Chromatography Solutions

Knowledge note #0019

Avantor® ACE® UltraCore and Solid Core Technology

INTRODUCTION

Solid Core (also known as superficially porous (SPP) or fused-core®) particles are a fairly recent introduction to liquid chromatography and can offer distinct advantages compared to traditional fully porous particles. In particular, they are able to offer performance similar to smaller sized fully porous particles at significantly lower back pressures. This Knowledge Note discusses the theory behind solid core technology and introduces the Avantor® ACE® UltraCore range of solid core columns.

SOLID CORE PARTICLES

Unlike the fully porous particles (FPP) generally utilised in liquid chromatography, solid core particles consist of a partially porous shell surrounding a non-porous silica core (Figure 1). The use of solid core particles is well established, but has really gained prominence in recent years as they are able to generate higher efficiencies than comparably sized fully porous particles and show less of a performance drop-off at higher flow rates.

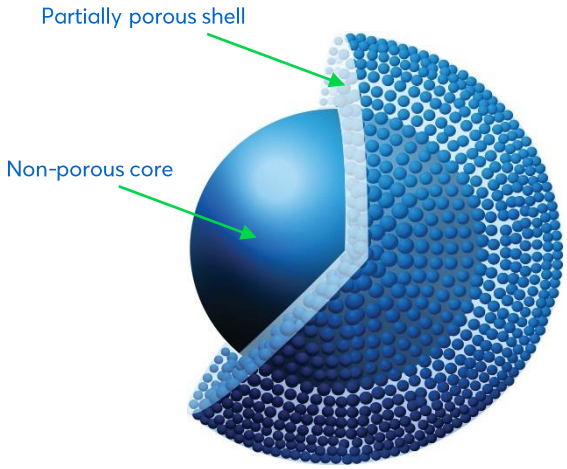


Figure1: Schematic structure of a solid core particle.

It has been shown that columns packed with 2.5-2.7 µm solid core particles are able to generate theoretical plate values comparable to 1.7 µm fully porous particles, with the advantage of significantly lower back pressure. This makes solid core particles an interesting option for increasing separation efficiency without the need to utilise UHPLC equipment and sub-2 micron particles.

AVANTOR® ACE® ULTRACORE

The ACE UltraCore range is manufactured from high purity solid core silica particles and is bonded using unique Encapsulated Bonding Technology (EBT™). This bonding increases ligand coverage and eliminates the negative effects of unbonded silanol groups, providing improved inertness, performance and stability. In addition, ACE UltraCore particles have an extended usable pH range (compared to traditional silica particles) of pH 1.5 – 11.0. Method developers can therefore utilise low, intermediate and high pH mobile phases to fully explore selectivity.

ACE UltraCore columns are available in two particle sizes (2.5 and 5 µm) and all common column lengths and internal diameters. Two bonded phases are available, the SuperC18 and the SuperPhenylHexyl. The ACE UltraCore SuperC18 provides analyte retention by hydrophobic interactions, whilst the SuperPhenylHexyl combines hydrophobic and aromatic interactions to provide alternative selectivity.

WHY ARE SOLID CORE PARTICLES MORE EFFICIENT?

The performance advantages of solid core particles, relative to their fully porous equivalents, can be understood by considering the van Deemter equation (see also Knowledge Note #0010) and its three composite terms:

$$HETP = A + \frac{B}{u} + C.u$$

A = Eddy diffusion (analyte paths, packing, wall effects)
B/u = Analyte longitudinal / axial diffusion
C.u = Analyte mass transfer between stationary & mobile phases

It was originally believed that improved mass transfer (i.e. the C-term) was responsible for the higher performance of solid core particles; however, more recent studies have shown that for small molecules (<500 Da) this contributes very little and that it is reductions in both the A and B-terms that are primarily responsible.[1-3] The reduction in eddy-diffusion (A-term) provides the largest contribution to the improved efficiency, potentially due to a more homogenously packed bed for

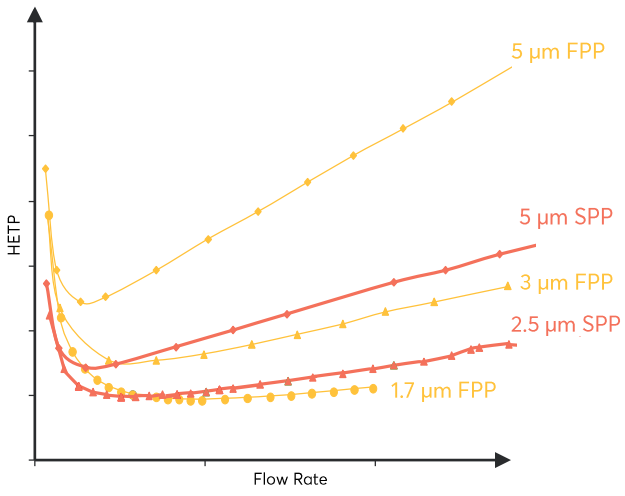


Figure 2: Comparison of van Deemter curves for ACE solid core particles and ACE fully porous particles.

