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Optimizing Microwave-assisted Crude Butter Extraction from Carabao Mango (*Mangifera indica*) Kernels

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ABSTRACT

Carabao mangoes are among the highly produced fruit crops in the Philippines. The processing and consumption of carabao mangoes leave a significant amount of waste seeds. Mango kernel butter extracted from waste seed kernels is a potential additive to cosmetic products or as a cocoa butter substitute. This study determined the pretreatment conditions that produce optimum yield prior to the mechanical extraction of the crude butter. Moreover, this study provided a general sensory evaluation of the finished product. Microwave power (160, 500, and 850 W), microwave exposure time (2.0, 3.5, and 5.0 min), and size levels (1.5, 3.0, and 4.5 mm) were tested for their effects on the yield of the mechanically extracted crude butter in wet basis percentage. The optimization procedures resulted to optimum pretreatment conditions of 160 W, 4.25 min, and 1.5 mm. Size level was the most significant factor in the crude butter yield. Sensory evaluation of the crude butter extracted at optimum pretreatment conditions through acceptance test by a test panel resulted to below neutral scores in visual appearance and odor,

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and above neutral score in texture, indicating the potential of mango butter as a good substitute to cocoa butter in cosmetic products.

Keywords: Microwave assisted crude butter extraction from Carabao mango kernels, optimization

LAYMAN'S ABSTRACT

This paper discusses the microwave-assisted mechanical screw extraction of mango butter from waste carabao mango seed kernels resulting from the processing and consumption of carabao mangoes. Moreover, the physical properties of the extracted butter, as well as its sensory properties as evaluated by the sensory taste panel, were also examined. The study explored the effects of the following on the microwave-assisted screw pressing of mango seed kernels: microwave power (160, 500, and 850 W), exposure time (2.0, 3.5, and 5.0 min), and size levels (1.5, 3.0, and 4.5 mm). The optimum extraction conditions were identified by the RSM methodology as 160 W, 4.25 min, and 1.5 mm. Sensory evaluation of the mango butter determined that it has a high overall acceptability as a substitute to cocoa butter in pharmaceutical, cosmetic, and skin care products.

INTRODUCTION

The distinct proportions of different fatty acids and the presence of their minor components characterize the physical and chemical properties of oils (Institute of Shortening and Edible Oils 2006). Vegetable oils are plant-derived triglycerides used for various purposes, including food, but not limited to fuels, cosmetics, lubrication, medicinal, and other industrial applications. Almost 80% of worldwide vegetable oil consumption is for food and related purposes, while the remaining 20% is used in the industrial and biodiesel sector with recently increasing usage due to energy security concerns (Rosillo-Calle et al. 2009). Mulimani et al. (2012) sought out the potential of deriving significant quantities of biodiesel from vegetable oils, such as using transesterification processes on plants that can be produced by smallholders and in rural areas, such as cotton, Mahua seed oil, and Neem seed oil.

Techniques in extraction are widely adapted for the isolation of bioactive compounds and vegetable oils from raw plant materials (Azadmard-Damirchi et al. 2011). The nature of the applied technique primarily determines the quality (not the inherent) of the resulting yield, and hence, discrepancies among different extraction methods and conditions can exist. Cold pressing, which subjects the plant material to pressure, is the simplest and cheapest method to release crude oil bound within the matrices of the plant material. In terms of quality, cold pressing is best in preserving the natural properties of the extracted oil. Hot pressing involves the introduction of heat and pressure to ease the flow of extracted oil. Most of the time, this method also requires heat treatment and size reduction to increase its efficiency. Residual meal from pressing can serve as source of animal feed and other food components, or can be subjected to solvent extraction, which involves the immersion of the treated or non-treated raw material to a heated solvent, to further dissolve the oil. The resulting solution can be freed from the solvent via distillation to obtain the crude oil. In terms of oil yield, cold pressing is the least efficient, followed by hot pressing, and solvent extraction. Although solvent extraction is superior in production levels, it poses several disadvantages, including plant security issues, high operation costs, emission of volatile and harmful solvents, quality degradation, and the relative complexity of the process. However, the method is practical to use on plant materials with oil contents of less than 20%, such as soybeans (Azadmard-Damirchi et al. 2011).

Mango is a perennial crop native to south Asia, primarily in India and Pakistan, and cultivated in tropical to subtropical areas. *Mangifera indica,* or widely known as Indian mango, is the most cultivated mango (UNCTAD 2003). The characteristic features of the fruit include its oval shape, smooth and leathery rind, fleshy to fibrous pulp, and its stony seed. At least 60 known varieties of mangoes with fruit colors ranging from red to yellow can be identified (Nzikou et al. 2010). The crop is mainly produced for the consumption of its fleshy pulp. Other products derived from the pulp include mango puree, mango juice, dried mangoes, concentrates, frozen mangoes, mango glaze, edible parts, mango in brine, and mango preserves (BOI 2011).

The Philippines produces the world's sweetest and less fibrous varieties of mangoes—an example of which is the Carabao mango from the provinces of Cebu and Guimaras. The *Pico* variety also offers the sour-sweet flavor comparable to the *Carabao* when ripe. While being more fibrous and smaller than the *Carabao*, *Pico* mangoes are still widely known because of their availability and cheaper price in the market.

The high demand for mangoes consequently promotes the increase in production through efforts in developing technologies and maximization of land and resource usage. Although a seasonal crop, several means and techniques are available for application, in order to produce the fruit in any season and to continuously sustain the demand for the fruit at lesser price inconsistencies all year round. The annual production capacity of the Philippines is approximately one million metric tons in average over the last few years. Moreover, mango is the third top agricultural commodity exported by the country, next to coconut and banana (BAS 2010).

Since the major use of mango is the consumption of the sour-sweet pulp being eaten raw or for different kinds of food preparation and processes, the seed and the skin, which comprise 30% to 50% of the weight depending on the variety, are typically treated as waste and immediately disposed without further utilization (Philippine Mango Seedling Farm Corp. [date unknown]). The vast amount of wastes generated from the fruit after consumption can be estimated to provide a potential and sustainable raw material for further processing (Kaphueakngam et al. 2009). In recent years, efforts have been made to find ways to utilize these wastes with acceptable economic and commercial feasibility. Peels and seeds can become sources of oils and other organic compounds for food, cosmetic, and alternative medicinal applications (Ashough and Gadallah 2011). Other research finds ways to use these by-products as sources of biodiesel for internal combustion engines. The fuel properties of mango kernels were analyzed in the study by Agbede et al. (2010). The low iodine value of the mango kernel oil limits its application as non-drying oil for lubrication purposes. However, the observed fuel properties still possess the potential for biofuel production, and further research on the improvement of these properties can make them into suitable biofuels.

The mango kernel is rich in carbohydrates, fats, proteins, and minerals (Anand and Maini 1997; Kittiphoom 2011). The kernel can also be used for extraction of edible oils and as a supplement to wheat flour (Kittiphoom 2011; Tandon and Kalra 1989). Ashoush and Gadallah (2011) reported that the potential antioxidant properties and high phenolic content of mango kernels and peel powders can help improve the nutritional quality and antioxidant properties of foods, such as biscuits. Among these promising applications, mango kernel oil has been bringing commercial benefits, especially in India, where mangoes are among the widely produced fruits. The oil from the kernel of the mango seed is typically light yellow in color with semi-solid texture and thick consistency at room temperature. It is also known as mango butter because it exhibits nearly solid consistency at room temperature and melts in contact with a warmer surface, such as the human skin. It is a good alternative natural ingredient or additive in various body and skin care products, such as lotions, sun protection creams, moisturizer, and soaps (Pioneer Enterprise [date unknown]). Interests on natural oils, such as from seeds, for cosmetic application are steadily increasing. In mango-based products like soaps and lotions, 3% to 12% of mango kernel oil is added by the manufacturers (BOI 2011).

