Storage of Ingeo™ Preforms

NatureWorks LLC

This bulletin is intended for use only as a tool to help provide information and address questions that may pertain to the time preforms made from Ingeo biopolymer are stored before being blown on a reheat stretch blow molding (RSB) machine. An experimental approach may be needed to evaluate any storage or fit-for-use requirements.

Injection stretch blow molded (ISBM) bottles made from Ingeo biopolymer have similar attributes to bottles made from PET. They look and feel similar to each other with regard to clarity and strength. Also, bottles made from these two resins are made essentially the same way. First, a preform is injection molded. Then, the amorphous preform is conditioned or reheated by infrared lamps in the blow molding machine and subsequently blown into a bottle mold by the use of a stretch rod and high pressure air. Inflation of the heated preform by high pressure air causes the polymer to stretch and orientate, allowing for stress induced crystallization to occur. Because of polymer orientation, the bottles have improved physical properties which allow it to be used in a wide variety of bottle packaging applications.

Despite similarities between Ingeo biopolymer and PET, there are some differences. One key difference is processing temperature. Typical melt processing temperatures for Ingeo biopolymer range from 200-230°C versus 270-290°C for PET. The glass transition temperature of Ingeo biopolymer is also about 15°C lower than typical bottle grade PET. This lower glass transition temperature means that an Ingeo preform requires a lower temperature for blowing than PET. This difference in glass transition temperature also means that preforms made out of Ingeo biopolymer are affected by environmental storage conditions in a similar, but slightly different, manner as compared to PET preforms.

Storage Conditions

Resin

Ingeo biopolymer is hydroscopic and should be stored in an environment designed to minimize moisture uptake. The product should be stored in a cool place, out of direct sunlight, at temperatures below 122°F (50°C). Boxed material should be kept in sealed containers until ready for loading into the blending and/or drying system. Bulk resin stored in silos or hoppers for extended periods (more than six hours) should be kept purged with dry air or nitrogen to minimize moisture uptake. In the case of outside storage, if the product is supplied in boxes or other non-bulk containers, the unopened container should be brought into the warehouse or production area and allowed to equilibrate for a minimum of 24 hours before opening to eliminate potential condensation. Refer to our “Crystallizing and Drying” bulletin for more information on proper resin drying and storage.

Preforms

Temperature and humidity are important parameters affecting a plastic packaging article’s ability to retain and hold its shape and integrity during shipping and storage. This is especially true of preforms, since after injection molding parts are amorphous. For example, uncrystallized Ingeo resin becomes soft, tacky, and clumps when its temperature reaches approximately 55-60°C (131-140°F). This is Ingeo biopolymer's glass transition temperature, the point at which the amorphous material begins to soften. This softening point is similar for amorphous preforms. The onset of this thermal change actually occurs lower than this temperature, and will also be affected by the humidity or moisture level in the air.

Preforms made out of Ingeo biopolymer should be stored in a cool place and out of direct sunlight, at temperatures below 104°F (40°C). Humidity will also have an impact on how the preforms will respond to deformation. The biggest concern is preforms at the bottom of the gaylord, where weight and compression could cause high load and deform the preforms. This is especially true at higher temperatures and humidities. The preform finish is somewhat more susceptible to deformation due to it being typically less thick than the body. Deformation of the finish could cause ovalization to occur. Ovalization occurs when the circular finish of a preform becomes slightly or moderately oval in shape due to high temperature, compressive forces, and/or humidity. If the ovalization is significant enough, the round blow nozzle on the blow molding machine cannot fit into the internal diameter of the finish. This could result in preforms with cracked finishes after blowing or preforms not being able to fit on the mandrels.

NatureWorks has done internal studies at elevated temperatures and humidity’s to simulate storage conditions preforms may encounter during shipping or in a warehouse environment. These tests were done on preforms that are larger in weight, with the finish being larger in diameter than a conventional water type finish or something like a 28 mm PCO. However, the results of the simulations are presented here for reference.

Preform Lab Storage Study

Experimental

A preform storage experiment was done to simulate conditions of temperature, humidity, and compression using an environmental chamber, a hollow cylinder, and some weight.

Preforms were placed in the test lab cylinder to a specific height and a pre-determined amount of weight was placed on top of the cylinder and preforms. The amount of weight used was calculated based on simulating the compressive forces preforms my see in a gaylord size container. This testing apparatus was then placed into an environmental chamber set at specific temperature and humidity conditions. Storage times were done over a one week period, where preforms were exposed at these conditions 24 hours a day. This test was done on a preform that is larger in weight, with the finish being larger in diameter than a conventional water type finish or something like a 28 mm PCO.

