Analysis of Polyvinylchloride using Gel Permeation Chromatography

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Abstract

Polyvinylchloride (PVC) can be divided into two main groups—hard and soft PVC, which is used for the production of for example tubings, cables, cars, furniture, foils, artificial leather and the covering of wall papers. In 1990, 11.4 million tons were used worldwide. The mol masses vary from 30000 to 130000 g/mol. Chlorinated PVC with a maximum concentration of 73 % of chloride is used whenever the plastic material needs to have drastically increased solubility compared to normal PVC. Chlorination also improves thermal stability and mechanical stability. Examples of products made from this modified PVC are resins, foil and fibers. 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.

Method Performance

Figure 1 shows the signal traces of 3 different batches of polyvinylchloride. The production process started with a normal PVC. In the second step this PVC was chlorinated and in the final process it was formed into tubes. MW data from these 3 production processes were evaluated. The differences in MW data are shown in table 1. It is interesting to note that the UV absorption increases during the manufacturing process, see figure 2.

PVC type	Mw data	Mn data	Polydispersity
PVC	111852	53648	2.085
Chlorinated PVC	107355	52145	2.059
Chlorinated PVC as tube	124378	61005	2.039

Table 1
Analysis of PVC with refractive index detector

Influence of flow and temperature stability

The following in which PVC was analyzed, demonstrates the importance of stable flow rates and stable oven temperatures.

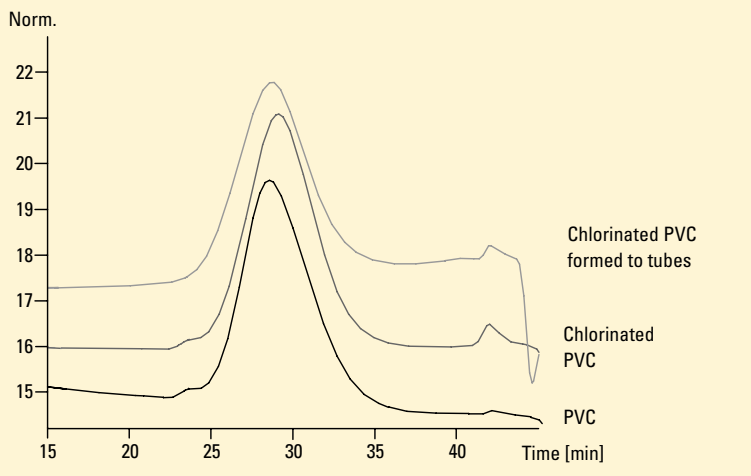

Figure 1
Analysis of PVC with refractive index detector

Conditions

Column 3 ˜ PSS GPC, 8 ˜ 300 mm, 5 µm 10^6,10^5,10^3 A
Mobile phase Tetrahydrofurane (THF)
Flow rate 0.8 ml/min
Oven Temp 20 °C
Injection vol 10 µl
UV DAD 254/100 nm
Refractive index detector
Sample preparation
Sample dissolved in 1 ml THF, filtered with 0.45 µm filter. Polystyrene standards from PSS were used for narrow standard calibration
Method performance
Precision of weight: average molecular weight (rsd of Mw) = < 1 %
Precision of number weight: average molecular weight (rsd of Mn) = < 1 %

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The precision of MW data, measured by relative standard deviation of for example Mw and Mn is mainly influenced by the stability of flow rate. To demonstrate the importance of stable flow rates and constant oven temperatures, experiments were done where for each parameter slight changes were made. It soon became obvious that flow changes even smaller than 0.5 % had an influence on the precision, whereas temperature changes below 1 °C did not have a major influence (see figure 3). Consequently the precision of the flow rate should be better than 0.1 %. 20 consecutive injections were made and the precision of the Mw data were < 0.4 %. (see figure 4.) Data was obtained using an HP 1090 Series HPLC system.

