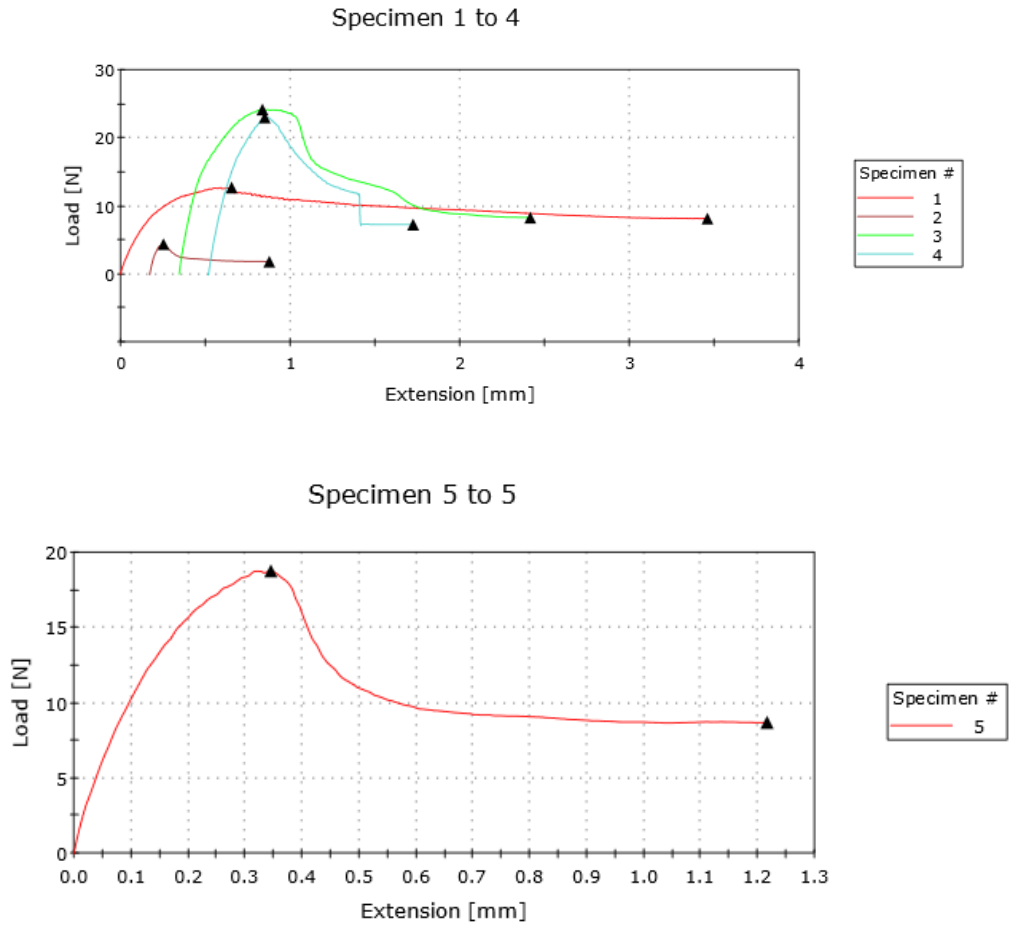


Figure 34. Instron Graph Results – Control Samples Sample 5

	Specimen label	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [%]	Load at Break (Standard) [N]	Tensile stress at Break (Standard) [MPa]	Tensile strain (Extension) at Break (Standard) [%]
1	Control 5	12.72	0.04	2.33	8.18	0.02	12.34
2	Control 5_2	4.43	0.01	0.41	1.88	0.01	3.52
3	Control 5_3	24.18	0.07	2.45	8.32	0.02	10.34
4	Control 5_4	22.99	0.06	1.66	7.32	0.02	6.02
5	Control 5_5	18.76	0.05	1.82	8.71	0.02	6.39
Coefficient of variation		49.08277	49.08277	47.01931	41.29253	41.29253	46.00539
Maximum		24.18	0.07	2.45	8.71	0.02	12.34
Mean		16.61	0.05	1.73	6.88	0.02	7.72
Median		18.76	0.05	1.82	8.18	0.02	6.39
Minimum		4.43	0.01	0.41	1.88	0.01	3.52
Range		19.74	0.05	2.05	6.83	0.02	8.82
Standard deviation		8.15447	0.02265	0.81498	2.84167	0.00789	3.55284
Mean + 1 SD		24.77	0.07	2.55	9.72	0.03	11.28
Mean - 1 SD		8.46	0.02	0.92	4.04	0.01	4.17

