Development of Superficially Porous Silica with Polyfunctional C18 Bonding Technique for Reversed Phase HPLC

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Abstract

Brand columns packed with superficially porous particles have been available for some time. The superficially porous media or so called core-shell media offers significant improvements such as higher efficiency and lower pressure drop for existing HPLC operations without having to replace existing HPLC systems with UHPLC systems.

In this study, core-shell silica bonded with C18 using polyfunctional bonding technique (SunShell C18) was evaluated. A 2.6 µm core-shell silica with a non-porous core approximately 1.6 µm in diameter and a superficially porous layer of 0.5 µm was used to be bonded with hexamethyloctadecyltetrasiloxane (HMODTS) and end-capped with trimethylsilane.

Proposed core-shell C18 yielded efficiencies close to totally porous sub-2 µm or 2 µm C18 and showed less than half the pressure drop to compare with the sub-2 µm or 2 µm C18. Stability under both acidic and basic pH conditions as well as peak shape of basic, acidic and metal-chelating compounds were evaluated and compared with other core-shell type columns. Proposed core-shell C18 was more stable than the other core shell C18s under acidic and basic pH conditions.

Loading capacity of amitriptyline was examined in acid, low-ionic-strength mobile phases such as a mixture of 0.1% formic acid and acetonitrile and 20 mM phosphate buffer pH7.0 and acetonitrile.

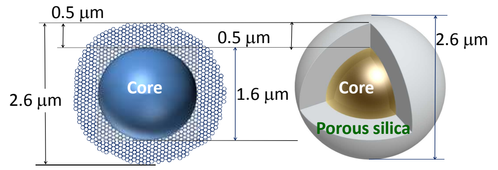


Figure 1. Schematic diagram of a core shell silica particle

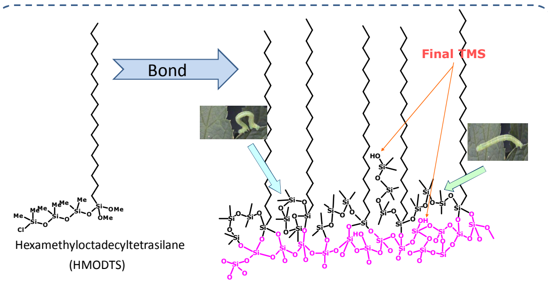


Figure 2. Schematic diagram of bonding of SunShell C18（Core Shell particle）

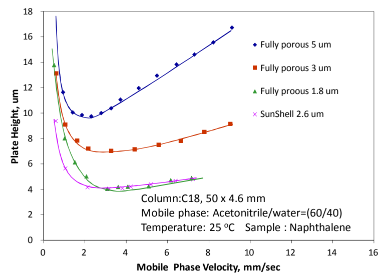


Figure 3. Comparison of Plate Height Plots

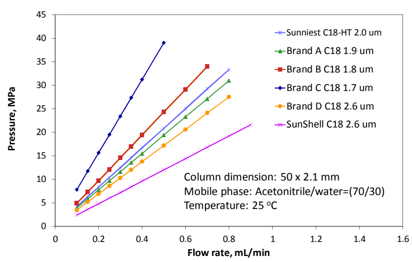
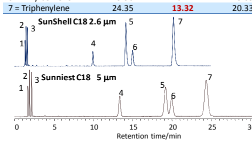


Figure 4. Comparison of Back Pressure for High Throughput Columns

Table 1. Comparison of retention between fully porous silica C18 and core shell silica C18s

	Fully porous silica C18 Sunniest C18 5 µm		Core shell silica C18 SunShell C18 2.6 µm		Core shell silica C18 Kinetex C18 2.6 µm	
Specific surface area	340 m²/g		150 m²/g		Effective 200 m²/g	
	Retention time (t_R)	Retention factor (k)	Retention time (t_R)	Retention factor (k)	Retention time (t_R)	Retention factor (k)
1 = Uracil	1.70	0	1.35	0	1.36	0
2 = Caffeine	1.90	0.12	1.47	0.09	1.49	0.10
3 = Phenol	2.17	0.28	1.65	0.22	1.61	0.18
4 = Butylbenzene	13.35	6.85	10.01	6.41	6.19	3.55
5 = o-Terphenyl	19.19	10.29	14.24	9.55	8.15	4.99
6 = Amylbenzene	19.96	10.74	15.09	10.18	8.75	5.43
7 = Triphenylene	24.35	13.32	20.33	14.06	9.44	5.94