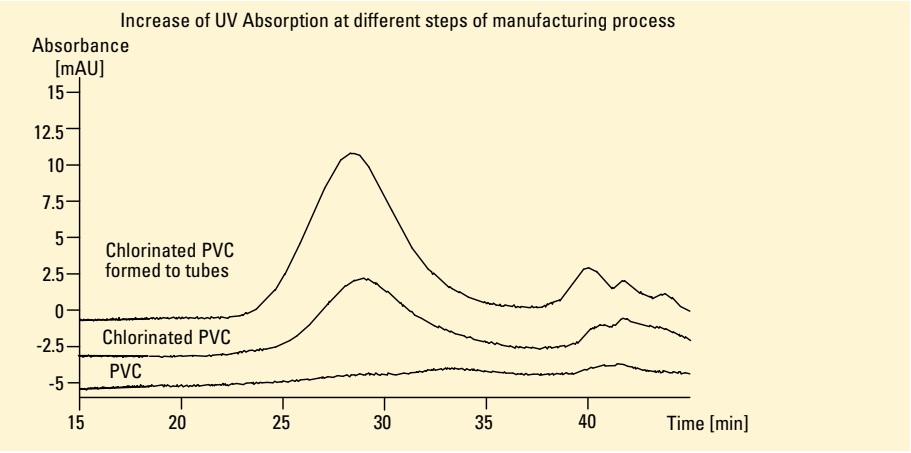

Figure 2
Different PVC types analyzed with UV DAD

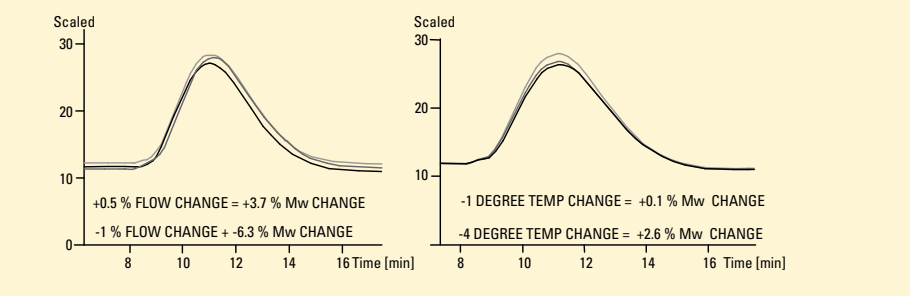

Figure 3
Influence of flow and temperature variations on precision of MW data

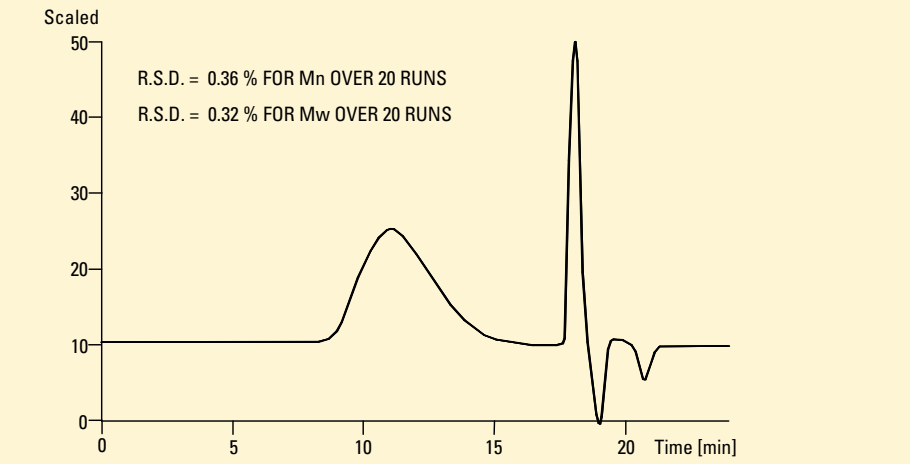

Figure 4
Precision for optimized conditions

Conditions

Column 7.5 ˜ 300 mm, 10^4 PLGel
Mobile phase
Tetrahydrofurane (THF)
Flow rate 0.2 ml/min
Oven Temp 40 °C
Injection vol 40 µl

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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