

Supercritical Fluid Application Notes

SFE544: Polymer Impregnations of Medical Implants using Supercritical CO₂

Introduction

Supercritical fluids have unique properties for the enhanced processing of polymeric materials. The ability of supercritical carbon dioxide to swell and plasticize polymers is critical to the extraction, impregnation, and modification of polymeric materials. In addition, polymer plasticization reduces polymer viscosity and shear stresses.

Supercritical carbon dioxide (scCO₂) is the most widely used supercritical fluid for polymer processing. CO₂ is inexpensive, nontoxic, and nonflammable and has a relatively low critical point. In addition, CO₂ is a gas under ambient conditions which makes for easy removal from polymeric matrices. This avoids the costly processes of drying or solvent removal from processed polymers.

The sorption of scCO₂ into polymers results in their swelling and changes the mechanical and physical properties of the polymers. The most important effect is the reduction of the glass transition temperature (*T_g*) of glassy polymers subjected to scCO₂, often simply called plasticization.

Impregnation of Polymers

Supercritical CO₂ is a solvent which can dissolve and carry small MW nonpolar compounds into a polymer and then precipitate the dissolved compound in the polymer by a reduction in pressure of the supercritical fluid. The CO₂ gas can then easily diffuse out of a polymer once the pressure is reduced to ambient. In addition, there are no solvent residues left in the impregnated polymer sample.

Polymers which have been impregnated using scCO₂ include:

- Polystyrene
- poly(methylmethacrylate) (PMMA)
- poly(vinyl chloride) (PVC)
- polycarbonate
- polyethylene
- poly(tetrafluoroethylene) (PTFE)
- poly(chlorotrifluoroethylene) (PCTFE)
- poly(4-methyl-1-pentene) (PMP)
- nylon
- poly(oxymethylene)
- poly(ethylene terephthalate) (PET)
- poly(dimethylsiloxane) (PDMS)
- polyimides

Solutes used in impregnating polymers range from metal carbonyl complexes, organic dyes to alpha -Tocopherol.

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Impregnation of Medical Implants

Hip and knee implants are typically made of titanium and contact points with bone are made of ultra-high molecular weight polyethylene (UHMW-PE) to reduce friction with bone. Alpha tocopherol is added to the polymer to reduce oxidative degradation of materials. Cross linked ultra-high molecular weight polyethylene (CL-UHMW-PE) offers increased wear resistance when compared to UHMW-PE. Unfortunately, alpha tocopherol cannot be mixed with UHMW-PE before cross linking of the polymer because it hinders the cross-linking reaction and the vitamin is degraded significantly in the crosslinking process.

Therefore, vitamin addition must be impregnated into the UHMW-PE after the cross-linking process and ideally the concentration of the vitamin should be similar over the implant cross section.

Using supercritical fluid technology, alpha tocopherol can be impregnated into UHMW-PE nearly homogeneously across the polymer implant.

Materials

UHMW-PE Hip Cups- Zimmer

DL-Alpha Tocopherol- Hoffmann LaRoche

Equipment



Applied Separations Supercritical Extraction Equipment: *Spe-ed* SFE Basic, *Spe-ed* SFE 2, *Spe-ed* SFE 4, Helix, Pilot and Production Plants.



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Procedure

Place the CL- UHMW- PE sample on a center support rack and place the rack in a 100 ml to 1-liter stainless steel vessel. Add a measured amount of alpha tocopherol into the bottom of the vessel. Purge the vessel of oxygen by flowing CO₂ gas through the vessel. Heat the vessel to the operating temperature and pressurize the vessel to the selected pressure. Hold the operating conditions for a specified time and allow the supercritical CO₂ to dissolve the vitamin and diffuse into the polymer sample. After the exposure time has expired slowly depressurize the vessel to prevent damage to the sample.

Impregnation Conditions

Extraction vessel: 100 mL – 1000 ml vessel

Sample: CL-UHMW Hip Cup

Pressure: 300 Bar

Temperature: 170 °C

Impregnation time: 12 hours

CO₂ Depressurization Rate: 1 to 5
Bar/minute (experimentally
determined)

Collection: 60 mL pre-weighed vial

Analysis: FTIR, HPLC

Conclusion

In summary, supercritical CO₂ deposition of alpha tocopherol into CL- UHMW- PE implants was achieved without any degradation of the alpha tocopherol. In addition, the vitamin E was uniformly

distributed throughout the polymeric hip cup and no toxic or dangerous solvents or chemicals were used in the impregnation process.

References

Gamse T, Marr R, Wolf C, Lederer K. Supercritical CO₂ impregnation of polyethylene components for medical purposes. *Hemijaska industrija*. 2007;61(5):229-32.

Wolf C, Maninger J, Lederer K, Frühwirth-Smounig H, Gamse T, Marr R. Stabilisation of crosslinked ultra-high molecular weight polyethylene (UHMW-PE)-acetabular components with α -tocopherol. *Journal of Materials Science: Materials in Medicine*. 2006 Dec;17:1323-31.