MonoSol Technical Bulletin: Thermoforming Considerations for Meeting European Union Regulations

There is concern about the accidental ingestion of liquid laundry detergent capsules by children. Because of this concern, the European Union (EU) amended CLP (Classification Labeling Packaging) Regulation 1272/2008 with EU Regulation 1297/2014. EU Regulation 1297/2014 was published on December 6, 2014 and it went into effect on December 26, 2014. Companies producing liquid laundry detergent single unit dose capsules must comply by June 1, 2015. If companies produced and shipped non-compliant capsules prior to June 1, 2015, then they have until December 31, 2015 to remove the non-compliant capsules from store shelves.

EU Regulation 1297/2014 adds three new CLP requirements to the soluble film of the liquid laundry detergent capsule. First, the soluble film shall "contain **an aversive agent** (such as a bittering agent) in a concentration which is safe, and which **elicits oral repulsive behavior within a maximum time of 6 seconds.**" Second, the capsule shall "**retain its liquid content for at least 30 seconds** when the soluble packaging is placed in water at 20C." Third, the capsule shall "**resist mechanical compressive strength of at least 300 Newton** under standard test conditions."

With regards to the first requirement, MonoSol, LLC offers water soluble films with denatonium benzoate, a well-known aversive and bittering agent reported to meet CLP requirements.

With regards to the second requirement, it is well established that thicker films will take more time to dissolve than thinner films. It is also reasonable to assume that thin areas in the capsule wall should be avoided so that the 30 second requirement can be met. Therefore the process of making thermoformed capsules must be well controlled so that there are no thin areas in the capsule walls.

With regards to the third requirement, it is reasonable to assume that thinner capsule walls will rupture more easily than thicker capsule walls. Therefore the process of making capsules must be well controlled so that there are no thin areas in the capsule walls. Furthermore, weak seals will rupture more easily than strong seals. Therefore, strong seals between the top and bottom films of the capsule, via heat or water solution processes, are also necessary for meeting the 300 Newton requirement.

Practitioners skilled in the art of making thermoformed capsules routinely control the following parameters in order to make capsules that do not contain thin spots:

- **Temperature of the film:** "The baseline objective for all heating is to create uniform heating throughout the sheet but this is hard to achieve." (Throne, 2008, p.101). Non-uniform temperatures in the film will lead to non-uniform stretching, which will lead to thick areas and thin areas in the capsule walls. Also, "when the film is heated, the plastic material will yield to natural thermal expansion, loss of crystallinity, and relief of molecular orientation, all of which tend to cause sheet movement and sheet distortion." (Florian, 1996, p.46) Therefore, one must choose the temperature of the film carefully; warm enough so that the film can be stretched to form the pocket but not too warm or else sheet movement and sheet distortion can occur. The temperature of the film should be set to provide the appropriate balance of viscous (non-recoverable) and elastic (recoverable) deformation.

