APPLICATION BRIEF

Liquid Chromatography

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HPLC Analysis of Ibuprofen in Water Using a Quasar SPP C18 Column

Introduction

Pharmaceuticals are continuously entering our waterways via human excretion of incompletely absorbed medication and improper disposal of unused drugs via drains and toilets.[1] A report found that the six most consistent highest reported concentrations in finished drinking water for active pharmaceutical ingredients (APIs) were from ibuprofen, triclosan, carbamazepine, phenazone, clofibric acid, and acetaminophen.[2]

Ibuprofen (Figure 1) is a widely used analgesic and antipyretic for adults and children and since it's one of the most reported pharmaceutical in finished drinking waste, it is important to determine the amount removed by different water treatment processes.[2,3] HPLC is an essential technique to determine the quantity of ibuprofen in water and help optimise a process for its removal. This application brief describes the use of a Quasar superficially porous particles (SPP) C18 column for the analysis of ibuprofen in water.

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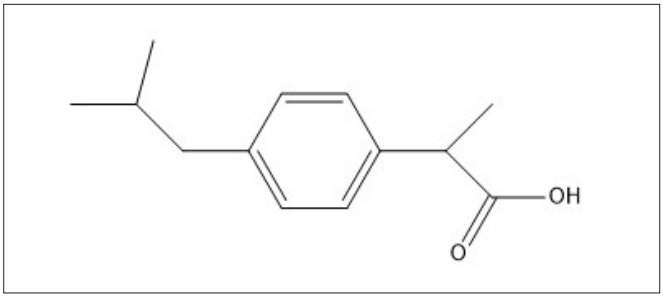

Figure 1. Chemical structure of ibuprofen.

Experimental Conditions

Method Parameters

Table 1. HPLC method parameters.

Instrument	PerkinElmer Flexar™ with PDA Plus™ Detector
Column	Quasar SPP C18 150 x 4.6 mm, 2.6 µm P/N: N9308910
Mobile Phase	60:40, ACN : H_2O (0.1 % phosphoric acid)
Flow Rate	0.6 ml/min
Temperature	25 °C
Wavelength	220 nm
Injection Volume	3 µL
Analyte	Ibuprofen

Solvents and Reagents

All solvents and reagents used were HPLC grade. A stock solution of ibuprofen (1 mg/mL) was prepared in acetonitrile. From this solution, a working standard solution was prepared (0.2 mg/mL) by dilution with distilled water. Before injection into the HPLC system, the standard was filtered using a 0.45 µm PTFE syringe filter (P/N: 02542885).

Results

Superficially porous particles, SPP, (also called: shell, fused-core, core-shell, partially porous, pellicular) are made of a solid, non-porous core surrounded by a shell of a porous silica material that has properties similar with those of the fully porous silica materials conventionally used in HPLC. The terminology of "fused-core" was introduced by Jack Kirkland. As the name implies, fused-core particles are manufactured by "fusing" a porous silica layer onto a solid silica particle (Figure 2). Such phases can be used on standard HPLC instrumentation, without worrying about high backpressure or compromising column longevity. With a shorter diffusion path with the SPP particle itself, coupled with a uniform packed bed and ultra-inert silica surface, reductions in run times can be observed. Use of these SPP columns also often results in higher kinetic efficiencies and savings in solvent consumption.

The analysis of ibuprofen in water using a Quasar SPP C18 (150 x 4.6 mm, 2.6 µm) column is shown in Figure 3. Ideally suited to the analysis of small molecules, the Quasar SPP C18 phase provides high efficiency for the analysis of ibuprofen in water (17,011 N using the Foley-Dorsey approximation method). Good peak shape is also observed due to the ultra-high purity silica base and low residual silanol activity. The run time of the method could be reduced by increasing the flow rate.