In some countries like India, mango kernel oil is sometimes used as a substitute for shea and cocoa butter. Only a few countries can cultivate cocoa on a commercial scale, that consequently leads to an unstable supply of cocoa butter, the primary ingredient in making chocolates. Cocoa butter is one of the most expensive vegetable oils, prompting many industries to seek out for alternative vegetable fats. Cocoa butter equivalents or CBEs are compounds that can be substituted to cocoa butter because of their almost similar fatty acid composition, causing minimal softening or hardening effects on the product where they are incorporated. Only six vegetable fats can be substituted to cocoa butter (EU Regulations 2003), and mango kernel oil is one of them, as it contains mango seed almond fat (MAF) that is high in stearic acid content. When mango kernel oil is blended with palm oil, their resulting mixture contains palmitic, stearic, and oleic acids, similar to that of the cocoa butter. The blend of 80% kernel oil—20% palm oil has a melting behavior and slip melting point that is closest to that exhibited by cocoa butter. The mango kernel butter itself can also be used as edible oil (Kaphueakngam et al. 2009). Mango kernel butter reduces skin cell degeneration, protects the skin against ultraviolet radiation from the sun, and restores skin elasticity. The stearic acid content of mango kernel butter makes it suitable as a preservative in different cosmetic products, and the high unsaponifiable matter content (4.58%) guarantees the use of such oils in the cosmetic industry. It is also being developed for medicinal applications and for food items, such as confectionery and blended oils (Nzikou et al. 2009). The kernel is about 75% of the seed weight, which is about 50% of the waste that can be derived from an average mango fruit. Mango seed kernels contain about 10% to 13% oil, depending on the variety and the climatic conditions where it is grown.

There are two intertwined extraction methods for mango kernel butter extraction: mechanical and solvent extraction. Mechanical methods, such as hydraulic pressing and screw pressing, are generally cheaper in terms of acquisition and operation costs than solvent extraction. However, the screw pressing procedure generally produces smaller oil yields compared to solvent extraction upon use of the same amount of raw material. Some processing plants in India employ both methods, wherein the by-product cake from the mechanical extraction process is further subjected to solvent extraction (Mahale and Goswami-Giri 2011). The industry studies by BOI (2011) presented a brief overview of the potential of establishing processing units, that do not exist at the present, for mango kernel oil production in the Philippines. The abundance of discarded seeds from processed and raw fresh mangoes is a potential cheap supply for a processing plant. Patterned after the processing schemes in India, the process can be manual, or semi-mechanized and mechanized with imported designs and fabricated units, such as driers, decorticators, mills, solvent extraction unit, finishing section equipment, and packaging units. With existing laws and government policies, as well as continuing research, the industry can be established feasibly for continuous operation in the country.

Up to date, there are 28 potential suppliers of mango seed kernels from different regions in the country, and 36 potential local manufacturers of mango kernel oil for cosmetic applications, including several multinational companies, such as Procter and Gamble, Unilever, and Sanofi-Aventis. Oil extraction technology for mango kernels is still under development in the country. However, several international companies involved in the extraction, manufacture, and distribution of mango seed oil, such as the American entities Jeen International Corporation and Protamine Chemicals Inc., already identified markets for mango oil in the Philippines.

Microwave (MW) pretreatment of the raw material for oil extraction, especially pulps and seeds, recently became a suitable pretreatment process for increasing the yield of the extracted oil. Moreno et al. (2003) revealed that MW pretreatment in avocados undergoing solvent extraction resulted to an increase in oil extraction efficiency from 54% in Soxhlet-hexane extraction to 97% in MW-assisted Soxhlet-hexane extraction. Uquiche et al. (2008) conducted MW pretreatment that significantly modified the microstructure of the substrate tissue, and helped in the increase of mass transfer of oils and other lipids of Chilean hazelnuts which will undergo mechanical extraction. Additionally, MW treatment further ruptured the cell wall of the treated sample, making cell permeability and oil movement throughout the cell walls more efficient. They also claim the MW pretreatment brought positive effects on the oil quality by providing higher stability to oxidative deterioration. Their data also showed that the time of exposure to microwave radiation has a more profound and significant effect ($p<0.05$) on the extraction yield compared to the potency of microwave power. Similarly, Momeny et al. (2012) studied the significant effects of MW pretreatment on the oil yield of an unidentified variety of mango kernels. The use of 300-W MW power and 180-sec exposure time resulted to an increase in oil yield to as much as 8.9% wet basis, in contrast to the 5.65% oil yield of the untreated sample, which was finely ground prior to extraction to increase the surface area in contact with the solvent. After treatment at 450 W for 180 sec, the sample was burnt, indicating that microwave heating of finely ground samples may exceed tolerance limit for extraction and analysis.

Using the conventional methods of solvent extraction in mango kernels, the BOI (2011) projected a percentage of annual profit of investment of about 27% if an oil extraction and processing facility for mango kernels will be built in the Philippines. The introduction of an optimal MW pretreatment to the local supply of mango kernels should theoretically increase the yield returned per investment and consequently increase the profit of the investor. Today, no study that focuses on the optimization of MW pretreatment for crude butter extraction from the mango kernels of the Philippines' locally produced mangoes and conducts a sensory analysis on crude butter from *Carabao* mango kernel exists.

Physical and Chemical Properties of Mango Kernel Butter and its Applications

Fresh mango butter is a thick and solid fat with the following properties: pale yellow in color at room temperature (Mahale and Goswami-Giri 2011), slight fatty odor and a bland taste, thick consistency at room temperature but readily melts at temperature of around 31 to 39 ^{o}C (Pioneer Enterprise 2006), and specific gravity of around 0.885 to 0.925, making it less dense than water. Kittiphoom (2011) cited that mango kernel oil has stearic (27%-38%) and palmitic (6.5%-10%) acids as its primary saturated fatty acid constituents, and oleic (42.6-48.3%) and linoleic acids (2.4-5.3%) as its major unsaturated fatty acids. Such fatty acid proportions make them suitable as cocoa butter substitute and for applications in other industries without further neutralization of the oil. In addition, mango kernel oil is more stable than other vegetable oils that are also rich in unsaturated fatty acids. The applications of mango kernel oil can extend into incorporation with widely produced vegetable oils, stearin manufacturing, and the confectionery and soap industries.

Mechanical Extraction and the Microwave Pretreatment Process

Mechanical extraction is accomplished by exerting compressive force or pressure on the raw material to release the crude oil. This method is widely employed on plant materials, such as coconut and other vegetables, which provide edible oil. There are three major steps in mechanical screw extraction method, namely pretreatment, pressing, and oil clarification. Pretreatment, which involves the application of heat and/or size reduction, is usually applied to increase the yield.

