



JOURNAL OF PHARMACEUTICAL SCIENCE AND BIOSCIENTIFIC RESEARCH (JPSBR)

(An International Peer Reviewed Pharmaceutical Journal that Encourages Innovation and Creativities)

Core-Shell / Solid core Particles: A Practical Substitute to Sub-2 Micron Particles: An overview

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ABSTRACT:

This review describes an short impression of the recent trends in core shell HPLC columns. Core-shell technology provides an well-designed solution to the problem of maximizing column performance without generating extreme column back-pressure. They consist of an impermeable inner core surrounded by a layer of fully-porous silica and thus are morphologically quite different from conventional fully-porous silica particles.



Figure 1

Figure 1 : Core shell silica and porous silica Particles

The sub-2 micron particle size advantages are available in many publications. The use of these columns only on ultra-High pressure liquid Chromatography (UHPLC) Instrumentation having pump capacity is more than 900 bar. However, such instruments may not be readily available to every analyst. Core-Shell / Solid core practical alternative has been introduced which propose better efficiencies with using conventional HPLC.

KEY WORDS: Core-Shell, UHPLC, porous silica, column performance.

Article history:

Received 01 Feb 2015

Revised 03 Dec 2015

Accepted 10 Dec 2015

Available online 01 Jan 2016

Citation:

Ramdharane S. M., Vyas K. B., Nimavat K. Core-Shell / Solid core Particles: A Practical Substitute to Sub-2 Micron Particles: An overview *J Pharm Sci Bioscientific Res.* 2016 6(1):162-167

INTRODUCTION:

In the earlier time there has been continuous drive to develop chromatographic stationary phases to perform fast HPLC separations, as sample quantity can be increased and therefore time and cost per sample reduced. The theory of chromatography expects that the efficiency of a HPLC separation increases with decreasing particle size. (Figure 2)¹

As such, most columns currently used for fast HPLC are packed with particles in the sub-2µm internal diameter region. The small particle diameter

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improves the separation and reduce run time and therefore efficiency, but at the expense of increased operating backpressure (Figure 3)²

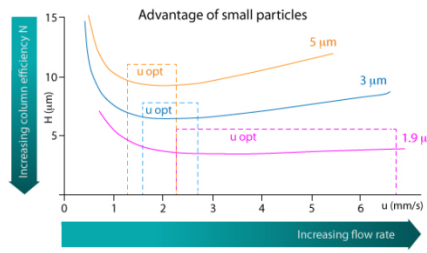


Figure 2: Van Deemter curves for various particle diameters.

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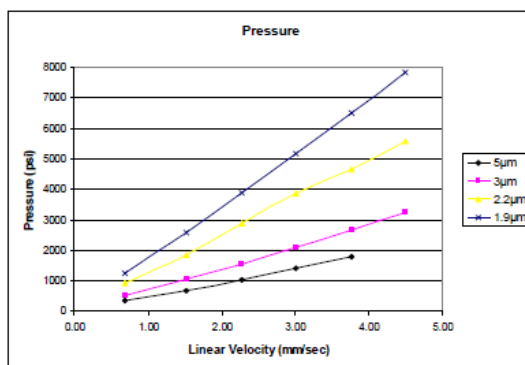


Figure 3 : Empirically measured column backpressures for the columns at various linear velocities.

Column backpressure is direct relation with the porosity of the packing material and also relation with speed of analysis.. Lately, approaches of HPLC technologies have interested in increasing the speed of analysis.^{2,3,4}

The shell-core particle technology, also named superficially porous or solid core or fused core particles, was first developed by Jack Kirkland where it was intended to achieve faster separation with higher sample throughput with maintaining column reliability^{5,6}.

This approach should be run in balance with low back pressure and maximum column efficiencies.

Theory of core-shell/solid core particles⁸

The general resolution equation relates the separation power of the chromatographic support to its efficiency, selectivity and retention capacity, which are dependent on Particle size and quality of the packing, bonded phase chemistry and surface area Respectively. Efficiency is solute independent (i.e. is an inherent function of the

physical properties of the column), whereas retention factor and selectivity are not.

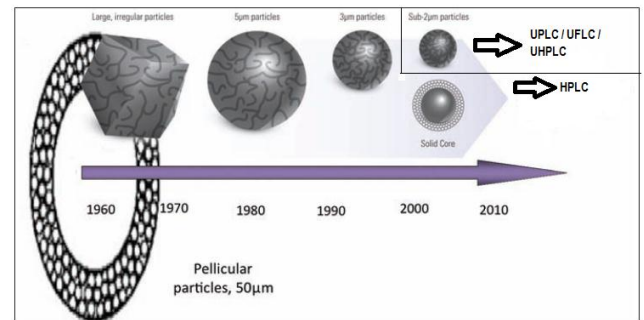


Figure 4⁷: Particle evolution: packing materials have changed from large pellicular particles via smaller totally porous particles to spherical particles with diameters of less than 2μm, to 2.6μm core shell particles

Equation 1

$$R_S = \frac{1}{4} \sqrt{N} \left(\frac{\alpha-1}{\alpha} \right) \left(\frac{k'}{1+k'} \right)$$

