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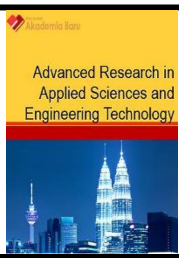

Effect of Pressure and Temperature on Solid Oil Particles Yield of Grind Ginger Rhizome using Rapid Expansion Supercritical Solution (ress) Carbon Dioxide

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ABSTRACT

Recently RESS method has been used by researchers to produce fine particles for pharmaceutical drug substances. Since RESS technology acknowledges a lot of benefits compare to conventional method of ginger extraction. Therefore, the objective of this research is to produce direct solid oil particles formation from ginger rhizome which contains valuable compounds by using RESS-CO2 process and to study the effect of pressure and temperature on extraction yield. RESS experiments were carried out at various extraction pressure of 3000, 4000, 5000, 6000 and 7000psi and at different extraction temperature of 40, 45, 50, 55, 60, 65 and 70°C, respectively for 40 minutes extraction time and constant flowrate (24ml/min).From the studies conducted, it was found that extraction pressure is an important factor that can affect particle size and extraction yield. By increasing the extraction pressure, the extraction yield was found to increase with increasing of pressure at constant temperature, but extraction yield was found to decrease with increasing temperature. The highest extraction yield is 2.41% obtained at extraction pressure of 6000psi and constant extraction temperature of 65°C.

Keywords:
Ginger; yield; RESS; supercritical carbon dioxide

1. Introduction

Ginger (Zingiberofficinale, Rosc.) in natural form, ginger oleoresin and ginger essential oil is widely used in pharmaceuticals, cosmetics as well as in foods and beverages industry. Research in the application of ginger extract received a lot of attention due to the increasing in the application of ginger extract product in the pharmaceutical products. Many studies acknowledge high amount of antioxidant content as a valuable compound in the ginger extract [1]. Recently, many scientific

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studies were focused on addressing the powerful of antioxidants for various health benefits such as stress, ageing, pathogen infestation and neurological disease [2]. In previous studies regarding antioxidant properties for ginger species, researchers had been focused mainly to the ginger rhizomes instead of the leaves that also used for food flavouring and traditional medicine [3].

Using natural medicine extracted from natural plant is not a new concept; in fact it has been used extensively for centuries. The early civilization has already employed natural medicines taken from plants, roots and leaves to cure the sickness, infection and wound. Zingiberofficinale contains a number of antioxidants such as beta-carotene, ascorbic acid, terpenoids, alkaloids, and polyphenols such as flavonoids and flavones glycosides [3]. The main constituents found in the extracts obtained using supercritical carbon dioxide are α-zingiberene, β-sesquiphellandrene, α-farnesene, geranial, β-bisabolene and β-eudesmol [4]. Gingerol, a major pungent ingredient of ginger, also has potent antioxidant activity [5]. Zancan *et al.*, [6] has studied extraction of ginger using supercritical carbon dioxide and found that the major constituents present in the ginger extracts were α-zingiberene, gingerols and shogaols. They also found out temperature, pressure and solvent used in extraction process affected the amount of the constituents.

Some of the artificial antioxidants are related to health damage such as kidney edema[6]. Some of the pungent constituents present in ginger have great antioxidants, anti-inflammatory and anticancer properties [7,8]. Manju and Nalini [9] had found out ginger can reduced number of tumour and incidence of colon cancer. Ginger rhizomes and their extracts also can be used to treat some number of illnesses such as cholesterol level reduction, gastric, nausea and flu, migraines, headaches, colds, fever, diabetes, stomach upset, menstrual problem, motion sickness, diarrhoea, muscle strains, anti-tumour, sore throat and persistent cough [10,11].