The following conditions were used:

Temperature (°C)	30	35	40	45
Relative Humidity (%)				
15%	Pass	Pass	Pass	All slightly failed, not noticed with naked eye.
65%	Pass	Pass	1 out of 5, showed very slight failure	Fail
100%	Pass	Pass	1 out of 5, showed very slight failure	Fail

Summary

From the table above it can be seen that humidity does have a significant impact on how the preforms withstand an external load at a given temperature. A temperature of 40°C is the maximum temperature the preforms should be exposed, so as to avoid deformation of the finish during storage.

From this study, it was found that above 40°C at 15%, 65%, and 100% humidity, that failure occurred. At 40°C at various humidity's (15%, 65%, and 100%), there was no failures at 15% humidity and 1 out of 5 were only very slight failures at 65% and 100% humidity. (For reference, the failure rate is based on if calipers were able to fit inside the preforms around the entire circumference. If so, then it was given a "pass." If not, it was considered to fail the test.). At 35°C, all the preforms passed at the 15%, 65%, and 100% humidity conditions.

Preform Bulk Storage Study

Experimental

Since temperature is an important parameter in terms of affecting a packaging article's ability to retain and hold its shape and integrity during shipping and storage, various Ingeo preforms were subjected to the following temperature and storage scenarios for simulation to help understand heat distortion effects:

- To a temperature of 66°C (150°F), without load, in order to determine how the heat would affect the shape of a single preform(s)
- To simulate load deformation when multiple preforms would be stored in a large volume container, weights were added to the top of a specified number of preforms while in the oven.

- A larger-scale simulation was also performed using a 55 gallon fiber storage drum to try and simulate real-life load conditions preforms may see in a shipping/storage environment.

For a non-load scenario, single preforms were stood upright in the oven for thermal exposure.

To simulate a load, preforms were placed in a test lab cylinder to a specific height and a pre-determined amount of weight was placed on top of the cylinder and preforms. The amount of weight used was calculated based on simulating the compressive forces preforms my see in a gaylord size container. This testing apparatus was then placed into an environmental chamber set at specific temperature and humidity conditions.

For the above simulations, a Thermotron laboratory oven was used. The oven test consisted of subjecting the preforms to elevated temperatures. The temperature program took the preforms from 30°C to 66°C, using a heating rate of 2°C/min and then holding the preforms for an hour at the 66°C temperature. After, the samples were cooled and then taken out for observation. To understand how the preforms would hold up, the samples were tested at couple of other temperatures.

For the larger scale bulk simulation, Ingeo and PET preform temperature stability was compared using a barrel oven heater. Approximately 85 lbs of Ingeo preforms were put into a fiber drum and placed in the oven at 50°C, along with 85 lbs of PET performs placed in a separate fiber drum. The performs remained at this temperature for approximately 48 hours. After 48 hours, the performs were evaluated for heat deformation. After being evaluated, the performs were returned to the oven at 55°C. This procedure (a 5°C increase in temperature after 48 hours) was followed until the performs began showing significant heat distortion.

Summary

The data indicated that preform shrinkage at 66°C is minimal on freestanding preforms, without load. Higher temperatures were not run for this free-standing preform heat test.

However, lab-scale load tests on preforms showed deformation occurring. Chart 1 illustrates the number of preforms that deformed as a function of temperature. Temperatures were increased from 30°C to three different temperatures at a 2°C/minute heating rate and then held at the final temperature for an hour under a simulated load. The data indicates that the majority of the deformation starts to occur in the 50-60°C temperature range. This temperature region corresponds to Ingeo biopolymer's glass transition temperature (Tg), which is 55-60°C. The Tg is a thermal transition event in which amorphous chain mobility starts to significantly increase. At Tg, the plastic material changes from a glassy, rigid state to a more mobile, rubbery state. In general, this is how many thermoplastic materials respond when their Tg is approached. This physical property change not only occurs in preforms, but also other plastic, amorphous-type articles.

