SUPERCRITICAL CO_2 EXTRACTION OF JACKFRUIT SEEDS

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***Abstract*—Various extraction technique had been used but no research on Supercritical CO_2 extraction (SC-CO_2) of jackfruits seeds has been done. The extraction equipment used in this research is SFT-100 with vital operating variables to be examined were at temperature (50°C, 60°C) with pressure (4000psi, 5000psi, 6000psi). While the extract was tested using Gas Chromatography-Mass Spectrometry (GC-MS), Varian GCMS 450 GC 240 MS Ion Trap. The highest oil yield obtain was 0.31%. There's 6 major medicinal constituent obtained after analyzed. Jackfruits seeds have the medicinal abilities namely acne treatment, antimicrobial, anticancer, antioxidant, anti-inflammatory and others medicinal values.**

Keywords— Jackfruit seeds, Medicinal Value, Supercritical CO_2 extraction,

I. INTRODUCTION

Jackfruits (*Artocarpus heterophyllus*) is a delicious yellowy fruit and Malaysian accepted it as 'Nangka'. Malaysian well-liked is Tekam Yellow (Nangka Madu) as a result of its texture was less juicy however tasted sweeter than Mantin and Mastura [1]. Jackfruit is one amongst the economical plantation in Malaysia. Jackfruit's pulp were consumed for many reasons likes eaten up directly, canned in brine, created into varied native delicacies like 'dodol' and crackers. Whereas the Jackfruit seeds will always be treated as wastes. Jackfruit has numerous medicinal uses particularly antioxidant (phenolic contents), anti-inflammatory, antimicrobial, anti-cancer and anti-fungal activity [2]. Methanolic extracts from jackfruit seed contain flavonoids, tannins and saponins (terpenoids) and so making this the initial study to prove that jackfruit seed potential as a candidate for future anticancer medical care [3]. Moreover, it showed potential in inhibitor, receptor binding activity with RBCs, in vitro medicament activities, antifungal activities and medicine activity since polyphenols and flavonoids present in the testa [4].

Typical solvent extraction operate by using dangerous organic solvent, environmental issues, health considerations and of cause effect on the nature of extract. These days a lot of advance and harmless extraction method had been discovered, that is SC-CO_2 extraction. It's known as supercritical fluids (SCFs) as a result of the fluid since its operating parameters (temperature and pressure) is changing from normal to critical condition. The price for the SC-CO_2 extraction equipment extremely expensive however it produces a clean oil and sustainable method. It is also environmental friendly methods of extraction by depressurizing CO_2 solvent before releasing to atmosphere [5]. SC- CO_2 extraction is apply in food, pharmaceutical and medical industries since it can be consider as the most promising as it offers to produce higher extraction yield with high quality of nutritional and bioactive compounds, lastly is SC-CO_2 extraction required shorter operating time than other extraction, roughly about 40min to obtain contain yield. [6]

The production of Jackfruit in Malaysia is indeed a must for us to be proud off but to date, Jackfruit is taken into account to be an underutilized fruit wherever most of the fruit seeds get wasted due to unawareness. This study helps in enhancing consumption of Jackfruit seeds by general public through promoting its medicinal values and offers likelihood to develop price additional product from jackfruits rather than throw it away [7]. Therefore, the objectives of this research were determine the highest oil yield from Jackfruit seeds using SC-CO_2 extraction beside to characterize the medicinal components in jackfruits seeds oil using GC-MS.