Usually, extraction of ginger rhizome will produce a ginger extract in liquid oil form. Recent developments in extraction technology allowed extraction of bioactive components of herb to be produced in the form of solid oil particle instead of liquid oil that need to undergo further conventional techniques for particle formation. Previous studies on ginger extraction used supercritical fluid extraction (SFE) and conventional extraction process such as solvent extraction and steam distillation. These techniques will produce ginger liquid oil that need to undergo conventional technique for particle formation [12]. The disadvantages of conventional technique for particle formation are due to the fact that, extraction product may contaminated with unwanted residue, excessive toxic organic solvents use and dispose, thermal and chemical solute degradation for certain biological substances, change of the solute structure, and difficulties in controlling the solute particle size [13].In addition, the conventional process for particle formation is time consuming and costly [14]. Furthermore, by using conventional methods, it is not possible to recover both aromatic and pungent components simultaneously from the ginger rhizome [15]. In the solvent extraction process, essential oil or the volatile part that responsible for ginger aroma will loss during the evaporation process of the solvents. Ginger oleoresin, pungent principle of the ginger can be extracted by solvent extraction process. Steam distillation process was used to extract essential oil and it cannot recover the pungent components due to main pungent component, gingerol is thermally degraded by high temperature. Therefore, blending process need to prepare a flavour containing both components of oleoresin and essential oil to get both aroma and pungent components of ginger [15]. The disadvantages of conventional technique for ginger extraction are large amount of solvent is required of solvent, time consuming, low extraction efficiency, low yields due to losses of volatile components, produce toxic residual solvent that can be harmful to environment and chemical structure for thermally degraded compound, can be destroyed at high temperature [16].

Therefore, in this study a clean extraction method which is RESS-CO2 technology was introduced for the extraction of powdered ginger to direct produce solid oil particle of bioactive components. RESS method offers a lot of benefits compared to conventional method. RESS-CO_2 offers a lot of beneficial compared to conventional extraction method such as nontoxic, environmental friendly method, easy to separate the solvent, mild operating conditions, short extraction time, eliminate conventional technique for particle formation and offers high quality of final product.

Solvent use in Rapid Expansion Supercritical Solution (RESS) method is supercritical fluid carbon dioxide (CO2) due to its environmental friendly properties such as nontoxic, non-flammable, easy to recycle, inexpensive, easily available, low critical temperature (Tc=31.05°C) and moderate critical pressure (Pc=7.38Mpa). Moreover it can reduce production cost, and eliminate environmental problem because the process did not require conventionally purification process. It also offers higher quality of final product than those extract produced using organic solvents.

To date, there is a growing interest in studies on natural product to direct solid oil particle formation in the pharmaceuticals industries [17]. Development of solid oil particle will increase the efficiency of drug delivery, reduced drug dosage and improve bioavailability of the drug [18]. RESS method have been used by researchers to produce fine particles for pharmaceutical drug substances [19]. RESS has successfully employed to produce small size contaminant free particles for heat sensitive materials such as aspirin and carbamazepine [20,21].

2. Methodology

Ginger Preparation

The Zingiberofficinale, Rosc rhizome used in this study was purchased from local market. Fresh ginger rhizomes were washed thoroughly with tap water to remove the dirt. The skin of the ginger rhizome was peeled and then cut into cross section slices with 2 to 3 mm thick. An amount of 500 g ginger slices was weighed and undergo oven dried in an oven model Memmert UFE 500 to remove moisture content until it remains in the sample at 10 to 12% [5,22].Dried ginger sample then was ground using mechanical grinder Retsch model SM100 for 3 minutes through a plate of 1mm pore size to obtain ginger the powder. The sample was then stored in the refrigerator at 4°C until further use.