Table xx. Instron Raw Data – Control Sample 6

Control Sample 6

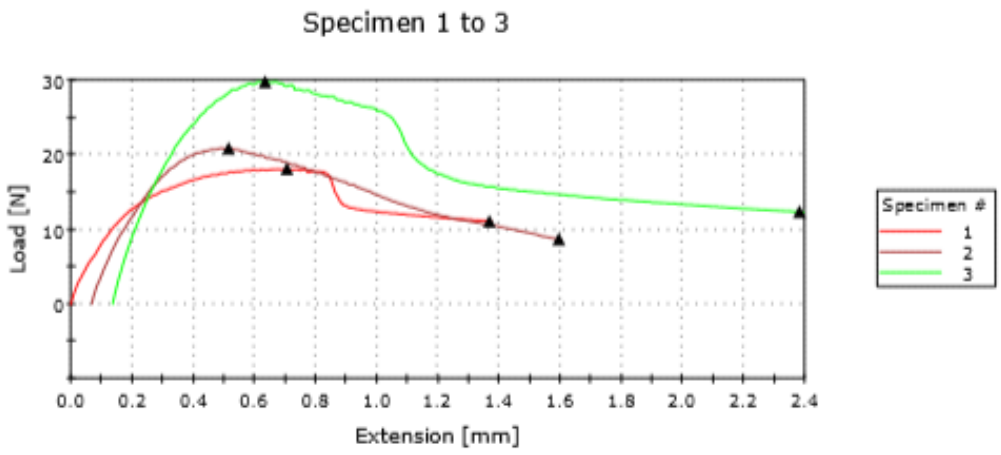


Figure 34. Instron Graph Results – Control Samples Sample 6

	Specimen label	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [%]	Load at Break (Standard) [N]	Tensile stress at Break (Standard) [MPa]	Tensile strain (Extension) at Break (Standard) [%]
1	Control 6_1	18.12	0.05	3.71	11.12	0.03	7.19
2	Control 6_2	20.86	0.06	1.60	8.74	0.02	5.44
3	Control 6_3	29.73	0.08	1.78	12.38	0.03	8.00
Coefficient of variation		26.51164	26.51164	49.68885	17.21955	17.21955	19.02011
Maximum		29.73	0.08	3.71	12.38	0.03	8.00
Mean		22.90	0.06	2.36	10.75	0.03	6.88
Median		20.86	0.06	1.78	11.12	0.03	7.19
Minimum		18.12	0.05	1.60	8.74	0.02	5.44
Range		11.62	0.03	2.12	3.64	0.01	2.56
Standard deviation		6.07226	0.01687	1.17406	1.85051	0.00514	1.30868
Mean + 1 SD		28.98	0.08	3.54	12.60	0.03	8.19

