

Supercritical Fluid Extraction of Irganox 1076 and Irgafos 168 from Polyethylene

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Abstract

Supercritical fluids can rapidly dissolve and extract common additives and unreacted monomers and oligomers from a polymer matrix. Since the presence and amount of additives may affect a polymer's performance, it is necessary to monitor additive concentrations for quality control.

Traditionally, additives are extracted from the polymer by methods that are labor intensive and rely on large volumes of solvent, such as methanol. Supercritical fluid extraction is an alternative technique to standard solvent extraction procedures and reduces the use and disposal of hazardous solvents.

Overview

In this poster, the parallel supercritical CO₂ extraction of various polymer samples for additives and oligomers was compared to standard techniques. Additives, including Irganox 1076 and Irgafos 168 and various oligomers, were extracted from polyethylene samples using supercritical carbon dioxide.

Materials

- Methanol
- Methylene Chloride
- Carbon dioxide – welding or industrial grade with cleanup column
- C18 SPE Cartridge – 500mg/6mL (#12006)
- *Spe-ed* Glass Wool (#7953)
- Ottawa Sand (#10548)

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Equipment

- Applied Separations' *Spe-ed*TM SFE Supercritical Extraction System
- SFE Modifier Pump
- Polymer Grinder – Cryo Grinder



Method

1. Grind 1.0 gram of polymer sample under liquid nitrogen and sieve.
2. Mix sample with 15 grams of sand and place in a vessel. Install the vessel into the *Spe-ed* SFE.
3. Place a 6mL C18 SPE cartridge on the discharge fitting.
4. Extract sample according to the specified extraction conditions.
5. Remove SPE cartridge and elute with 5mL of methanol/methylene chloride (1:1).

Extraction Conditions for Polyethylene

Extraction vessel	24mL
Sample	1.0g
Pressure	7000psi
Temperature	140°C
Valve temperature	140°C
CO₂ Flow rate	4L/min. gas
Collection	C18 SPE cartridge
Dynamic time	30 minutes
SPE elution	5mL methylene chloride/methanol (1:1)

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Analyte Recovery

Elute SPE cartridge with 5mL of methanol/methylene chloride (1:1) and analyze via HPLC

Results

Analyte	SPE (ppm)	Liquid Extraction (ppm)
Irganox 1076	1642	1638
Irganox 1010	1557	1540
Irgafos 168	1582	1576
Ethanox 398	1021	1027

Conclusion

The supercritical carbon dioxide extraction of additives from polymers offers a viable alternative to solvent based procedures. The accuracy and precision of the results were comparable to the standard method while extraction times were reduced. In addition, the use of hazardous solvents was significantly reduced.

References

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