Microlute® CSi: Evaluation of a Novel Composite Silica Technology for Solid Phase Extraction

James Edwards

Solid phase extraction (SPE), LC-MS, pharmaceutical, composite silica, C18, Microlute® CSi

Introduction:

Solid phase extraction (SPE) is the gold standard for sample preparation in chromatographic analysis. It is used to capture, clean and concentrate samples while removing interfering compounds to obtain reliable and sensitive chromatographic data.

Traditionally, SPE products are in a loose packed format with active resin between porous frits. However, these products suffer from issues such as channelling, inconsistent resin mass and voiding which lead to reduced recovery and poor reproducibility.

The Microlute® CSi products use a novel composite technology which uses a blend of porous plastic and chromatographic SPE resin. This technology has been designed to eliminate issues caused by inherent issues and inconsistent packing of loose packed SPE plates.

The data in this application note compares SPE results from a C18 10 mg loose packed plate with a C18 10 mg composite plate (Microlute® CSi) which were both created using the same batch of C18 resin.

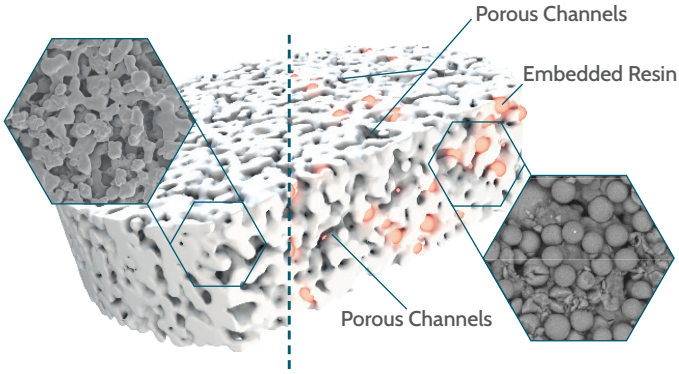

Figure 1. Schematic illustration of composite technology

Materials and methods:

A range compounds were chosen consisting of acidic, basic and neutral analytes. Each was selected to due to their intrinsic physical and chemical properties such as pKa and LogP. Using a wide range of compounds enabled a more detailed comparison between each plate format. A total of six replicates were tested from each compound class with an appropriate SPE method.

The SPE followed a typical 10 mg reversed phase method:

Condition: 500 µL MeOH
Equilibration: 500 µL of H_2O
Load: 500 µL sample*
Wash: 500 µL of H_2O
Elute: 2 x 250 µL MeOH**

*: pH adjusted to neutralise any charge on acidic and basic compounds

**: pH adjusted to charge the acidic and basic compounds

The eluted samples were dried down and reconstituted before being analysed with an Agilent 1260 HPLC attached to an Agilent Single Quadrupole MS/MSD.

Compound	LogP	pKa	Analyte Type
Atenolol	0.16	9.60	Basic
Pindolol	1.75	9.25	Basic
Dexamethasone	1.83	-3.30, 12.42	Neutral
Corticosterone	2.09	-0.26, 13.86	Neutral
Carbamazepine	2.77	-3.80, 15.96	Neutral
Ketoprofen	3.12	4.45	Acidic
Naproxen	3.18	4.15	Acidic
Niflumic acid	4.43	5.30	Acidic
Propranolol	3.48	9.42	Basic
Nortriptyline	3.90	9.70	Basic
Desipramine	4.90	10.40	Basic

Table 1: Properties of the analytes used in testing [1]

Results and Discussion:

Recovery

The data obtained showed that both plates performed well in analyte recovery across all 11 compounds. Average recoveries of 91% and 88% using the composite and the loose packed plates respectively, demonstrates the utility of the SPE technique in sample preparation for chromatography.

