UNIVERSITI PUTRA MALAYSIA

FORMULATION AND EVALUATION OF EFFERVESCENT JOSEPHINE PINEAPPLE TABLET

FARIDATUL AIN MOHD ROSDAN

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By

FARIDATUL AIN MOHD ROSDAN

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

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

FORMULATION AND EVALUATION OF EFFERVESCENT JOSAPHINE PINEAPPLE TABLET

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FARIDATUL AIN MOHD ROSDAN

June 2014

Chairman : Assoc. Prof. Yus Aniza Yusof, PhD

Faculty : Engineering

There is an increasing demand in commercialization of native fruits for utilization as functional foods and medicinal extracts. Josapine pineapple is used in this study since it is a delicious and popular fruit due to its good aroma, flavour, juiciness, sweetness and texture. This study is mainly undertaken to design and optimize an effervescent tablet formulation of the Josapine pineapple by using the D-optimal experimental design methodology. This thesis is presented in two major parts namely, an evaluation of physicochemical properties of Josapine pineapple pulp and freeze-dried powder using Principal Component Analysis (PCA) and the optimization of an effervescent pineapple tablet formulation by using mixture design. At the beginning of this study, the physicochemical properties of Josapine pineapple pulp and freeze-dried powder were investigated because an understanding on the physicochemical properties between Josapine pineapple pulp and freeze-dried powder provides valuable information in order to develop an effervescent tablet formulation. PCA was then used to analyze the variations of physicochemical and sensory properties of the Josapine pineapple under different treatments (pure, 10 % or 20 % maltodextrin and 10 % or 20 % sugar) under two different conditions (pulp and freeze-dried powder). Josapine pineapple powder, citric acid, sodium carbonate and stevia were used in the formulations as independent variables. Tablets were prepared by direct compression method and evaluated for their disintegration time and sensory properties which were regarded as responses in a D-optimal design. From this study, it was found that freeze-dried pineapple powder with addition of maltodextrin and sugar can be differentiated based on physicochemical properties data and analysis. The optimum formulation contained pineapple powder, citric acid, sodium carbonate and stevia at 49.59 %, 20 %, 11.96 % and 18.45 %, respectively. In addition, optimum formulation has a very fast disintegration time and quite high overall acceptability which represents the consumer approval. The observed values of the responses obtained from the optimized formulation were very close to the predicted values where the Euclidean distance calculated for optimum formulation was equal to 0.26. This study reveals that the effervescent pineapple tablet has a wide potential for future development and can be enhanced for commercialization. This work may contribute towards Malaysia's economic growth especially in the food and beverage industry.

