UNIVERSITI PUTRA MALAYSIA

MODELLING AND OPTIMIZATION OF PRODUCTION OF COFFEE-LIKE POWDER AND BREW FROM PALM DATE SEEDS BY ROASTING AND GRINDING

IBRAHIM MOHAMED FIKRY MOHAMED AHMED

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By

IBRAHIM MOHAMED FIKRY MOHAMED AHMED

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

August 2019

DEDICATION

I dedicate this work to

My great father and mother

My sweetheart wife (**Walaa**)

My beloved kids: **Adam** and **Leenah**

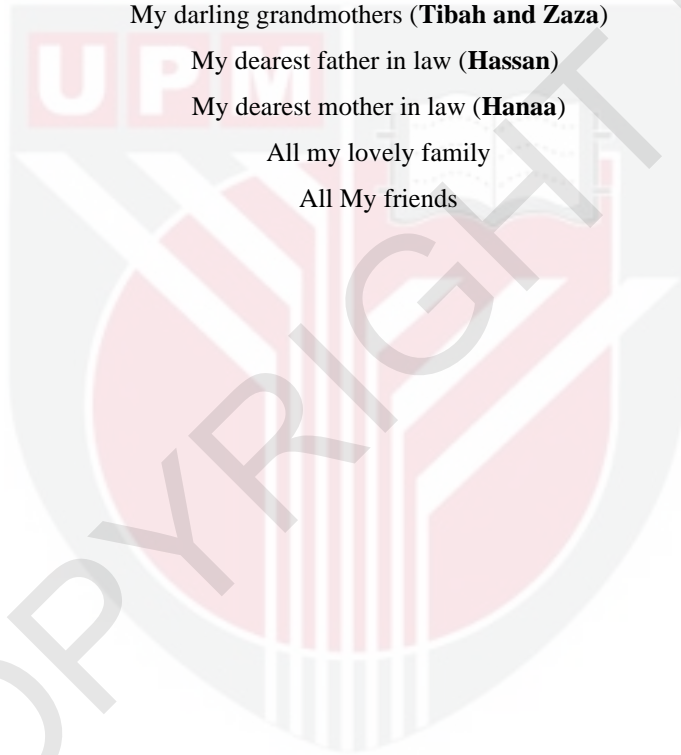My beloved brothers and sisters

My darling grandmothers (**Tibah and Zaza**)

My dearest father in law (**Hassan**)

My dearest mother in law (**Hanaa**)

All my lovely family

All My friends

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of
the requirement for the degree of Doctor of Philosophy

**MODELLING AND OPTIMIZATION OF PRODUCTION OF COFFEE-LIKE
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August 2019

Chairman: Prof. Ir. Yus Aniza Yusof, PhD
Faculty: Engineering.

A significant quantity of date seeds is being generated annually from palm date industry.
This by-product is usually discarded or used as an animal fodder. However, palm dates
seeds can be a potential source of nutritious elements and antioxidants which could be
used for human consumption in a powder form and decaffeinated coffee-like brew.

Hence, the study investigated the thin layer drying characteristics of date seeds at three
roasting temperatures (160,180, and 200 ℃) and modelled the changes in color,
hardness, total specific grinding energy and the oil extraction yield of date seeds during
roasting process. Besides, the study aimed to investigate the influence of roasting
temperature and time on the physical and sensory properties of the full fat roasted date
seeds powder (RDSP) and defatted roasted date seeds powder (DRDSP).

Additionally, the effect of roasting temperature and time on antioxidants, total phenolic
contents, quality and sensory attributes of the full fat and defatted brews was
investigated. Further, development of predictive models to estimate the properties of
both RDSP and DRDSP and the proposed brews, as well as the optimization of the
roasting conditions for preparation of the RDSP and DRDSP and brews were conducted.

The effects of the storage conditions on the physical and flowability properties of the
RDSP and DRDSP as well as the quality and sensory attributes of their brews were
studied and the shelf life of the powders and brews by integrating of quality and sensory
data was assessed.