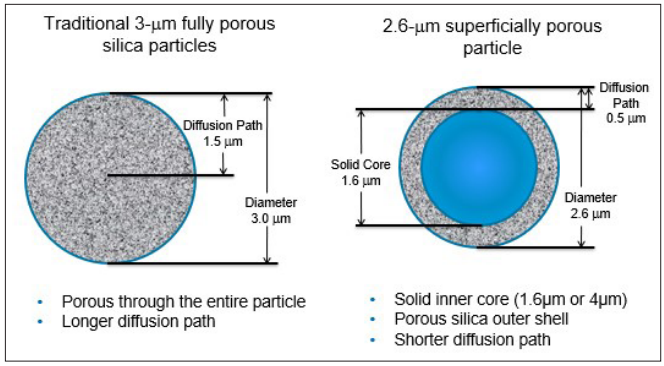

Figure 2. Comparison of a conventional fully porous particle (left) and a superficially porous particle (right).

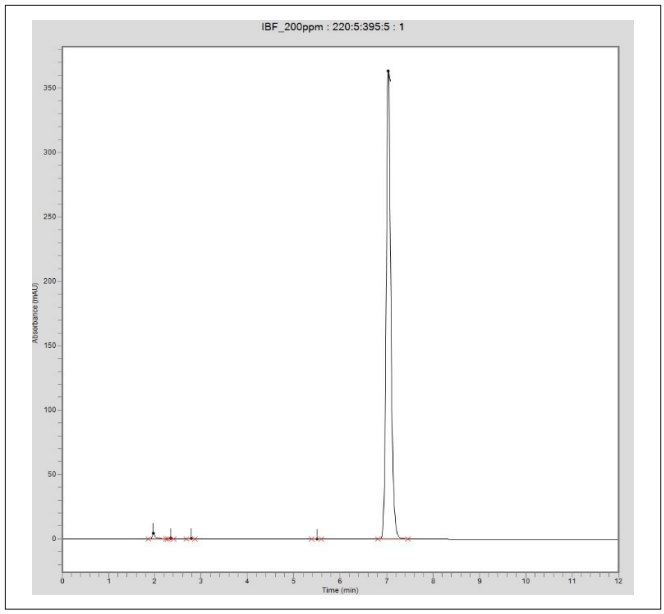

Figure 3. HPLC analysis of ibuprofen.

Conclusion

- Good retention and peak shape for the analysis of ibuprofen in water is observed on the Quasar SPP C18 phase.
- Run time could be reduced by using a faster flow rate.
- SPP columns provide faster run times in comparison with conventional fully porous particle columns, as well as higher kinetic efficiencies and savings in solvent consumption.
- The ultra-high purity silica base and low residual silanol activity yields excellent peak shape.

References

1. J. Bound and N. Voulvoulis, Predicted and measured concentrations for selected pharmaceuticals in UK rivers: implications for risk assessment, Water Research, 40, 2006, 2885-2892.
2. C.G. Daughton, Pharmaceutical Ingredients in Drinking Water: Overview of Occurrence and Significance of Human Exposure. Chapter 2, R. Halden (ed.), Contaminants of Emerging Concern in the Environment: Ecological and Human Health Considerations. American Chemical Society, Washington, DC, 1048:9-68, (2010).
3. R. Bushra and N. Aslam, An Overview of Clinical Pharmacology of Ibuprofen, Oman Medical Journal, 25, 2010, 155-1661.

Consumables Used

Component	Description	Part Number
Column	Quasar SPP C18 (150 x 4.6 mm, 2.6 µm)	N9308910
HPLC Vials	2 mL, 9 mm Screw Top Vial with Write-on Patch and Fill Lines (100/pack)	N9307802
HPLC Vial Caps	9 mm Screw Top Blue (polypropylene) Cap with PTFE/Silicone pre-slit Septa (100/pack)	N9306203
Syringes	Syringe 1 mL BD Luer-Lok Disposable, Pack of 100	02542890
Syringe Filters	0.45 µm PTFE syringe filter	02542885
PEEK Fittings	Fingertight for 1/16" OD PEEK tubing	09920513

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