# DEVELOPMENT OF HALALAN TAYYIBAN QURANIC MIXED FOOD EFFERVESCENT TABLET (QMFET) BY USING MULTIVARIATE ANALYSIS

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A thesis submitted in fulfillment of the requirements for the award of the Doctor of Philosophy in Science

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This thesis is for the sake of Allah SWT, my Creator and Master, my source of inspiration, wisdom, knowledge, and insight and to Muhammad SAW (may Allah bless him), my great teacher and messenger, who taught us the meaning of life. This thesis is also dedicated to:

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### ABSTRACT

Quranic food such as pomegranates, raisins, honey, dates, and figs known as food mentioned in Quran that beneficial to human. Effervescent tablet is one of interesting type of nutraceutical product to be developed. However, manual effervescent tablet development incure more time and high cost. Application of multivariate analysis helps in minimise the time and cost. The challenge in effervescent tablet development as a nutraceutical product lies in improving its physical and chemical qualities, consumer acceptability, and therapeutic properties. Therefore, this study is aimed to develop Halalan Tayyiban Quranic mixed food effervescent tablets (QMFET) by using multivariate analysis. The phytochemicals in Quranic foods were screened, and the flavonoid content of Quranic mixed food (QMF) was optimised by simplex centroid mixture design. Then, QMFET was developed by demand forecasting study, powder physicochemical test, objective sensory test, quality test, antioxidant activity test, cytotoxicity test, and Halal Critical Point (HCP) identification. In demand foracasting study, a strong correlation (r > 0.7) was determined between demographic and perceived product quality towards QMFET. A QMF powder had good flowability quality. Consequently, QMFET formulation containing 35.77% QMF powder, 26.68% Lascorbic acid, 26.68% sodium bicarbonate, 3.75% sweetener, 3.74% food flavour, and 2.27% food colorant exhibited high overall sensory acceptability. The quality of QMFET met the United States Pharmacopeia (USP) standards and the antioxidant capacity are within strong range (>30 µg/mg). QMFET was also not toxic to human normal cell line (HS27). QMFET had comply with the Halalan Tayyiban principles and four HCP were identified and established. In conclusion, QMFET have a high potential as a Halalan Tayyiban nutraceutical product to be commercialised.



### ABSTRAK

Makanan al-Quran seperti delima, kismis, madu, kurma, dan buah tin dikenali sebagai makanan yang disebut dalam al-Quran dan bermanfaat kepada manusia. Pil mudah larut adalah salah satu jenis produk nutraseutikal yang menarik untuk dibangunkan. Walau bagaimanapun, pembangunan pil mudah larut manual memerlukan lebih banyak masa dan kos yang tinggi. Aplikasi analisis multivariat membantu meminimumkan masa dan kos. Cabaran dalam pembangunan pil mudah larut sebagai produk nutraseutikal adalah untuk meningkatkan kualiti fizikal dan kimia, kebolehterimaan pengguna dan sifat terapeutiknya. Oleh itu, kajian ini bertujuan untuk membangunkan pil mudah larut makanan campuran al-Quran (QMFET) Halalan Tayyiban dengan menggunakan analisis multivariat. Fitokimia makanan al-Quran telah disaring dan kandungan flavonoid makanan campuran al-Quran (QMF) dioptimumkan menggunakan reka bentuk campuran simpleks sentroid. Kemudian, QMFET dibangunkan melalui kajian ramalan permintaan, ujian fizikokimia serbuk, ujian sensori berobjektif, ujian kualiti, ujian aktiviti antioksidan, ujian kesitotoksikan dan penentuan Titik Kritikal Halal (HCP). Dalam kajian ramalan permintaan, korelasi yang kuat (r > 0.7) antara demografi dan kualiti produk dapat dilihat terhadap QMFET. Serbuk QMF mempunyai kualiti kebolehaliran yang baik. Hasilnya, formulasi QMFET yang mengandungi 35.77% serbuk QMF, 26.68% asid L-askorbik, 26.68% natrium bikarbonat, 3.75% pemanis, 3.74% perisa makanan, dan 2.27% pewarna makanan menunjukkan kebolehterimaan sensori yang tinggi. Kualiti QMFET adalah mematuhi piawaian United States Pharmacopeia (USP) dan mempunyai kapasiti antioksidan pada tahap tinggi (>30 µg/mg). QMFET juga tidak toksik kepada sel normal (HS27). QMFET mematuhi prinsip Halalan Tayyiban dan empat HCP telah dikenalpasti. Kesimpulannya, QMFET mempunyai potensi tinggi untuk dikomersialkan sebagai produk nutraseutikal Halalan Tayyiban.