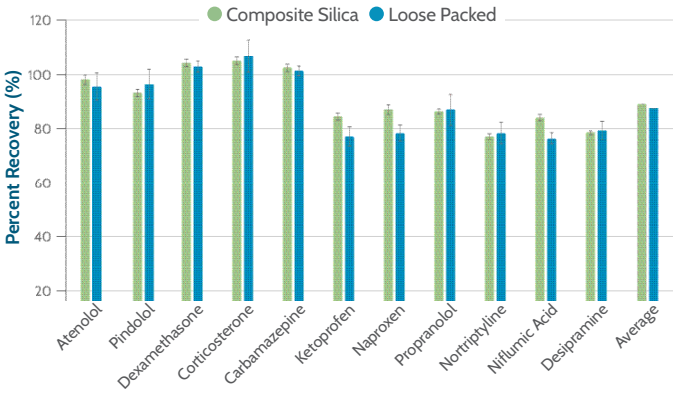

Figure 2: Percent recovery for each analyte using an average recovery calculated from six replicates.

Reproducibility

The difference in reproducibility between the two plates was significant with a substantial improvement when using the composite plate. This is clearly demonstrated by calculating an average percentage relative standard deviation (% RSD) across the compounds tested; the loose packed plate had an RSD of approximately 6% compared to less than 2% for the composite plate (Microlute® CSi).

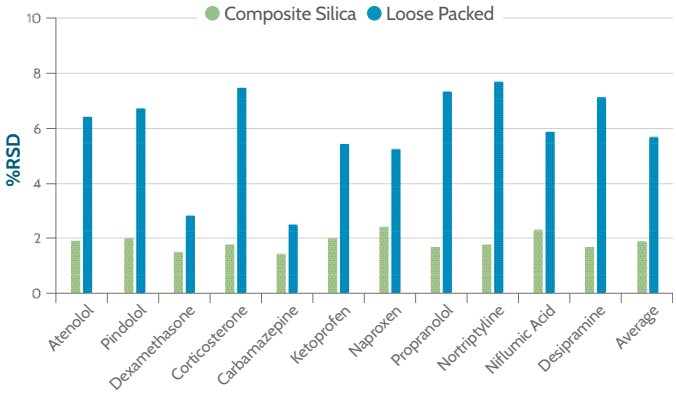

Figure 3: Percent relative standard deviation for recovery for each analyte using six replicates.

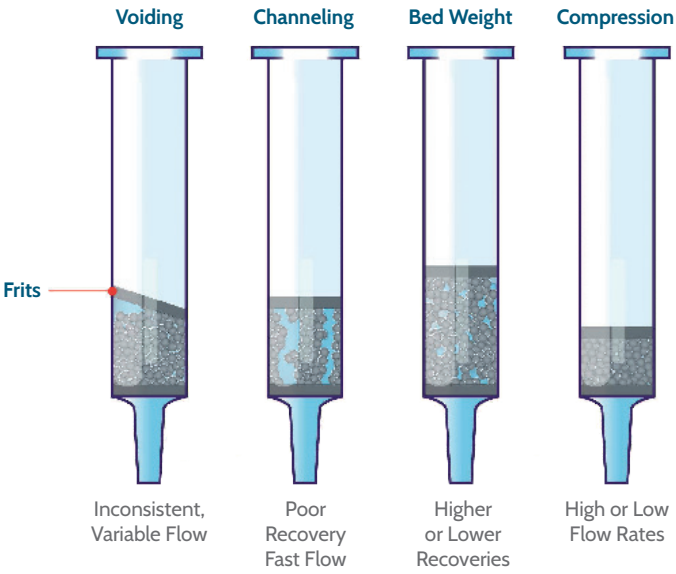

Figure 4: Common problems with traditional loose packed products.

As can be seen from the above data the composite silica plate had noticeably greater reproducibility across all 11 compounds tested. Lower recoveries and poorer reproducibility in the loose packed plate can be attributed to inconsistencies in the packing of chromatographic media inherent to this process (see figure 4 for details). The unique immobilisation technology used in Microlute® CSi ensures consistent bed weights and optimal liquid flow through every well, plate after plate. This leads to predictable interaction of solutions and analytes with the active chromatography resin.

Conclusions:

The 10mg Microlute® CSi C18 composite silica plate for SPE gives excellent reproducibility and recovery across all compounds tested in this study. The uniformity of the composite technology enables an even distribution of active media and maximises interactions throughout the structure leading to exceptionally reproducible data. As demonstrated in this short study the novel composite format enhances recovery and can provide up to 3 x times better reproducibility than traditional loose packed plate formats.

Citation: 1. "Drugbank," Drugbank, [Online]. Available: https://go.drugbank.com/. [Accessed 2024]