Finding a Solution to Crosslinking during Melt Flow Analysis

On occasion, routine tests can become anything but routine, yielding surprising results that require an extra degree of analysis and interpretation. This was the case for Teel Analytical Laboratory (TAL) during what appeared to be routine melt flow analysis.


Melt flow analysis testing device

TAL frequently performs melt flow analysis for customers wanting to determine a polymer's melt flow rate (MFR), a measure of its molecular weight or grade. Knowing a material's MFR can help customers understand its properties and its behavior under different conditions. Melt flow analysis involves heating and applying weight to the material to measure the rate at which the melt flows out of a die, measured in grams of polymer per 10 minutes (g/10 min). Different materials respond to the application of heat and weight differently depending on their molecular weight or structure, which is then used to determine MFR. Plastics with lower molecular weight, shorter polymer chains, and less molecular branching flow easier and faster. TAL can also develop custom method conditions to answer a variety of customer questions about a material's properties and melt flow rate, such as what parameters are required to achieve a specific flow rate with a given material. Depending on the information the customer is looking for, melt flow testing can be completed in a few hours or less.

The Problem


Customer-provided polypropylene resin

TAL assumed this would be the case when a customer requested a set of tests on a polypropylene material and samples made from it. The customer provided some virgin material (technically referred to as polypropylene rubber-based thermoplastic vulcanized resin) and four different manufactured samples. The customer first wanted to see if TAL could find the heat and weight parameters to achieve a melt flow rate of 5-25 g/10 min with the virgin material. The customer then requested TAL use the same parameters to test the four processed samples to see how their processing methods affected the melt flow rate of the parts compared to the virgin material.

Discovering and Solving Crosslinking

The ASTM standard for polypropylene material suggested 230°C and 2.16 kilograms of pressure to achieve the customer's requested melt flow rate, so TAL started with those parameters. Surprisingly, the melt flow rate under these conditions turned out to be very slow – less than 1 g/10 min.

TAL decided to add successively higher weight to try to increase the flow while maintaining the 230°C temperature. TAL tried testing at 3.8, then 5, and then 10 kilograms. Instead of creating a successively higher melt flow rate as would be typical, the melt flow rate stayed the same or even decreased at the higher weights. Clearly, something unusual was happening.

TAL then decided to return to the original weight of 2.16 kilograms and successively increase the temperature instead. TAL tried 240°C, then 250°C, and then even 300°C (at which point the material should have been entirely liquid). At each new temperature, again, the resin's MFR stayed the same or even decreased.

Given its strange behavior under the parameters tried thus far, TAL concluded the material must be undergoing the unusual process of crosslinking, something the lab had never experienced in the course of testing before. Crosslinking is a chemical reaction in polymers that causes their typically separate molecular strands to link together. When linked, the strands make the material stronger and more resistant to deformation. Crosslinking can be brought on in any polymer by the right combination of pressure and heat. In this case, TAL needed to find the right combination to avoid it.

TAL hoped to isolate one element, heat or pressure, that it could determine was primarily responsible for the crosslinking and adjust it. TAL started by decreasing the temperature to 190°C and using the ASTM-suggested weight of 2.16 kilograms. Under these conditions, the material started to melt and flow. It was apparent that high heat was the factor primarily causing the crosslinking. All TAL had to do at this point was to increase the weight to 12.5 kilograms at 190°C to achieve the customer's desired flow rate of 5-25 g/10 min.

The last step was to test the customer's provided samples under the same conditions (190°C and 12.5 kilograms) to see if their processing methods affected flow rate. Below are the results of the testing for the virgin resin and each of the four samples.

Melt Flow Rate of Virgin Material and Processed Samples

Sample	Melt Flow Rate (g/10 min)
Virgin PP Resin	9.647
Sample 1	0.760
Sample 2	1.207
Sample 3	2.853
Sample 4	3.873

The processing methods used on the sample parts had an evident impact on flow rate, decreasing it under the established parameters significantly.

Conclusion

TAL was able to not only to achieve the customer's desired melt flow rate and provide them the information about the processed parts they were looking for, but also alert them to the tendency of the material to crosslink under certain conditions. Instead of taking the usual time of several hours, this melt flow analysis took several days, requiring TAL to apply the standard melt flow analytical tools and methods in new ways to solve the crosslinking problem for the customer. TAL further gained experience adjusting parameters in counterintuitive ways to overcome crosslinking that can help during root cause analysis in the future.