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### LIST OF SYMBOLS AND ABBREVIATIONS

- °C Degree Celsius
- h Hours
- mL Millilitre
- g Gram
- % Percent
- mg Milligram
- nm Nanometre
- L Litre
- Eq. Equation
- cm Centimetre
- μg Microgram
- TFC Total flavonoid content
- TPC Total phenolic content
- DPPH 2,2-Diphenyl-1-picrylhydrazyl
- ABTS 2,2'-Azino-bis-(3-ethylbenzothiazoline-6-sulfonic acid)
- FRAP Fluorescence recovery after photobleaching
- MTT 3-(4,5-Dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide
- MS Malaysian Standard

JAKIM	-	Jabatan Kemajuan Islam Malaysia
QMF	-	Quranic mixed food
USP-NF	-	United States Pharmacopeia. National Formulary
ROS	-	Reactive oxygen species
QMFET	-	Quranic mixed food effervescent tablet
HDL	-	High-density lipoprotein
LDL	-	Low-density lipoprotein
DNA	-	Deoxyribonucleic acid
FTIR	-	Fourier transform infrared spectroscopy
DS	-	Dietary supplement
FDA	-	Food and Drug Administration
NIH	-	National Institutes of Health
FSQD	-	Food Safety and Quality Division
DCA	-	Drug Control Authority
NPRA	_	National Pharmaceutical Regulatory Agency
CODEX	EDI	Codex Alimentarius collection of food standards
PCA	Rr	Principle component analysis
КМО	-	Kaiser–Meyer–Olkin
HCA	-	Hierarchical cluster analysis
НССР	-	Halal critical control point

Hazard analysis critical control point



HACCP -

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### LIST OF PUBLICATIONS

Salbi, N.M., Muhammad, N. & Abdullah, N. (2021). Optimizing flavonoid-rich Quranic Mixed Food (QMF) formulation with simplex-centroid mixture design. Food Research 5 (2) 80 – 87. https://doi.org/10.26656/fr.2017.5(2).470 (Scopus)

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### **CHAPTER 1**

### INTRODUCTION

### **1.1 Background of study**

In the current era, the fast rhythm of modern life has affected our eating habits, which in turn causes negative effects on health. Concurrently, there is a high demand for nutraceutical products, which are associated with the acceptance of food-like substances as being harmless when compared to conventional pharmaceuticals (Yusof, Etti, & Chin, 2015). Considering this, effervescent tablet is an interesting form of functional food that has a potential for innovation. This is due to the characteristics of such food, which have a complex active composition or nutrients that are beneficial to people. The development of nutraceuticals is mostly to improve quality, especially the active ingredients, during processing, storage, packaging, and transportation. Conversely, in pharmaceuticals, most of the innovation is to solve the problem of poor acceptability of medicine and improve medicinal function (Eyjolfsson, 2014). There are a few stages of evaluation throughout the tablet development process: formulation, pre-compression evaluation, tablet evaluation, *in vivo* or *in vitro* assessment, and dissolution tests that affect the physical, chemical, biological, and sensory quality of the tablet.

Additionally, there is a strong trend in the market for consumers to choose natural products and select those with some specific beneficial functions for the human body. In this context, the selection of functional ingredients with the required phytochemical content for the intended use is the primary challenge in formulating nutraceutical products (Malek *et al.*, 2017). In the food development science and advanced chemical synthetic capacity, food mixtures have become more established and sophisticated, and their reach continues to grow. Consequently, as bioactive



reactions are incredibly complex, numerous studies have been conducted on biological systems, such as those by Gouvinhas *et al.* (2017), Guiné and Barroca (2014), Mahmoudi *et al.* (2018), and Pourghayoumi *et al.* (2016). In addition, various techniques and methods for studying synergism and antagonism have been highlighted by previous studies (Chiou, 2007; Hidalgo *et al.*, 2010; Kurin *et al.*, 2012; Rahim *et al.*, 2020). The use of multiple foods can confer multiple therapeutic benefits (Farooq & Sehgal, 2019), and this practice is becoming increasingly popular and may be the industry's best option. Using multiple foods with various mechanisms or modes of action will also steer the effect against a single target or disease and manage it better (Amin *et al.*, 2018).

