

Introduction

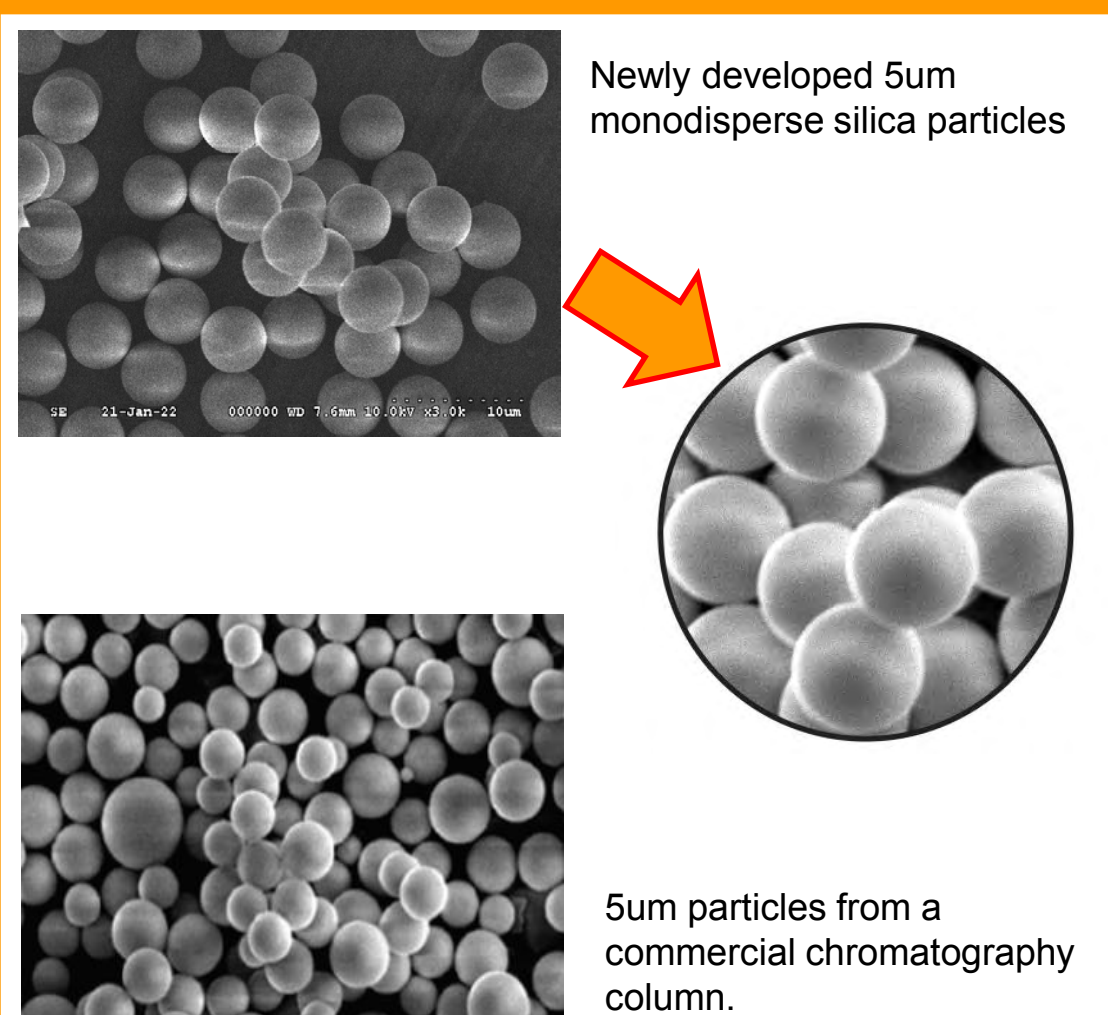
There are many developments in the silica particles used in liquid chromatography that have been well documented over the years. The move from irregular silica to spherical silica, the decrease in particle size from >5µm in HPLC to sub 2µm particles in UHPLC, the improved silica purity of type B silica over that of type A silica, and more recently the adoption of superficially porous particles versus traditional fully porous particles.