Table xx. Instron Raw Data – Control Sample 6

Control Sample 7

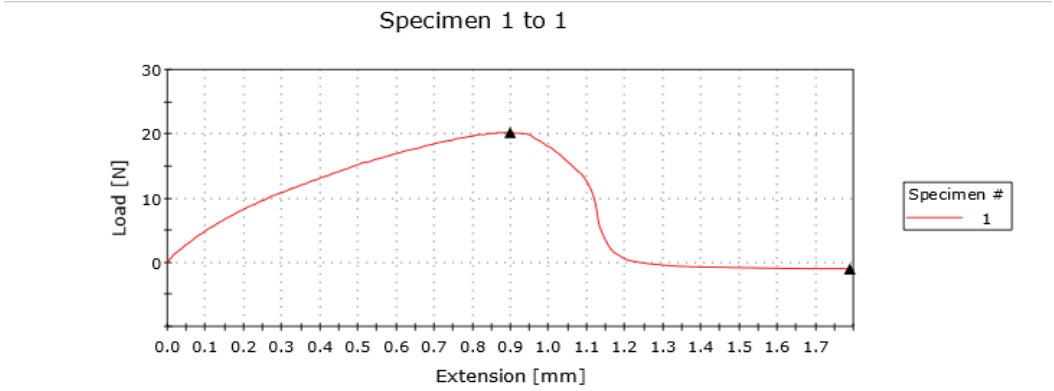


Figure 35. Instron Graph Results – Control Samples Sample 7

	Specimen label	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [%]	Load at Break (Standard) [N]	Tensile stress at Break (Standard) [MPa]	Tensile strain (Extension) at Break (Standard) [%]
1	Control 7	20.22	0.10	2.99	-0.91	0.00	5.96
Coefficient of variation		-----	-----	-----	-----	-----	-----
Maximum		20.22	0.10	2.99	-0.91	0.00	5.96
Mean		20.22	0.10	2.99	-0.91	0.00	5.96
Median		20.22	0.10	2.99	-0.91	0.00	5.96
Minimum		20.22	0.10	2.99	-0.91	0.00	5.96
Range		0.00	0.00	0.00	0.00	0.00	0.00

Table xx. Instron Raw Data – Control Sample 7

Control Sample 8

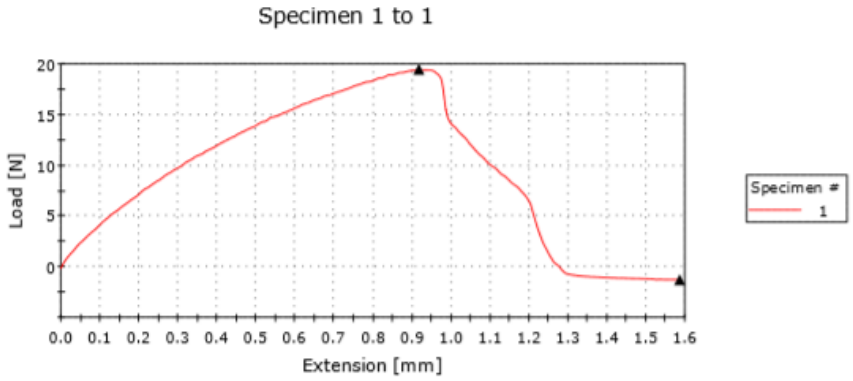
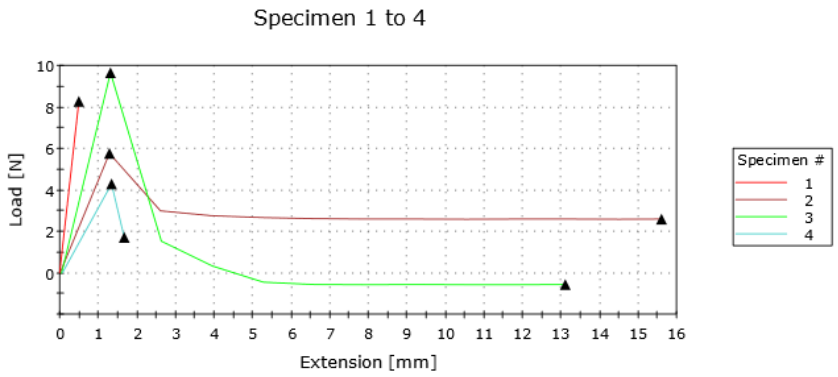


Figure 36. Instron Graph Results – Control Samples Sample 8

	Specimen label	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [%]	Load at Break (Standard) [N]	Tensile stress at Break (Standard) [MPa]	Tensile strain (Extension) at Break (Standard) [%]
1	Sample 8	19.45	0.10	3.05	-1.20	-0.01	5.28
Coefficient of variation		-----	-----	-----	-----	-----	-----
Maximum		19.45	0.10	3.05	-1.20	-0.01	5.28
Mean		19.45	0.10	3.05	-1.20	-0.01	5.28
Median		19.45	0.10	3.05	-1.20	-0.01	5.28
Minimum		19.45	0.10	3.05	-1.20	-0.01	5.28

