STUDIES ON LIQUID AND SUPERCRITICAL CARBON DIOXIDE EXTRACTION OF INDIAN SPICES

By

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CERTIFICATE

This is to certify that the thesis entitled **"STUDIES ON LIQUID AND SUPERCRITICAL CARBON DIOXIDE EXTRACTION OF INDIAN SPICES"** being submitted by **MR. ASHOK KUMAR** to the **Indian Institute of Technology, Delhi** for the award of **DOCTOR OF PHILOSOPHY** is a record of bonafide research work carried out by him under our guidance and supervision in conformity with the rules and regulations, of Indian Institute of Technology, Delhi.

The results contained in this thesis have not been submitted, in part or in full, to any other University or Institute for the award of any degree or diploma.

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ABSTRACT

Liquid and Supercritical Fluid Carbon dioxide extraction has emerged as a potential separation technology for natural products due to its inherent advantages such as low temperature extraction, superior quality, low energy inputs, reduced process time and no solvent residue in final product.

Moreover carbon dioxide is an inexpensive, odourless, colourless, non-toxic, non-flammable, non-corrosive, free of health hazards, environmentally safe and suitable for extraction of botanicals.

In view of this, status of liquid and SCF extraction process, its applications and future scope have been critically reviewed with special reference to essential oil food flavours and pharmaceutically active compounds. The merits and demerits of conventional extraction procedures (hydrodistillation and solvent extraction) have also been discussed.

India is famous as the "Home of Spices" and is still the leading producer and exporter of major spices like Ajowan, Celery, Coriander, Dill, Ginger, Turmeric, Black pepper, Cardamom and Clove. These spice extractives, essential oils and oleoresins play a major role in processed food industry World over and the export demand for quality spice extractives of low solvent residue, higher percentage of active constituents (pungent principles) from India is ever increasing. The small scale Indian manufacturers are facing difficulties to meet the stringent specifications of importing countries.

The present study was aimed to establish the feasibility of using carbon dioxide extraction process. For this purpose, some of the widely used spices of Indian origin like Ajowan, Caraway, Ginger and Turmeric were selected for extraction studies. These are used in food, pharmaceuticals (Ayurvedic formulations), perfumary and cosmetics preparations.

Crushed samples of the botanicals, were taken for CO_2 extraction and steam distillation. Extraction using CO_2 as a solvent was carried out in laboratory scale Liquid/Supercritical fluid extraction apparatus. Studies were carried out at 60, 100, 150, and 200 bar and 10°, 20°, 30° and 40°C. Steam distillation experiments were conducted for comparison.

The extracts were analysed by thin layer chromatography and Gas chromatography. Thymol, a solid crystalline compound separated from Ajowan essential oil was identified by IR, Mass and NMR spectroscopy.

The percentage yield (w/w) of extracts was measured and percentage of active constituents present in the extracts (like Thymol in Ajowan, ar-turmerone in Turmeric, α-Zingiberene in Ginger and Carvone in Caraway essential oils) were determined by GC. The composition of the extracts obtained by steam distillation are also given for comparison. It was observed that a larger quantity of extract is obtained with Carbon dioxide as compared to that by steam distillation. Also the CO_2 extract contains a larger quantity of major active compounds. Effect of pressure, temperature, time of extraction and particle size on yield and quality of essential oils in both the processes have been discussed.

The optimum experimental conditions of CO_2 extraction were used for unsteady state mass transfer studies. The rate of extraction with Carbon dioxide was analysed mathematically and the values of diffusion parameters (D/r_o^2) were calculated from the experimental data.

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