Mobile phase: Methanol/water(75:25)
Temperature: 40°C
Column dimension: 150 x 4.6 mm
Flow rate: 1.0 mL/min

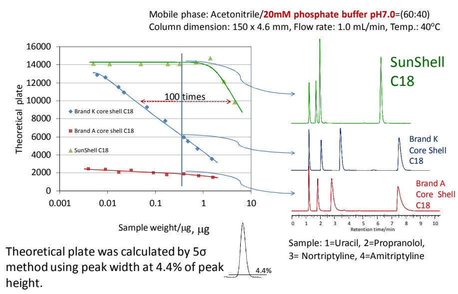


Figure 5. Loading capacity of amitriptyline I

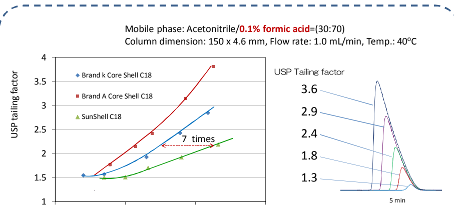


Figure 6. Loading capacity of amitriptyline II

In the case of using acetonitrile /0.1% formic acid as a mobile phase, amitriptyline peak shows more tailing because a loading capacity decreases in an acidic, low-ionic-strength mobile phase.

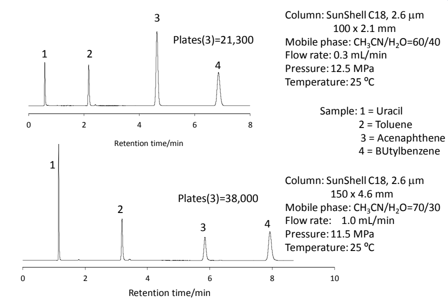
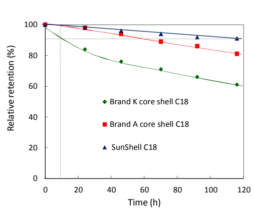


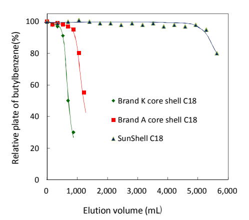
Figure 7. Chromatogram of standard samples of SunShell C18



Durable test condition
Column size: 50 x 2.1 mm
Mobile phase: CH_3CN/1.0% TFA, pH1=10/90
Flow rate: 0.4 mL/min
Temperature: 80 °C

Measurement condition
Column size: 50 x 2.1 mm
Mobile phase: CH_3CN/H_2O=60/40
Flow rate: 0.4 mL/min
Temperature: 40 °C
Sample: 1 = Uracil
2 = Butylbenzene

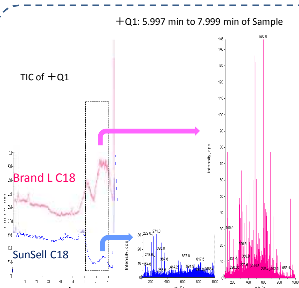
Figure 8. Stability under acidic pH condition



Durable test condition
Column Size: 50 x 2.1 mm
Mobile phase: CH_3OH/20mM Sodium borate/10mM NaOH=30/21/49 (pH10)
Flow rate: 0.4 mL/min
Temperature: 50 ºC

Measurement condition
Column Size: 50 x 2.1 mm
Mobile phase: CH_3OH/H_2O=70/30
Flow rate: 0.4 mL/min
Temperature: 40 ºC
Sample: 1 = Butylbenzene

Figure 9. Stability under basic pH condition



Column size: 50 x 2.1 mm
Mobile phase:
A) 0.1% acetic acid
B) CH_3CN
Gradient:
Time: 0min 1min 5min 7min
%B: 5% 5% 100% 100%
Flow rate: 0.4 mL/min
Temperature: 40 °C
MS : ABI API-4000
Ionization: Turboionspray (cation)
Measurement mode: Q1 Scan m/z 100-1000

Figure 10. Bleeding test using LC/MS

Conclusion

*Core shell particle bonded with polyfunctional C18 (SunShell C18) showed 2 times higher retention factor than Kinetex C18.
*SunShell C18 had 7 to 100 times higher loading capacity than the other core shell C18.
*Furthermore, SunShell C18 was not only stable under acidic and basic pH conditions to compare with the other core shell C18s, but also showed less bleeding under a typical LC/MS condition.