The study outcomes revealed that the thin layer drying characteristics of date seeds were
satisfactorily described by the modified Henderson & Pabis model. While, the color
parameters (L*-value and b*-value) and the hardness of the roasted date seeds can be
fittingly described by the first-order equation, while a*-value and ΔE were adequately

defined by the zero-order model. A decrease in total specific grinding energy and an increase in oil yield were recorded as the roasting temperature and time were increased. The physicochemical, flow, quality and sensory attributes of the products were found to be significantly influenced by the roasting temperatures and time. The models proposed could satisfactorily describe the changes in the physicochemical, flow, quality and sensory attributes of RDSP, DRDSP and the brews. The optimum conditions of the roasting process obtained using the superimposed contour plot were 199.7 °C and 10.2 min to produce RDSP and 200 °C and 11.2 min to prepare DRDSP. Also, the optimal roasting temperature and time were 197.98°C and 12.8 min, respectively for preparing the full fat brew. While, the optimum points for preparing the defatted brew were 199.19°C and 22.7 min. Regarding the storage and shelf life study, the results showed that the storage conditions have a significant effect on the properties of RDSP, DRDSP and brews. Descriptive models have been developed for describing the different properties and the shelf life of the powders and brews.

Interestingly, the powders and brews are microbiologically stable during the storage process. The study outputs are beneficial for dryers and roaster designs, for quality control of the final product, and for determination of the suitable storage conditions. So, there is an ample scope for the emergence of new bio industries in the date growing countries towards maximum utilization of the palm date seeds in addition to efficient and effective palm date fruit waste management.

Abstrak tesis yang disampaikan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PEMODELAN DAN PENGOPTIMUMAN PENGHASILAN KOPI-SEPERTI SERBUK DARI BIJI KURMA DENGAN MEMANGGANG DAN KISARAN

Oleh

IBRAHIM MOHAMED FIKRY MOHAMED AHMED

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Banyak biji bijian yang dijana setiap tahun dari industri kurma. Produk sampingan ini biasanya dibuang atau digunakan sebagai makanan haiwan. Walau bagaimanapun, biji kurma ini boleh menjadi sumber yang berpotensi untuk unsur-unsur berkhasiat dan antioksidan yang boleh digunakan untuk penggunaan manusia dalam bentuk serbuk dan minuman beralkohol kopi tanpa kafein.

Oleh itu, kajian ini menyiasat ciri-ciri pengeringan lapisan nipis biji kurma pada tiga suhu pemanggang (160,180 dan 200 °C) dan memodelkan perubahan warna, kekerasan, jumlah tenaga pengisaran khusus dan hasil pengeluaran minyak dari benih kurma semasa proses pemanggangan.

Selain itu, kajian ini bertujuan untuk mengkaji pengaruh suhu dan masa pemangganan pada sifat fizikal dan sensori serbuk biji kurma penuh lemak (RDSP) dan serbuk biji kurma yang dibuang lemak yang dibakar (DRDSP). Di samping itu, kesan suhu dan masa pemangganan terhadap antioksidan, jumlah kandungan fenolik, kualiti dan ciri-ciri kualiti dan sensori minuman seakan kopi yang dihasilkan menggunakan serbuk biji kurma penuh lemak dan serbuk biji kurma yang dibuang lemak turut dikaji.

Tambahan pula, pembangunan model ramalan untuk menganggarkan sifat-sifat RDSP dan DRDSP serta cadangan yang dicadangkan, serta pengoptimuman keadaan pemanggang untuk penyediaan RDSP dan DRDSP dan minuman telah dijalankan. Kesan keadaan penyimpanan pada sifat fizikal dan sifat kebolehaliran RDSP dan DRDSP serta sifat kualiti dan deria dari minuman telah dikaji dan jangka hayat serbuk dan minuman dengan mengintegrasikan data kualiti dan deria ditaksir.

