How to determine the real cause for production failure using advanced analytical techniques?

Method Development for a Superplasticizer Production Failure Investigation

Executive Summary

There are various sources for production failures in polymer industry that results in poor performance in the produced polymer. Production failure in production design and development stage is rather common, but it is much costlier when it happens because of unoptimized polymerization processes for developed products. The cost includes the cost for disposal or re-use of the failed batches, product liability, loss of brand credibility & competitive edge, expensive recalls and warranty claims. Modern analytical techniques can be used to determine the real cause for production failure not only in design and development stage but also to optimize the polymerization process and consequently minimize the related cost of production failure. This article demonstrates how analytical scientists in the Expert Capability Center Deventer (ECCD) are solving cutting edge analytical challenges by case study of superplasticizer production failure investigation.

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A superplasticizer is a copolymer mixture which is used as dispersant in, for example, concrete and mortars, to avoid particle segregation and to improve the flow characteristics. Polycarboxylate ethers, or PCEs, are a new generation of superplasticizer, and consist of an acrylic polymer backbone, grafted with polyethylene glycol side chains. The rheological properties induced by a PCE superplasticizer are dependent on the copolymer composition, functional groups and size and molecular shape. Therefore, careful control over the production process is of paramount importance and identifying the cause of production failures is key to maintaining high-quality products. The challenge is to choose the right analytical techniques to identify the root cause of production failures.

During the production of a PCE product, one batch was produced in specification, but appeared to have poor application performance. Scientists in ECCD have analyzed the molecular weight distribution by triple detection Size Exclusion Chromatography and the monomer composition by NMR. The results of the investigation showed noticeable differences in the molecular weight distribution and monomer composition of the sample and reference. Based on the combined findings of the NMR and SEC analyses it was recommended to the production team to re-adjust the polymerization process to achieve product consistency.

The advanced multi-analytical technique method development process includes several steps

Multi-analytical approach for superplasticizer production failure investigation

1) What are PCE superplasticizers

Polycarboxylate ethers, or PCEs, are composed of an acrylic polymer backbone grafted with polyethylene glycol side chains (Figure 1). The acrylic polymer backbone contains carboxylate groups that act as anchoring groups on the surface of the particles present in the cement matrix. The performance properties of such superplasticizers depend on several parameters, such as the ratio of the acrylic and polyethylene glycol chains, the molecular weight distribution of the PCE's, and the number of carboxylate groups. During production, the product quality is monitored by continuous analysis of general product specifications, such as acid value and viscosity, that can be measured in a relatively short time.

Finding the cause for differences in application performance properties between product batches that meet the product specifications requires more in-depth analysis of the product batches, which are not part of the routine QC control.

Two batches of the same product were found to have different application properties, although both batches were produced within specification. The origin of the different application properties was investigated in the ECCD laboratories. The chemical compositions and molecular weight distributions of both batches were analyzed and compared.

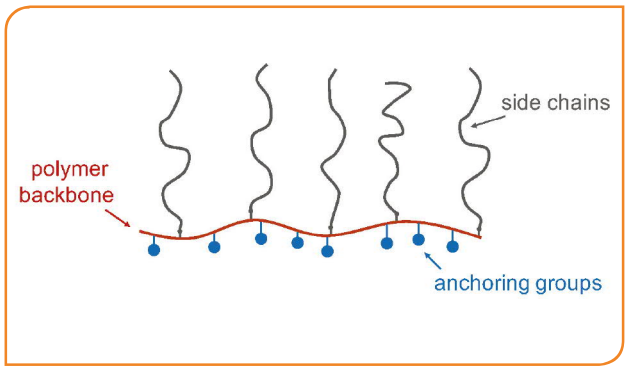

Figure 1: Polycarboxylate ether superplasticizer

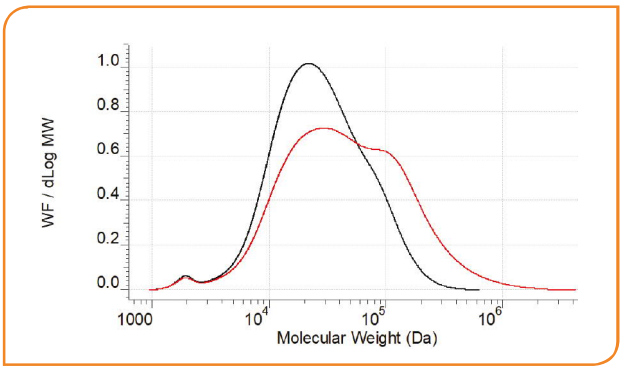

Figure 2: Overlay of the MWD of the sample (red) and the reference sample (black) as determined by triple detection SEC.

2) Analysis of molecular weight distribution

As the copolymer is easily soluble in water, aqueous Size Exclusion Chromatography (SEC) with triple detection is the method of choice to determine the molecular weight distribution of the polymer. With triple detection SEC both absolute molecular weights and structure information of the polymers are obtained. The SEC analysis showed that the failed sample has a higher average molecular weight than the reference batch, and contains a fraction with high molecular weight, which is absent in the reference batch (Figure 2).

3) Analysis of the chemical composition

Comparing the chemical compositions of the wrong PCE batch with the good batch by 1H NMR showed a difference in composition between the two batches, with a considerable lower amount of EO groups in the failed batch.
Further analysis by DOSY NMR revealed that the high-MW fraction in the failed batch consisted largely of polyethylene glycol grafted to the acrylic polymer, clearly indicating that the amount of initiator used was not correct.

Table 1 Results of the SEC and NMR analyses.

SAMPLE	TRIPLE SEC MN / MW (G/MOL)	WEIGHT RATIO EO / METHACRYLIC	HYDRODYNAMIC RADIUS (NM)	INTRINSIC VISCOSITY (DL/G)
Production batch	20700 / 83900	54 / 46	8.5	0.692
Reference	17000 / 38700	50 / 50	6.0	0.434

Conclusion

Investigating the root cause of production failures can be very challenging detective work. The ECCD has an excellent track record in developing effective and accurate failure analysis methods as shown in this superplasticizer production case study. These types of method development projects require a deep knowledge of organic chemistry, polymer science and analytical science. This white paper exemplifies that ECCD scientists have the right knowledge and technical capability to make detailed production failure investigations possible and deliver the real cause so the customer can rapidly take the best corrective actions.

Recommendation to the customer

To adjust the level of polymerization initiator and monomer ratio in the production process.

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