UNIVERSITI PUTRA MALAYSIA

FEASIBILITY OF EXTRACTION METHODS OF ESSENTIAL OIL FROM PINEAPPLE PEELS

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FBSB 2019 20

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

July 2018

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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July 2018

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Faculty : Biotechnology and Biomolecular Sciences

The pineapple industry produces substantial amount of solid wastes where the value of waste generated was reported as high as 75-80%. Peels, cores, stems, crowns and pulp were removed and becoming waste during the processing. Since disposal of these wastes is expensive due to high transportation cost and a limited availability of landfills, they are mostly being unscrupulously dumped and openly burnt. The current practice of dumping the pineapple wastes can cause microbial spoilage and environmental problems due to high contents of moisture and sugar. Pineapple wastes including peel composed of aromatic compound such as esters, alcohols, acids, aldehydes and ketones, in which it can be extracted as essential oil. The essential oil plays an importance use in industrial applications such as in aromatherapy, fragrances and flavors in food and beverages as well as personal care. However, up to date, there are very limited studies on the extraction methods of essential oil from pineapple wastes. Considering the value of pineapple peel as a feedstock for essential oil extraction, the present study was conducted to identify a feasible method for the extraction of essential oil from pineapple peel. Three methods have been carried out in this study; (1) hydro-distillation (HD), (2) hydro-distillation with enzyme-assisted (HDEA), and (3) supercritical fluid extraction (SFE). Among the methods used, only SFE resulted in the formation of essential oil with 0.17% (w/w) yield, whereas HD and HDEA methods only produced the hydrosol with the yield of 70.65% and 80.65%, respectively. The microscopic observation using scanning electron microscope (SEM) of the sample's cell wall substantiated that only SFE method showed the rupture of the essential oil gland after the extraction process. The gas chromatography-mass spectrometry (GC-MS) analysis showed that the major volatile compounds identified in the essential oil produced through SFE method were propanoic acid ethyl ester (40.25%), lactic acid ethyl ester (19.35%), 2-heptanol (15.02%), propanal (8.18%), 3-hexanone (2.60%) and butanoic acid ethyl ester (1.58%). These important volatile compounds indicate the potential of pineapple peel to be used as a substrate for essential oil production. This study suggested that the SFE is a feasible method for the extraction of essential oil from pineapple peel which produced the major important volatile compounds.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Sarjana Sains

KAJIAN KESESUAIAN KAEDAH PENGEKSTRAKAN MINYAK PATI DARIPADA KULIT NANAS

Oleh

NURSHAZANA MOHAMAD

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Industri nanas menghasilkan sejumlah besar sisa pepejal di mana nilai sisa yang terhasil adalah dilaporkan sebanyak 75-80%. Kulit, empulur, batang, mahkota dan pulpa dibuang dan akan menjadi sisa ketika pemprosessan. Memandangkan pelupusan sisa-sisa ini mahal kerana kos pengangkutan yang tinggi dan ketersediaan tapak pelupusan yang terhad, kebanyakan sisa ini dilupuskan secara tidak berhemah dan dibakar secara terbuka. Amalan semasa dengan membuang sisa nanas boleh menyebabkan kerosakan mikrob dan masalah alam sekitar disebabkan kandungan kelembapan dan gula yang tinggi. Sisa nanas iaitu: kulit mengandungi sebatian aromatik seperti ester, alkohol, aldehid, asid dan keton, yang boleh diekstrak sebagai minyak pati. Minyak pati memainkan peranan penting dalam penggunaan industri melalui aplikasinya dalam aroma terapi, pewangi dan perasa dalam makanan serta minuman dan juga barangan penjagaan peribadi. Walau bagaimanapun, sehingga kini, kajian mengenai kaedah pengekstrakan minyak pati dari sisa nanas adalah sangat terhad. Mempertimbangkan nilai kulit nanas sebagai bahan mentah untuk pengekstrakan minyak pati, kajian ini telah dijalankan untuk mencari kaedah yang sesuai untuk pengekstrakan minyak pati dari kulit nanas. Tiga kaedah yang telah dijalankan dalam kajian ini ialah (1) hidro-penyulingan (HP), (2) hidro-penyulingan dengan bantuan enzim (HPBE), dan (3) pengekstrakan cecair genting lampau (PCGL). Diantara kaedah-kaedah yang digunakan, hanya PCGL menghasilkan minyak pati dengan hasil 0.17% (w/w), manakala kaedah HP dan HPBE hanya menghasilkan hydrosol dengan hasil sebanyak 70.65% dan 80.65%. Kajian menggunakan mikroskop elektron pengimbasan (MEP) terhadap dinding sel sampel membuktikan bahawa hanya kaedah PCGL mengakibatkan kelenjar minyak pati pecah selepas proses pengekstrakan. Analisis kromatografi gas-spektrometri jisim (KG-SJ) menunjukkan bahawa sebatian aromatik mudah meruap yang dikenalpasti dalam minyak pati yang dihasilkan melalui kaedah PCGL ialah etil ester asid propanoik (40.25%), etil ester asid laktik (19.35%), 2-heptanol (15.02%), propanal (8.18 %), 3-hexanone (2.60%) dan ester etil asid butanoik (1.58%). Sebatian-sebatian penting ini menunjukkan potensi kulit nanas untuk digunakan sebagai bahan mentah untuk penghasilan minyak pati. Kajian ini mencadangkan bahawa PCGL adalah kaedah yang sesuai untuk pengekstrakan minyak pati dari kulit nanasyang menghasilkan sebatian aromatik meruap yang penting.