In screw pressing, the pretreated raw material is fed to the hopper of the screw extruder and then to the screw conveyor where it is pressed. The freshly pressed oil contains several other components, such as water, fibers, dust, and other impurities from the raw material, which contribute to the unclear appearance of the oil. Clarification, or the process of letting the oil stand for some time to form a purer oil layer, can remove these impurities. Moreover, the oil can be cooked to remove the moisture and separate the oil. However, since the process extracts oil only through applied shear pressure (and temperature for an expeller with a heater), the recovery of the raw material from the matrix and the characteristics of its natural perforations, primarily the material cell wall, are severely limited. To address this problem, one possible and practical approach is the adoption of microwave pretreatment or microwave-assisted extraction to improve the extracting capability of an oil extraction method by making perforations larger (Mandal et al. 2007).

In contrast to solvent extraction methods executed alone, microwave or MW pretreatment prior to solvent extraction can bring several advantages in an oil extraction venture, such as improved yield and quality, direct extraction capability, lower energy consumption, faster processing time, and reduced solvent levels (Azadmard-Damirchi et al. 2011). Microwaves are non-ionizing electromagnetic waves with frequency between 300 MHz to 300 GHz, which are between the X-ray and infrared rays in the electromagnetic spectrum. Microwaves can be created from electric and magnetic fields oscillating at perpendicular fields. In MW pretreatment, heating occurs in a localized manner, resulting to minimal loss of heat to the surroundings.

In studies involving microwave-assisted Soxhlet apparatus, extraction time can be as low as thirty minutes compared to the conventional solvent extraction system accomplished in long hours. The practical application of microwave energy in pretreatment lies in the frequency of 2450 MHz, the common frequency found in domestic microwave ovens. The use of frequencies lesser or greater than 2450 MHz will result in the non-heating of the treated material. Any plant material, such as seeds, can contain minute amounts of moisture in their interstices at atmospheric pressures. The microwave heats and evaporates the locked moisture from the plant material, giving immense pressure to the cell walls that bind the moisture, eventually causing their swelling and bursting. This allows the oil to move freely out of the matrices of the plant material, making it easier for the surrounding solvent during extraction to dissolve it. There are several factors that significantly affect the performance of microwaves for oil extraction, including microwave irradiation power, exposure time, nature and quantity feed material, matrix characteristics of the material to be extracted, and the temperature of the treatment

(Letellier and Budzinski 1999; Zuloaga et al. 1999; Huie 2002; Wang and Weller 2006; Mandal et al. 2007).

Other Effects of Microwave Pretreatment

Aside from the oil extraction yield, MW pretreatment also affects the major and minor components, the physicochemical properties, and the overall quality of the oil produced. Oleic and linoleic acids present in mango kernel oil are more dramatically affected by microwave pretreatment than palmitic and stearic acid. Antioxidative phenolic content and tocopherol levels significantly increased after microwave pretreatment prior to extraction, primarily due to the damage and rupture of the plant cell membrane. In the MW pretreatment of sunflower oil, an increase in the exposure time caused the decrease in the refractive index, unsaponifiable matter, and the iodine values; and the increase in the saponification value and the free fatty acid content. Increased heating times, temperature, and power settings can result in a higher degree of oil deterioration. However, microwave pretreatment of olive, sunflower, and rapeseed oils led to increased stability because of the increase in released antioxidants, such as tocopherols (Yoshida et al. 1995; Veldsink et al. 1999; Anjum et al. 2006; Azadmard-Damirchi et al. 2011).

Optimization by Response Surface Regression

Response surface regression is an analysis used to fit a polynomial regression model with cross product terms of variables, which may be raised up to the third power, and to calculate the minimum or maximum value of the surface (NCSS 2014). Many industrial experiments that seek out optimization solutions from a given set of factor variable employ response surface methodology. If a factor is measured at three or more values as determined by the experiment, a quadratic response surface can be estimated with the use of least squares regression. If the surface generated has a hill or valley-like shape, an optimal value can be determined and predicted. However, if the shape becomes more complicated or if the offered optimum solution is far from the range of experimentation, the shape of the surface can still be used for an analysis to create recommendations as to how new experiments should be performed (SAS Institute 2014).

Statistical software packages like STATISTICA 10, SAS 8.0, andDesign Expert 7.0 can analyze and process optimization multiple response processes by response surface methodology via desirability functions. Desirability functions approach results by obtaining an optimum set of quantitative values from multiple responses, by assigning a score to a set of responses, and by selecting the factor setting that shows the relatively maximum or optimal score. It is one of the widely used methods for determining a set of conditions from tested and actual values from experimentation expected to yield a desired maximum output (NIST/SEMATECH 2012). Desirability functions for each response can produce a desirability profile. The overall desirability can be obtained by determining the geometric mean of the desirability for each response. This is not only limited to maximization, but it can also find the set of responses that can achieve a minimum response score. Statistical computer softwares can be used to compute for desirability functions and can provide a desirability profile for optimization studies (SAS Institute 2014). Crude butter fat yield (Y_1) is expressed as the following by response surface regression (RSM): $Y_1 = f(X_{1,} X_{2,} X_3)$. The true relationship between Y_1 and $X_{1,} X_2$, X_3 can be complicated, and in most cases, unknown. However, second-degree quadratic polynomial equations can represent them in the range of interest (Annadurai and Sheeja 1998).

Sensory Evaluation and Acceptance Test

IFT (1975) defined sensory evaluation as "a scientific discipline used to evoke, measure, analyze, and interpret reactions to those characteristics of food and materials as they are perceived by the senses of sight, smell, taste, touch, and hearing". Since food properties relevant to consumer perception studies, such as flavor, odor, and texture, are hardly quantified by product marketing, the use of the senses of a group of actual people with minimal bias as much as possible is applied to quantify properties in a predetermined scaling.

The three broad methods of sensory evaluation include descriptive, discrimination, and acceptance. Descriptive test (Cernohous [date unknown]) is the sensory method that objectively identifies attributes and qualities of a food or a product using human subjects (panelists) specifically trained for this purpose. Discrimination test is designed to measure the likelihood that two products are perceptibly different. Acceptance test or affective test measures actual user or end user response in terms of likeliness or acceptability (Gengler [date unknown]). Acceptance tests, which are commonly used for new products for release in the market, do not require trained or professional panelists like in the other two methods, but necessitate the participation of larger number of people that represents the consumer body. The 9-point Hedonic scaling quantifies human responses. Other factors that can affect the outcome of sensory evaluation include health, interest, availability and communication skill of the panelists, and the subjective nature or bias responses of the panelists (Gengler [date unknown]).

Significance of the Study

The Philippines contributes an average of one million tons (MT) to the 35-MT annual worldwide production (FAO 2009). Local production of fresh mangoes is tallied at 825423 metric tons, with 20114 metric ton of exports including processed forms and 4,200 tons of oil extracts (BAS 2010). Estimates in India reveal that 30000 tons of oil extracts come from their total annual harvest of 7 MT (BOI 2011). The figures in actual yield of mango kernel butter by solvent extraction change across the varieties, sizes, maturity, pretreatments, and environmental growth conditions of the mangoes. According to existing literature, the pure oil content of mango oil from the kernel is about 815% dry basis.

Objectives of the Study

This study explored the microwave-assisted mechanical screw extraction and optimized crude butter extraction from microwave pretreated *Carabao* mango kernels. Specifically, this study: (1) evaluated the effects of size reduction, microwave power, and exposure time parameters for crude butter extraction during microwave-assisted mechanical screw extraction; (2) performed sensory evaluation through acceptance tests on mango kernel butter extracted at optimal conditions; and (3) determined the optimal pretreatment conditions prior to microwave-assisted mechanical screw extraction.

