A MICRO DIFFERENTIAL VISCOSITY DETECTOR FOR POLYMER SEPARATION SYSTEMS

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ABSTRACT

In this paper we present the first micromachined viscosity detector suitable for coupling to conventional, commercially available polymer separation systems. The μ-viscometer (viscochip) has a reduced dead volume compared to conventional viscometers. It is shown that this results in better chromatographic resolution.

Keywords: viscosity detector, separation systems, GPC/SEC, polymers

1. INTRODUCTION

In this paper we present the first micromachined viscosity detector suitable for coupling to conventional, commercially available polymer separation systems (GPC/SEC). In Gel Permeation Chromatography the mass distribution of a polymer sample is determined by separation of the different masses, followed by detection by one or preferably more detectors, such as refractive index (RI), light scattering (LS) and viscosity detectors. Viscosity detection is based on sensitive measurement of the specific viscosity change η_{sp}, produced by the dissolved polymers. Since small viscosity variations must be measured, a differential technique is applied using a Wheatstone bridge (figure 1) [1].

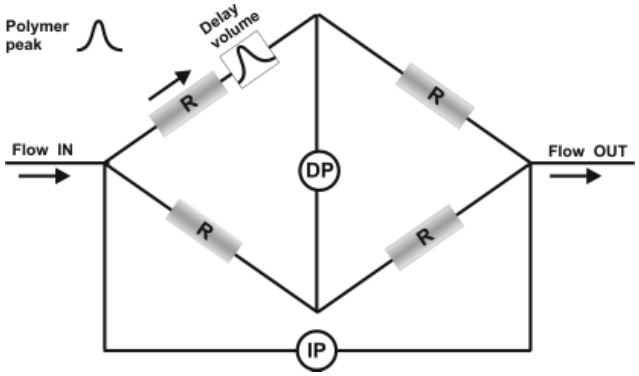

Figure 1: a polymer viscosity peak from a separation column will be absorbed by the delay volume in one branch, thus unbalancing the bridge. The specific viscosity η_{sp} is given by the pressures *DP* and *IP*: $\eta_{sp} = 4DP/IP$

The only comparable micro-device in literature has been presented at μTAS 2002 by the same author [2]. This was however developed specifically for a micromachined hydrodynamic chromatography system. For this system, operating a detection volume two orders of magnitude smaller than the current viscochip, a micromachined differential pressure transducer had to be employed, which limited resolution and thus practical use. This paper presents a device in which conventional pressure transducers are applied.

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2. SYSTEM DESIGN

The most commonly used detector configurations in a 3-detector system are shown in figure 2. All detectors have 1.0 ml/min as their optimum flow rate. As the typical detection volume is 8 µl for an RI, 8 µl for a LS and 30 µl for a viscometer, it is clear that the viscometer has the largest extra-column contribution to peak broadening. Narrow peaks are important as these are necessary for accurate determination of molecular weight distribution in GPC. Compared to a stand-alone viscometer the situation is worse in configuration A, where the RI and LS detectors add additional peak broadening to the viscometer signal. Circumventing this by splitting the flow as in configuration B means RI and viscometer do not run at an optimum flow rate, thus reducing system performance.

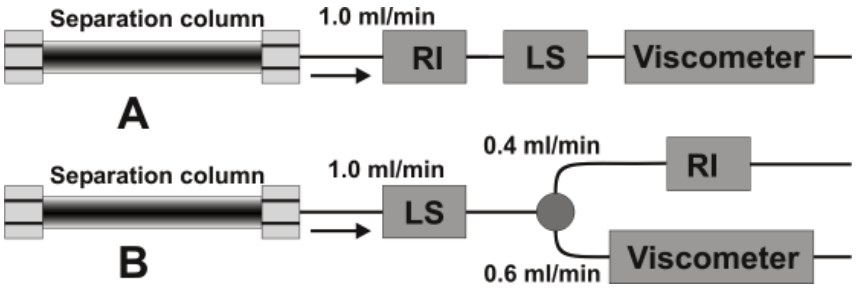

Figure 2: The most commonly used 3-detector configurations. In A the RI and LS detectors add peak broadening to the viscometer signal. In B the flow is split, meaning both RI and viscometer run at a flow rate below their optimum.

In figure 3 therefore the conventional viscometer has been replaced by a glass-glass Wheatstone bridge viscochip, with a detection volume of 2 µl. Designed for an optimum flow rate of 0.1 ml/min, the split flow setup allows all detectors to be run at near-optimum flow rates. Additionally, the ratio of detection volume to flow rate – which can be seen as an averaging time, decreasing system resolution – is improved using the viscochip: 1.2 seconds instead of 1.8 seconds.