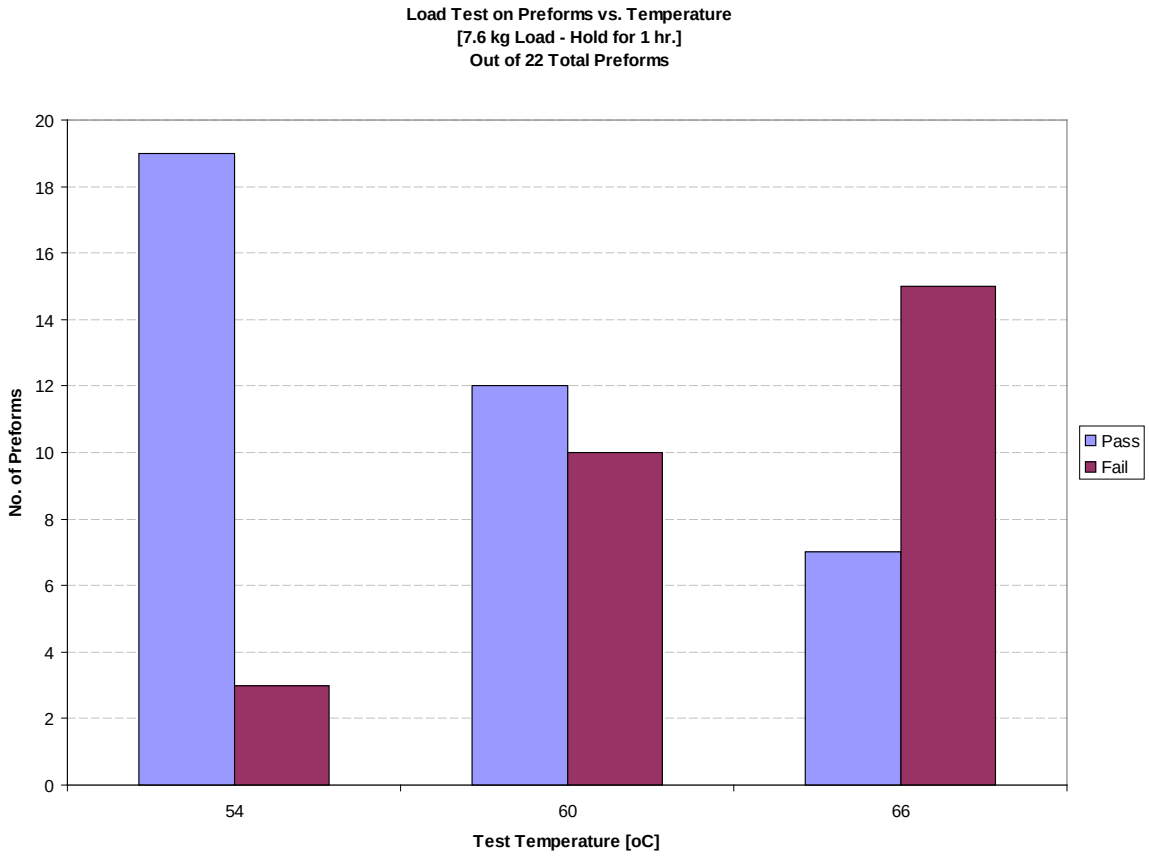

Chart 1 – Thermotron oven test on Ingeo preforms with load simulation

For the larger-scale bulk storage simulation, the Ingeo preform deformation results are similar to the Thermotron oven laboratory results. After 48 hours, the Ingeo and the PET performs were heat stable at both 50°C and 55°C. However, the PET preforms were also stable at 60, 65, and 70°C. The PET preforms began to fail at about 75°C, with about 2% failing - primarily around the finish area of the preform. At about 80°C, 60 lbs of the PET preforms were bad, and 25 lbs were still considered acceptable.

The Ingeo preforms began to significantly fail at about 60°C. At this temperature, the total weight of the fiber drum was 85 lbs. and 35 lbs. of the performs were found to have failed – a 41% failure rate. No significant preform deformation was noted at 55°C. A qualitative evaluation revealed performs at the bottom of the drum were affected the most by heat distortion. Deformation was mostly around the finish area. The finish area became deformed and was no longer a perfect circle. The perform height diminished by 2 to 3 mm on some samples. At 65°C for 48 hours, all the Ingeo performs failed.

Preform Storage & Aging

Aging is common to all polymers and storage temperature and humidity affects how fast this process occurs. Aging is the process whereby the polymer molecules are trapped or “frozen” when the plastic cools and solidifies during injection molding. The polymer chains are trapped in this non-equilibrium state, and over time, the polymer chains continue to pack and free volume relaxation occurs. With this relaxation, plastics undergo physical property changes such as an increase in polymer chain density, and increases in tensile and flexural yield stress, and a decrease in elongation and impact strength. Depending on storage conditions, this aging process occurs over different times and can affect the material’s physical properties, going from ductile to brittle behavior.

Preforms also start to absorb moisture after they are injection molded. The amount of moisture absorbed can have an effect how the Ingeo preforms stretch during the reheat-stretch-blow molding process. The increase in moisture can increase the

natural stretch ratio (NSR) of Ingeo preforms. During blow molding, this effect can be off-set by decreasing the blow molding temperature or the temperture the preforms are exposed to, to help bring the NSR back in line. However, if the preforms have too high a moisture level or are too old, then it may become very difficult, if not impossible, to establish a robust and consistent blow molding process.