Hasil kajian mendedahkan bahawa ciri-ciri pengeringan lapisan nipis biji kurma telah memuaskan dengan diterangkan oleh model Henderson & Pabis yang telah diubahsuai. Selain itu, parameter warna (L * - nilai dan b * - nilai) dan kekerasan biji kurma panggang

boleh digambarkan dengan tepat oleh persamaan pesanan pertama, manakala * - nilai dan ΔE didefinisikan secukupnya oleh perintah sifar model. Pengurangan jumlah tenaga pengisaran khusus dan peningkatan hasil minyak direkodkan kerana suhu dan masa pemanggangan meningkat. Ciri-ciri fizikokimia, kebolehaliran, kualiti dan deria dari produk didapati sangat dipengaruhi oleh suhu dan masa panggang.

Model-model yang dicadangkan dapat menggambarkan perubahan sifat-sifat fizikokimia, kebolehaliran, kualiti dan deria RDSP, DRDSP dan minuman. Kondisi optimum proses panggang yang diperoleh menggunakan plot kontur superimposed adalah 199.7 ℃ dan 10.2 min untuk menghasilkan RDSP dan 200 ℃ dan 11.2 min untuk menyediakan DRDSP. Juga, suhu pemanggang yang optimum dan masa adalah 197.98 ° C dan 12.8 min, masing-masing untuk menyediakan minuman penuh lemak. Walau bagaimanapun, keadaan optimum untuk menyediakan minuman tanpa lemak adalah 199.19 ° C dan 22.7 min. Mengenai penyimpanan dan kajian hayat , keputusan menunjukkan bahawa keadaan penyimpanan mempunyai kesan yang signifikan terhadap sifat RDSP, DRDSP dan minuman. Model deskriptif telah dibangunkan untuk menerangkan ciri-ciri yang berbeza dan jangka hayat serbuk dan minuman. Menariknya, serbuk dan minuman stabil secara mikrobiologi semasa proses penyimpanan. Keluaran kajian adalah berguna untuk pengering dan reka bentuk pemanggang, untuk kawalan mutu produk akhir, dan untuk menentukan keadaan penyimpanan yang sesuai. Oleh itu, terdapat ruang lingkup yang mencukupi untuk kemunculan industri bio baru di negara-negara yang semakin berkembang ke arah penggunaan maksimum biji kurma selain pengurusan sisa buah kurma yang cekap dan berkesan.

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In the Name of Allah, the Most Merciful, the Most Compassionate all praise be to Allah, the Lord of the worlds; and prayers and peace be upon Mohamed His servant and messenger.

First and foremost, I must acknowledge my limitless thanks to Allah, the Ever-Magnificent; the Ever-Thankful, for His helps and bless. I am totally sure that this work would have never become truth, without His guidance.

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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 Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS AND NOMENCLATURE

Symbol	Definition	Symbol	Definition
Abs	Absorbance	MC	Moisture content
A_{gg}	The corresponding geometric surface area of this ellipsoid.	MR	Moisture ratio
A_g	The corrected surface area	K_G	GAB model constant
A_o	Frequency factor	K_p	Water vapor permeability
A_p	The surface area of the pocket	k	Drying coefficient or reaction rate constant
ALP	Aluminium foil laminated polyethylene	L^*	Lightness
ANOVA	Analysis of variance	L_0^*	Initial L*
a^*	Red/green	l_1	Length
a_0^*	Initial a*	l_2	Width
a	Constant	l_3	Thickness (the minimum diameter)
a_w	Water activity	l_m	The mean diameter
b	Constant	N	The number of the experimental data
b^*	Yellow/blue	n	An exponent
b_0^*	Initial b*	p^*	The saturation vapor pressure of water
BI	Browning index	$PE \%$	Mean relative percent error
c	Constant	RDSP	Full fat roasted date seeds
C	The measured property value	RH	Relative humidity
C_G	GAB model constant	R^2	Coefficient of determination
C_o	The initial C	R_g	Gas constant
DRDSP	Defatted roasted date seeds	SPSS	Statistical Package for Social Sciences