MATERIALS AND METHOD

Materials and Equipment

The materials and equipment used in the study consisted of the following: mango kernels from ripe Carabao variety, microwave-safe bowls, kitchen grater (diameters of 1.5 mm, 3.0 mm, and 4.5 mm)[5], Promac™ Microwave Oven (Model SVC-005038), scissors, plastic bags, electronic balance, funnel, large flat pan, domestic stove, small-scale motor-powered expeller, glass rod, evaporating dish, and a pair of pliers.

Experimental Design

The experiment used three independent variables, namely microwave power in watts (W), size in millimeters (mm), and microwave exposure time in minutes (min). A three-level, three factorial Box and *Behnken* experimental design was formulated using the essential regression software. The design is an independent quadratic design, which does not contain embedded factorial or fractional factorial design. In addition, treatment combinations are at the midpoints of edges of the process space and at the center (NIST/SEMATECH 2012). This study used 15 basic experimental runs depicted in Table 1.

Table 1. Independent and dependent parameters of the study

Independent Parameter	Symbol Code			Dependent Parameter
	-1	0	1	
Microwave Power Level (W)	160	500	850	Oil yield, wet basis
Microwave Exposure Time (min)	2	3.5	5	and dry basis
Size Reduction Level (mm)	1.5	3	4.5	

Fresh mango seed samples weighing 10 grams were used in each run. The microwave power settings used were 160, 500, and 850 W; size levels were 1.5, 3.0, and 4.5 mm; and the exposure time settings were 2.0, 3.5, and 5.0 min. Table 1 shows the matrix of the experimental runs. Table 2 shows the combinations of the independent and dependent parameters for the pretreatment conditions of mango kernel butter extraction.

Preparation and Dry Weight Determination of Samples

Ripe mangoes of the same sizes and batch were purchased from the Angono Public Market, Philippines. The fruits were manually depulped to extract the stones. The fresh stones were immediately washed and decorticated using a pair of pliers to determine the suitability of the kernel inside for processing. As shown in Figure 1, the collected kernels were then washed and stored in the vegetable compartment of a domestic refrigerator for preservation prior to pretreatment. The dry weights were determined using 10 grams of fresh sample in a pre-weighed tin container placed in an air oven for 12 hours. The weight of the tin can and the dried sample was measured, and from this, the weight of the tin can alone was deducted to determine the actual dry weight of the sample that will be used for the dry basis computation of the crude butter yield. Moisture content of the fresh mango kernel sample was computed using the equation $\%MC = \frac{W_1 - W_2}{W_1}$ 100%, where MC is the actual moisture content (%), W_1 is the initial weight of the fresh sample (10 g), and W_2 is the final weight of the oven-dried sample (g).

Table 2. Matrix of independent and dependent parameters

Run #	$X_{1, MICROWAVE}$ Power (W)	$X_{2, MICROWAVE}$ Exposure Time (Mins)	$X_{3, SIZE}$ Reduction Level (Mm)	Y_1 Crude Butter Yield %[6]
1	160	2	3	
2	850	2	3	
3	160	5	3	
4	850	5	3	
5	160	3.5	1.5	
6	850	3.5	1.5	
7	160	3.5	4.5	
8	850	3.5	4.5	
9	500	2	1.5	
10	500	5	1.5	
11	500	2	4.5	
12	500	5	4.5	
13	500	3.5	3	
14	500	3.5	3	
15	500	3.5	3	

[6] Based on wet basis

Figure 1. Hot Water bath set-up for the evaporation of moisture.

Microwave Pretreatment and Crude Butter Extraction

The mango kernels were reduced in sizes using kitchen graters of different sizes as stated in the experimental design. Approximately ten grams of the reduced fresh sample was placed in a bowl, in such a way that the fresh sample beneath is equally distributed to the floor of the bowl. A total of 30 mL of boiling water was poured in the bowl prior to the microwave pretreatment to properly distribute the effects of the microwave treatment during different exposure times and to eliminate the error of heat distribution that will only be used in boiling the water. Hot water is also a safe solvent for the better dissolution of crude butter from the kernel, especially when combined with mechanical extraction. Crude butter was then placed inside the domestic microwave Promac™ Model SVC-005038 with a five-power level adjustable knob which provide specific power outputs convertible to watts. The mixture was cooked inside the microwave for a specific exposure time. The cooked mixture was then expelled from its solid constituents in the small-scale expeller. The water-butter mixture from each experimental run was placed in plastic bags as temporary containers for the mixture, in order to minimize the losses in transferring prior to evaporating the water from the mixture.

Evaporation of Water

The water-butter mixture from the plastic container was transferred into a re-weighed evaporating dish. A large flat pan was filled with ample amount of distilled water and placed onto a kitchen stove. The evaporating dish was then placed in the center of the pan as in a hot water bath as shown in Figure 1. This process of moisture evaporation is much safer than direct heating and prevents excessive physical and chemical damage to the crude butter. Crude butter contains high amounts of carbohydrates that are very susceptible to damage by high temperature. The water in the pan was consistently replaced until all free-flowing moisture inside the evaporating dish disappeared. The weighing of the evaporating dish with the dried butter inside followed. The dry weights of the crude butter used in subsequent computations were calculated as follows: $DBW = WD_2 — WD_1$, where DBW is the crude butter dry weight (g), WD_2 is the weight of the evaporating dish and butter after the hot water bath (g), and WD_1 is the weight of the evaporating dish (g).

Crude Butter Yield

Using the oven-dry weight of the fresh sample, the crude butter yield in dry basis was computed using $CBY_{DB} = \frac{DBW}{W_2} * 100\%$, where CBY_{DB} is the crude butter yield in dry basis (%), DBW is the dry weight of the crude butter (g), and W_2 is the final weight of the oven-dried sample (g). On the other hand, using the fresh sample weight prior to pretreatment, the crude butter yield in wet basis was computed using $CBY_{WB} = \frac{DBW}{W_1} * 100\%$ (5), where CBY_{WB} is the crude butter yield in wet basis (%), DBW is dry weight of the crude butter (g), and W_1 is the initial weight of the fresh sample (g).

Statistical Analysis

Results from the experiment were analyzed using the SAS 9.0 software to determine the effects of the independent parameters to the crude butter yield in wet basis. Contour and surface plots were also generated from the same program. Optimization analysis was performed using the STATISTICA 10 software under polynomial regression to determine the pretreatment conditions that will theoretically result to an optimum yield. A desirability profile was also generated using the analysis. Trial runs using the determined optimum pretreatment conditions from the polynomial regression analysis were also conducted. The predicted and actual values were compared and analyzed to determine if there exist significant differences between the predicted and actual values by Duncan Multiple Range Test (DMRT) at 95% level of significance.

Sensory Evaluation of the Crude Butter Extracted

The crude butter sample in the optimal pretreatment condition derived from the statistical analysis underwent sensory evaluation through acceptance test. Ten men and ten women college students aged 17 to 24 years participated in the tests. To reduce the subjective responses of the participants, the test was performed in areas with adequate lighting and free from other odors that can be present in the area. The criteria for the sensory analysis were overall visual appearance, odor, and texture. They were also oriented that the sample will be used as a cosmetic additive. The 9-point Hedonic scale was used for the acceptance test of the mango butter by the sensory panel (Table 3). DMRT analysis of the results from the evaluation determined the significant difference in the mean scores among panelists. The crude butter was evaluated according to its visual appearance, odor, and texture when applied to the palm of the hand.