One area of development that has been discussed less and is still open to debate is the particle size distribution (psd) of these chromatographic materials. In this article we discuss the move towards improved monodispersity of silica particles for the use in high performance liquid chromatography (HPLC) and how the use of more monodisperse stationary phases can impact the resulting chromatographic parameters such as column efficiency.

Particle Morphology

Using a modified Stober manufacturing process we have produced silica particles that have an extremely high degree of monodispersity with a uniform smooth surface. Monodispersity generates high efficiency HPLC columns due to the reduced flow path dispersion (Eddy diffusion). Subsequent SEM imagery of the Evosphere® particles in comparison with traditional particles highlights the much narrower size distribution (figure 1).

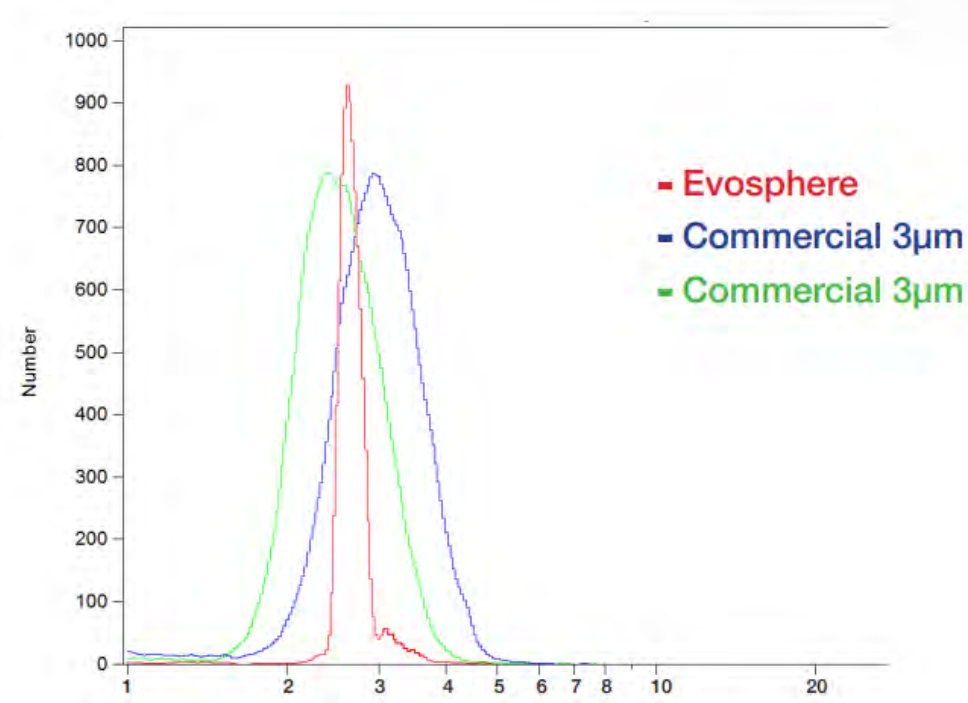
FIGURE 1. SEM Imaging of HPLC Particles



Particle Size Measurement

The silica particle size and distribution were measured using a Coulter Multisizer 3 along with samples from two different commercial 3µm silicas. As can be seen in figure 2 both commercial silicas have a much broader size distribution than the new monodisperse material, with one having a slightly smaller mean particle size and the other a slightly larger mean particle size.

FIGURE 2. Particle Size Distribution



	Monodisperse Silica	Commercial 3µm Silica-A	Commercial 3µm Silica-B
Mean Particle size (d50)*	2.66µm*	2.48µm	2.97µm
SEM Particle Size	3.0µm	2.8µm	3.2µm
D90/10	1.12	1.58	1.61
Pore Volume	0.89	0.88	0.89

* Measured by Coulter Counter

* Fortis® and Evosphere® are registered trademarks of Fortis Technologies Ltd.
 * Fortis Technologies recognises the trademarks of all other manufacturers.
 * All columns are original manufacturers packed columns.