ACKNOWLEDGEMENTS

Alhamdulillah, all the praise to the Almighty Allah for giving me opportunity to conduct a research entitled “Feasibility Study on the Extraction Methods of Essential Oil from Pineapple Peel” in fulfillment of the requirement for the degree of Master of Science in Industrial Biotechnology. This research project would not have been possible without the support of many people. My appreciation goes to my main supervisor, Dr Mohamad Faizal Ibrahim and supervisory committee, Dr Norhayati Ramli and Prof Dr Suraini Abd Aziz from Department of Bioprocess Technology, Faculty of Biotechnology and Biomolecular Sciences, Universiti Putra Malaysia.

Deepest gratitude to all the members of Environmental Biotechnology Research Group, Universiti Putra Malaysia, for their guidance and assistance. Finally, an honourable mention to my husband, Ahmad Nazroll Mohd, my daughter, Izzah Amani, and all of my family members for their fully understandings and supports for me to complete this project. Without their strong helps and supports, I would face many difficulties while doing this project.
Thank you.

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

ADF	Acid detergent fiber
ADL	Acid detergent lignin
CO_2	Carbon dioxide
DNS	Dinitrosalicylic acid
FPA	Filter paper assay
FPU	Filter paper units
g	gram
GCMS	Gas chromatography-mass spectrometry
h	hour
HD	Hydro-distillation
HDEA	Hydro-distillation with enzyme-assisted
HMF	5-hydroxymethylfurfural
MARDI	Malaysia Agriculture Research and Development Institute
min	minute
mL	millilitre
MPIB	Malaysia Pineapple Industry Board
NDF	Neutral detergent fiber
RM	Ringgit Malaysia
SEM	Scanning electron microscopy
SFE	Supercritical fluid extraction
USD	US Dollar

CHAPTER 1

INTRODUCTION

Malaysia is ranked within top 20 as world leading countries in the production of pineapple (MPIB, 2014), and 7th in Asia after Thailand, Philippines, Indonesia and Vietnam. Malaysia produced 315,977 tonnes of pineapple in 2013 and statistic showed that the trend of pineapple production in Malaysia has been increasing since 2010 to 2015 (MPIB, 2015). Although it is considered to be relatively small as compared to other commodity in Malaysia such as palm oil and rubber, the pineapple industry still play an important role in the country's socio-economic development. The export value of pineapple in Malaysia was RM 155 million a year and expected to achieve RM 320 million in 2020 (Othman, 2017). The forecasted increment in the export value becomes a great opportunity to many pineapple farmers and plantation owner to generate more income through pineapple farming.

The pineapple industry produces substantial amount of wastes during the processing. Peels, cores, stems, crowns and pulp are removed during planting, harvesting and manufacturing which turn into wastes. As much as 30-50% of the total fruit weight is discarded in the canning process (Lun *et al.*, 2014). With the increasing of total production, it is estimated that the amount of waste generated will increased. Therefore, a proper way of managing the pineapple waste is very essential. Improper disposal of the pineapple waste can cause microbial spoilage and environmental problems due to the waste material containing high moisture and sugar content (Lun *et al.*, 2014). The pineapple wastes are usually shredded or ploughed back into the mineral soil after cropping and before replanting. However, the inherent nature of peat that does not allow shredding or ploughing of these wastes into the soil before replanting resulted in recycling practice through open burning (Ahmed *et al.*, 2002).