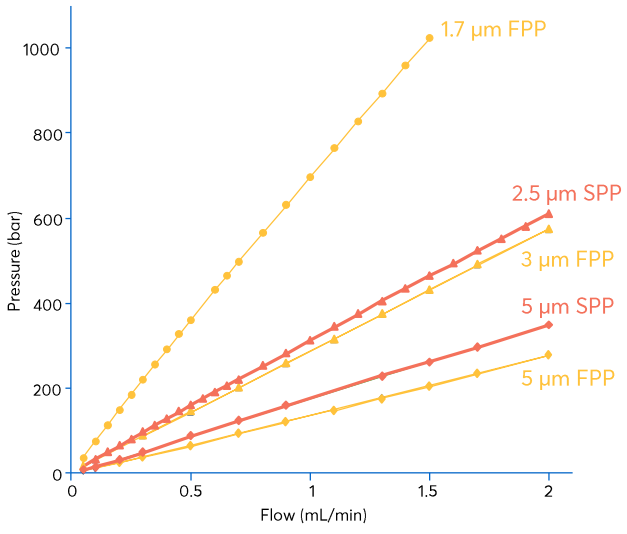


Figure 3: Plot of flow rate vs total backpressure for 50 x 2.1 mm columns packed with ACE solid core and fully porous particles.

columns packed with solid core particles. The B-term contribution also improves performance as the presence of a solid core reduces longitudinal diffusion of the analyte band.

IMPROVED CHROMATOGRAPHIC PERFORMANCE

Two options are typically considered to improve the efficiency of a separation and therefore the resolution achieved between peak pairs. Firstly, column length can be increased to increase the efficiency of the column. This results in a proportional increase in run time and back pressure. The second option is to reduce the particle size of the packing material. Using this approach, the column length can be reduced to obtain a faster analysis whilst still maintaining separation efficiency. However, this will result in a significant increase in backpressure and may therefore not be possible on some HPLC instruments due to pressure limitations.

Solid core technology provides a third option. Figure 2 shows a comparison of the van Deemter plots obtained for 2.5 and 5 µm solid core particles and fully porous equivalents. By comparing the 5 µm solid core and 5 µm fully porous particles, it can be seen that a dramatically lower height equivalent to a theoretical plate (HETP) value is obtained. This directly equates to a significant increase in column efficiency. Figure 3 demonstrates that this performance gain is delivered at approximately the same back pressure as the fully porous equivalent. Columns packed with 5 µm ACE UltraCore particles can therefore be utilised on any standard HPLC system to improve chromatographic performance over traditional 5 µm fully porous particles, without encountering back pressure issues (Figure 4). In this example, the run time is also significantly reduced due to the fact that solid core particles have a lower porosity and therefore lower surface area for analyte interaction and hence are less retentive. Due to the narrower peaks and smaller peak volumes, sensitivity also increases markedly.

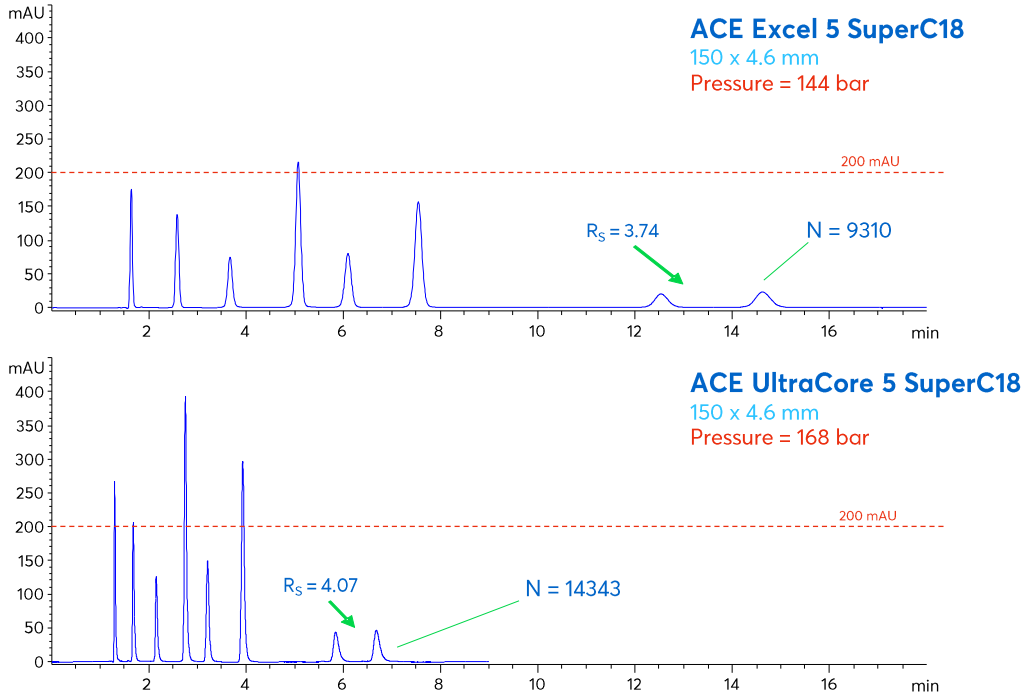


Figure 4: Separation of antihistamines on the fully porous ACE Excel 5 SuperC18 and the solid core ACE UltraCore 5 SuperC18.