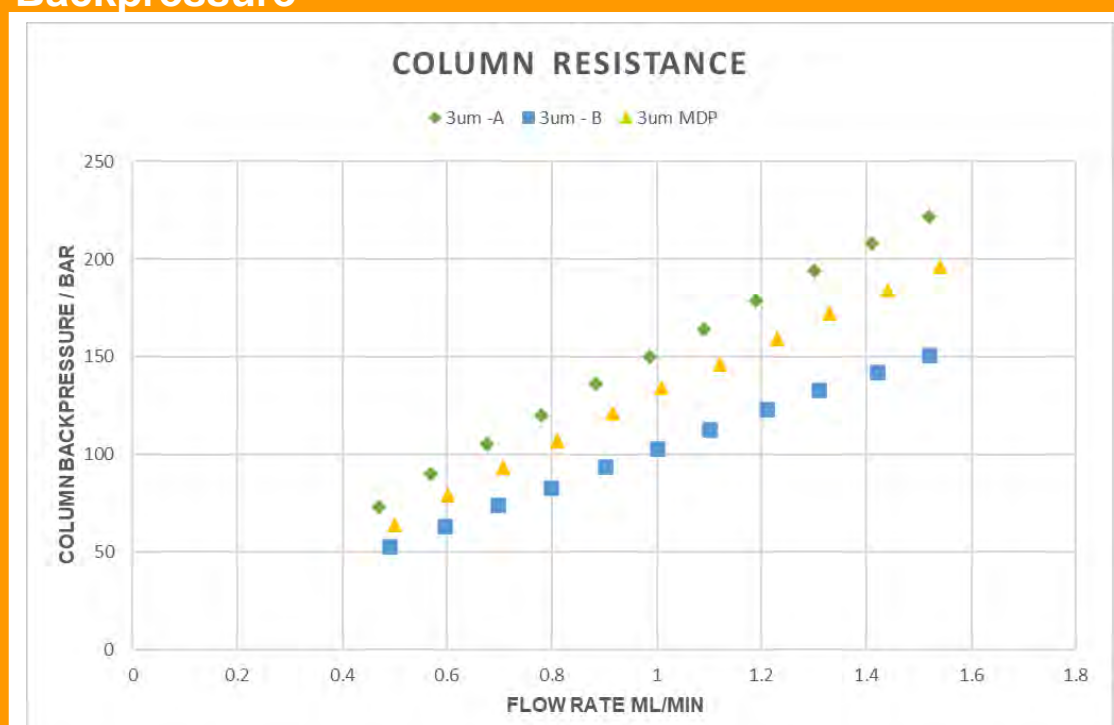
D90/10 Measurement

When assigning a measurement to characterise a particle size distribution the ratio of D90/10 is often quoted, and as such can be used to gauge the degree of size uniformity of the particles. The parameter D90 signifies the point in the size distribution, up to and including which, 90% of the total volume of material in the sample is 'contained'. For example; if the D90 is 6µm, this means that 90% of the sample has size of 6µm or smaller. The definition for D50, is then the size point below which 50% of the material is contained. Similarly, the D10 is the size below which 10% of the material is contained. This description has long been used in size distribution measurements. As the particle size distribution for chromatographic silica moves towards monodisperse then the D90 and D10 values become closer together and the D90/10 value tends towards a value of 1.

Column Backpressure

When we then studied the column backpressure of columns packed under identical conditions containing these three different silicas (figure 3), we could see that although the mean particle size affects the resulting backpressure the degree of monodispersity (d90/10) does not.