- **Temperature of the mold:** The mold temperature must be as uniform as possible. Hot spots in the mold could lead to thin areas in the capsule walls, while cold spots in the mold could prevent stretching of the film.
- **Sharp bends in the mold:** Sharp bends in the mold should be minimized as much as possible. When the film is formed or pulled into the mold, a bending moment is applied on the film when the film encounters a bend in the mold. "As the bending moment further increases, plastic zones develop in the member." (Beer, Johnston, DeWolf, Mazurek, 2012, p.256) The more extreme the angle, the weaker the film becomes. Weak points in soluble film can lead to faster dissolving times.
- **Shape of the mold:** Unique characteristics of the mold (other than sharp bends) can also cause thinning of the walls. "The portion of the product that is formed last is the thinnest, most oriented, and weakest" which can explain the excessive thinning in the bottom corners of some products. (Throne, 2008, p.11) In corroboration, "the final thermoformed structure tends to have thick walls in the places where the sheet touched the mold first and thin walls where it touched last." (Szegda, 2009, p.22) Therefore, the mold should be designed so that the film forms into the mold uniformly and simultaneously in order to avoid thin spots.
- **Film restraints:** "The unheld and unsupported side of the sheet will usually distort and will tend to pull away from the clamped area. This causes a shift in material thickness, with thickening and gathering that concentrates towards the center of the sheet." (Florian, 1996, p.46) Therefore, one must understand how the film is restrained during thermoforming so that wall thickness inconsistencies can be minimized.
- **Vacuum (when it is applied and how much):** "The rushing air movement created by the suction could very well cause chilling of the heated thermoplastic sheet, which would affect its stretching, and ultimately, forming results." (Florian, 1988, p.91) Therefore, while vacuum is very useful or even essential in pulling the film into the cavity to form a pocket, vacuum can also lead to inconsistencies in temperature, and in turn, inconsistencies in capsule wall thicknesses.
- **Vent holes:** "Incorrect placement or slightly clogged vent holes result in trapped air pockets. The trapped air will cause different amounts of stretching in different areas," resulting in variation of wall thickness. (Florian, 1988, p.283) Vent holes (together with operating conditions) should be selected (in size and location) to allow for adequate air flow rates and not be prone to secondary thermoforming or stress concentrations.
- **Vacuum holes:** The water soluble film is sometimes pulled into the vacuum hole during the production of the capsule. If so, then a thin area or a weak spot is formed, leading to a lower

chance of meeting EU Regulation 1297/2014. Careful control of the vacuum conditions is required.

There are many patents and patent applications that specify the range of conditions for successful thermoforming and capsule production. Temperature ranges, mold shapes specifications, vacuum conditions, and vent hole placements can be found in the prior art. Examples of patents and patent applications include EP 1311429 B1 and EP 1375637 A1.

Water soluble films are a complex mixture of polymer, water, and additives. Practitioners skilled in the art of making capsules deliberately select the water soluble film that is suitable for their equipment and for their specific liquid laundry detergent formulation. Practitioners skilled in the art carefully control environmental conditions so that film properties do not change. If film properties change, then capsule production could be negatively affected or capsules may no longer meet the EU Regulation 1297/2014. Controlling the variation in ambient relative humidity is especially important in the successful use of water soluble films.

- **Moisture content:** A water soluble film with variable moisture content will exhibit variable properties. "Materials with different initial moisture content will produce different stress-strain curve in tensile test, thus the elastic modulus, yield strength, and tangent modulus will be different." (Szegda, 2009, p.128) In addition, "moisture loss affects the deformation … moisture loss results in material shrinkage and this will affect the resultant shape or thickness distribution." Therefore, it is critical to control the moisture content of the film and the capsule.
- **Crystallinity:** "A broad softening range gives a wide processing window for forming into a final part, without excessive sag in the oven. The abrupt melting of a crystalline plastic leads to a much narrower temperature window over which thermoforming can occur." (Rosen, 2002, p.23) The liquid laundry detergent inside the capsule and/or the environment outside of the capsule can affect the crystallinity of the film, therefore both must be controlled.
- **Thermal expansion:** Thermal expansion is a characteristic that can affect thickness. Uneven thermal expansion can result in uneven film thickness. The presence of processing aids, plasticizers, solvents, or dissolved gases can increase thermal expansion. Crystallinity, orientation, hydrogen bonding, or crosslinking can reduce thermal expansion. (Throne, 2008, p.36) The liquid laundry detergent inside the capsule and/or the environment outside of the capsule can affect the thermal expansion of the film, therefore both must be controlled.

In summary, producers of single unit dose liquid laundry detergent capsules by the thermoforming process can vary a number of processing parameters so that capsules can meet EU Regulation 1297/2014. Producers can look into the prior art or consult with their suppliers for guidance on processing parameters. Producers must also control the environmental conditions of their processing plant so that the properties of the water soluble films and single unit dose liquid laundry detergent capsules are not negatively affected.

References

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