Table 3. 9-Point Hedonic scale (Gengler [date unknown])

Dislike	Dislike	Dislike	Dislike	Neither	Like	Like	Like	Like
Extremely	Very much	Moderately	Slightly	Like nor dislike	Slightly	Moderately	Very much	Extremely

RESULTS AND DISCUSSION

Effects of the Independent Parameters on Crude Butter Yield

Table 4 illustrates the crude mango butter extracted from mango kernels at different experimental runs as influenced by selected independent parameters at various levels. The extracted crude butter ranged from 1.2$\%_{wb}$ to 26.4$\%_{wb}$ or 2.26$\%_{d}$ to 48.85$\%_{d}$, which is way below the yields of 60% to 90% when using the Soxhlet-hexane extraction method. Crude mango kernel butter is a combination of

Table 4. Crude butter yield (%) from the experimental runs

Trial	Dependent Parameters			Final Crude Butter Dry Weight (grams)	Extracted Crude Butter (% wet basis)	Extracted Crude Butter (@ dry basis)
	Power Level (W), X1	Exposure Time (min), X2	Size Reduction Level (mm), X3			
1	160	2	3	0.65	6.5	12.03
2	850	2	3	1.5218	15.3	28.18
3	160	5	3	0.811	8.1	15.02
4	850	5	3	0.765	7.7	14.17
5	160	3.5	1.5	2.638	26.4	48.85
6	850	3.5	1.5	2.072	20.1	38.37
7	160	3.5	4.5	1.1369	11.4	21.05
8	850	3.5	4.5	0.41	4.1	7.59
9	500	2	1.5	1.2782	12.8	23.67
10	500	5	1.5	2.275	22.8	42.13
11	500	2	4.5	0.122	1.2	2.26
12	500	5	4.5	0.359	3.6	6.65
13	500	3.5	3	0.512	5.1	9.48
14	500	3.5	3	0.591	5.9	10.94
15	500	3.5	3	0.481	4.8	8.91

oil, proteins, and carbohydrates, such as starch. The effectiveness of the extraction relies on the suitable combinations of the pretreatment procedure and the mechanical extraction. Mechanical extraction processes alone are inefficient because of the inherent stickiness or the sap-like texture of the fresh butter. The fresh butter adheres to the solid cake or to the inside walls of the screw conveyor in a screw-press expeller. Application of direct heat to the expeller will usually burn the rich carbohydrate content of the kernel and will result in a stickier sap-like substance in the cake with no yield at all of low quality butter. The butter extracted from the mango kernel is readily biodegradable. If left in average room temperature, it may spoil in the course of a week. Therefore, the storage of extracted butter in refrigerated spaces is highly advisable to prolong its shelf life in its fresh form.

Contrary to the reports in literature about the density of the butter, the extracted butter from the experimental runs had densities of over 1 g/cc. This is primarily due to the rich carbohydrate and protein content of the crude butter, which had not gone any refining process after its extraction. Figure 2 shows the crude butter yield in both wet and dry basesin percent. As seen in the graph, the trends of the wet and dry bases do not differ, because of the same oven dry weight used in the computation of the dry basis per run. Run 5 employing the parameters 160 W, 3.5-min exposure time, and 1.5-mm size reduction level produced the highest percent yield among the trials. Run 5 was followed by Run 10 at 500 W, 5-min exposure time, and 1.5-mm size reduction level. The third highest in oil yield is Run 6 at

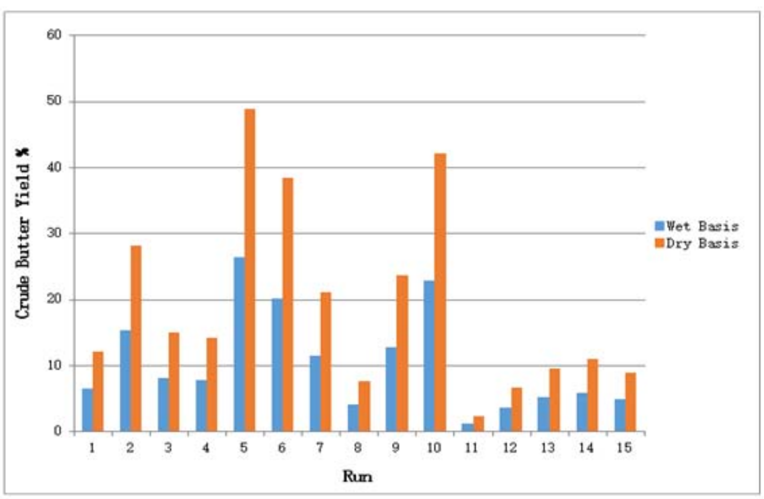


Figure 2. Crude butter yield (%) profile from mango kernels in wet and dry basis by run.

850 W, 3.5-min exposure time, and 1.5-mm size reduction level. Smaller particle sizes of the fresh sample prior to microwave pretreatment increased the butter yields. Size reduction of the sample increased the yield by incrementing the surface area affected by the applied pretreatment process. The increased surface area also helped the mechanical extraction by easing the flow of the water-butter mixture in the interstices.

The lowest yield occurred at Run 11 at 500 W, 2-min exposure time, and 4.5-mm size reduction level. Such result could be attributed to the small surface area in contact with the hot water and the microwave power. Since the fresh sample is in hot water during the microwave exposure, only the outermost area of the sample is affected by the heating process, which removes the crude butter from its solid constituents with the help of water acting as a solvent. The solubility of butter to water is significantly affected by temperature: the warmer the water, the more soluble the butter will be. Figure 6 shows the settling of the butter in cool temperatures of around 4^{o}C. Increased microwave power can also bring additional effects to the physical and chemical properties of the crude butter. Table 5 shows the brief sensory evaluation of the trials as affected by microwave power.

After the pretreatment, the mango butter, which was exposed to high microwave power, released strong characteristic odor, as opposed to the odorless description of mango butter in the literature and product catalogs for mango butter. This odor can bring undesirable properties to the finished product especially when used in cosmetic applications normally applied near olfactory organs. High microwave power in combination with long exposure times can also result in the darkening of the crude butter from pale yellow to dark yellow. The increased surface area of the samples also makes them more susceptible to the changes brought by increased microwave power. Figures 3 to 5 show the contour plots of the effects of the combination of two independent parameters on the crude butter yield.

Table 5. Color and smell evaluation* of trials based on microwave power level

Microwave Level (watts)	Sensory Evaluation
160	Almost odorless, with slight yellowing of the crude butter.
500	With a characteristic odor with slight yellowing of the crude butter.
850	Strong characteristic odors with darkening of the crude butter.