FIGURE 3. Effect of Size Distribution on Column Backpressure



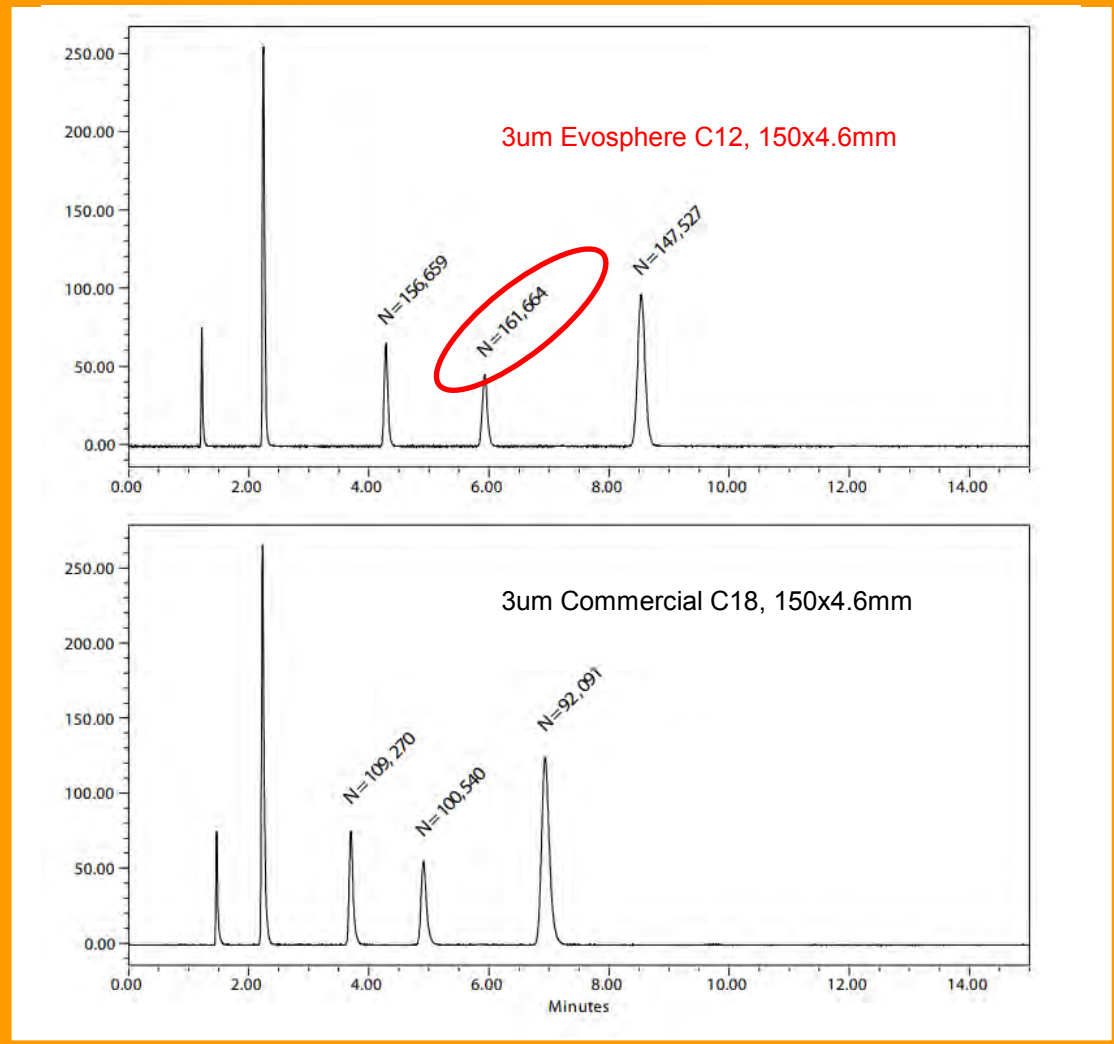
Efficiency Gains

The efficiency of columns packed with the new monodisperse particles were then compared against a number of existing commercial (non-monodisperse) columns and the results can be seen in figures 4 and 5.

