SUBCRITICAL WATER EXTRACTION OF 6-GINGEROL AND 6-SHOGAOL
FROM Zingiber Officinale

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To my mum, Ramlah Ibrahim, my dad Md Sarip Baba, my wife Nor Hasmi Abd Ghani and my family members.

With all love and gratitude
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Nowadays, natural products extract as a nutritional supplement becomes a part of healthy lifestyle. However, numerous scientific evidences suggested that the processing methods that mainly use organic solvents in extraction processes may result an undesired toxic residues in the product. Thus, the so called ‘green’ solvent that is water seems to be best alternative to substitute the organic solvent in the natural herb extraction process. In this study, the effect of subcritical water extraction (SWE) was employed for the extraction of bioactive compounds from *zingiber officinale* namely 6-gingerol, 6-shogaol and 10-gingerol. Two types of SWE equipment which are ASE 200 and CLEAR SWE prototype had been utilized. The ASE 200 with the capacity of 24 ml was used to evaluate the performance of the CLEAR SWE prototype with capacity of 1000 ml. Three parameters were manipulated in the SWE optimization process that are extraction temperature (100 to 200°C), static extraction time (10-60 minutes) and solvent to sample ratio (28/3-28/1 ml/g) at a fixed pressure of 3.5 MPa. The analysis was done using the High Performance Liquid Chromatography. Two main bioactive compounds namely 6-gingerol and 6-shogaol were extracted with the traces of 10-gingerol. The extraction and fractionation of 6-gingerol and 6-shogaol were obtained at the lower and higher temperature; respectively. The optimum conditions for the 6-gingerol was at the temperature of 130°C, in 30 minutes and solvent to sample ratio of 28/2 ml/g with the overall mass transfer coefficient of 8.1179 x10^{-7} m/s. Meanwhile the optimum condition for the 6-shogaol was at the temperature of 170°C, in 20 minutes and solvent to ratio of 28/2 ml/g with the overall mass transfer coefficient of 18.3764 x 10^{-7} m/s. It is found that the ginger bioactive compounds will be started to degrade at a temperature above 180 °C.
ABSTRAK

Kini, pengambilan ekstrak produk herba sebagai makanan tambahan adalah kebiasaan dalam amalan gaya hidup sihat. Namun, banyak bukti saintifik menunjukkan bahawa kaedah pemprosesan yang kebanyakannya menggunakan pelarut organik dalam proses pengekstrakan boleh mengakibatkan sisa toksik yang tidak diingini dalam produk tersebut. Oleh itu, pelarut teknologi hijau, iaitu air menjadi alternatif terbaik untuk menggantikan pelarut organik dalam proses pengekstrakan herba semula jadi. Dalam kajian ini, pengekstrakan sub lampau genting (SWE) telah dikaji untuk mengekstrak sebatian bioaktif, iaitu 6-gingerol, 6-shogaol dan 10-gingerol daripada zingiber officinale. Dua jenis peralatan SWE, iaitu ASE 200 dan prototaip CLEAR SWE digunakan. ASE 200 yang berkapasiti 24 ml digunakan untuk menilai prestasi prototaip CLEAR SWE yang berkapasiti 1000 ml. Tiga parameter telah dimanipulasi dalam proses pengoptimuman SWE iaitu suhu (100 hingga 200°C), masa (10-60 minit) dan nisbah pelarut kepada sampel (28/3-28/1 ml / g) pada tekanan tetap iaitu 3.5 MPa. Hasil ekstrak dianalisa dengan menggunakan Kromatografi Cecair Prestasi Tinggi. Dua sebatian bioaktif utama iaitu 6-gingerol dan 6-shogaol dan hanya sedikit 10-gingerol dapat diekstrak. Dengan menggunakan proses SWE, pengekstrakan dan pemisahan 6-gingerol dan 6-shogaol dapat dicapai pada suhu yang berbeza. Keadaan optima untuk pengekstrakan 6-gingerol adalah pada suhu 130°C, dalam masa 30 minit dan nisbah pelarut kepada sampel adalah 28/2 ml/g dengan nilai pekali pemindahan jisim keseluruhan, iaitu 8.1179 x10 \(^{-7}\) m/s manakala keadaan optimum untuk 6-shogaol pula adalah pada suhu 170°C, dalam masa 20 minit dan nisbah pelarut kepada sampel adalah 28/2 ml/g dengan nilai pekali pemindahan jisim keseluruhan iaitu 18.3764 x 10 \(^{-7}\) m/s. Sebatian bioaktif halia mula merosot pada suhu melebihi 180°C.