Table xx. Instron Raw Data – Control Sample 8

Control and Room Temperature Processed Samples



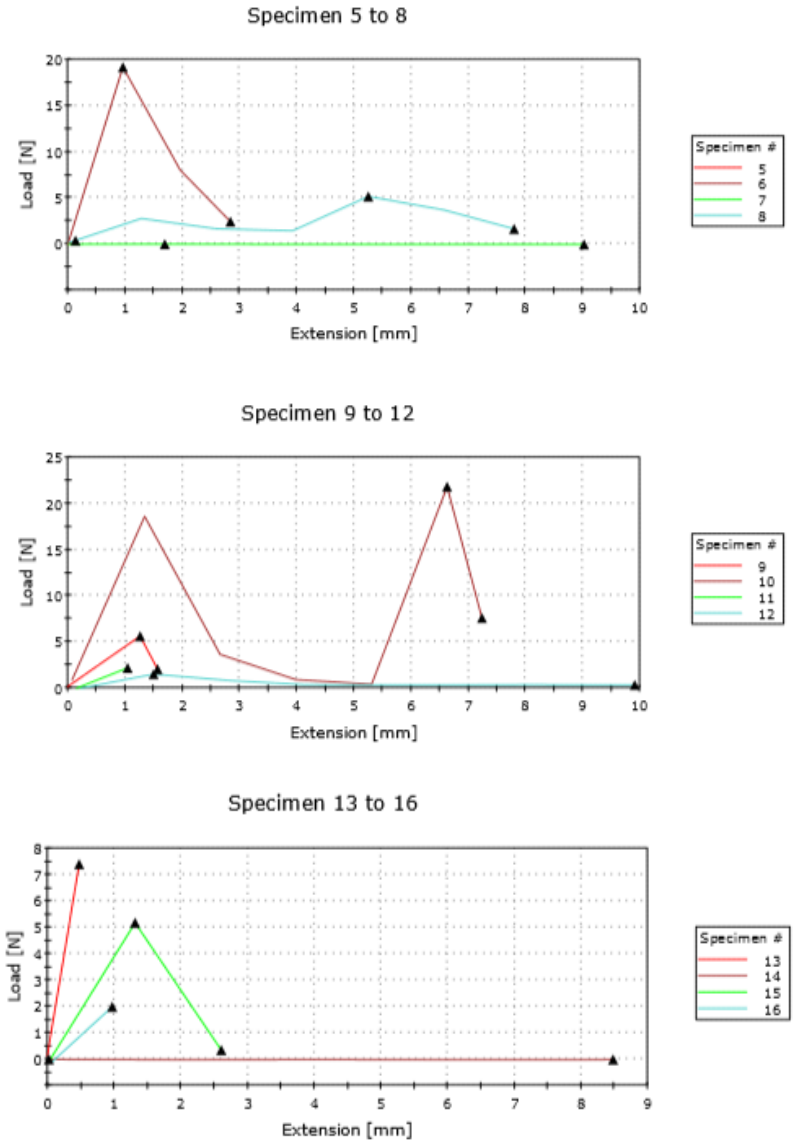


Figure 37. Instron Graph Results – Control Samples and RT samples

	Specimen label	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [%]	Load at Break (Standard) [N]	Tensile stress at Break (Standard) [MPa]	Tensile strain (Extension) at Break (Standard) [%]
1	PEO - Untreated - Sample 1	8.27	0.51	1.89	8.27	0.51	1.89
X 2	PEO Untreated Sample 1_2	5.77	0.36	4.80	2.61	0.16	58.95
X 3	PEO Untreated Sample 1_3	9.65	0.60	4.80	-0.54	-0.03	49.42
4	PEO Untreated Sample 1_4	4.30	0.27	4.81	1.73	0.11	6.06
5	PEO Untreated Sample 1_5	0.40	0.02	0.52	0.40	0.02	0.52
6	PEO RT Sample 1	19.11	0.75	3.20	2.44	0.10	9.46
7	PEO RT Sample 1_2	0.01	0.00	6.38	-0.01	0.00	34.10
8	PEO RT Sample 2	5.17	0.32	19.80	1.66	0.10	29.45
9	PEO RT Sample 3	5.65	0.40	4.80	2.09	0.15	5.95
10	PEO RT Sample 2_2	21.79	1.53	24.81	7.62	0.53	27.10
11	PEO RT Test 3_3	2.21	0.16	3.38	2.21	0.16	3.38
12	PEO RT Sample 3_4	1.55	0.11	4.80	0.39	0.03	36.61
13	PEO Untreated Sample 4	7.38	0.46	1.81	7.38	0.46	1.81