- Rs – resolution
- N – efficiency
- α – selectivity factor
- k' – retention factor

Equation 2

$$HETP = A + \frac{B}{\mu} + C_m \mu + C_s \mu$$

- HETP – height equivalent to a theoretical plate
- μ - Linear velocity of mobile phase
- A – Eddy diffusion constant
- B – Longitudinal diffusion constant
- C_m – Resistance to mass transfer in the mobile phase
- C_s - Resistance to mass transfer in the stationary phase

The height equivalent to a theoretical plate (HETP) is generally used as a measure of efficiency when comparing columns. HETP is related to linear velocity through the column via the van Deemter equation. In this equation A, B and C (both components) are constants that describe contributions to band broadening through Eddy diffusion, longitudinal diffusion and resistance to

mass transfer respectively. Peak or band broadening is the consequence of several mass transfer processes that occur as the analyte molecules migrate down the column. The A-term, Eddy diffusion, is dependent on particle size and the homogeneity of the packed bed. Smaller particles reduce the A-term and therefore improve efficiency. The average particle size distribution of a spherical chromatographic medium is generally defined through the ratio $d_{90}/10$; the closer this value is to 1 the less spread there is on the average diameter of the particles. The Accucore material has a $d_{90}/10$ of 1.12 whereas most fully porous particles have a $d_{90}/10$ around 1.50. The schematic on Figure 5 illustrates the effect of the average particle size distribution on the homogeneity of the chromatographic packed bed.

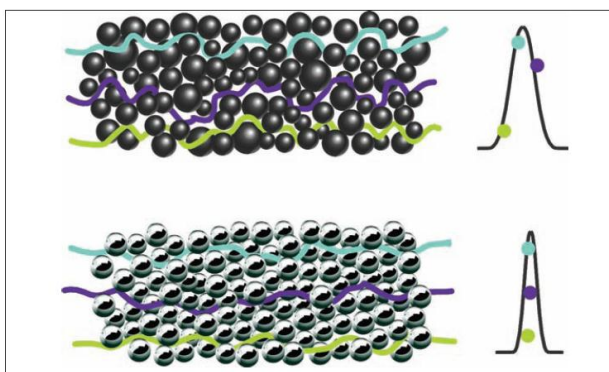


Figure 5 : Effect of the average particle size distribution

Whereas the A-term is independent of the linear velocity of the mobile phase through the column, the C-term, resistance to mass transfer, is proportional to it and therefore an important consideration when working with fast separations. The C-term has two contributors:

- resistance to the mass transfer in the stationary phase C_s
- resistance to the mass transfer in the mobile phase C_m .

The first occurs when the analyte molecule diffuses in and out of the pores of the stationary phase particle. With solid-core particles the diffusional path of the analytes is limited by the depth of the outer porous layer, and therefore analytes do not have the propensity to have greater diffusional lengths within the more limited pore structure of the solid-core material. This results in less band broadening and more efficient peaks. The resistance to mass transfer in the mobile phase is caused by the fact that the liquid is flowing in the channels

between particles and analytes have to diffuse through the liquid to reach the stationary phase. This effect is equivalent to the longitudinal diffusion, however whereas with the longitudinal diffusion increasing the flow reduces the band broadening, increasing the flow will have an adverse effect on the homogeneity of the flow in a radial direction. Analytes that are in the centre of the flow will have a longer diffusional path to the particle than analytes that are at the edge nearer to the particle. Better packing and smaller particles result in a more uniform diffusional path in the liquid mobile phase. From the discussion above we may expect solid-core particle packed columns to be more efficient than fully porous particle packed columns of the same average particle diameter. Both the A and C-terms are reduced, and therefore H is reduced which equates to higher efficiencies. It would also be expected that the drop off in efficiency that is seen with increasing flow rates will be less with solid-core material than with fully porous material due to a lesser contribution from the resistance to mass transfer terms. The next section will investigate the experimental findings found when comparing porous and solid-core particles.

Core-shell / Solid core Advantage⁹

Core-shell / Solid core columns provide comparable efficiency (more resolving power) with fully porous sub 2μ columns. Can be suitable in conventional HPLC instrumentation

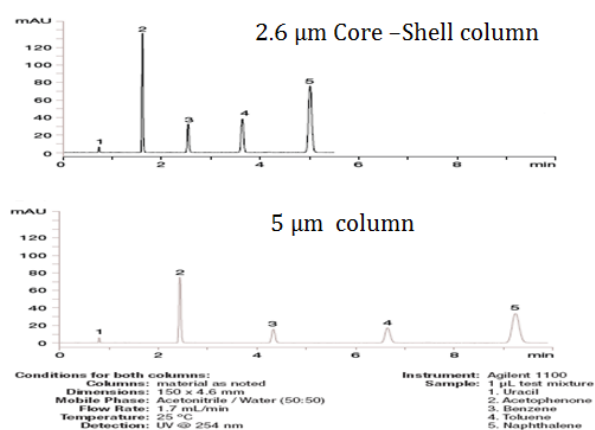


Figure 6: one of the application HPLC vs Core- Shell⁹

Core-shell / Solid core columns having increase peak capacities, Narrow chromatographic peak widths and improve resolution and improved sensitivity (better signal to noise ratios) Band broadening in the column is

significantly reduced with these chromatographic using conventional HPLC instrumentation. technology. Run time is reduce as compare to UHPLC

