Solid Core 4 µm HPLC Column Comparison to Fully Porous 3 µm and 5 µm Columns: Efficiency and Pressure

Luisa Pereira, Thermo Fisher Scientific, Runcorn, Cheshire, UK

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Solid core, fused core, superficially porous, pressure, efficiency, impedance

Abstract
In this technical note, the chromatographic performance of solid core 4 µm particle packed HPLC columns is compared with that of fully porous 5 µm and 3 µm particle packed columns. Parameters compared are column pressure, efficiency, and impedance.

Introduction
The use of partially porous particles, with a diameter between 2 and 3 µm, is gaining momentum, as these provide similar efficiency to sub-2 µm particles but with significantly lower column backpressures.

Often, however, solid core particles are packed into short, narrow-bore columns, which has implications in terms of the system set up. System dead volume and operating parameters have to be optimized to get the best possible performance out of the column.

It is demonstrated herein that by using a solid core 4 µm particle packed in conventional column dimensions, significant improvements in the assay performance can be achieved without the need to make changes to the operating parameters or system set up. With Thermo Scientific™ Accucore™ XL 4 µm HPLC columns, it is possible to dramatically improve separation efficiency, and therefore resolution and sensitivity over those obtained with conventional fully porous 5 µm and 3 µm particle packed columns. These improvements are obtained with only a 40% increase in backpressure over the 5 µm and a reduction in backpressure compared to the 3 µm material. The Accucore XL 4 µm solid core HPLC columns exhibit significantly lower impedance than fully porous materials.

Equation 1, known as the Burke-Plummer equation, shows the dependency of the pressure drop across the column on a variety of experimental parameters. The pressure is directly proportional to the column length, flow rate, and mobile phase viscosity and is inversely proportional to the square of the particle size diameter and the square of the column internal diameter ID. The interstitial porosity (the spaces between the particles that are accessible by the mobile phase) has a more complicated relationship to the pressure. There are other operating parameters that have an impact on the overall system pressure, such as the ID and length of the connecting tubing in the LC system, detector setup parameters such as flow cell volume in UV or the ID and length of the capillary components in ESI or APCI sources in LC/MS.

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Equation 1

$$\Delta P = a \frac{(1 - \varepsilon_i)^2}{\varepsilon_i^3} \frac{F L \eta}{d_c^2 d_p^2}$$

where

ΔP = pressure drop across the column
a = constant (dependent on packing, normal values in the range 150-225)
ε_i = interstitial porosity of the packed bed
F = flow rate through the column
L = length of the column
η = viscosity of the mobile phase
d_p = particle diameter
d_c = column internal diameter

The conventional approach to compare the chromatographic performance of columns is to plot a normalized efficiency (HETP - height equivalent to a theoretical plate) as a function of mobile phase flow rate or linear velocity, often referred to as a van Deemter plot. This approach does have limitations, since it does not account for analysis time or pressure restrictions of the chromatographic system. Kinetic plots [1] are an alternative method of plotting the same experimental data but allow other parameters, such as pressure, to be incorporated. Therefore, we can infer the kinetic performance limits of the tested chromatographic materials. There are a variety of ways in which this data can be presented, and all of these plots are referred to as kinetic plots. In one of the most useful forms of kinetic plots, a term called impedance is used. Impedance (Equation 2) defines the resistance a compound is subjected to as it moves down the column, relative to the performance of that column. This term gives a true measure of the performance of the column as it incorporates efficiency, time, and pressure, which are critical practical considerations of a chromatographic separation.

Equation 2

$$E = \frac{\Delta P\, t}{\eta\, N^2}$$

where E = impedance
t = dead time of chromatographic system
ΔP = pressure drop
η = kinematic viscosity of mobile phase
N = efficiency

In kinetic plots, the linear velocity, conventionally plotted on the x-axis in the van Deemeter plot, is transformed into the pressure drop limited plate number. Using a maximum pressure drop for the system, any experimental set of data of HETP- linear velocity obtained in a column with arbitrary length and pressure drop can be transformed into a projected efficiency (N)-t_0. This represents the plate number and t_0-time, which could be obtained if the same chromatographic support was used in a column that was long enough to provide the maximum allowed inlet pressure for the given linear velocity.

