Analysis of Epoxy Resins using Gel Permeation Chromatography

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Polymer /chemical industry

Abstract

Epoxy resins are produced through the reaction of bisphenol A and epichlorohydrin to macromolecules, (figure 1). These products are then used in the production of duroplasts, which are highly resistant against oil and fuel. Examples of products made from this material include, casting resins for the electrical industry, laminates for cars and airplanes, inner coatings for containers and tubings for the chemical industry. In 1987 85 000 tons were used in Germany.

To ensure the highest quality, molecular weight (MW) data have to be evaluated for each batch of produced polymer. Gel Permeation Chromatography is an analytical tool used to characterize polymers which are soluble in organic solvents.

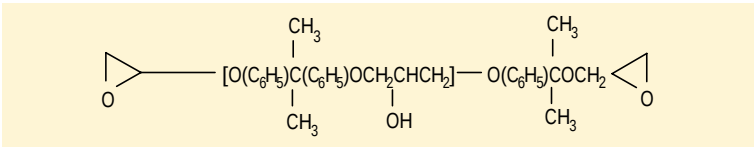

Figure 1
Epoxy resins

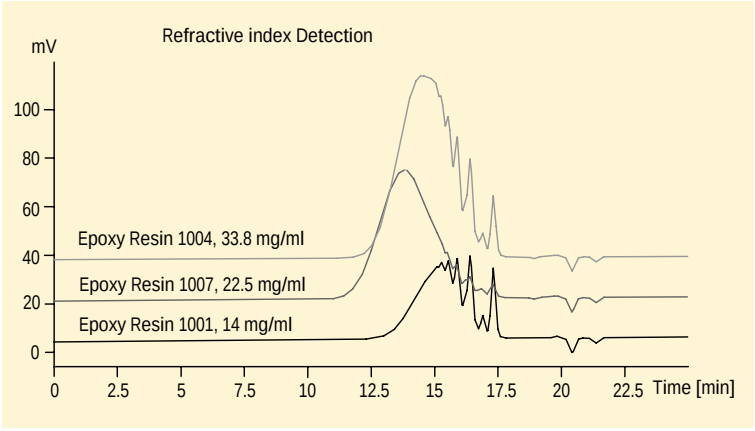

Figure 2
Analysis of three different epoxy resin qualities

Conditions

Column
2 ˜ PLgel mixed-D, 7.5 ˜ 300 mm, 5 µm
Mobile phase Tetrahydrofurane (THF)
Flow rate 1 ml/min
Oven Temp 20 °C
Injection vol 20 µl
UV detector
DAD 254/100 nm, reference 360/100 nm
Refractive index detector

Sample preparation
Sample dissolved in 1 ml THF
Polystyrene standards from PSS were used for narrow standard calibration

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Method Performance

Figure 2 shows an overlay of 3 different batches of epoxy resin. It can be seen that the epoxy resin 1001 has a relatively low molecular weight compared to the others. The epoxid 1007 has a high molecular weight whereas Epoxid 1004 falls in the middle. The molecular weight data of these three batches are combined in table 1. The precision of the molecular weight data was tested for batch 1001, and the MW data of 10 consecutive runs was evaluated. Figure 3 shows an overlay of the chromatograms. The rsd of Mw and Mn was calculated and found to be:

rsd of Mw over 10 runs = 0.66 %

rsd of Mn over 10 runs = 2.5 %

Method performance

Precision of weight: average molecular weight (rsd of Mw) = < 1 %
Precision of number weight: average molecular weight (rsd of Mn) = < 3 %

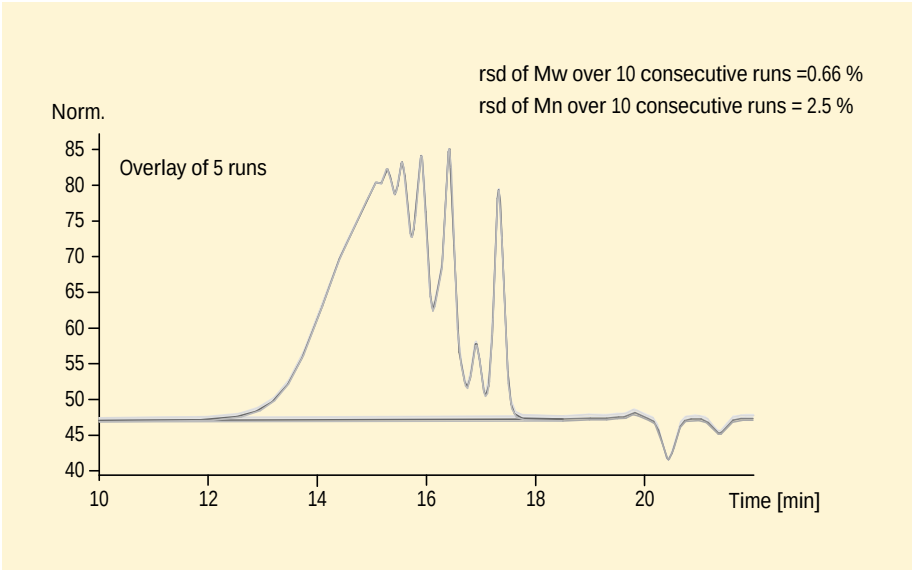
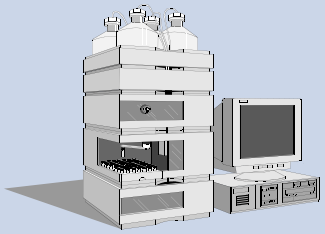


Figure 3
Precision of molecular weight data

Equipment

Agilent 1100 Series:
- isocratic pump
- degasser (recommended)
- autosampler
- thermostatted column compartment
- diode array detector and/or HP 1047A refractive index detector

Agilent ChemStation
+ software
+ polymer labs GPC software



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