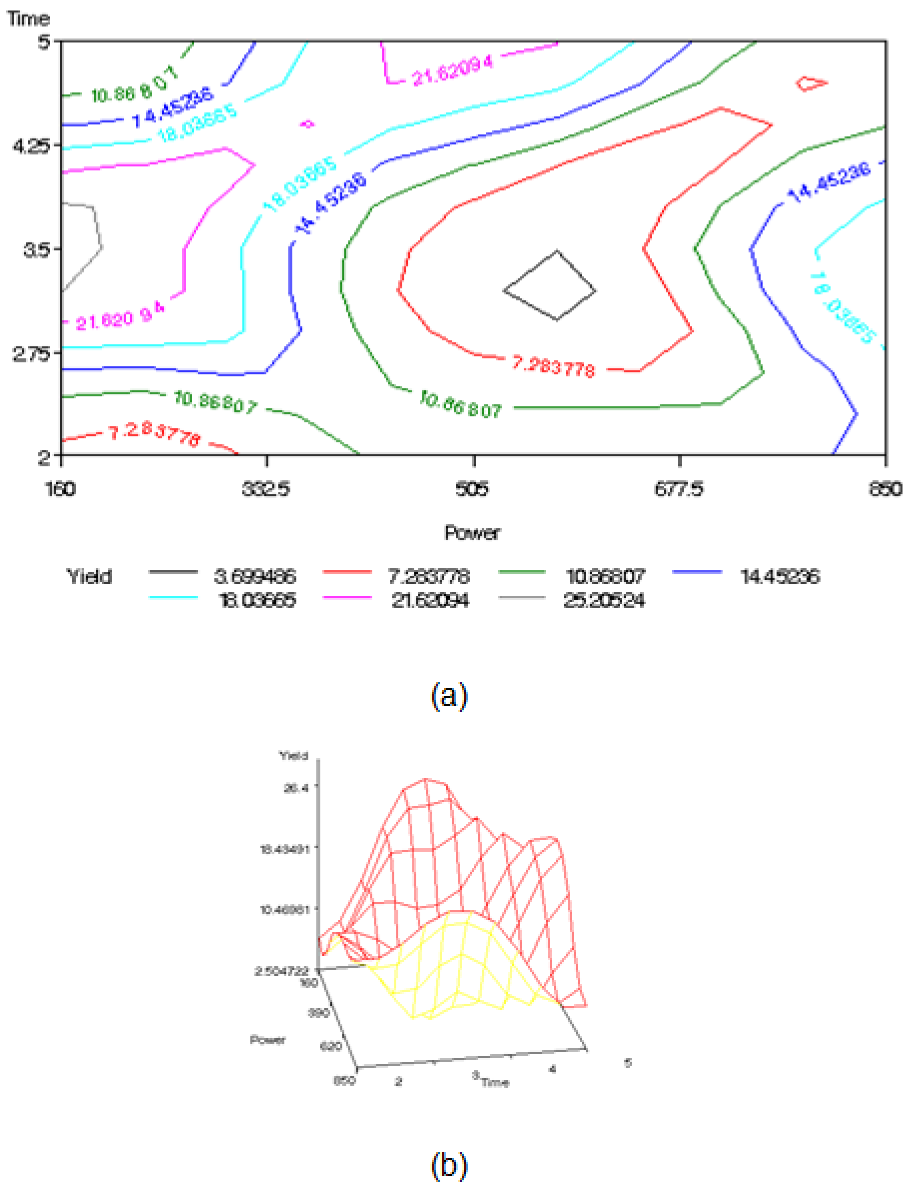


Figure 3. Contour (a) and surface (b) plots of crude butter yield (%) by microwave power (W) and exposure time (min).

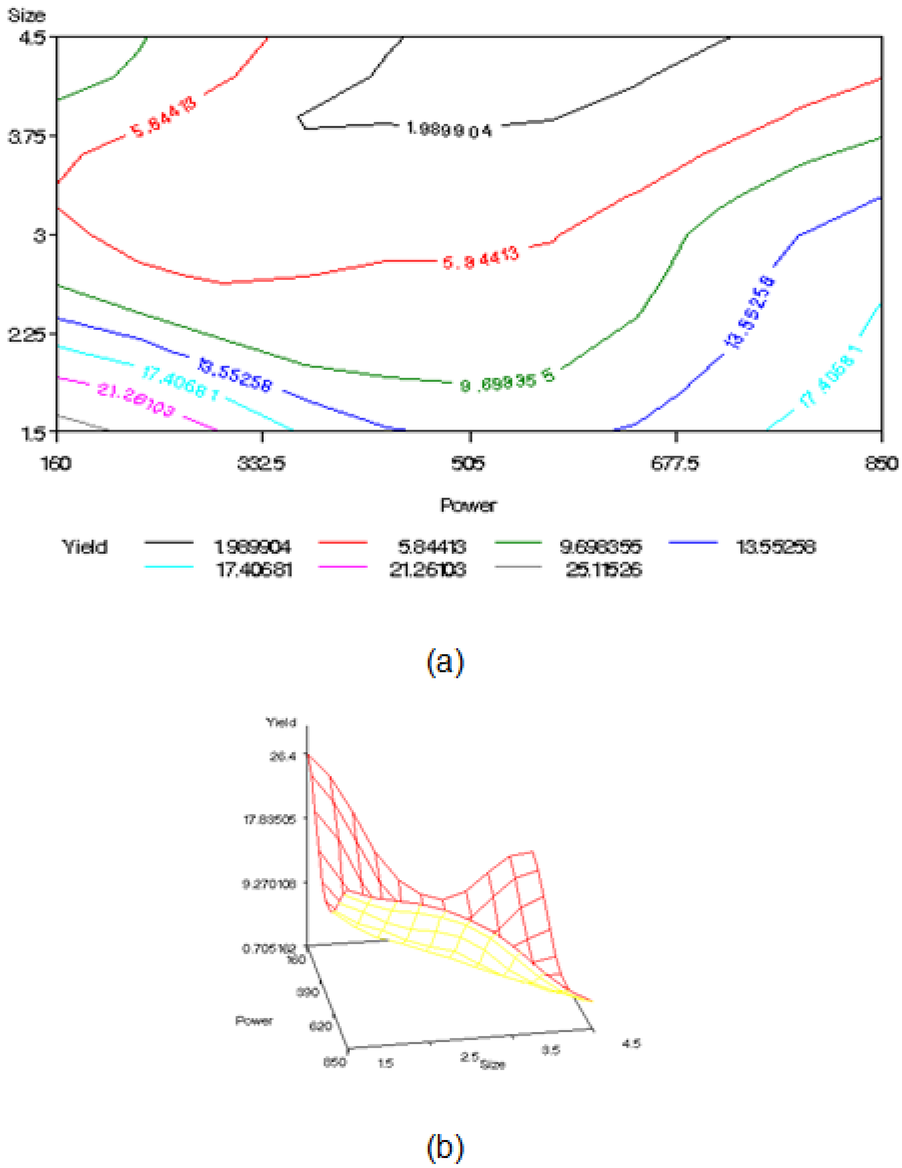
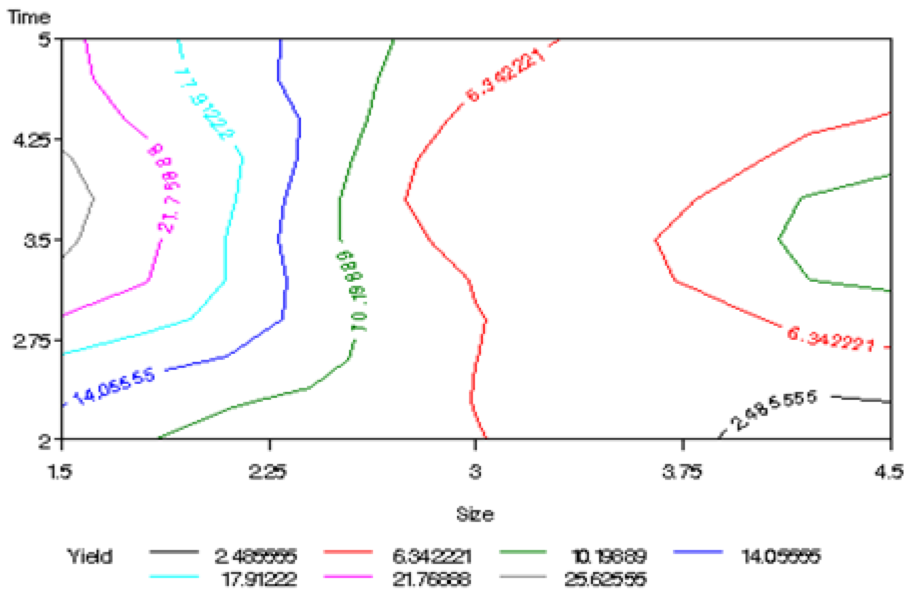
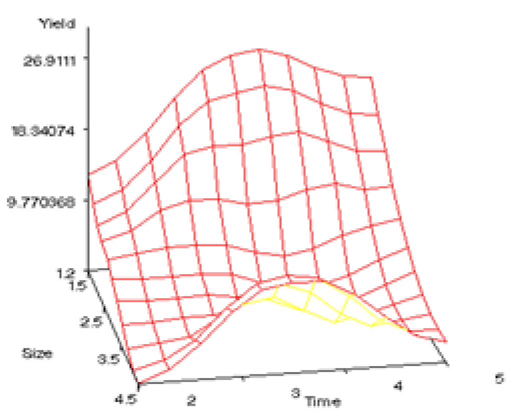