D_{eff}	Effective moisture diffusivity	SSE	Sum of the squared errors
D_e	Equivalent spherical diameter	T	Temperature
D_o	Diffusion consistency coefficient	T_{abs}	Absolute temperature
E_a	Activation energy	TR	Temperature ratio
E_t	Total specific grinding energy	T_o	Initial product temperature
E_{sc}	Specific crushing energy	T_s	Surrounding temperature
E_s	Specific grinding energy	t	Time
f_e	Shape factor	TPC	Total phenolic contents
H	Hardness	U	Eccentricity of the ellipsoid
H^+	Hydrogen ion concentration	V	The voltage
HR	Hausner ratio	V_g	Pycnometric volume
I	The current	V_{gg}	Geometric volume
CI	Compressibility (Carr) index	V_b	The volume of the powder without tapping
M_p	The mass of the powder	X_{exp}	The experimental moisture content
M_r	Amount of powder remained on the sieve after sieving	X_{pred}	The predicted moisture content
WSI	Water solubility index	$\overline{X_{exp}}$	The average moisture content
w	The weight of the silica gel	y_n	The response value
x	Predictors	ΔE	Total color difference
X	The moisture content at any time	$\propto_e$	Thermal diffusivity
X_c	Critical moisture content	ρ_B	Bulk density
X_o	The initial moisture content	ρ_T	Tapped density
X_e	The equilibrium moisture content	β	Constant coefficients
X_m	The monolayer moisture content	θ	Angle of repose

CHAPTER 1

GENERAL INTRODUCTION AND RESEARCH OBJECTIVES

1.1   Palm date production and industries

The palm date (*Phoenix dactylifera* L.) tree is one of the most cultivated trees which is commonly found in the Afro-Asiatic dry-band, stretches from North Africa to the Middle East. As shown in Figure 1.1, the total harvested area increased by around one million hectares over the period 1980-2016 as well as the world production of palm date fruits increased accordingly to be approximately 8.5 Million tons (FAO, 2016). Date fruit, which can be seen in Figure (1.2a), is a berry consisting of a fibrous, parchment-like endocarp, a fleshy mesocarp and pericarp (skin) which represent around 85 to 90% of the fruit. It has a single seed (Figure (1.2b)), which constitutes between 10 to 15% of the total date fruit weight (Hasnaoui et al., 2012; Hussein et al., 1998). Dates are usually consumed at the Rutab (semi-ripe) and Tamr (fully-ripe) stages, with or without processing. There are many value-added date processing plants such as production of date juice concentrates (spread, syrup and liquid sugar), date honey, date jam, date vinegar, and date pastes for different usage purposes (bakery, confectionary and etc.) (Al-Farsi and Lee, 2008; Hossain et al., 2014). Unfortunately, wastes such as seeds or pits are generated from these processes and usually discarded (Besbes et al., 2004a). Figure (1.3) indicates the wastes resulted from palm date industry starting from harvesting to the consuming (Siddiq and Greiby, 2013). As it can be concluded from Figure (1.3), date seeds represent the major waste of human consumption or processing.