The moisture content was expressed as the amount of water that presents in a moist sample. The moisture content determination for ginger rhizome was specified based on the Palm Oil Research Institute of Malaysia (PORIM) test method [23]. An amount of 10 g of ginger slice was weighted accurately in a dried glass dish at 55ºC for 6 to 7 hours. Previously, glass dish undergoes drying process in an oven and cooling at room temperature before the mass of the empty dried dish was weighted. After that, the dish was removed and undergoes cooling process. Then, the cooled dish was weighted until it achieved constant weight. Moisture content expressed in percentage of the equivalent ratio of water content with the total mass of the sample was calculated using equation (1).

$$Moisture\ content, \% = \frac{m_1 - m_2}{m_1 - m_0} \quad (1)$$

Where; m_0= mass of the dish (g)
m_1= mass of the dish with sample before drying (g)
m_2= mass of the dish with sample after drying (g)

Rapid Expansion Supercritical Solution (RESS) Carbon Dioxide

RESS experiments were done using RESS equipment modified from Supercritical Fluid Technologies Model SFT-100 equipment. Each run will be conducted using 8 g sample of ground ginger based on observation on the maximum mass of ground ginger that can be loaded and fitted in 25 ml of the extraction vessel provided by the Supercritical Fluid Technologies Model SFT-100. If the mass of the sample is higher than 8 g, it will cause some loose fitting at the top of the extraction vessel seal that will cause a fault or leaking of CO2 when the extraction process in progress.

Extraction pressure and temperature was set at the desired value, and ginger sample was inserted in a cotton bag before putting in the extraction vessel. The seal on the top of an extraction vessel was Extraction pressure and temperature was set at the desired value, and ginger sample was inserted in a cotton bag before putting in the extraction vessel. The seal on the top of an extraction vessel was sealed tightly before closing the top cover. Dynamic valve was opened and restrictor valve was tightly closed during the extraction time. Glass vial was inserted in the expansion chamber and tightly seal the expansion vessel. When the extraction temperature achieved the desired value, SFT-10 constant flow pump (flowarte:24 mL/min, pressure 0 to 10000psi) was run to feed high pressure liquid CO2 (99 % purity provided by MOX Linde Gases Sdn.Bhd) continuously in the extraction vessel at a fixed solvent flowrate of 24 ml/min. Basically, liquid CO2 will be converted to a supercritical condition when it is pump into the extraction vessel (heated zone). To achieve the desired pressure set point, CO2 pump will continuously actuate.

After 40 minutes of extraction, the restrictor valve was quickly open to depressurize the supercritical solution for the separation of solute from the solvent through the coaxial expansion nozzle with internal diameter of 1.0 mm and distance of 80 mm from the nozzle to the collection bottle. Depressurized CO2 at the ambient pressure will convert into gaseous form and purged into the ambient. Extraction product will be collected in collection vials.

Percentage of Solid Oil Yield Determination

To determine the extraction yield, weight of the collection vials will be measured before and after RESS process. The extraction product was weighed using the analytical balance model Mettler Toledo AB204-S to ginger solid oil yield determination. The extraction yield is defined as the mass of the extracted solid oil particle divide by the mass of the ground sample loaded in the extraction column. The solid oil yield was determined using equation (2).

$$Yield\ (\%) = \frac{mass\ of\ solid\ oil\ particle\ (g)}{Initial\ ginger\ sample\ (g)} \tag{2}$$

3. Results and Discussion

Effect of Pressure on Solid Oil Particle Yield (%)

In this work, the effect of different extraction pressure on solid oil particle yield (%) was investigated at 3000, 4000, 5000, 6000 and 7000psi while holding constant at a different extraction temperature of 40, 45, 50, 55, 60, 65 and 70°C, respectively for 40 minutes extraction time.

Figure 1, shows sample of the SEM images for solid oil ginger particle obtained from RESS carbon dioxide method at extraction temperature of 45°C and extraction pressure of 5000psi. The effect of pressure on the yield at constant temperature , graphically shows in Figure 2. The experimental results refer to the total solute extract yield per sample (8 g) of ginger powder.