FIGURE 4. Efficiency Gains Provided by Monodisperse Silica



FIGURE 5. Efficiency Gains

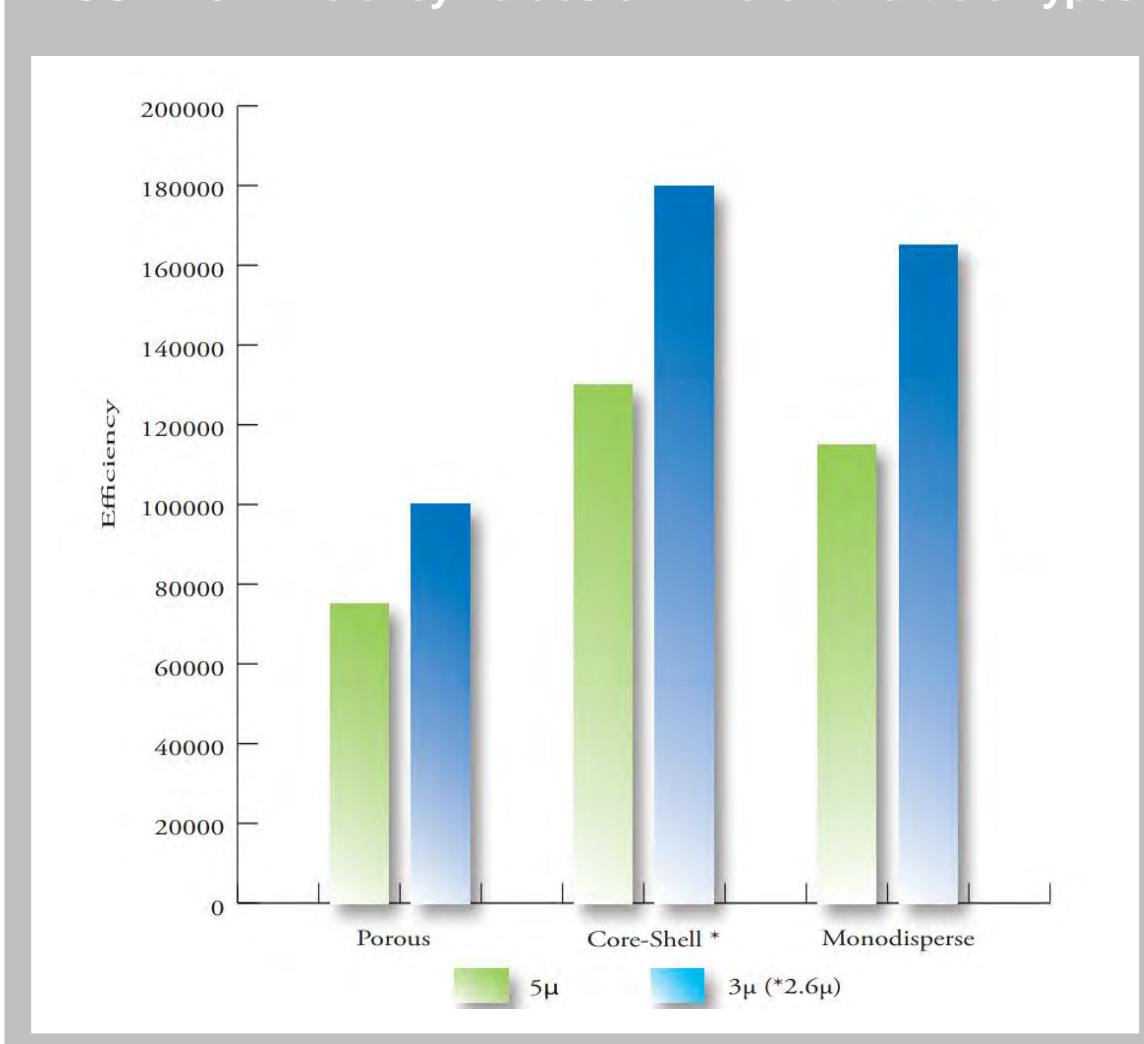


SPP (Core-shell) versus MFPP

The recent adoption of superficially porous particles (SPP) in HPLC has been mostly due to the high column efficiencies achievable without generating the high backpressures that require UHPLC instrumentation. This performance is generated due to the low Eddy diffusion (A-term) coupled with rapid mass transfer (C-term).