II. METHODOLOGY

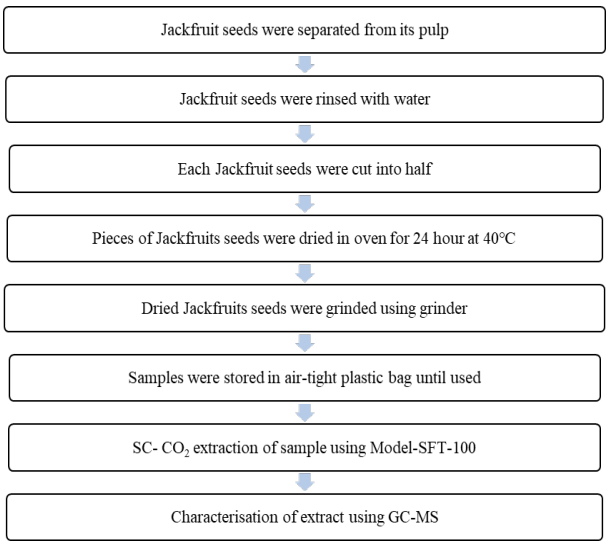

Figure 1 : Process Flow of SC-CO_2 Extraction Experiment

Figure 1 shows the overall method on performing this experiment. Samples of jackfruits seeds' powder had been prepared. After obtaining ripe Tekam Yellow Jackfruit from supermarket, the Jackfruit is take out from its rags and the seeds were separated from the pulps. Then, the jackfruits seeds were cleanly washed with distilled water. The seeds is carefully cut into half and dried in oven for 24 hr at 40°C. After that, the seeds were grinded using grinder with the size of sieving approximately at 250

µm and store in air-tight plastic bag. About 10g of samples were inserted into extraction vessel using cloth bag. Dynamic extraction were applied for 40 min and every 10 min extract will be collected. In this extraction experiment, particle size were kept constant at 250µm. Pre-treatment to alter moisture content of a sample produce insignificant effect on extraction yield [8]. Furthermore, solvent flow rate on extraction yield equally to give insignificant effect [9]. Therefore the moisture content was neglected and the solvent flow rate were kept constant at 5ml/min. The vital operating variables to be examined were at temperature (50°C, 60°C) with pressure (4000psi, 5000psi, 6000psi). For every parameters, two trials were conducted. Next, the extract was diluted with 0.5ml Hexane solvent to be analyzed using GC-MS.

III. RESULTS AND DISCUSSION

A. Percentage of Oil Yield Calculation

$$\%\ Extraction\ Yeild = \left(\frac{Mass\ of\ Extarcted\ Oil}{Dry\ Mass\ Basis\ (10g)}\right) \times 100\%$$

$$= \left(\frac{10.02 - 9.57}{10.02}\right) \times 100\%$$

$$= 0.32\%$$

Equation 1: Percentage of Oil Yield Calculation

Equation 1 illustrate an example of calculating percentage of extraction of the highest oil yield. The mass of extracted oil was calculated by calculating the differences between the initial mass of cloth bag before extraction and final mass of cloth bag after extraction. Then the mass of oil yield obtain was divided by initial mass of cloth before multiplying with 100%.

B. Jackfruit Seeds Oil Extracted

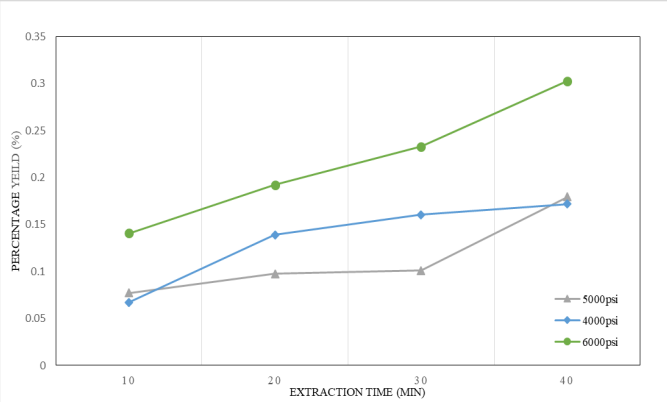

Figure 2: Graph of extraction yield versus time at constant temperature 50°C varies with pressure

