Chlorinated Compounds in Linseed Oil Analyzed with Agilent EcoSpheres

Application Note

Food Testing and Agriculture

Author

Greg Saunders and
Ben MacCreath
Agilent Technologies, Inc.
2850 Centerville Road
Wilmington, DE 19809-1610
USA

Introduction

Linseed is the seed of the flax plant Linum usitatissimum L., Linaceae. Its oil is derived from the seed by cold-pressing. Because linseed oil can polymerize into a solid form it is used as a drying oil in, for example, wood finishing, as a binder in oil paints, and as a hardener in putty. The oil is also edible and has a minor role in human nutrition. Although industrial use of linseed oil has declined because of the emergence of synthetic alkyd resins, it continues to be sold as a nutritional supplement. For this reason, there is a need for analytical methods to ensure the oil is free of contamination, particularly pesticide residues. However, food samples can have a complex contamination profile and may require clean-up before attempts are made to isolate the contaminants of interest.

Gel permeation chromatography (GPC) with Agilent EcoSpheres is an ideal technique for the clean-up of samples from food sources. GPC uses a size-based separation to distinguish the species of interest from higher molecular weight matrices. The size exclusion separation is based on partitioning the components of the sample between a flowing liquid phase and stationary liquid trapped in the pores of a suitable material, typically porous crosslinked polymer beads. The pore size in the media is carefully controlled such that smaller molecules are able to enter the pores, whereas large molecules have restricted access. Larger molecules therefore elute from the column very quickly whereas smaller molecules are retained and elute later.

EcoSpheres is an economic, loose microporous material in a dry-powder form ready for swelling and gravity packing into glass columns. Microporous materials have low levels of crosslinking and no permanent pore structure. Instead, pores are generated when these materials swell in solvents. Microporous packings allow higher loadings, where high resolution is not required.

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Method and Results

A sample of linseed oil spiked with trichlorobenzene was used to test the use of GPC and EcoSpheres in the analysis of this nutritional supplement.

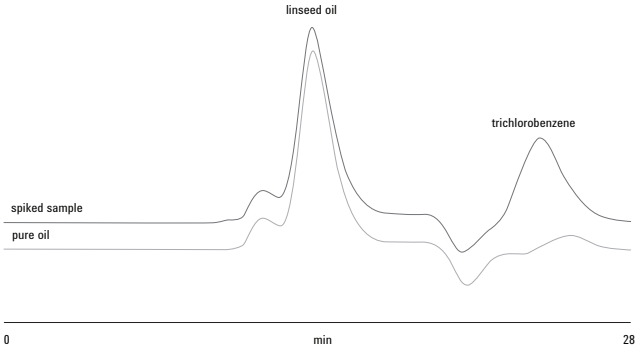

Figure 1. Overlaid chromatogram of samples of spiked and un-spiked linseed oil produced by gel permeation chromatography with EcoSpheres microporous media. The presence of trichlorobenzene is evident in the spiked sample.

Conditions

Column:	EcoSpheres glass column, 450 x 25 mm (EcoSpheres loose media (100 g) p/n PL1460-4M03; complete glass column p/n PL1310-0054)
Sample:	Linseed oil spiked with trichlorobenzene
System:	Agilent 1260 Infinity Isocratic Pump and Manual Injector
Eluent:	Tetrahydrofuran
Flow Rate:	5 mL/min
Detector:	Agilent 1260 Infinity Variable Wavelength Detector VL, 254 nm
Data collection/ analysis:	Cirrus GPC software and PL DataSteam data capture unit

Conclusion

A GPC system using EcoSpheres material is an easy and simple method for the analysis of food samples such as linseed oil. In this example, a chlorinated compound was detected within 24 min.

EcoSpheres beads are designed for the economic, low pressure separations of pesticides, chlorinated hydrocarbons and other small molecules from high molecular weight organic matrices. They are microporous, nominally 45 µm, beads of styrene lightly crosslinked with divinyl benzene. The result is a product virtually free from fines and therefore highly reproducible, column after column, batch after batch. Swelling in common organic solvents generates the pore structure of the EcoSpheres. When swollen, the material can be packed by gravity into Agilent glass columns, specifically designed to be leak-free and to avoid issues of voiding and channeling, which can seriously affect the results. EcoSpheres glass columns are easy to pack, and once the column is packed and equilibrated, are very stable, leading to reliable data throughout the column lifetime.

For More Information

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