Pressure Comparison

Figure 1 shows how the column backpressure of the Accucore XL 4 µm HPLC column compares with that of the fully porous 5 µm and 3 µm columns tested. On average (across the flow rate range tested), the Accucore XL 4 µm HPLC column gives 42% higher pressure than fully porous 5 µm and 13% lower pressure than fully porous 3 µm HPLC columns. Even when running the 150 x 4.6 mm Accucore XL 4 µm HPLC columns at a flow rate of 2 mL/min, the backpressure is only 200 bar.

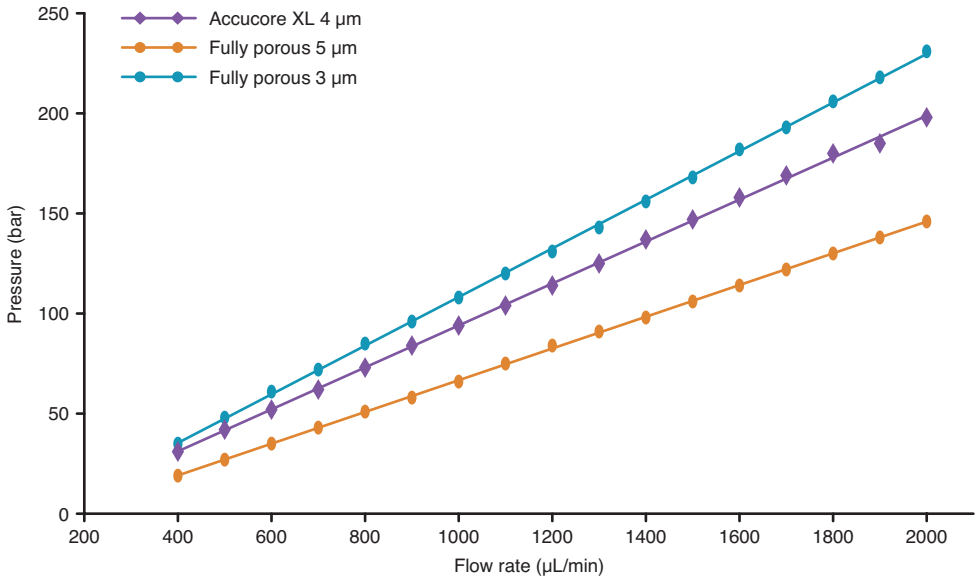

Figure 1: Comparison of column pressure for Accucore XL 4 µm HPLC column and fully porous 5 µm and 3 µm columns. All columns: 150 x 4.6 mm; test conditions: mobile water / acetonitrile (50:50 v/v); column temperature: 30 °C.

Efficiency Comparison

Figure 2 compares the efficiency of the Accucore XL 4 µm material with that of the fully porous 5 and 3 µm materials tested using a van Deemter plot. On average (across the flow rate range tested), the Accucore XL 4 µm material gives 75% more efficiency than fully porous 5 µm and 50% more efficiency than fully porous 3 µm. The curve for the Accucore XL 4 µm HPLC column is very flat; therefore, a wide range of linear velocities (or flow rates) can be used without losing chromatographic performance.