Figure 4. Contour (a) and surface (b) plots of crude butter yield (%) by microwave power (W) and size reduction level (mm).



(a)



(b)

Figure 5. Contour (a) and surface (b) plots of crude butter yield (%) by size reduction level (mm) and exposure time (min).

Figure 6. Settling of butter-water mixture prior to water bath. Crude butter solidified and settled at the bottom of the mixture at low temperature.

Analysis of Variance (ANOVA) of the Independent Parameters Affecting Crude Butter Yield

The RSRegression procedure of SAS 8.0 performed the ANOVA of the independent parameters to determine their significance to the crude butter yield from mango seeds. Table 6 shows the results of the ANOVA tests on the independent parameters. The most significant parameter is the size of the sample, followed by the microwave power at 95% confidence interval. The size of the sample also reflects the degree in which microwave power can penetrate the sample, as well as the degree of contact in the hot water acting as the solvent. Microwave power has a direct effect on the temperature of pretreatment, wherein higher temperatures ease the flow of the crude butter from its solid constituents. Microwave exposure time has the least significance among the parameters. This might have been caused by the soaking of the sample in hot water prior to the actual microwave pretreatment that caused initial dissolution of the crude butter to the water. Table 7 shows the significance of the response surface regression models for the crude butter yield in wet basis, as well as the R^2 value, and coefficient of variation.

Table 6. ANOVA of the independent parameters on the response variable (Yield % wet basis) at 95% confidence level

Parameter	DF	Sum of Squares	Mean Square	F Value	Pr > F
Microwave Power	4	1.108216	0.277054	1.40	0.3537ns*
Exposure Time	4	0.429478	0.107370	0.54	0.7125ns*
Sample Size	4	6.157077	1.539269	7.80	0.0224**

* Not significant

** Highly significant at 95% level of confidence; significantly affects crude butter yield based on DMRT at 95% confidence interval

Table 7. ANOVA showing the significance of the response surface regression model for the crude butter yield in wet basis.

Source	DF	Sum of Squares
Linear	3	4.944845ns*
Quadratic	3	1.941378ns
Cross Product	3	0.363926**
Total Model	9	7.250149ns
Lack of Fit	3	0.980424ns
Pure Error	2	0.006467
Total Error	5	0.986891
r^2	0.8802	
Coefficient of Variation	42.6365	

* Not significant

** Significant at 90% confidence level;

The R^2 value of this regression model is 0.88, whereas the coefficient of variation is 42.6365. These values explain the degree of the precision and reliability of the experiment. The high variation among the data can be attributed to the relatively small sample size used in the experiment, which is significantly susceptible to losses incurred. It is therefore recommended to use a larger sample size for further studies, in order to equalize the effects of microwave power and exposure time to larger sample sizes.

Optimization through Polynomial Regression via Desirability Functions

Figures 4 to 6 show the plot of the predicted desirability values based on the minimum and maximum values of the study's independent parameters. From this data, the predicted values of crude butter yield in wet basis per derived factor level were determined. As analyzed using the General Linear Model - Polynomial Regression in the STATISTICA 10 software, factor levels from the respective independent parameters with the highest desirability values were selected and considered as the optimal pretreatment conditions for crude butter extraction (Table 8). As highlighted, the optimal conditions are as follows: 160-Wmicrowave power, 4.25-minute exposure time, and size reduction level of 1.5 mm diameter grater size. As expected from the initial results, the finest sizes of the fresh samples were determined as optimal and the predicted values indicated a decrease in the increase rate in percent yield as the size of the sample increases (Figure 7). The trend for the effect of microwave power shows high desirability values in both minimum and maximum values. The trend for time shows relatively little changes in the predicted yields along its range. Equation (6) shows the polynomial equation generated from the analysis, with crude butter yield in wet basis as the response variable.

$$Y_1 = 48.4450 - 0.0424X_1 + 2.5037X_2^2 - 0.2815X_2^2 - 19.7278X_3 + 2.4296X_3^2$$

where:

Y_1 = Crude Butter Yield in Percent Wet Basis (%)

X_1 = Microwave Power (W)

X_2 = Exposure Time (min)

X_3 = Size Reduction Level (mm)

Table 8. Regression coefficients for the optimization of crude butter yield in % wet basis

Effect	Coefficient
Intercept	48.4450
Power	-0.0424
Power^2	0.0000
Time	2.5037
Time^2	-0.2815
Size	-19.7278
Size^2	2.4296