Figure 2 shown that percent of extraction yield was directly proportional to extraction time. The percentage yield of Jackfruit seeds oil increases as the time of extraction lengthen. The highest amount of oil yield were obtained at pressure of 6000psi followed by 5000psi and lastly 4000psi. Successfully, about 0.32% of Jackfruit seeds oils abled to be extracted at 6000psi while the least oil yield managed to be extracted was 0.17% at the lowest pressure. Density of CO_2 solvent increase when high pressure was supplied in the extractor block. Thereby, the solubility of Jackfruit seeds powder within CO_2 solvent rises resulted from the intensified interactions between solvent molecules and seeds oil [10]. The average percentage of extraction yield obtained at pressure 6000psi was 0.31% whereas 0.18% extraction yield resulted at 5000psi and lastly 0.17% at 4000psi.

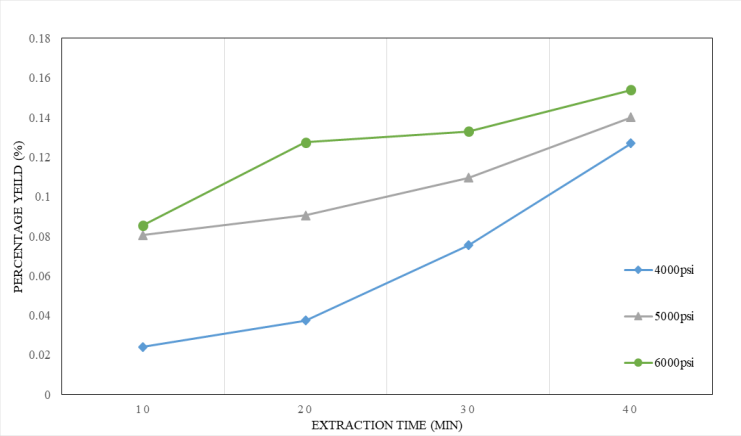

Figure 3: Graph of extraction yield versus time at constant temperature of 60°C varies with pressure

Figure 3 also shows the effect of different pressure on extraction yield except the extraction temperature was slightly higher, at 60°C. Clearly the percent of extraction yield was directly proportional to extraction time as well. As the time lengthen, the percentage yield of Jackfruit seeds oil increases too. The most effective extraction pressure parameter was at 6000psi followed by 5000psi and lastly 4000psi. About 0.16% of Jackfruit seeds oils effectively extracted at 6000psi while the least oil yield managed to be extracted was only 0.13% at 4000psi. The same principle applied as previous experiment. High pressure supplied at 6000psi increases the density of CO_2 solvent. As a result, the solubility of Jackfruit seeds powder within CO_2 solvent rises [10]. The average percentage of extraction yield obtained at pressure 6000psi was 0.15%, 0.14% at pressure 5000psi whereas 0.13% extraction yield resulted at pressure of 4000psi.

Among all extraction's operating parameter, the utmost oil yield produce was at temperature 50°C and pressure 6000psi about 0.32%. There was a 12% more of oil yield obtained at this particular operating parameters than undergoing extraction at 60°C and 6000psi. As pressure increase the oil yield said to be increase as well at a particular temperature. From figure 3 and 4 the oil yield the oil does not increase with temperature since oil yield will increases as vapor pressure of component became the main factor. The result obtained from this experiment was different. Where temperature increase does not increase the extraction yield. A study also had shown similar result. During the temperature of 313 K, it exhibited a more independent behavior, and the effect of CO_2 density is more pronounced than that of vapor pressure of the components [11]. There was a large different between the oil yield at (60°C, 5000psi) and (60°C, 6000psi). Increasing the pressure by 1000psi allowed to produce 13% more oil than previous operating pressure of 5000psi at the same temperature, 50°C.