Based on the world production quantity of date fruit, around 846,000 tons of date seeds could be produced annually (i.e. considering 10% of the total fruit weight). This might constitute an environmental problem and require additional handling cost. Earlier, it was confirmed that grinded date seeds were used at cattle feed since the early 1900s (Sirisena et al., 2015). Then lately, the chemical properties of date seeds have been investigated by many researchers including Al-Farsi et al. (2007); Al-Farsi and Lee (2008); Almana and Mahmoud (1994); Bouhlali et al. (2017); Fikry (2016); Ghnimi et al. (2015); Ghnimi et al. (2017); Habib and Ibrahim (2009b); Hamada et al. (2002c); Hussein et al. (1998); Rahman et al. (2007b).These reports aimed to exploit the potential use of date seeds for human consumption, pharmaceutical, nutraceutical and cosmetic applications. So, the lack of utilization of this by-product as food human consumption constitutes a huge economic loss because palm date seeds have high mineral content, dietary fibers, phenolic compounds, antioxidants, and importantly, but devoid of caffeine and are useful as a coffee alternative brew. Hence, it is suggested that this by-product could be a very useful raw material to the food processing industry for developing new products such as powders and brews that contains significant amount of nutrient, especially when the current marketing trend is focusing on natural supplements.
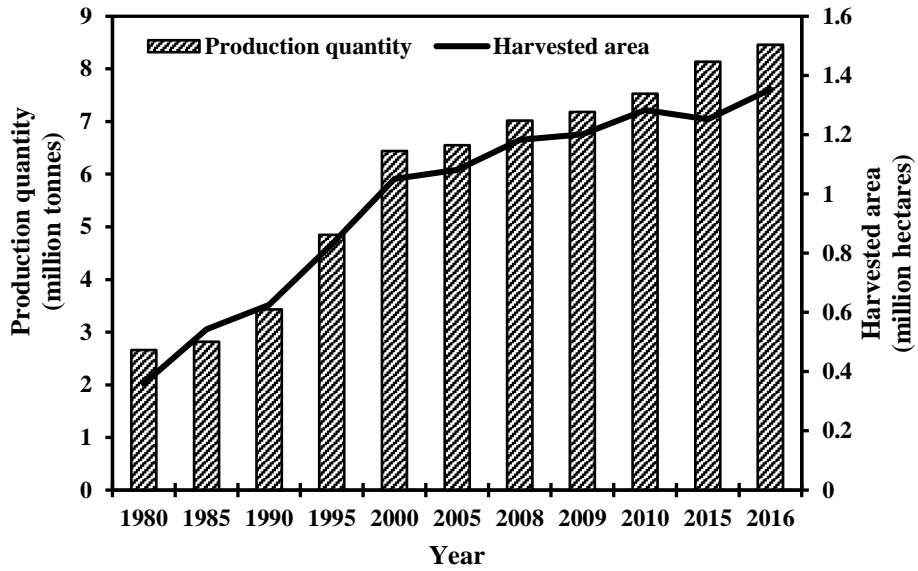
Figure 1.1 :   World production and harvested area of palm date trees in the period 1980-2016 (FAO, 2016)
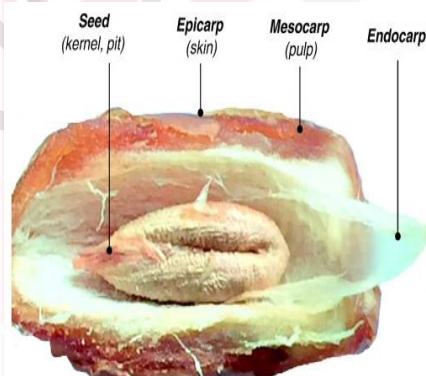
(a)   (b)

Figure 1.2 :   Photos of palm date tree with whole date fruit (a) and the structure of the palm date fruit (b) (Ghnimi et al., 2017)
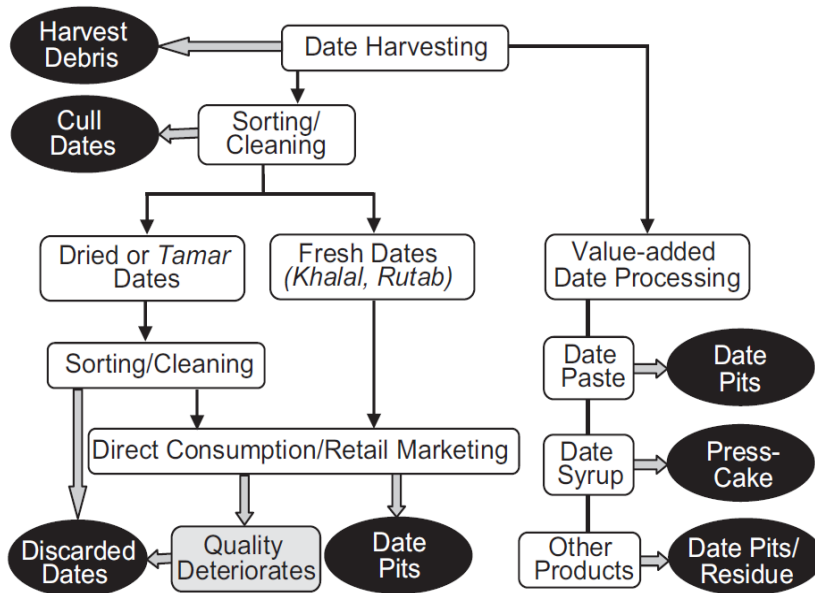
Figure 1.3 : The obtained by-products of palm date industry from harvesting to consuming