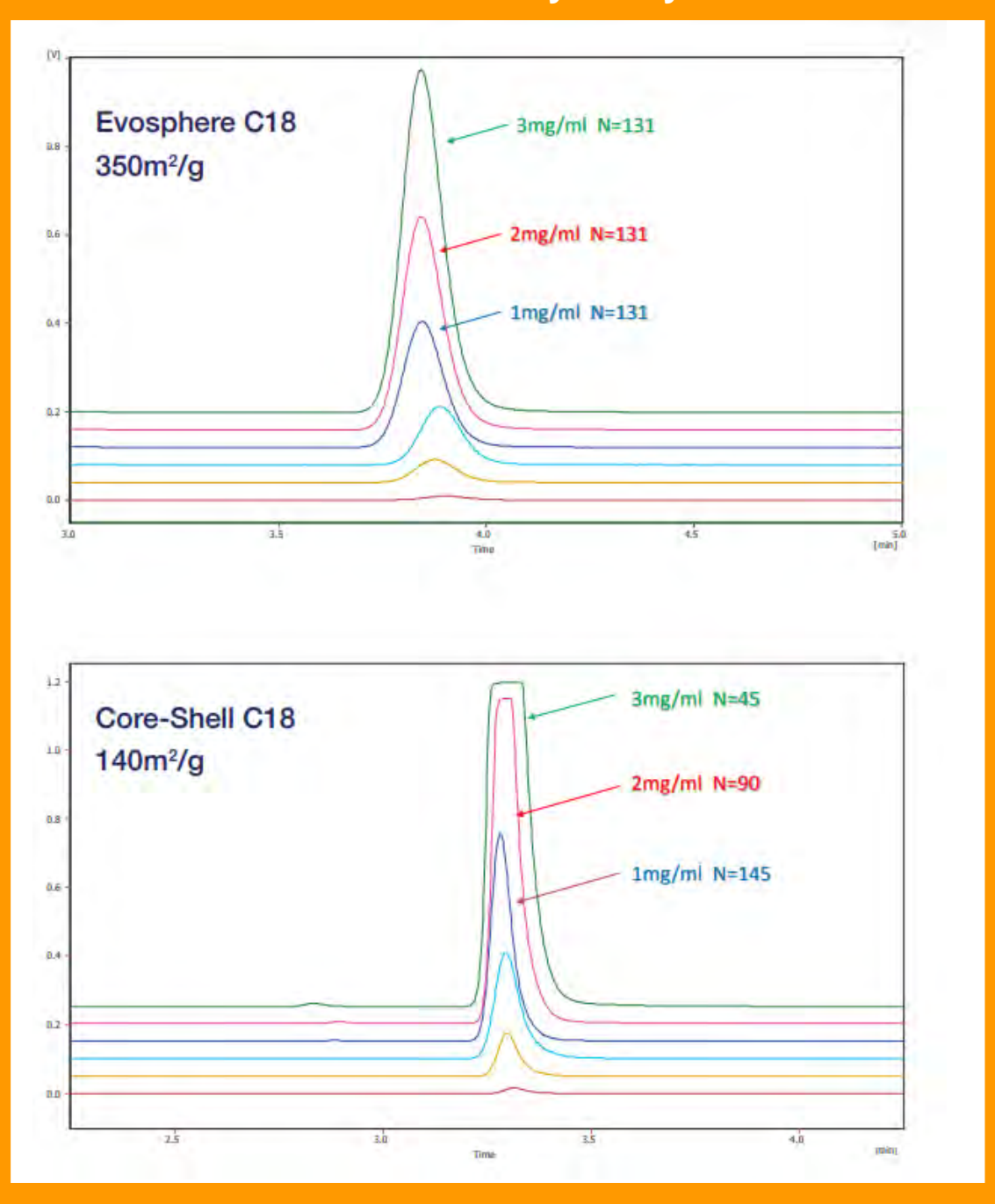
When we compare SPP efficiencies with those of MFPP, that have a similar Eddy diffusion, we can see that for the first time fully porous particles can perform to a similar level as SPP (see figure 6).

FIGURE 6. Efficiency Values of Different Particle Types



Where MFPP come into their own is the ability to scale up processes in terms of size and loadability. Due to the much higher surface area of monodisperse fully porous particles sample loading can be increased without the loss of peak shape and efficiency as seen with SPP (see figure 7).

FIGURE 7. Column Loadability Study



Another consideration when looking to move a chromatographic method from the laboratory to the production facility is the cost and availability of the materials involved. Due to the processing costs of SPP these materials can be prohibitively expensive or not available to the customer in larger scale. Whereas the MFPP discussed in this poster are fully scalable and produced at traditional cost levels.

Conclusion

Our new silica particles have been manufactured to provide a high degree of monodispersity with a uniform smooth surface. Monodispersity generates high efficiency HPLC columns due to the reduced flow path dispersion (Eddy diffusion). SEM imagery of the Evosphere® in comparison with traditional particles highlights the much narrower size distribution. Combining this monodisperse particle with a range of surface chemistries can provide the ultimate in terms of sample resolution and sensitivity when developing new and improved HPLC methods.