Levasil® colloidal silica improving properties of polyesters

In patents and scientific literature, it is shown that adding small amounts of colloidal silica in the polymerization of polyesters can improve the properties of products made from the resulting resins. Improved abrasion, scratch, and heat resistance as well as improved barrier properties have been reported for PET films and bottles. Improved mechanical properties and better processability is claimed for e.g. fiber applications. Here we present results from a screening study showing that our silane modified product Levasil® CC301 is the optimal grade for this application.

The importance of good dispersion

Silica can be found in many forms and can be used both as a dry powder as well as in dispersions. Levasil® is a colloidal silica and thus manufactured and delivered in dispersion. Since it has never been dried it contains discrete nano particles, free from agglomerates, which is very important to achieve a good dispersion in the final application. Dry silica powders can be incorporated in a polyester resin afterwards, by a separate compounding process. However, to get the best result with a high impact using a low dosage, we recommend using a colloidal dispersion as part of the ingredients of the polymerization process. It can be concluded from literature that this strategy will maximize dispersion, as during polymerization the particles will be grafted with polyester chains, preventing agglomeration.

Screening study

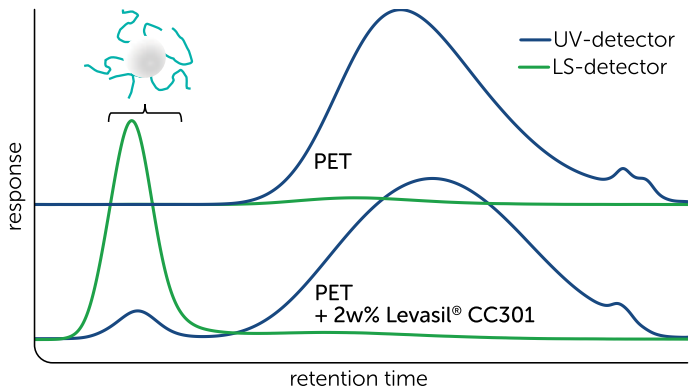
To better understand the underlying mechanisms and to identify the optimal product for this application, we have conducted a screening laboratory study on the use of Levasil® colloidal silica as an additive in PET synthesis. Three products were evaluated: a standard grade sodium stabilized colloidal silica, a deionized product and a silane modified colloidal silica, Levasil® CC301.
The PET-samples were prepared analogously as in industrial practice, starting with a dispersion of terephthalic acid in ethylene glycol, but now including a certain amount of colloidal silica, subsequently distilling of the water and the excess of ethylene glycol.

In this screening study, the resulting products were only characterized using Size Exclusion Chromatography (SEC) and Differential Scanning Calorimetry (DSC).

In figure 1, a typical result of the SEC-analysis is shown. Based on the response of the UV-detector, highlighting the presence of terephthalic acid moieties, and the LS detector, highlighting the presence of high density scatterers, we can conclude that we indeed deal with a polyester resin containing individual silica-particles that are grafted with polyester chains. All samples made yielded similar results.

In figure 2, DSC-curves are shown for a PET-resin made without a Levasil® and a PET-resin containing 2w% of Levasil® CC301, illustrating that the presence of a small amount of silica has a large influence on the crystallization. The other Levasil® grades tested also influenced the crystallization but clearly to a lesser extent. Another remarkable finding was that the crystallization was significantly more affected when using 2w% of a Levasil® grade as compared to 4w%.

Figure 1 - SEC-analysis for a **PET-resin made without colloidal silica and a PET-resin containing 2w% of Levasil® CC301**



The unique function of Levasil® CC301

The difference in performance between the silane modified Levasil® CC301 and the other grades is related to the fact that it provides a far more stable linkage with the polyester chains upon reaction with the C-OH groups. The other grades yield Si-O-C linkages, which are notoriously sensitive towards hydrolysis.

Conclusions

We have clearly shown that addition of colloidal silica to the PET polymerization will result in particles being linked to the polymer and that this influences the crystallization of the polymer. An interesting finding was also that the effect was stronger when using less silica.

Because most properties are directly related to the crystalline-amorphous morphology of polyesters, the effect of incorporating silica on crystallization is the main reason why such a low dosage of an additive can significantly influence the material properties, as claimed in various patents.

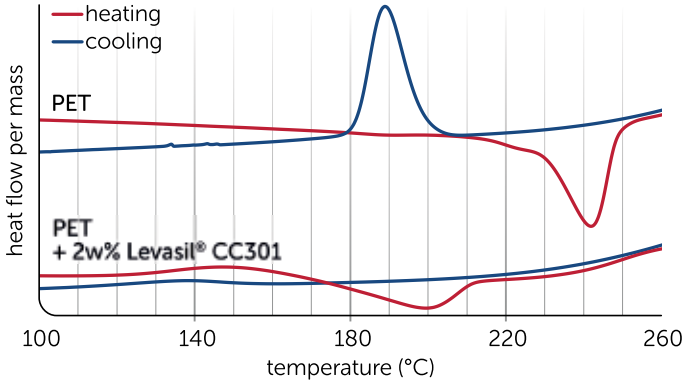
The next step - effect on material properties

This screening study shows the potential of using Levasil® in tuning polyester properties. Since it yields hydrolytically stable linkages with the resin, Levasil® CC301 is the best choice.

The effect on the properties of an end-product is strongly dependent on the processing conditions. Hence, to be able to draw further conclusions on the technical potential, additional application focused studies are needed in cooperation with partners having the right expertise and infrastructure.

Please contact us, if you are interested to learn more on the results of our screening studies and consider exploring the potential of this technology for your applications.

Figure 2 - **DSC-curves for a PET-resin made without colloidal silica and a PET-resin containing 2w% of Levasil® CC301**



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