

Supercritical Fluid Application Notes

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Oil Reduction of Whole Pecans by Supercritical Fluid Extraction



Introduction
Pecans with a high oil content (around 50-70% by weight) have a short shelf life. It has been observed that oxygen in the air causes the oxidation of oil in the nuts, which in turn leads to rancidity.

SFE is an extraction technology using supercritical carbon dioxide that can be used to decrease the oil in pecans and thus increase their shelf life. Supercritical CO₂ is an all natural and ecologically safe way to extract analytes from a variety of matrices. It eliminates the use, exposure to, and disposal of hazardous solvents required by conventional solvent processes, while providing comparable extraction results in less time.



Equipment

- ✓ Applied Separations' *Spe-ed*TM SFE or Helix Supercritical Extraction System
- ✓ Analytical Balance

Materials

- ✓ Native pecan halves
- ✓ *Spe-ed* Glass Wool (Cat. #7953)
- ✓ Carbon dioxide –Coleman grade with dip tube

Method

Weigh out 20 g of pecan halves and spray kernels with water using an atomizer spray bottle to an 11.6% moisture content. Drain excess water off kernel surfaces and place sprayed pecans in Zip-LocTM freezer bags. Seal bags and keep pecans at room temperature for 48 hr for optimum moisture equilibration. Store pecans in Zip-LocTM freezer bags at 4 °C until ready for extraction. Ensure that the pecans are brought to room temperature (around 25 °C) for at least 30 minutes before extraction. When ready to begin extraction process, place a plug of *Spe-ed* Wool into an extraction vessel and pour 20 g of pecan halves into the vessel using a funnel. Then, place a plug of *Spe-ed* Wool on top. Compress the sample with a tamping rod, fill the void volume with another plug of *Spe-ed* Wool, then seal the vessel. Install the vessel into the *Spe-ed* SFE. Place a predried and preweighed collection vial containing a plug of *Spe-ed* Wool on the discharge tube. Extract sample according to the specified extraction conditions. Remove collection vial and dry residual moisture from oil to constant weight (AOAC Method 926.12).

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Extraction Conditions

Extraction vessel:	300 mL
Sample:	20 g
Pressure:	620 Bar
Temperature:	75 °C
Valve temperature:	100 °C
CO ₂ Flow Rate:	3 L/min (gas)
Collection:	60 mL pre-weighed vial
Dynamic time:	60 minutes
Depressurization time:	20 minutes

Conclusion

The supercritical carbon dioxide extraction of pecan oil 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 eliminated. Pecan breakage was minimized by pre-spraying of the kernels with water and controlled rate of depressurization.

References

Li, M.; Bellmer, D.D.; and Brusewitz, G.H. "Pecan Kernel Breakage and Oil Extracted by Supercritical CO₂ as Affected by Moisture Content." *Journal of Food Science*. Volume 64, No.6, 1999.

Alexander, W.S.; Brusewitz, G.H.; and Maness, N.O. "Pecan Oil Recovery and Composition as Affected by Temperature, Pressure, and Supercritical CO₂ Flow Rate." *Journal of Food Science*. Vol 62, No.4, 1997.

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