Oil extraction from squid viscera by supercritical carbon dioxide and ethanol for fractionation of fatty acids and phospholipids

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Abstract
Squid viscera, non edible parts of squid were used for oil extraction by supercritical carbon dioxide (SCO₂) and ethanol for fractionation of fatty acids and phospholipids. The supercritical fluid extraction was carried out at temperatures ranging from 35-45°C and pressures ranging from 15-25 MPa for 2.5 hours. The CO₂ flow rate (22 g/min) was constant throughout the extraction period. The amount of oil extracted was increased with the increasing pressure and temperature. SCO₂ extracted almost lipids without phospholipids from squid viscera at 45°C and 25 MPa. The amount of oil extracted by SCO₂ at low pressure and temperature was low. The extracted oil of squid viscera was fractionized by GC for fatty acids components. In all extraction conditions, the amount of palmitic and docosahexaenoic acid (DHA) was high. The highest amount of DHA (15.47%) and palmitic acid (17.08%) was found in squid viscera oil extracted at 25 MPa/45°C and 15 MPa/35°C, respectively. The amount of oleic and eicosapentanoic acid (EPA) was above 7% in all extraction conditions. The highest percentage of oleic acid (9.09%) and EPA (8.73%) was found in squid viscera oil extracted at 20 MPa and 45°C. Myristic, palmitoleic, stearic, arachidonic, eicosanoic and eicosatenoic acid were present significant amount in extracted oil from squid viscera. SCO₂ extracted squid viscera residues were used for phospholipids isolation by ethanol extraction. The ethanol extracted lipid was used for HPLC analysis for phospholipids compositions. Phosphotidylcholine and phosphatidic acid were found to be present in higher amount in squid viscera.

Materials
Squid viscera were collected from F & F Co., Ltd., Busan, Korea.

Methods
Supercritical CO₂ extraction
A laboratory scale supercritical fluid extraction unit was used for extracting oil from squid viscera. After SCO₂ extraction, the squid viscera residues were stored at −80°C until further use.

Fatty acids analysis by GC
Hewlett Packard gas chromatograph (5890 Series II GC system).
Column: Agilent DB-Wax capillary column (30 m length x 0.250 mm internal diameter, 0.25 μm of film)
Carrier gas: N₂
Initial temperature: 130°C. Final temperature: 240°C
Standard fatty acid methyl esters: Supleco, USA

Phospholipid analysis by HPLC
Silica column
Mobile phase: Chloroform : Methanol : Water
Detector: ELSD
Carrier gas: N₂
ELSD temperature: 60°C

Results
Oil extraction yield increased with increasing extraction pressure and temperature. The amount of oil extracted was highest at 45°C and 25 MPa. The amount of oil yields depend on density of CO₂. Pressure and temperature changes greatly affect the density of CO₂.

Fatty acid compositions
The important polyunsaturated fatty acids- EPA and DHA are present at higher amount in squid viscera. Among the saturated fatty acids, palmitic (C16) acid is found to be present in higher amounts.

Phospholipid
Phospholipids has been measured after SCO₂ extraction. The SCO₂ extracted squid viscera residue was again extracted with ethanol. Phosphotidylcholine was present highest amount.

Conclusion
SCO₂ extracted almost oil from squid viscera at higher pressure and temperature. The amount of polyunsaturated fatty acid was higher in SCO₂ extracted oil comparing to soxhlet extraction by hexane. Squid viscera contained higher amounts of EPA and DHA. Among the phospholipid, Phosphotidylcholine was found to be highest.

References