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

FORMULASI DAN PENILAIAN TABLET NENAS JOSAPINE BERBUIH

Oleh

FARIDATUL AIN MOHD ROSDAN

Jun 2014

Pengerusi : Prof. Madya Yus Aniza Yusof, PhD

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Terdapat permintaan yang semakin meningkat dalam pengkomersilan buah-buahan asli untuk penggunaan sebagai makanan berfungsi dan juga ekstrak untuk perubatan. Nenas Josapine digunakan dalam kajian ini kerana ia adalah buah yang lazat dan popular kerana aroma, rasa, jus, kemanisan dan tekstur yang bagus. Kajian ini dijalankan terutamanya untuk mereka bentuk dan mengoptimumkan formula tablet berbuih nenas Josapine dengan menggunakan kaedah reka bentuk eksperimen D-optimum. Tesis ini dipersembahkan dalam dua bahagian utama iaitu , perbandingan sifat fizikokimia antara pulpa dan serbuk beku-kering nenas Josapine menggunakan Analisis Komponen Utama (PCA) dan pengoptimuman formulasi tablet nenas berbuih dengan menggunakan reka bentuk campuran. Pada awal kajian ini, sifat fizikokimia pulpa dan serbuk beku-kering nenas Josapine disiasat kerana pemahaman tentang sifat fizikokimia antara pulpa dan serbuk beku-kering nenas Josapine menyediakan maklumat yang berharga dalam usaha untuk membangunkan formulasi tablet berbuih nenas Josapine. PCA kemudiannya digunakan untuk menganalisis variasi sifat fizikokimia nenas Josapine dan analisis derianya di bawah rawatan yang berbeza (tulen , 10% atau 20% maltodekstrin dan 10% atau 20% gula) di bawah dua keadaan yang berbeza (pulpa dan juga beku-kering serbuk) . Serbuk beku-kering nenas Josapine, asid sitrik, natrium karbonat dan stevia telah digunakan dalam formulasi sebagai pembolehubah bebas. Tablet telah disediakan menggunakan kaedah mampatan langsung dan dinilai dari segi masa perguraian dan juga ciri deria yang dianggap sebagai tindak balas menggunakan reka bentuk D-optimum. Formulasi optima mengandungi serbuk beku-kering nenas, asid sitrik, natrium karbonat dan stevia pada 49.59 %, 20 %, 11.96 % and 18.45 % masing-masing. Di samping itu, formulasi optima mempunyai masa perguraian yang sangat cepat dan penerimaan keseluruhan agak tinggi yang juga mewakili penerimaan pengguna. Nilai yang diperhatikan daripada tindak balas yang diperolehi daripada formulasi optimum adalah sangat dekat dengan nilai-nilai yang diramalkan di mana jarak Euclidean dikira untuk formulasi optima iaitu 0.26. Kesimpulan secara keseluruhan, kajian ini menunjukkan bahawa tablet nenas berbuih mempunyai potensi yang luas untuk dibangunkan pada masa hadapan dan boleh dipertingkatkan untuk dikomersialkan. Kerja ini boleh menyumbang ke arah pertumbuhan ekonomi Malaysia terutama dalam industri makanan dan juga minuman.

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Thank you so much.

I certify that an Examination Committee has met on 5th June 2014 conduct the final examination of Faridatul Ain Mohd Rosdan on her degree thesis entitled " Formulation and Evaluation of Effervescent Josapine Pineapple Tablet" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the student be awarded the degree of Master of Science.

Members of the Examination Committee were as follows:

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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

AIDS	Acquired Immunodeficiency Syndrome
ANOVA	Analysis of variance
ARVs	Anti-retrioval drugs
a_w	Water activity
CI	Carr Index
D	Diameter
DDGS	Dried Distiller Grains with Soluble
DE	Dextrose equivalent
DOE	Design of experiment
Ed	Euclidean distance
F	Crushing forces or loads [MPa]
H	Thickness [m]
H_2SO_4	Sulphuric acid
HIV	Human Immunodeficiency Virus
HR	Hausner Ratio
IC_{50}	50 % inhibition of the viability of all the experimental human cancer cells
ICH	International Conference on Harmonization
LSD	Least significant different test
MARDI	Malaysian Agricultural Research and Development Institute
NaOH	Sodium hydroxide
NAP 3	The Third of National Agriculture Policy
Obs_i	Observed values
PC1	First principal component

PC2	Second principal component
PCA	Principal Component Analysis
PCs	Principal Components
$Pred_i$	Predicted values
Qbd	Quality by design
RSM	Response surface methodology
SAS	Statistical Analysis Software
SD	Standard deviation
SEM	Scanning electron microscope
T	radial tensile strength [MPa]
TA	Titratable acidity
T_g	Glass transition temperature
TSS	Total soluble solids
U.S FDA	United States Food and Drugs Administration
WHO	World Health Organization

CHAPTER 1

INTRODUCTION

1.0 Introduction

This chapter introduces the background of this study based on the raw material which is the Josapine variety of pineapple including the benefits for human health and the application of the pineapple in the nutraceutical industry in Malaysia specifically. The problem statement then follows, along with the objectives of this study.