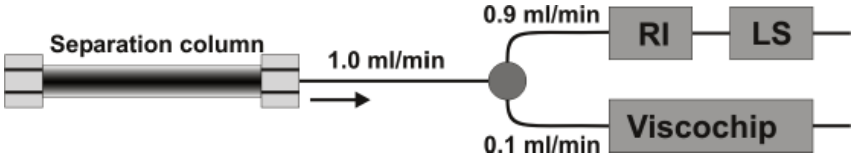

Figure 3: Using a viscochip, the detector configuration from figure 2B can be adapted giving a near-optimum flow rate through all detectors.

3. RESULTS

The effect on system chromatographic resolution is shown in figures 4 and 5, for both the viscochip and a conventional viscometer. Signal input for all detectors was the same by using a flow split. For a broad standard (figure 4) there is virtually no difference in peak shape, demonstrating the performance of the viscochip. For narrow polydispersity material (figure 5) the viscochip peak width in figure 5 is more than 20% narrower than for the conventional viscometer connected as in figure 2A, giving a much more realistic peak shape. The results demonstrate the improvement in performance of the viscochip compared to conventional viscometers at 1.0 ml/min, and also its capability for miniaturised GPC systems running at 0.1 ml/min, which is desirable for continuous process monitoring.

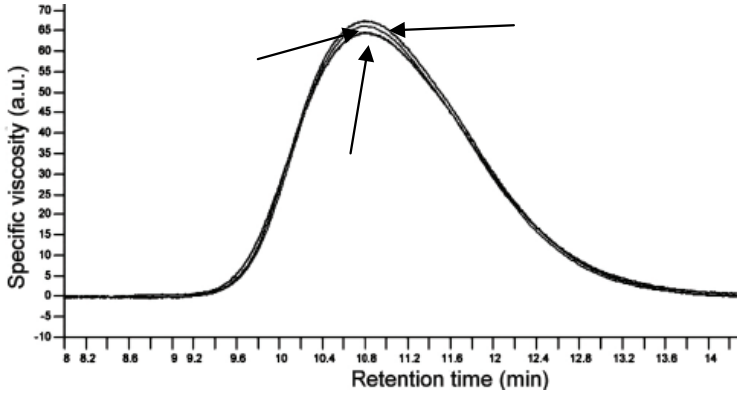

Figure 4: Response to a broad polystyrene polymer standard. Signals from a viscochip (1, running at 0.1 ml/min), and a conventional viscometer (running at 1.0 ml/min) are shown, the latter both as a single detector (2) and behind a RI and LS detector (3) as in figure 2A.

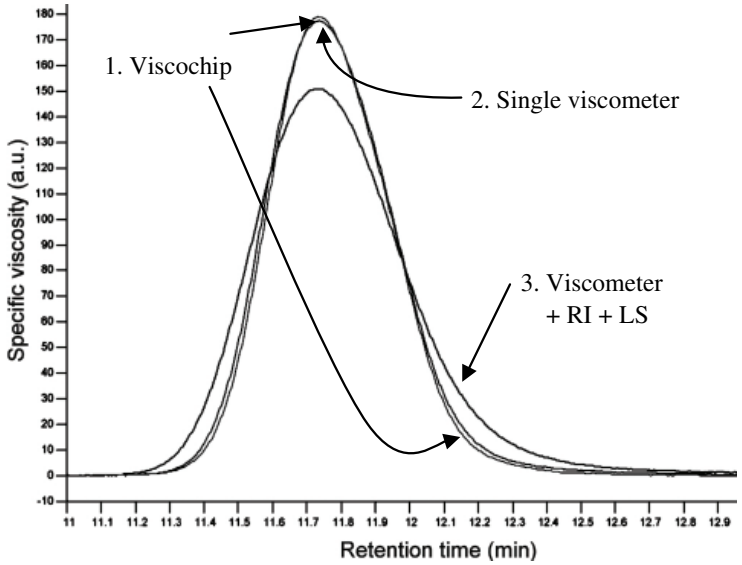

Figure 5: Setup as for figure 4, using a narrow 195 kDa PMMA standard. The viscochip trace clearly has the smallest peak width. The small difference between 1 and 2 will be larger for a viscometer running at 0.6 ml/min as required in figure 2B.

4. CONCLUSIONS

A micromachined viscosity detector was presented, suitable for coupling to GPC/SEC systems, showing reduced band broadening compared to conventional viscometers.

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