	Specimen label	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [%]	Load at Break (Standard) [N]	Tensile stress at Break (Standard) [MPa]	Tensile strain (Extension) at Break (Standard) [%]
14	PEO Untreated Sample 4_2	0.00	0.00	0.00	-0.01	0.00	32.02
15	PEO Untreated Sample 4_3	5.16	0.32	4.80	0.35	0.02	9.70
16	PEO Untreated Sample 4_4	1.99	0.12	3.42	1.99	0.12	3.42
Coefficient of variation		113.27816	113.28867	119.23135	111.98031	115.37272	96.69239
Maximum		21.79	1.53	24.81	8.27	0.53	36.61
Mean		5.93	0.35	6.03	2.61	0.16	14.39
Median		4.73	0.29	4.11	1.86	0.11	7.76
Minimum		0.00	0.00	0.00	-0.01	0.00	0.52
Range		21.78	1.53	24.81	8.27	0.53	36.08
Standard deviation		6.71402	0.40149	7.18878	2.92098	0.19015	13.91274
Mean + 1 SD		12.64	0.76	13.22	5.53	0.35	28.30
Mean - 1 SD		-0.79	-0.05	-1.16	-0.31	-0.03	0.48

Table xx. Instron Raw Data – Control samples and RT samples

55°C Samples

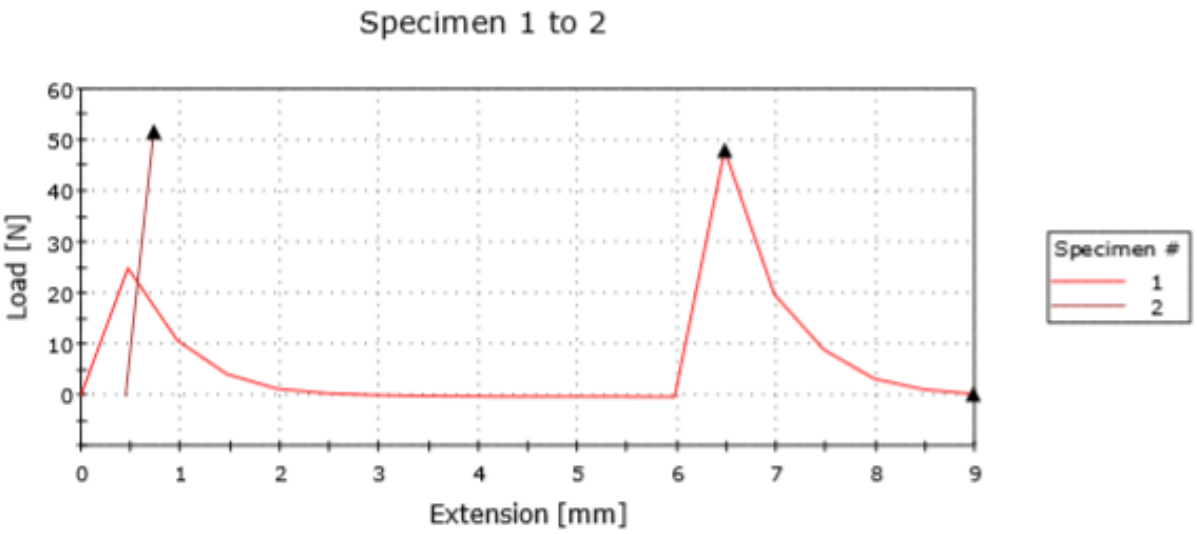


Figure 38. Instron Graph Results – 55°C

	Specimen label	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [%]	Load at Break (Standard) [N]	Tensile stress at Break (Standard) [MPa]	Tensile strain (Extension) at Break (Standard) [%]
1	Sample 3 - 55C	48.04	2.40	21.57	0.33	0.02	29.91
2	Sample 3 - 55C again	51.60	2.58	0.94	51.60	2.58	0.94
Coefficient of variation		5.05184	5.05183	129.55564	139.64067	139.64067	132.76035
Maximum		51.60	2.58	21.57	51.60	2.58	29.91
Mean		49.82	2.49	11.26	25.97	1.30	15.43
Median		49.82	2.49	11.26	25.97	1.30	15.43
Minimum		48.04	2.40	0.94	0.33	0.02	0.94
Range		3.56	0.18	20.63	51.28	2.56	28.96
Standard deviation		2.51707	0.12585	14.58714	36.25877	1.81294	20.47897
Mean + 1 SD		52.34	2.62	25.85	62.22	3.11	35.90
Mean - 1 SD		47.31	2.37	-3.33	-10.29	-0.51	-5.05