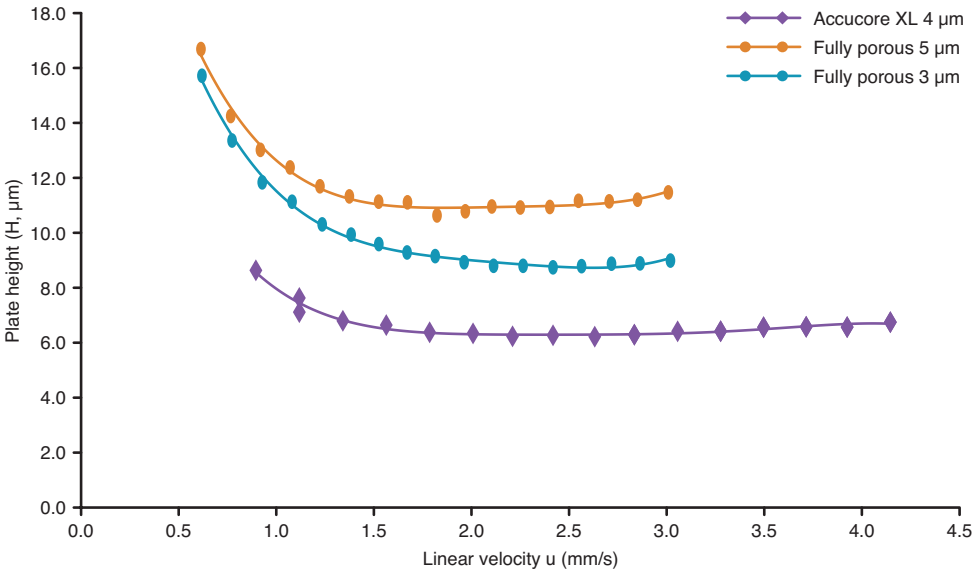

Figure 2: Efficiency comparison using van Deemter plots for Accucore XL 4 µm HPLC column and fully porous 5 µm and 3 µm columns. All columns: 150 x 4.6 mm; test conditions: water / acetonitrile (50:50 v/v); column temperature: 30 °C; test probes: o-xylene and theophylline (t_0 marker).

Impedance Comparison

Impedance is a term that gives a true measure of the performance of the column, as it incorporates efficiency, time, and pressure, which are critical parameters for chromatographers. Lower impedance values indicate faster chromatography and generation of narrower peaks at lower backpressures. The solid core particles, tight control of particle diameter, and automated packing processes used in Accucore HPLC columns all contribute to low impedances. On average (across the flow rate range tested), the Accucore XL 4 µm HPLC column provides the following:

- 59% more efficiency per unit time than fully porous 5 µm and 53% more efficiency per unit time than fully porous 3 µm (Figure 3).
- 79% lower impedance than fully porous 5 µm and 72% lower impedance than fully porous 3 µm (Figure 4).