Health and wellness are among the core segments of the fast-moving consumer goods industry, with an ever-increasing health consciousness among consumers around the world (Nazir et al., 2019). At present, nature-based nutraceutical food products with targeted health benefits are at the heart of research and development activities (De Vries et al., 2018). In conjunction with these matters, several studies have been conducted on exploring the unique medicinal properties and chemical constitution of foods mentioned in the Holy Quran, which are known as Quranic foods (Sheikh & Dixit, 2015). It is undeniable that Quranic foods have therapeutic properties and have been used in preventive medicine for decades (Western et al., 2009). Quranic foods known to have health benefits are dates, raisins, honey, pomegranates, and figs (Muhammed & Shamsi, 2016; Al-Habsi & Al-Khusaibi, 2018). Moreover, Quranic foods are known for their phytochemicals, especially flavonoids, that are related to their curative properties, which have been utilised for the treatment of various ailments, including cancer prevention due to their high antioxidant properties (Muhammed & Shamsi, 2016). Besides flavonoids, other polyphenols in these foods have the potential to be biological modifiers that could grant health benefits to consumers (Rahmani et al., 2014; Asaduzzaman et al., 2015; Azahar et al., 2017).

Multivariate statistical and chemometric techniques established for analytical chemistry have been widely utilised in food science and technology in the recent decade. Chemometrics is typically used when a dataset is huge and complex in terms of sample numbers, types, and reactions. Multivariate techniques (Sergio *et al.*,



2017) can be used to optimise the formulation or process. It involves three main steps: analysing to select the fitted model, optimisation (Adiba et al., 2011; Salem et al., 2011; Bose et al., 2013; Saifullah et al., 2016; Azahar et al., 2017), and validation (Banala et al., 2013; Rahim et al., 2018). The simplex-centroid approach is one of the mixture designs types that is used in response surface methodology to construct, improve, and optimise mixtures (Patel et al., 2017). Currently, the main chemometric methods in food studies are used to investigate the impact of process variables on chemical composition and the authentication using chemical markers. While supervised multivariate statistical approaches are utilised for authentication, pattern recognition methods such as principal component analysis and cluster analysis are used to connect the level of bioactive components with in vitro functional characteristics. Chemometrics is a useful tool when dealing with large, multiple, and complicated real-life situations in a multifactorial and holistic setting. When high-dimensional data is involved, chemometrics should undoubtedly be employed by organisations or industries that need to check the quality of foods, raw materials, and processes.



Halalan Tayyiban products have gained attention not just among Muslims, but throughout the world. Pertaining to food consumption, Amir (2016) introduced seven principles underlying the Halalan Tayyiban products, which are halal, *shubhah*-free, hygiene, beneficial, safe, clean, and desirable. The aspects that underline these principles should be implemented in each area or process of food development. Malaysia aims to be the world's halal hub and has established standards and guidelines to be referred to by industry players to align with the concept of Halalan Tayyiban. The most relevant standard is MS 1500: 2009, which outlines the general guidelines for halal food manufacturing, focusing on production, preparation, handling, and storage of the food. This standard is not established by itself but is based on other standards such as MS 1514: Good Manufacturing Practices, MS 1900: Shariah-Based Quality Management System, and MS 1480: Food Safety According to HACCP System. In parallel with this standard, JAKIM has established a manual for halal certification that provides a detailed explanation of the requirements that are addressed in the Malaysian Standard.

In conjuction with those standards, this study aims to produce Halalan Tayyiban Quranic mixed food effervescent tablet (QMFET) by using multivariate analysis. Therefore, the Halalan Tayyiban principles were implemented in the OMFET development in this study. Multivariate analysis was applied throughout the process of QMFET development. The simplex-centroid design method was utilised in this study to optimise the flavonoid content of the formulation. Furthermore, discriminant and interaction analyses were performed on the mixture formulated from pomegranate, raisin, date, fig, and stingless bee honey. The acceptable by nature principle indicates that the food should not just be accepted by humans but also conform to the related standard. Therefore, demand forecasting and sensory evaluation were conducted. Beside overall acceptance, the compliance of the effervescent tablet quality with the related standard was also evaluated. Furthermore, the health benefit of the product was also evaluated in terms of antioxidant and anticancer properties. At the end, the application of Halalan Tayyiban principles in - roint the effervescent tablet development process was evaluated and Halal Critical Point was established.