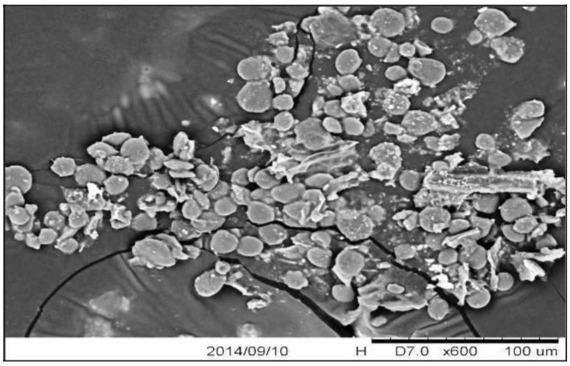

Fig. 1. SEM Image of Ginger Particles Produced By RESS-CO2 Obtained at Extraction Temperature of 45°C and Extraction Pressure of 5000psi

Referring to Figure 2, the highest extraction yield occurred at pressure 6000psi and the yields are 2.05, 2.18, 1.73, 2.41, and 2.08%, respectively. And at constant temperature of 45°C and 55°C, the highest extraction yield occurred at pressure 5000psi and 7000psi, respectively. From all the different pressure studies at constant temperature, the highest extraction yield is 2.41% found at 65°C and 6000psi. For all the test constant temperature, increasing extraction pressure from 3000psi to 6000psi, clear rises in extraction yield can be seen, but when further increase extraction pressure to 7000psi, extraction yields start to decrease; except for temperature of 55°C, where extraction yield show a small increment from 1.75% to 2.04%. Increase in extraction pressure is associated with increasing of saturation and solubility of ginger. Increasing the extraction pressure, higher ginger solute solubility produced, hence resulted in higher amounts of extraction yield [20]. The extraction yield increased with increasing of pressure at constant temperature. The results might be the cause of the increased dissolving power of supercritical CO2 due to increase density caused by an increase of pressure at the constant temperature.

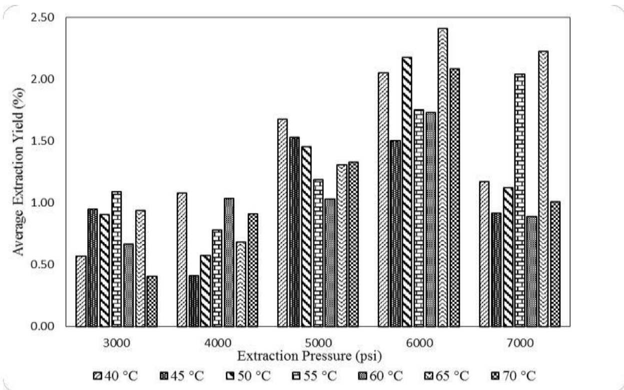

Fig. 2. Effects of Different Pressure on Solid Oil Particle Yield at Constant Temperature

Effect of Temperature on Solid Oil Particle Yield (%)

In this study, the effect of different extraction temperature on solid oil particle yield (%) was investigated varied from 40, 45, 50, 55, 60, 65 and 70°C by fixing extraction pressure to be constant at 3000, 4000, 5000, 6000 and 7000psi respectively for 40 minutes extraction time. The effect of temperature on the extraction yield at constant pressure , clearly demonstrated in Figure 3.

Referring to Figure 3, the highest extraction yield for all different extraction temperature (40, 45, 50, 55, 60, 65 and 70°C) occurred at constant pressure 6000psi for most of the extraction temperature, except for at extraction temperature of 45°C, where at constant pressure of 5000psi, the extraction yield is 1.53%, compared to extraction yield at 6000psi is 1.50%, and at extraction temperature of 55°C, where at constant pressure of 6000 and for 7000psi, the extraction yield is 1.75% and 2.04%, respectively.

Based on the overall performance, expanding extraction pressure from 40°C to 70°C, did not affect much in increasing of extraction yield. For example, at constant pressure 3000psi extraction yield at temperature of 40°C is 0.57% and at 70°C is 0.41%. According to work done by Tozuka, Miyazaki, & Takeuchi (2010), by increasing extraction temperature, at constant extraction pressure, the extraction yield will reduce. But in our study, by rising the extraction temperature at constant pressure, fluctuation of extraction yield occurred [24]. But for overall, increasing the extraction temperature from 40°C to 70°C, the extraction yield reduced. Extraction yield at 40°C is higher compare to at 70°C.