1.1 Pineapple

Pineapple is the most important representative of the Bromeliaceae family and is cultivated in tropical and subtropical countries including Malaysia, Hawaii, South Africa, Philippines and Thailand for local consumption and also international export (Elss et al., 2005). Pineapple holds the third rank in world tropical fruit production only preceded by the banana and citrus fruits (De Poel et al., 2009). The Malaysian Pineapple Industry Board (MPIB) reported that for 2008, Johor produced the highest yield of pineapple with 143963 metric tons followed by Kelantan and Kedah with 8209.6 and 1121.7 metric tons, respectively (MPIB, 2010). Based on its potential economic and commercial value, pineapple has been identified as one of the priority commodities to be developed for the domestic and international markets in The Third National Agriculture Policy – NAP 3 (Samah, 2004). Additionally, Malaysia is in 11th place in the ranking of the world's pineapple exporters and aims to move up beyond the current ranking. This can be achieved by increasing the export value through technology and production (Zolkepli, 2010). Therefore, current research into the pineapple is focused on the development of new varieties that provide benefits during plantation, harvesting, postharvest and also product development.

Pineapple is a unique tropical fruit having outstanding juiciness, vivacious flavour, delicious taste and immense health benefits. Pineapple contains significant amounts of calcium, dietary fibre, potassium and copper. Furthermore, it has a small amount of fat and cholesterol and is a good source of vitamins such as vitamin B1, vitamin B6, and vitamin C. Much research has been done and is still ongoing which looks at the therapeutic effects of pineapple fruit especially with regard to human health.

1.2 Problem Statement

According to Francis (1982), about 70 % of pineapple is consumed as fresh fruit. The problem with fresh pineapple is how best the fruit can be stored at room temperature in order to minimize postharvest losses. Nevertheless, some of the fruit is wasted at the production points due to lack of sufficient storage, transportation and processing facilities. In association with the climate problem, most of the local fruits present high water content, making them more susceptible to decomposition by microorganisms, chemical and also enzymatic reactions. Therefore, they are

extremely perishable and cannot be marketed or exported as fresh produce. Additionally, freshly expressed juice is highly susceptible to spoilage due to a lack of protection by skin or cell walls, and the fluid components are thoroughly mixed with air and microorganisms from the environment. Thus, unheated juice is subject to rapid microbial, enzymatic, chemical and physical deterioration. All these problems result in the adoption of various processing techniques to extend the shelf life of the fresh fruits so as to ensure all year round availability of the fruit in different forms. The goal of processing is to minimise these undesirable reactions while still maintaining, and in some cases enhancing, the inherent qualities of the starting fruit (Bates et al., 2011).

Hence the development of an effervescent pineapple tablet formulation is a suitable solution in the current situation. The fresh pineapple will processed into powder, formulated and compacted into tablet. The effervescent pineapple tablet allows longer periods of storage, minimise packaging requirements and also reduce shipping weight. Thus, it can lower the production cost and provide advantages for the industry to be marketed locally or overseas while maintaining the quality of the fresh pineapple. However, difficulties might occur during the production process, for example, during the freeze drying process to obtain pineapple powder as a main ingredient in the formulation. The powder obtained might be sticky, hygroscopic and have lower solubility due to the presence of low molecular weight sugars and acids in the pineapple pulp which leads to a low glass transition temperature (Bhandari and Howes, 1999). These problems can be solved by the addition of drying agents such as polymers and gum. The drying agents are normally used for microencapsulation to protect sensitive food components against unfavourable ambient conditions, to mask or preserve flavours and aromas, reduce the volatility and reactivity and also to provide additional attractiveness for the merchandising of food products (Gharsallaoui et al., 2007).

1.3 Objectives of the Study

The overall objectives of this research are:

1. To determine and analyze the physicochemical and sensory properties of pineapple pulp and freeze-dried pineapple powder.
2. To optimize an effervescent pineapple tablet formulation using mixture design.