1.2   Problem statement

Several prospective uses of palm date seeds have been revealed and reported in the literatures. For instance, palm date seeds were utilized for including in food products such as ground beef (Amany et al., 2012), bakery products (Almana and Mahmoud, 1994; Bouaziz et al., 2010; Platat et al., 2015), chocolate (Bouaziz et al., 2017) and drinks (Abdillah and Andriani, 2012; Ghnimi et al., 2015; Mirghani, 2012; Venkatachalam and Sengottian, 2016). Recent studies trends are to extract oil from palm date seeds for incorporation in different applications such as the production of mayonnaise (Basuny and Al-Marzooq, 2011) and it could be used for cosmetic and pharmaceutical purposes (Afiq et al., 2013; Al-Shahib and Marshall, 2003; Besbes et al., 2004a; Besbes et al., 2004b; Devshony et al., 1992). However, it must be noticed that such applications still at the research and development stages and their commercial manufacture has not yet been hard done.

In view of the fact that people everywhere continue to consume large quantities of coffee daily due to its pleasant flavor, unique aroma, and its caffeine content (20 – 40 %) (Venkatachalam and Sengottian, 2016; Weisse, 2015). Delgado-Andrade and Morales (2005) stated that coffee could minimise the oxidation of human low-density lipoprotein, thereby decreasing the risk of atherosclerosis. Notably, these effects can be attributed to the presence of phenolic compounds and antioxidants (Parliment et al., 2000). However, negative health impacts could occur due to the coffee's high caffeine content which is considered a psychoactive drug.

Several studies have shown that consuming caffeine can cause raised blood pressure, panic attacks, hypertension, gout flare-up, insomnia, indigestion, infertility, inhibition of collagen creation in skin as well as depression and anxiety symptoms (Diego et al., 2008; Faupel et al., 2004; Kristjansson et al., 2013; Venkatachalam and Sengottian, 2016). Alternatively, a brew made from roasted palm date seeds can be safely consumed and served to people who are sensitive to caffeine and prefer to enjoy the characteristic flavor and aroma of caffeine-free coffee without the adverse effects (Al-Farsi et al., 2007; Baliga et al., 2011; El Sheikh et al., 2014; Hamada et al., 2002c; Rock et al., 2009). This brew can be obtained in the powdered form after roasting and grinding of date seeds, which gives extra flavor and aroma. Making style of these brews (coffee-like beverages) is like that of the traditional Turkish coffee, which is described as follows. A certain amount of roasted date seeds powder is mixed with water in a special coffee vessel, stirred occasionally, and let it boil slowly until a foam is occurred on the top, which takes about a few minutes (Sekeroglu et al., 2012). Interestingly, roasted palm date seeds have similar aromatic compounds (alcohols, aldehydes) which exist in Arabica coffee brews (Saafi‑Ben Salah et al., 2012; Somporn et al., 2011).

Unsurprisingly, people in the Arabian region such as the Kingdom of Saudi Arabia (KSA) and the United Arab Emirates (UAE) are already using full fat roasted and powdered date seeds as coffee alternatives and coffee-like brews for a long time (Baliga et al., 2011; Ghnimi et al., 2015; Rahman et al., 2007b), believing that date seed coffee have memory-enhancing properties (Sekeroglu et al., 2012). Furthermore, this powder is currently available in local and international markets and it is a source of choice for people who prefer the decaffeinated coffee (Baliga et al., 2011; Ghnimi et al., 2015).