Mobile Phase A: 0.1% Formic acid in H_2O, B: 0.1% Formic acid in MeCN; Gradient: 20-70% B in 20 minutes; Flow rate: 1.0 mL/min; Temperature: 40 °C; Injection volume: 10 µL; Detection: UV, 254 nm; Sample: 1) Aspirin, 2) Phenacetin, 3) Sulindac, 4) Tolmetin, 5) Naproxen, 6) Nimesulide, 7) Flurbiprofen, 8) Diclofenac, 9) Phenylbutazone, 10) Meclofenamic acid.

Similarly, Figure 2 shows that the 2.5 µm solid core particles can deliver significantly higher performance than a 3 µm FPP and is similar to the performance of a 1.7 µm UHPLC particle. However, as shown by Figure 3, this performance is not associated with the high backpressures generated by 1.7 µm UHPLC particles. In fact, the backpressure generated by the 2.5 µm solid core particles is only marginally higher than that of the 3 µm FPP. It is therefore possible to obtain chromatographic performance similar to a 1.7 µm UHPLC particle at more modest HPLC pressures, utilising a 2.5 µm ACE UltraCore column. The higher efficiencies of solid core particles mean that shorter column lengths can be used, leading to reduced run times. Figure 5 shows how the ACE UltraCore 2.5 SuperC18 can be utilised to provide a large increase in performance at HPLC pressures. In this example, the USP method for Naproxen was moved from a 5 µm fully porous column to a shorter column packed with ACE UltraCore 2.5 µm particles. Despite the shorter column length, an increase of 81% in efficiency and 55% in resolution was obtained.

In addition, solid core particles also have a higher optimum linear velocity and flatter van Deemter curve, meaning that higher flow rates can also be utilised to reduce run times further.

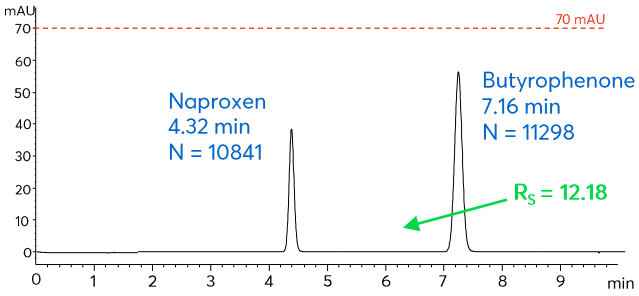
CONCLUSION

The Avantor® ACE® UltraCore range of columns can be utilised to achieve the benefits of solid core particles, including higher efficiencies and faster analysis than equivalent fully porous particles, without the disadvantage of high backpressure.

REFERENCES

1. G. Guiochon, F. Gritti, J. Chromatogr. A, 1218 (2011) 1915-1938
2. F.Gritti, G. Guiochon, LCGC N. America, 30 (2012) 586-595
3. S. Fekete, D. Guillarme, M. Dong, LCGC N. America, 32 (2014) 420-433

ACE 5 C18
150 x 4.6 mm
Pressure: 100 bar



ACE UltraCore 2.5 SuperC18
100 x 4.6 mm
Pressure: 202 bar

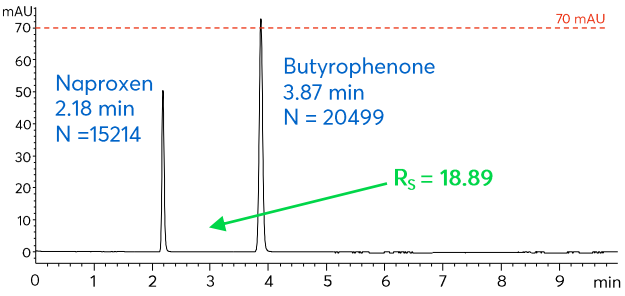


Figure 5: USP method for Naproxen run on the fully porous ACE 5 C18 and solid core ACE UltraCore 2.5 SuperC18.

Mobile Phase: H_2O with 2% glacial acetic acid/MeCN (50:50 v/v); Flow Rate: 1.2 mL/min; Temperature: Ambient (22 °C); Injection: 20 µL (150 x 4.6 mm), 11.6 µL (100 x 4.6 mm); Detection: UV, 254 nm.