A. Medicinal Values

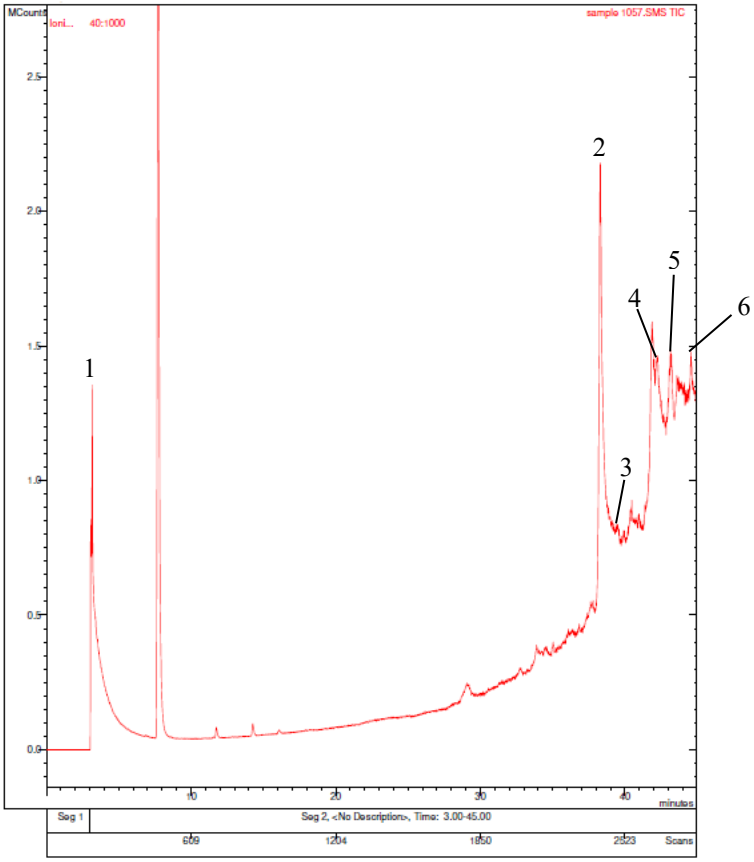

Figure 4: GCMS analysis of Jackfruit Seeds extract

Table 1: Characterization of Medicinal Values of Jackfruit Seeds Identified by Using GC-MS

Number	Medicinal Compounds	Molecular Formula	Retention Time	Peak Area %	Medicinal Value
1	(1-Methoxy-pentyl)-Cyclopropane	$C_9H_{18}O$	7.710	21.63	1. Anesthesia[12] 2. Spirocyclization Process [13]
2	Dodecanoic acid	$C_{12}H_{24}O_2$	38.279	18.10	1. Acne Treatment [14] 2. Increasing HDL cholesterol level in blood stream.[15] 3. Antimicrobial [16]
3	n-Hexadecanoic acid	$C_{16}H_{32}O_2$	39.46	2.01	1. Anticancer and antioxidants. [17] 2. Antioxidant and antimicrobial [18]
4	Vitamin A aldehyde	$C_{20}H_{28}O$	41.837	6.279	1. Growth and development, immune system and good vision. [19] [20]
5	Cholestan-3-ol, 2-methylene-, (3á,5à)	$C_{28}H_{48}O$	42.214	6.146	1. Anticancer [21] 2. Anti-inflammatory [22]
6	1-Heptatriacotanol	$C_{37}H_{76}O$	43.61	1.43	Antihypercholesterolemia [23]

The most obtained medicinal compound from extract was (1-Methoxy-pentyl)-Cyclopropane at about 21.63% at retention time of 7.71min. Cyclopropanes is an important molecule in Spiro cyclization (rigidifying of a bioactive molecule) in medicinal chemistry's field. For example, diastereoselective synthesis of Spirobarbiturate-Cyclopropanes through Organobase-Mediated Spirocyclopropanation of Barbiturate-Based Olefins with Benzyl Chlorides [13]. Water and Schmidt managed to introduce Cyclopropane as anesthetics agent to replace Ethylene and Nitrous Oxide to be applied on surgery's patients [12].