1.4 Scope of Study

Optimization is a special technique developed to increase the desirable quality parameters by analyzing the various components of individual factors such as disintegration time, sweetness and flavour required in relation to sensory evaluation of detailed descriptors. Optimization consists of few steps for obtaining the optimum condition or result under a given set of constraints. Optimization is very useful especially in food research with different systematic experimental designs for product process or formulation (Stone et al., 1974). Expected high desirable scores and low disintegration time in an effervescent fruit tablet are computed by using an integrated approach such as analysis of variance (ANOVA) and response surface

modelling. There is still no report of an optimization study on effervescent fruit tablets in Malaysia.

Usually for experiments that involve a formulation, a two-level factorial is utilized as an alternative for the design of experiment (DOE) method. The two-level factorial consists of all combinations of each factor at its high and low range of levels. Using a two-level factorial this can decrease the number of experiments required because only a fractions of runs need to be completed to produce estimates of the main effects and simple interactions. In pharmaceutical, nutraceutical and food formulations where the conditions need the response to be dependent on the proportions of the ingredients, factorial designs may not make much sense. Therefore, mixture design is much more suitable in this study because it accounts for the dependence of response on the proportionality of the ingredients used. In mixture experiments, the factors are the components or ingredients of a mixture and consequently their levels are not independent (Montgomery, 2009).

1.5 Outline of the Thesis

Chapter 1 gives an introduction to the study which briefly reviews the materials used which is the Josapine variety of pineapple. A brief background and benefits to human health are explained. The problem statement is also clarified in this chapter in order to clarify the situation up to this current study taking place. At the end of the chapter, the overall objectives are described.

Chapter 2 gives details of the literature review. The literature review consists of the background theory and analysis that is involved in this research generally. The background theory explains the raw materials, the ingredients used in the effervescent tablet, and a few recently developed effervescent tablets especially from the pharmaceutical and nutraceutical point of view. Next, a brief description explains the physicochemical properties of the materials especially using proximate analysis (moisture content, ash, fibre, fat and protein), titratable acidity, water activity and sensory analysis are described as they are important in order to develop an effervescent pineapple tablet formulation. Further, the freeze drying process and additives used in order to produce a suitable freeze dried powder are also discussed in this chapter. In addition, some discussion on the principal component analysis (PCA) and optimization process is also incorporated. From the optimization process, the experimental work on the tableting of the formulation generated by the optimization process is described. The tableting process by using uniaxial die compaction is described to facilitate the production of effervescent pineapple tablets. Additionally, the mechanical behaviour of the effervescent pineapple tablet is included and discussed.

In **Chapter 3** information is provided about materials and methodology. First of all, raw materials will be introduced as well as other ingredients used in the study. Next, the method used in producing freeze-dried pineapple powder is explained. Then, the methodology of physicochemical analysis including colour values (L*, a*, and b*), titratable acidity (TA), total soluble solids (TSS), proximate composition (moisture, protein, ash, fibre and fat content), water activity (a_w), pH, and also the sensory evaluation are included. After that, the principal component analysis (PCA) is

explained. This statistical tools attempt to explore by using analysis of variance (ANOVA) and PCA if it is possible to distinguish the difference in taste between Josapine pineapple pulp and freeze-dried powders.

Chapter 4 discusses the development of the effervescent pineapple tablet formulation by adapting the effervescence effect as an advantage for fast dissolution in water. Moreover, the study is extended to develop a formulation of effervescent pineapple tablet that is acceptable to the consumer by using an optimization technique through mixture design. In addition, the mixture design facilitated by Design Expert 7.0 is used to develop a series of formulations. In the context of the development of the effervescent pineapple tablet formulation, a study is performed of the effect of the composition mixture on the sensory properties and disintegration time. The amount of pineapple powder, citric acid, sodium carbonate and stevia are manipulated in this study and denoted as independent variables.

Chapter 5 presents a summary of the overall study of this research including the recommended effervescent pineapple tablet formulation. In addition, recommendations for future work are also presented in this chapter.

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