Current popular available phases with brand name

Table -1

Company : Advanced Material Technology		Company : Sigma-Aldrich Co. LLC	
Brand : HALO ¹⁰		Brand : Ascentis® Express ¹¹	
Particle size 2.7 µm		Particle size :2.7 µm	
Name	Phase	Name	Phase
Octadecyldimethylsilane	C-18	Octadecyldimethylsilane	C-18
Octyldimethylsilane	C-8	Octyldimethylsilane	C-8
Phenyl Hexyl	Phenyl	Phenyl Hexyl	Phenyl
RP-Amide	Amide	RP-Amide	Amide
Pentafluorophenylpropyl	PFP	Pentafluorophenylpropyl	F5
ES-CN	CN	ES-CN	CN
Peptide ES-C18	C-18	Peptide ES-C18	C-18
Penta HILIC	HILIC	HILIC(Si)	HILIC
HILIC	HILIC		

Table-2

Company : Phenomenex Inc		Company : Fortis Technologies Ltd	
Brand : Kinetex ¹²		Brand : Speedcore ¹³	
Particle size 2.6 µm		Particle size :2.6 µm	
Name	Phase	Name	Phase
Octadecyldimethylsilane	C-18	Octadecyldimethylsilane	pH Plus C-18
Octadecyldimethylsilane	EVO-C18	Octadecyldimethylsilane	C-18
Octadecyldimethylsilane	XB-C18	Pentafluorophenylpropyl	PFP
Octyldimethylsilane	C-8	Biphenyl	Biphenyl
Phenyl Hexyl	Phenyl	Pentafluorophenylpropyl	F5
Pentafluorophenylpropyl	F5	HILIC	HILIC
Biphenyl	Biphenyl		
HILIC	HILIC		

Table-3

Company : YMC CO., LTD.		Company : Knauer	
Brand : Meteoric Core ¹⁴		Brand : Blueshell ¹⁵	
Particle size 2.7 µm		Particle size :2.6 µm	
Name	Phase	Name	Phase
Octadecyldimethylsilane	C-18	Octadecyldimethylsilane	C-18
Octadecyldimethylsilane	C18-BIO	Octadecyldimethylsilane	C-18A
Octyldimethylsilane	C-8	HILIC	HILIC
		Pentafluorophenylpropyl	PFP
		Phenyl Hexyl	Phenyl

Table-4

Company : Agilent Technologies		Company : ChromaNik Technologies Inc.	
Brand : Poroshell 120 ¹⁶		Brand : Sunshell ¹⁷	
Particle size 2.7 µm		Particle size :2.6 µm	
Name	Phase	Name	Phase
Octadecyldimethylsilane	EC-C18	Octadecyldimethylsilane	C18
Octadecyldimethylsilane	HPH-C18	Octadecyldimethylsilane	C18-WP
Octadecyldimethylsilane	SB-C18	Octadecyldimethylsilane	HFC18-16
Octyldimethylsilane	EC-C8	Octadecyldimethylsilane	HFC18-30

Octyldimethylsilane	HPH-C8	Octyldimethylsilane	C8
Octyldimethylsilane	SB-C8	Octyldimethylsilane	C8-30
Alkylamide	Bonus-RP	Phenyl Hexyl	Phenyl
HILIC	HILIC	Pentafluorophenylpropyl	PFP
Cyno	EC-CN	Butyl	C4-30
Alkyl reverse phase	SB- Aq	Alkyl reverse phase	RPAQUA
		2-Ethylpyridine	2-EP
		Amide	HILIC-Amide

Table-5

Company : Macerey Nagel		Company : Restek	
Brand : Nucleoshell ¹⁸		Brand : Raptor ^{TM19}	
Particle size 2.7 µm		Particle size :2.7 µm	
Name	Phase	Name	Phase
Octadecyldimethylsilane	RP-C18	Octadecyldimethylsilane	C-18
Octadecyldimethylsilane	RP-C18Plus	Octadecyldimethylsilane	ARC-18
Phenyl Hexyl	Phenyl	Biphenyl	Biphenyl
Pentafluorophenylpropyl	PFP		
HILIC	HILIC		

Table-6

Company : Thermoscientific		Name	Phase
Brand : Accucore ²⁰		Polar Premium	Polar Premium
Particle size 2.6 µm		Phenyl Hexyl	Phenyl
Name	Phase	Pentafluorophenylpropyl	PFP
Octadecyldimethylsilane	C-18	Phenyl -X	Phenyl -X
Octadecyldimethylsilane	aQ	Silane bonded phase	C-30
Octadecyldimethylsilane	150-C18	HILIC	HILIC
Octyldimethylsilane	C8	RP-MS	RP-MS
Urea-HILIC	Urea-HILIC	Butyl	150-C4
Amide	150-Amide-HILIC		

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