Table xx. Instron Raw Data – 55°C

62°C Samples

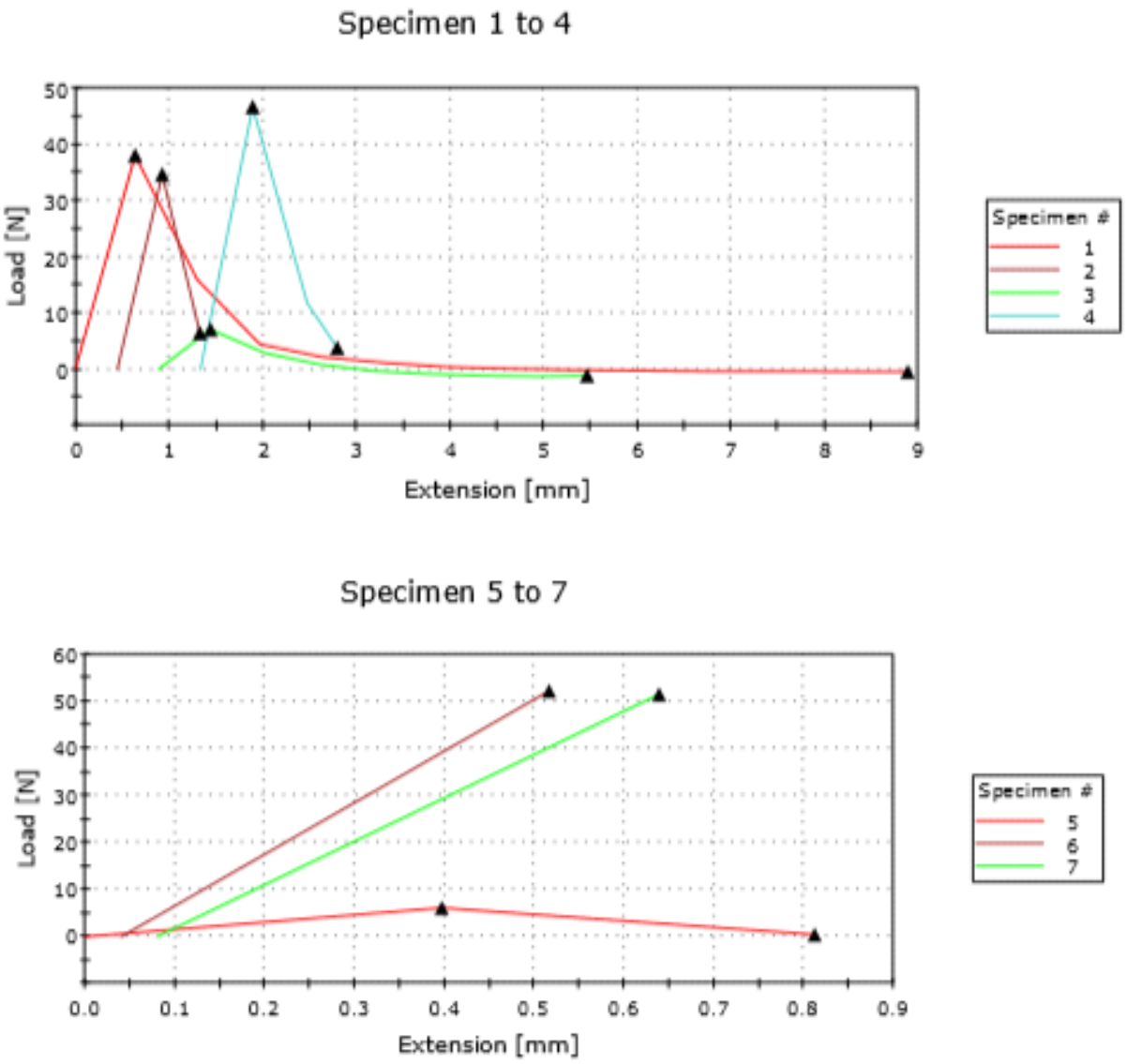


Figure 39. Instron Graph Results – 62°C

	Specimen label	Maximum Load [N]	Tensile stress at Maximum Load [MPa]	Tensile strain (Extension) at Maximum Load [%]	Load at Break (Standard) [N]	Tensile stress at Break (Standard) [MPa]	Tensile strain (Extension) at Break (Standard) [%]
1	Sample 1 - 62C	38.01	1.81	1.58	-0.38	-0.02	22.20
2	Sample 1 - 62C again	34.64	1.65	1.60	6.50	0.31	2.94
3	Sample 2 - 62C	7.16	0.34	1.57	-1.04	-0.05	13.06
4	Sample 2 - 62C again	46.62	2.22	1.59	3.88	0.18	4.17
5	Sample 2 - 2x again	6.12	0.29	1.59	0.43	0.02	3.24