Although, coffee-like beverage has been prepared from full fat roasted palm date seeds and characterized by (Abdillah and Andriani, 2012; Ghnimi et al., 2015), it was reported that the roasting at high temperature for long time could effect on the properties of beverages prepared from oilseeds leading to undesirable quality and sensory attributes (Maga, 1973).

1.3 Research Gap

To the best of the author`s knowledge, information regarding characterization of whole date seeds during roasting process, the effect of the roasting conditions on the physicochemical and sensory attributes of the powders and the brews produced from roasted full fat and defatted date seed as well as storage stability of the powder and brew prepared from the roasted full fat and defatted seeds and its shelf life correlated to the consumer acceptability have not been studied yet. Hence, the following seven shortcomings would be complemented to characterize the product through the present work**:**

1   The moisture, color, hardness kinetics, total energy requirements for grinding process as affected by roasting conditions (temperature and time) have not been evaluated yet.

2   Previous authors did not study the effect of roasting conditions on the total phenolic content and antioxidants of the brew as affected by the roasting conditions.

3   The previous works have not provided information on the optimum roasting conditions that can be used for coffee-like brew preparations.

4   The previous works did not investigate the kinetic changes during storage conditions of the roasted palm date seeds powder.

5   Storage stability and prediction of the shelf life of the roasted palm date seeds powder and brew at different storage periods and different storage methods were not examined to date.

6   Integration between the quality data and the sensory attributes of the brew from palm date seeds has not been done yet.

7   Comparison between the characteristics of full fat powder and defatted powders was not included in literature review.

Therefore, there is a need for complementation the lack existed in the literature review regarding date seeds brew through production and characterizing of this prospective product and optimize its production conditions according to the consumer preference.

1.4   Scope of The Study

Wastage reduction and developing a new product rich in nutritional elements from palm date seeds are the scope of this study. This study is providing information on the roasting process of the date seeds, grinding energy consumption, oil yield, the physical, flowability properties of the seeds powder, the quality properties of the brew made from date seeds and the shelf life of the powder and brews. These data can help the manufacturers of date seeds powder and brew to design the roasting and storage processes.

1.5   Objectives of The Study

The main objectives of this work are to produce and characterize coffee-like powder and brew from seeds of palm date fruit (*Phoenix dactylifera* L.), specifically;

1- To characterize the whole palm date seeds and to model the kinetic changes in their physical properties, grinding energy consumption and oil yield during the conventional roasting process.

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2- To study the effect of the roasting conditions (Temperature and time) on the physical, flowability and sensory attributes of the coffee-like powder from full fat and defatted palm date seeds.

3- To study the effect of the roasting conditions (Temperature and time) on some quality properties, antioxidants, total phenolic contents and the sensory attributes of coffee-like brew from full fat and defatted palm date seeds.

4- To assess the quality attributes and predict the shelf life of the date seeds powder and brew under different storage conditions (Temperature, relative humidity).

1.6   Structure of Thesis

This thesis was divided into seven chapters.

- Chapter one focuses on five components namely; the general introduction, problem statement, research gap, objectives and structure of the thesis.

- Chapter two provides a detailed literature review on the physical and nutritional properties of date seeds, the application of date seeds in food industry, the importance of roasting process, kinetics of roasting, the changes in the properties of date seeds during roasting and the storage stability of food powders and brew.

- Chapter three presents the general methodologies and statistical data analysis procedures used in this study.

- Chapter four represents the first technical chapter that discusses the characteristics of whole palm date seeds and kinetics modelling of the physical properties' changes during the conventional roasting.

- Chapter five presents information on the effect of roasting conditions on the physical, flowability and sensory attributes of coffee-like powders from palm date seeds.

- Chapter six centres on the effect of roasting conditions on antioxidants, total phenolic contents, some quality and sensory attributes of coffee-like brew from palm date seeds.

- Chapter seven discusses the quality assessment and shelf life prediction of coffee-like powder and brew from palm date seeds under different storage conditions.

- General conclusion and recommendations for future works have been presented in chapter eight.

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LIST OF PUBLICATIONS

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