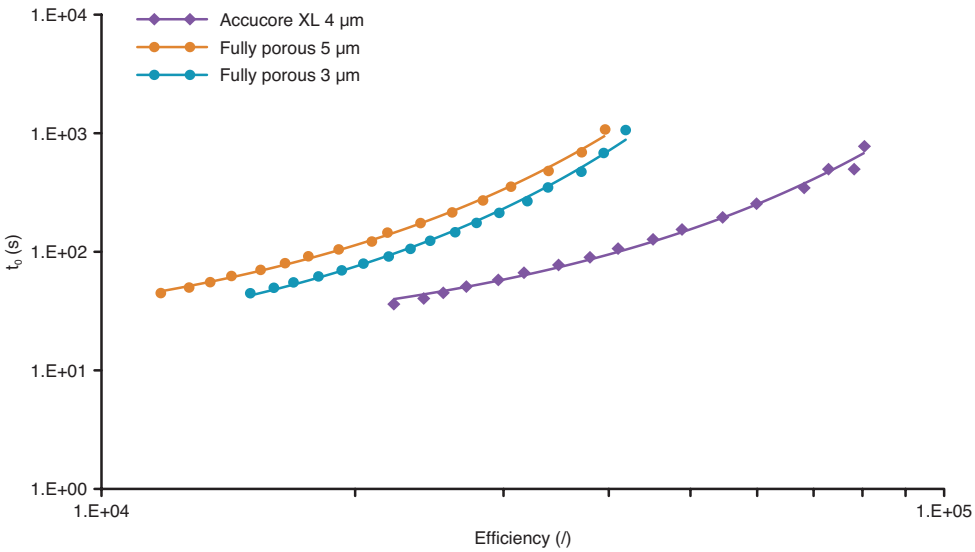

Figure 3: Performance comparison of Accucore XL 4 µm HPLC column and fully porous 5 µm and 3 µm columns using kinetic plots: efficiency per unit time. All columns: 150 x 4.6 mm; test conditions: water / acetonitrile (50:50 v/v); column temperature: 30 °C; test probes: o-xylene and theophylline (t_0 marker).

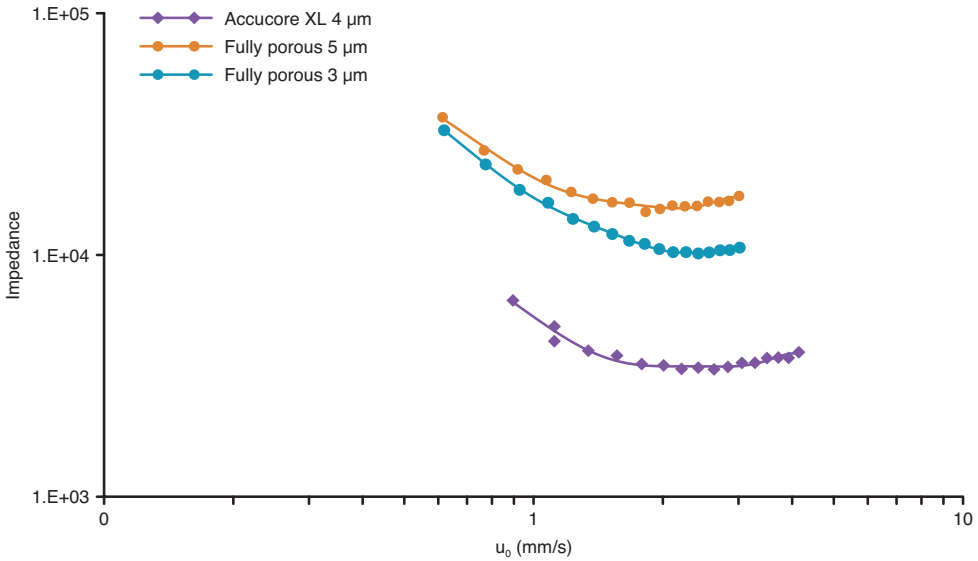

Figure 4: Performance comparison of Accucore XL 4 µm HPLC column and fully porous 5 µm and 3 µm columns using kinetic plots: column impedance (E) relative to linear velocity (u). All columns: 150 x 4.6 mm; test conditions: water / acetonitrile (50:50 v/v); column temperature: 30 °C; test probes: o-xylene and theophylline (t_0 marker)

Conclusion

- The Accucore XL 4 µm solid core HPLC columns provide improvements in efficiency in excess of 50% over fully porous 5 µm and 3 µm columns.
- The backpressure of the Accucore XL 4 µm solid core HPLC column is 42% higher than fully porous 5 µm, but 13% lower than fully porous 3 µm.
- The Accucore XL 4 µm solid core HPLC columns exhibit significantly lower impedance than fully porous materials.

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USA and Canada +1 800 332 3331
France +33 (0)1 60 92 48 34
Germany +49 (0) 2423 9431 20 or 21
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China +86 21 68654588 +86 10 84193588
+86 20 83145199 800 810 5118
India +91 22 6742 9494 +91 27 1766 2352
Australia 1 300 735 292 (free call domestic)
New Zealand 0800 933 966 (free call domestic)
All Other Enquiries +44 (0) 1928 534 050

Technical Support
North America +1 800 332 3331
Outside North America +44 (0) 1928 534 440

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