Accelerated aging of preforms and bottles occurs at elevated storage temperatures and relative humidity levels. In general, it is poor supply chain practice to store Ingeo preforms or bottles in hot, humid conditions. Storage temperatures greater than 40°C should be avoided, along with high humidity levels. Best results can also be achieved when storage times are minimized.

Follow NatureWorks SmartCare Guidelines

Besides converters and suppliers establishing their own best practices, NatureWorks has developed suggested guidelines and check lists to ensure safe handling and storage of Ingeo based packages.

- Avoid high temperatures (below 40°C).
- Keep out of direct sunlight.
- Keep it cool. Store on a lower rack.
- Handle with care.

For more information on “Smart Care,” please visit www.natureworksllc.com/care.

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Safety and Handling Considerations

Material Safety Data (MSD) sheets for Ingeo biopolymers are available from NatureWorks LLC. MSD sheets are provided to help customers satisfy their own handling, safety, and disposal needs, and those that may be required by locally applicable health and safety regulations, such as OSHA (U.S.A.), MAK (Germany), or WHMIS (Canada). MSD sheets are updated regularly; therefore, please request and review the most current MSD sheets before handling or using any product.

The following comments apply only to Ingeo biopolymers; additives and processing aids used in fabrication and other materials used in finishing steps have their own safe-use profile and must be investigated separately.

Hazards and Handling Precautions

Ingeo biopolymers have a very low degree of toxicity and, under normal conditions of use, should pose no unusual problems from incidental ingestion, or eye and skin contact. However, caution is advised when handling, storing, using, or disposing of these resins, and good housekeeping and controlling of dusts are necessary for safe handling of product. Workers should be protected from the possibility of contact with molten resin during fabrication. Handling and fabrication of resins can result in the generation of vapors and dusts that may cause irritation to eyes and the upper respiratory tract. In dusty atmospheres, use an approved dust respirator. Pellets or beads may present a slipping hazard. Good general ventilation of the polymer processing area is recommended. At temperatures exceeding the polymer melt temperature (typically 170ºC), polymer can release fumes, which may contain fragments of the polymer, creating a potential to irritate eyes and mucous membranes. Good general ventilation should be sufficient for most conditions. Local exhaust ventilation is recommended for melt operations. Use safety glasses if there is a potential for exposure to particles which could cause mechanical injury to the eye. If vapor exposure causes eye discomfort, use a full-face respirator. No other precautions other than clean, body-covering clothing should be needed for handling Ingeo biopolymers. Use gloves with insulation for thermal protection when exposure to the melt is localized.

Combustibility

Ingeo biopolymers will burn. Clear to white smoke is produced when product burns. Toxic fumes are released under conditions of incomplete combustion. Do not permit dust to accumulate. Dust layers can be ignited by spontaneous combustion or other ignition sources. When suspended in air, dust can pose an explosion hazard. Firefighters should wear positive-pressure, self-contained breathing apparatuses and full protective equipment. Water or water fog is the preferred extinguishing medium. Foam, alcohol-resistant foam, carbon dioxide or dry chemicals may also be used. Soak thoroughly with water to cool and prevent re-ignition.

Disposal

DO NOT DUMP INTO ANY SEWERS, ON THE GROUND, OR INTO ANY BODY OF WATER. For unused or uncontaminated material, the preferred options include recycling into the process or sending to an industrial composting facility, if available; otherwise, send to an incinerator or other thermal destruction device. For used or contaminated material, the disposal options remain the same, although additional evaluation is required. (For example, in the U.S.A., see 40 CFR, Part 261, “Identification and Listing of Hazardous Waste.”) All disposal methods must be in compliance with Federal, State/Provincial, and local laws and regulations.

Environmental Concerns

Generally speaking, lost pellets are not a problem in the environment except under unusual circumstances when they enter the marine environment. They are benign in terms of their physical environmental impact, but if ingested by waterfowl or aquatic life, they may mechanically cause adverse effects. Spills should be minimized, and they should be cleaned up when they happen. Plastics should not be discarded into the ocean or any other body of water.

Product Stewardship

NatureWorks LLC has a fundamental duty to all those that make and use our products, and for the environment in which we live. This duty is the basis for our Product Stewardship philosophy, by which we assess the health and environmental information on our products and their intended use, then take appropriate steps to protect the environment and the health of our employees and the public.

Customer Notice

NatureWorks LLC encourages its customers and potential users of its products to review their applications for such products from the standpoint of human health and environmental quality. To help ensure our products are not used in ways for which they were not intended or tested, our personnel will assist customers in dealing with ecological and product safety considerations. Your sales representative can arrange the proper contacts. NatureWorks LLC literature, including Material Safety Data sheets, should be consulted prior to the use of the company’s products. These are available from your NatureWorks LLC representative.

For additional information please contact NatureWorks via our website on the tab called FAQ’s or by clicking here.

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