Troubleshooting in SFC

By The Chromatography Helpdesk

The HelpDesk team occasionally are allowed out of the office, and on one such recent occasion took advantage of this to attend the most recent HPLC meeting in New Orleans. One of the posters drew the team's attention and this edition of the help desk will focus on some rather interesting data that the team from the Vrije Universiteit Brussel obtained [1]. After speaking to the author it would appear that the investigation was initiated after a piece of tubing was changed and the team observed some unusual results. As with all good scientists this resulted in a series of further experiments, duly followed by a sound interpretation of the data.

The existence of different types of flow regimes and in particular, turbulent flow in nature is something that is not disputed. However in the field of chromatography this is not the case. Indeed there are techniques which are referred to as being turbulent flow, but this is more of a name than any actual bearing on the nature of the fluidic flow in the column. That is not to say that it is not feasible to have turbulent flow in a packed bed, as with the correct flows, particle sizes and fluid viscosities it is feasible to have a turbulent flow. It should also be noted that there are many different types of flow that are possible within a packed bed and it is very likely that running at very high linear velocities will result in transitioning from one form of flow to another.

So what has this to do with changing a piece of tubing? This is where it gets interesting, and a little background information is required on how to determine if the flow regime is laminar or turbulent. In a piece of open tubing, the nature of the flow can be classified by calculating the Reynolds number.

$$Re = \frac{Inertial\ Forces}{Viscous\ Forces} = \frac{\rho D \upsilon}{\mu} = \frac{4\rho . flowrate}{\mu \pi D}$$

Where

Re – Reynolds number (a dimensionless measure of the nature of the fluid flow)

ρ – density of fluid (kg/m3)

D – diameter of open tubing (this is a scaling factor to allow Re to be dimensionless, m)

υ – average linear velocity of fluid in tubing (m/s)
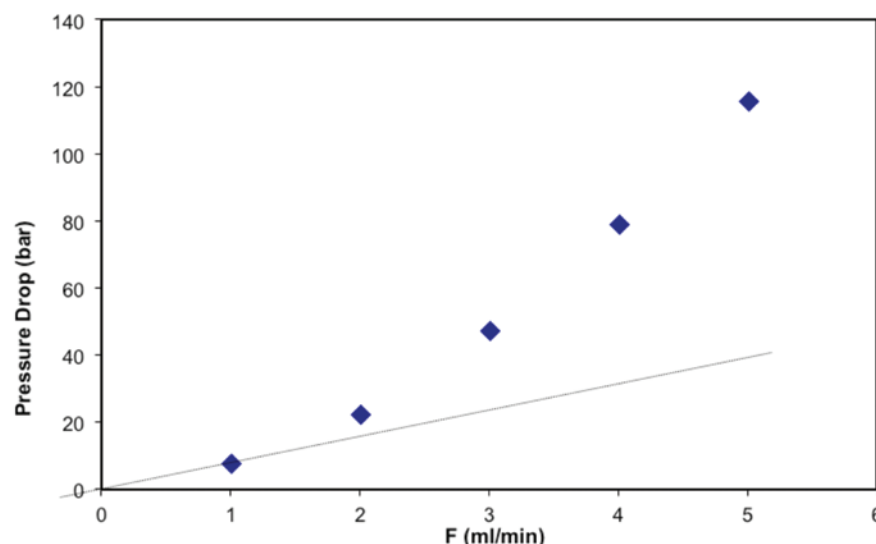
μ - fluid dynamic viscosity (Pa s)

Figure 1: Total system pressure drop with varying flow rate. Note that there is not a linear relationship.

It is agreed that the point at which turbulence is the dominant mode of fluid flow occurs at about a Re of 2000-4000. This transition point will depend on the topography of the tubing, with more rough tubing promoting the generation of turbulence. It can also be seen from the equation, that lowering the viscosity increases the Reynolds number, as does increasing the diameter of the tubing, assuming all other parameters (density, viscosity and velocity) are constant. However, when re-expressing the linear velocity in the form of the flow rate it shows that for a fixed flow rate, the Reynolds numbers increases when decreasing the tubing diameter.