6	Sample 2 - 62C 2x again	52.16	2.48	2.38	52.16	2.48	2.38
7	Sample 3 - 62C	51.47	2.45	1.39	51.47	2.45	1.39
Coefficient of variation		58.12816	58.12816	19.13186	151.80364	151.80364	109.66075
Maximum		52.16	2.48	2.38	52.16	2.48	22.20
Mean		33.74	1.61	1.67	16.14	0.77	7.06
Median		38.01	1.81	1.59	3.88	0.18	3.24
Minimum		6.12	0.29	1.39	-1.04	-0.05	1.39
Range		46.04	2.19	0.98	53.20	2.53	20.81
Standard deviation		19.61254	0.93393	0.31976	24.50821	1.16706	7.73727
Mean + 1 SD		53.35	2.54	1.99	40.65	1.94	14.79
Mean - 1 SD		14.13	0.67	1.35	-8.36	-0.40	-0.68

Table xx. Instron Raw Data – 62°C

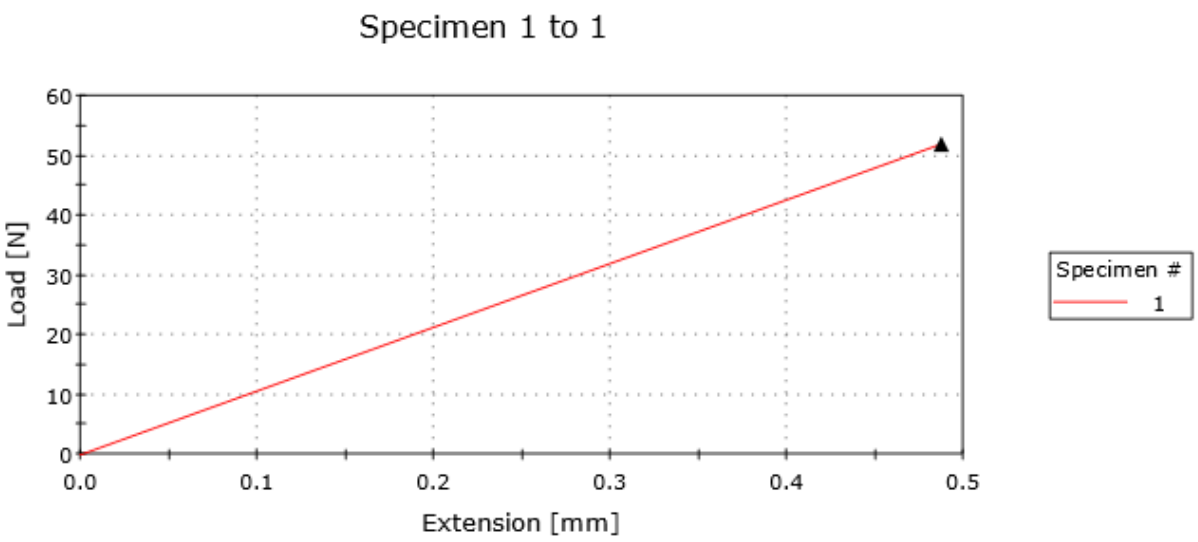


Figure 40. Instron Graph Results – 62°C