The highest extraction yield for all constant pressure occurred at temperatures below than 70°C. Where, at constant pressure 3000psi as can been seen inFigure 3, the highest extraction yield of 1.09% occurred at temperature of 55°C; at constant pressure of 4000psi, the highest extraction yield of 1.08% was found at temperature of 40°C; at constant pressure of 5000psi, the highest extraction yield of 1.68% occurred at temperature of 40°C; and at constant pressure of 6000psi and 7000psi, highest value of extraction yield of 2.41% and 2.22%, respectively were found at temperature of 65°C.

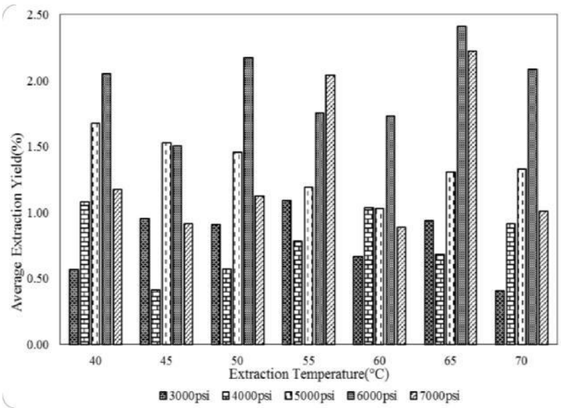

Fig. 3. Effects of Different Temperature on Solid Oil Particle Yield at Constant Pressure

As shown in Table 1, for ANOVA analysis on the effect of different temperature at constant pressure on the solid oil particle extraction yield, it showed significance difference, since all the P-Value, was below than 0.05. This value indicates that, the effect of different temperature in range of

40 to 70°C at constant pressure of 3000 to 7000psi give a significant effect and on the solid oil particle extraction yield

Table 1
ANOVA Test for the Effect of Different Temperature on Extraction Yield at Constant Pressure

		Extraction Yield			
Pressure (psi)	Temp(°C)	Min(%)	Std Deviation	F	P-Value
3000	40	0.57	0.02		
	45	0.95	0.04		
	50	0.91	0.02		
	55	1.09	0.04	140.2	1.10E-11
	60	0.67	0.04		
	65	0.94	0.04		
	70	0.41	0.04		
4000	40	1.08	0.04		
	45	0.41	0.09		
	50	0.57	0.03		
	55	0.78	0.02	66.30	1.78E-09
	60	1.04	0.04		
	65	0.68	0.05		
	70	0.91	0.06		
5000	40	1.68	0.2494		
	45	1.53	0.1938		
	50	1.46	0.0924		
	55	1.19	0.0811	4.61	8.74E-03
	60	1.03	0.0668		
	65	1.31	0.3046		
	70	1.33	0.0164		
6000	40	2.05	0.1289		
	45	1.50	0.0182		

	50	2.18	0.2040		
	55	1.75	0.2686	10.29	1.87E-04
	60	1.73	0.2398		
	65	2.41	0.0366		
	70	2.08	0.0786		
7000	40	1.17	0.0759		
	45	0.92	0.0513		
	50	1.12	0.0613		
	55	2.04	0.0831	164.6	3.66E-12
	60	0.89	0.0172		
	65	2.22	0.1056		
	70	1.01	0.0929		

4. Conclusion

The aim of this study is to extract the ginger solid oil particle from dried ginger rhizome using Rapid Expansion Supercritical Solution-CO_2 technique and to determine effect of extraction pressure and temperature on the extraction yield. The influence of pressure and temperature on extraction yield was investigated at 3000, 4000, 5000, 6000 and 7000psi; and at 40, 45, 50, 55, 60, 65 and 70°C. The highest extraction yield, 2.41%, occurred at extraction pressure of 6000psi and extraction temperature of 65°C. From the conducted studies, it was proven extraction pressure and extraction temperature were important factors that can affect extraction yield, since increasing the extraction pressure, the extraction yield increased. And increasing extraction temperature at constant pressure, the extraction yield will reduce.

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