So what happened in this case? Ruben De Pauw, a PhD student under the supervision of Ken Broeckhoven, an assistant professor at Vrije Universiteit Brussel, was altering the tubing in his SFC experiment and noticed that it had an effect on the experiment. The system pressure drop was seen to be increasing disproportionately with increasing flow rate and the pressure variations were also much larger than expected. The data that the team from Brussels obtained is shown in *Figure 1*. As all good practical chromatographers aspire to do, Ruben was looking at minimising the dead volume in his chromatographic system and a quick win here was to reduce the i.d. of the tubing that was being used. However in doing so as can be seen from *Figure 2* the fluid flow moved from a laminar flow regime to a turbulent one in the connection tubing.

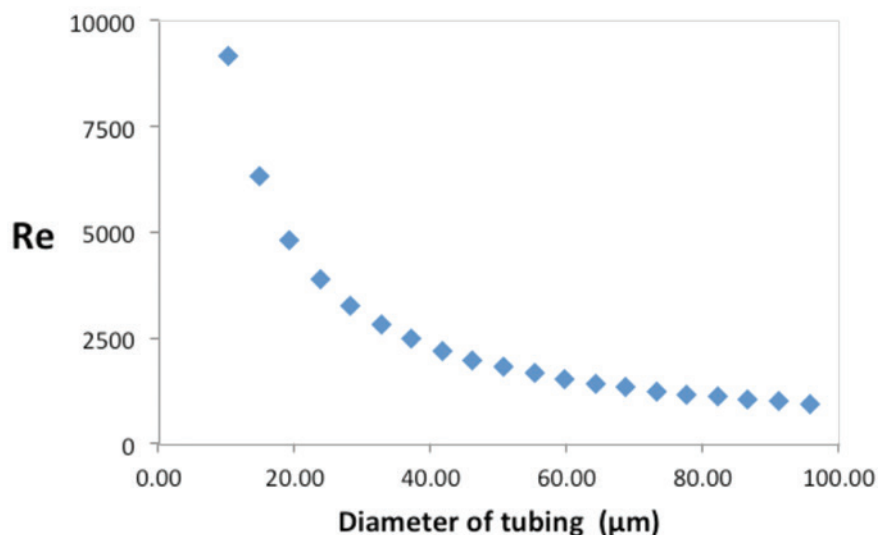The further set of experiments that Ken and his team performed demonstrated that

Figure 2: Effect of changing the tubing diameter on the Reynolds number

driven shift in the equilibrium in the stationary phase analyte complex that is formed during the separation process, resulting in a shift in the retention time.

This type of observation is more often found with low viscosity chromatographic systems and SFC has in general a lower viscosity than that obtained in HPLC at room temperatures. The effects both in terms of the retention time and system pressure drop indicate that when laboratories are moving into different areas of chromatographic science an open mind should always used to confront the inevitable challenges that ensue.

The Chromatography HelpDesk would like to thank Dr Broeckhoven for his invaluable input into the production of this article.

References

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it was not only the size of the tubing, but also the type of tubing that could cause a transition to turbulent fluidics. Ken and Ruben demonstrated that in changing the flow regime from laminar to turbulence that there is a change in the pressure drop relationship with the flow rate. For a turbulent flow system this is a quadratic function, which means that at the same nominal flow but simply altering the tubing i.d. two different flow regimes

can be experienced. This can have an effect on the pressure, which in turn can also have an effect on the retention time as has been demonstrated by many authors previously [2-4]. This is particularly the case within SFC where pressures gradients have been used to optimise the separation. The shift in retention time due to the change in the pressure is a phenomenon that is now well understood, and can be simply explained by a pressure