#### 1.2 Problem statement of study



Nutraceutical products with low demand will fail on the market and be unable to generate a profit for a company. Consumer demand for greater diversity and enhanced advantages from nutraceutical products made feasible by technology is on the rise. Thus, developing a product that is acceptable to the target consumer is the primary challenge in product development. Consumer demand is affected by consumer location and product trend, both of which influence acceptability. Also, it is challenging to obtain satisfactory acceptance for nutraceutical products (Faridatul et al., 2016). These difficulties should be taken into account when formulating the new product (Yusof *et al.*, 2015)

Failure to achieve the desired health benefit results in decreased customer satisfaction while also destroying the true purpose of nutraceutical product development. Quranic food had become the attractive ingredient in development of new nutraceutical product nowadays due to their medicianal properties. However,

there is limited research has been done on the health properties of Quranic food mixtures as normally multiple variation of Quranic food are mixed in a product, which leads to misunderstandings of Quranic food product (Muhammed & Shamsi, 2016). The selection of ingredient with certain phytochemical qualities was linked to a product's health benefit (Ververidis *et al.*, 2007). Quranic foods are known as polyphenol-rich ingredient especially flavonoid that contribute to high antioxidant and had potential as anticancer theraphy (Slatnar, Klancar, Stampar, & Veberic, 2011; Vallejo, Marín, & Tomás-Barberán, 2012). Phytochemoical screening helps in identified the polyphenol that is highly present in ingredient that useful for optimization process to achieve intended health benefit (Malek *et al.*, 2017). As mentioned previously, determination of phytochemicals and functional properties of ingredient and its mixture before combining with the effervescent agent is necessary (Aslani & Jahangiri, 2013; Hussein, 2015).

When multiple foods are mixed, there is a chance that substances will interact, which could lead to ingredient deterioration and altered functionality. Loss of functional qualities will result in a product's poor bioactivity, which will reduce its ability to serve as a supplement and benefit consumers. These problems are extremely serious when adding many bioactive compounds to a product (Sun-Waterhouse, 2011). There is less information available on the flavonoid content of Quranic foods when combined, which is relevant to the development of Quranic mixed food (QMF) products. According to previous studies, there are many diverse elements in plant products, and this diversity implies a high likelihood of interactions (Serpen *et al.*, 2012; Rahim *et al.*, 2018). As highlighted by Farooq and Sehgal (2019), challenges in multicomponent mixtures include changing the metabolites' synergistic or antagonistic natures.

It also reported that the issues of time consumption and the high cost of the formulation trial-and-error technique in identifying the novel mixture of ingredients for the intended nutraceutical use have been a challenge (Patel *et al.*, 2017). Zaki (2014) mentioned that the problems in nutraceutical tablets are related to their tablet powder formulation to ensure the end product of tablet meet the quality and standard. Then, Sreeramya *et al.* (2018) highlighted that end users are looking for organoleptic values in new nutraceutical food products. Hence, in formulation of effervescent



tablet, powder properties of raw material and sensory evaluation are important for the development of high quality and acceptable effervescent tablet. Less visible study partnership the powder physicochemical quality with sensory evaluation in formulating effervescent tablet. Coating material type and ratio influence the physicochemical quality of powder (Lourenco *et al.*, 2020) while product taste helps in maximum consumer acceptance (Sharif *et al.*, 2017).

Moreover, the effervescent tablet that fail to meet standard will prevent the product from receiving certification from an acknowledged body, which will lower the market confidence. As per the standard of United States Pharmacopeia (USP) and the National Formulary (NF) standard (USP.NF, 2008), effervescent tablets should have desirable hardness, thickness, friability, and pH, besides the effervescence time. Then, in order to comply with the stipulated standard, it is also necessary to manage the physicochemical characteristics and bioactivity of effervescent tables (Faridatul Ain et al., 2016). However, previous studies have mostly focused on the physical and chemical characteristics of the tablet (Ong et al., 2013; Prakash et al., 2011; Saifullah et al., 2014; Saifullah et al., 2016; Zea et al., 2013). The review by Sreeramya et al. (2018) highlighted that end users are seeking extra nutritional benefits and organoleptic values in new nutraceutical food products. Thus, the tablet developed should not just meet the quality standard but also be beneficial to health to increase the demand. Therefore, the determination of functional properties and safety such as antioxidant and cytotoxicity activity of effervescent tablets is conducted (Prakash et al., 2011; Thoke et al., 2013).



The global halal market has been expanding over time, and products that don't meet those standards will have limited market potential. Beyond the difficulties in processing, the main feature of Halalan Tayyiban must be taken into account in the development of new products, given the rise in demand for it. As the halal industry expands and Malaysia strives to become a global halal hub, this issue should not be overlooked in new effervescent tablets product development. The principle of Halalan Tayyiban can serve as a relevant guideline as it is fundamental in food development applications. As validation of halal ingredients is a critical parameter in the Halal Assurance System (Sunaryo, Mardiah, & Rahmawati, 2019), it is also critical for the processing and analysis method (Sugibayashi *et al.*, 2019).

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