Among the pineapple wastes generated, peels were reported as the largest portion generated which encountered about 30-42% (w/w) of the total waste generated (Rattanapoltee & Kaewkannetra, 2014). Besides, there are some aromatic flavours that can be smelled from pineapple peels due to the presence of a certain quantity of the essential oils, esters and organic acids. A study conducted by Barretto *et al.* (2013) showed that the pineapple residues (peels and leaves) constituted mostly of esters (35%), followed by ketones (26%), alcohols (18%), aldehydes (9%), acids (3%) and other compounds (9%). The presences of these compounds indicate that the pineapple peel has the potential to be extracted as aromatic essence in the form of essential oils.

Essential oils are concentrated volatile aromatic compounds produced by plants, also known as the easily evaporated essences that give plants their wonderful scents. These highly concentrated substances are found in special cells, glands or ducts located in various parts of plants such as flowers, leaves, stems, roots, seeds, bark, and resin or fruit rinds (Kumar & Tripathi, 2011). These oils are often used for their flavor therapeutic or

odoriferous properties, in a wide selection of products such as foods, medicines and cosmetics. The global essential oil market size exceeded USD 6.0 billion in 2015. Increasing applications in aromatherapy coupled with rising demand for fragrances and flavors in food and beverages and personal care is expected to drive market growth over the forecast period (GVR, 2016). Among the major types of essential oils that available in the market are citronella, pepper mint, lavender, orange, lime, clove, eucalyptus and many more. There are also some pineapple essential oils available in the market nowadays. However, most of the pineapple essential oil available in the market is actually fragrance oil. Pineapple fragrance oil offers a gentle scent of pineapple which is typically a combination of natural ingredients combined with synthetic compounds. It is widely used in the production of body lotion, bath salts, scented candles and other various personal care products (Fuller, 2016).

There are various methods can be used to extract essential oil from the plant matrix. The conventional extraction methods such as hydro-distillation (HD), steam-distillation and cold-pressing are the most commonly used methods for the extraction of essential oil (Abdul-majeed *et al.*, 2013; Galadima *et al.*, 2012). These conventional methods of extraction become preference because of the simple and cheap extraction set up. In addition, there are also several innovative green extraction methods that have been recently developed such as the microwave assisted extraction (Cansu *et al.*, 2011), CO_2 supercritical fluid extraction (SFE) method (Jusoh, 2014; Nautiyal & Tiwari, 2012; Pourmortazavi & Hajimirsadeghi, 2007) and enzyme-assisted extraction method (Sowbhagya *et al.*, 2011; Chandran *et al*., 2012). This recent trend of innovation is developing in parallel to meet the demands on the economical perspective, competition among the industries, eco-friendly, sustainability and good quality for industrial production.

Although there are various methods that can be used a feasible method to extract essential oil efficiently from pineapple peel is limited. Besides, exploration of the valuable compounds in the pineapple peels might be beneficial for future application such as in the various personal care applications. Therefore, three extraction methods (1) hydro-distillation (HD), (2) hydro-distillation with enzyme-assisted pretreatment (HDEA) and (3) supercritical fluid extraction (SFE) were investigated in this study in order to identify the feasibility of these methods to extract essential oil from pineapple peel.

Therefore, the objectives of this study are:

1. To extract essential oil from pineapple peel through hydro-distillation (HD), hydro-distillation with enzyme assisted (HDEA) and supercritical fluid extraction (SFE).
2. To determine the volatile compounds of essential oil from pineapple peel using gas chromatography- mass spectrometry (GC-MS)
3. To examine the cell wall structure of the pineapple peel before and after extractions under scanning electron microscopy (SEM)

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LIST OF PUBLICATIONS

Paper publication:

Mohamad, N., Abd-Aziz, S., Ramli, N. (2019). Comparison of hydro-distillation, hydro-distillation with enzyme-assisted and supercritical fluid for the extraction of essential oil from pineapple peels. *3biotech*, 9(6):234. DOI: 10.1007/s13205-019-1767-8

Conference proceeding:

Nurshazana Mohamad, Suraini Abd-Aziz, Norhayati Ramli and Mohamad Faizal Ibrahim. Feasibility study on the extraction methods of essential oil from pineapple peel. In the 5th SAES – International Symposium on Applied Engineering and Sciences (SAES2017) UPM – Kyutech, UPM, Selangor, Malaysia.
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