At 38.28min about 18.10% of Dodecanoic acid or standardly recognized as Lauric acid was discovered. It is a saturated fatty acid with strong bactericidal properties. *Propionibacterium acnes* (P. acnes) is a commensal bacteria that are normally present on the skin of a vast majority of human. This acnes becoming chronically inflammatory triggers when it meets favorable dermatophysiological terrain. Study shown that P. acnes is the most sensitive to Lauric acid among these bacteria thus proving that Lauric acid as antibiotic therapy of acne vulgaris [14]. Furthermore, Lauric acid brilliantly assisting in increasing the level of High-Density Lipoprotein (HDL) cholesterol or commonly referred as "good" cholesterol since it will eliminate excess

cholesterol in blood. Lauric acid able to increase serum total cholesterol better than other fatty acid [15]. Lauric acid was also extracted form *C. appendicigera* and *C. helenioides* and the antimicrobial activity were tested in vitro using the agar-well diffusion method with the microorganisms. Positive results were shown against Gram-positive bacteria in comparison to the Gram-negative bacteria [17].

About 2.01% of Palmitic acid was available in Jackfruit seeds at retention time of 39.46min. It can be a promising antitumor agent and also as the best antioxidant activity in the vegetable-origin products which directly related to the size of the hydrocarbon chain in the structure of the fatty acid molecule [18]. The Palmitic acid extracted from the Greek pollen showed antioxidant clinical able from observed free radical scavenging activity and the effects of pollen on human fibroblasts. Moreover, it have antimicrobial profile especially against Gram positive strains [19].

Besides that, at 41.84min' peak about 6.28% of Vitamin A aldehyde was analyzed. Vitamin A is fat-soluble and can be comprises of unsaturated nutritional organic compounds, for example retinol (alcohol), retinal (aldehyde), retinoic acid (irreversibly oxidized form of retinol) and several pro-vitamin A carotenoids (mainly β–carotene) [24]. Vitamin A are important to regulate immune system and good vision. It is also an important constituent of growth and embryonic development because it function as hormone-like growth factor (retinoic acid) for epithelial and other cells by irreversibly oxidized form of retinol [19]. Other effects of retinoid include anti-inflammatory effects, induction of apoptosis, and inhibition of tumor promotion. Vitamin A which is very useful for embryonic development in the womb because it is brilliant for the development of hematic blood tissues, eyes, skin, and hair of the fetus [20].

Cholestan-3-ol, 2-methylene-, (3á, 5à) is a steroid compound and it is available in extract at 42.42min and 6.15%. It medicinal activities are like anti-tumor and anti-inflammatory. Steroids have been used as anticancer agents since the 1940s in a wide variety of solid tumors, including breast and prostate cancer, and the lymphoid hematologic malignancies. If chemotherapeutic options have been abandoned, Cancer's patients can further consumed steroid to reduce this illness. Pass experiences showed that patients improved after 3 weeks and able to survive for another 15 more month [21]. Inflammatory diseases include asthma, rheumatoid arthritis and conjunctivitis. Steroid compounds will aid in interference of the inflammatory pathways and thus suppression of inflammation [22].

Finally at 43.61min about 1.43% of 1-Heptatriacotanol was shown from analyzed result. It is a long chain fatty acid alcohol same as Octacosanol. Research shown that long chain of fatty acid alcohol able to suppress lipid accumulation in rats fed on a high-fat diet [23].

I. Conclusion

SC- CO_2 extraction method is environmental friendly since no waste being produced and also time efficient as pure oil can be extracted efficiently in minutes of time. Temperature and pressure are the vital parameter leading this extraction. The optimum parameters obtaining the highest yield were at 50°C and 6000psi. Jackfruit seeds do contain multiples valuable medicinal compounds beneath it which have the tendency to cure patients with kinds of unthreatening diseases and reducing live threatening disease such as cancer. Medicinal compounds that are present are (1-Methoxy-pentyl), Palmitic acid, Lauric acid, Vitamin A aldehyde, Cholestan-3-ol, 2-methylene-, (3á, 5à) and 1-Heptatriacotanol.

Acknowledgment

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