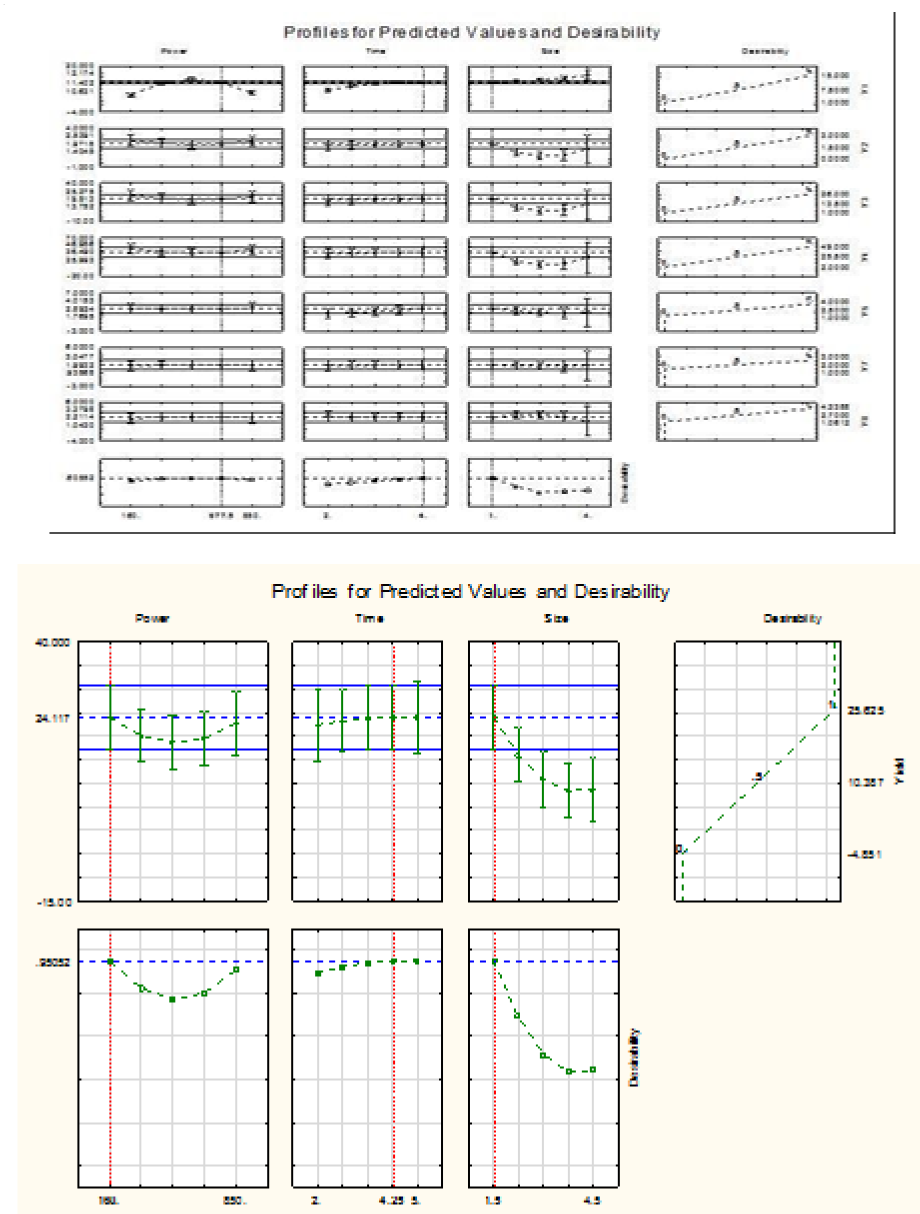


Figure 7. Desirability plot for the predicted values and desirability values of the dependent parameters for the extraction of crude butter.

Verification of the Optimal Pretreatment Condition

The optimum pretreatment conditions for the crude butter extraction determined by the polynomial regression analysis shown in Table 9 were tested by replicating the experiment in these conditions for three trials. The results of the verification test results are shown in Table 10.

Table 9. Predicted responses at each level of each factor while holding all other factors constant at their current settings

	Factor-Level	Predicted-Yield	Desirability-Value
Power	160.0000	24.11667	0.950520
Power	332.5000	20.20885	0.822293
Power	505.0000	18.68958	0.772442
Power	677.5000	19.55885	0.800965
Power	850.0000	22.81667	0.907864
Time	2.000000	22.44167	0.895559
Time	2.750000	23.31667	0.924270
Time	3.500000	23.87500	0.942591
Time	4.250000	24.11667	0.950520
Time	5.000000	24.04167	0.948059
Size	1.500000	24.11667	0.950520
Size	2.250000	16.15417	0.689247
Size	3.000000	10.92500	0.517663
Size	3.750000	8.42917	0.435768
Size	4.500000	8.66667	0.443561

Table 10. Comparison of the predicted and actual values using the predetermined optimal conditions

	Trial 1	Trial 2	Trial 3	Average
Observed Values %	23.54a[11]	21.96c[11]	22.15b[*]	**22.55b[*]**
Predicted Value %		**24.11667a**		
Percent Difference %		**6.5**		

[*] Values in a row followed by the same letters are not significantly different from each other using DMRT at 95% level

All verification values at optimum conditions were lower than the predicted values at optimum conditions, thereby requiring changes in the chosen independent parameters tested. This suggests that other combinations of factors may be more appropriate in producing optimum yields from microwave-assisted screw pressing of mango butter from seed kernels. Lower values of factor levels may be examined in the future to lower the cost of production. Lowering the power, exposure time, and particle size will reduce the total production costs. Although hexane extraction by Soxhlet method produces higher yields, the method is not recommended, in terms of the costs, product safety, and environmental considerations. Problems on health among workers will rise and the setup of a hexane waste treatment will be necessary if hexane extraction is employed in extracting crude butter from mango seeds.

The average percent difference between the actual and observed values is 6.5%. This error is primarily attributed to the observed losses incurred in the mechanical extraction of the butter, as well as in the transportation of the water-butter mixture from one container to another during laboratory analysis. The butter extracted from the verification runs had a yellowish white color with no significant odor. It is also vulnerable to spoilage when stored in average room conditions in less than a week.

Acceptance Test of the Crude Butter in Optimum Pretreatment Condition

Twenty untrained analysts evaluated the acceptance test of the crude butter processed from the optimal pretreatment condition using the 9-point Hedonic Scale. Table 11 shows the results of the experiment. Figure 8 depicts the radar graph of the sensory properties of the crude mango butter as graded by the sensory panel members. Converting the score from the Hedonic scale into numerical values, the significance between the mean values of the three sensory criteria were tested using the DMRT at 95% confidence interval on SAS 9.0. Results from the analysis show significant differences between the scores per criterion as perceived by the individual evaluators. The sensory characteristics of crude mango butter undergoing a consumer level acceptance test is generally subjective in nature and based on the individual preferences of each evaluator (Figure 8). However, comparison of the mean values between the three criteria shows that the mean texture score (above neutral) is significantly different from both visual appearance and odor mean scores (below neutral). Based on the Hedonic Scaling, two of the important criteria, namely visual appearance and odor, for the evaluation of a potential cosmetic product obtaineda below neutral average perception. Visual scores ranged from 4 to 12, odor ranged from 3 to 20, and texture scores ranged from 2 to 19. The values indicate that odor and texture are the primary considerations of the panelists in expressing the olfactory properties of the mango butter. The observations suggest that further processing or refining of crude mango butter kernel is recommended to improve these sensory properties if it is intended to be marketed as a fresh product.

Table 11. Acceptance test results of the crude Butter in the optimal pretreatment cond ition as determined by the test panelists.

Participant #	Visual Appearance A	Odor B	Texture C
1	Dislike Slightly	Neutral	Like Slightly
2	Dislike Slightly	Dislike Slightly	Like Slightly
3	Like Slightly	Dislike Slightly	Like Slightly
4	Like Slightly	Neutral	Like Moderately
5	Like Slightly	Neutral	Like Slightly
6	Like Moderately	Dislike Moderately	Dislike Slightly
7	Dislike Slightly	Dislike Slightly	Dislike Slightly
8	Dislike Slightly	Neutral	Like Slightly
9	Dislike Slightly	Neutral	Neutral
10	Like Slightly	Neutral	Like Slightly
11	Dislike Slightly	Dislike Slightly	Like Slightly
12	Like Slightly	Neutral	Like Moderately
13	Like Slightly	Like Slightly	Like Moderately
14	Dislike Slightly	Dislike Slightly	Like Slightly
15	Dislike Slightly	Dislike Slightly	Neutral
16	Dislike Slightly	Neutral	Neutral
17	Dislike moderately	Dislike Slightly	Like Slightly
18	Dislike Slightly	Like Slightly	Like Slightly
19	Neutral	Like Slightly	Like Moderately
20	Dislike Moderately	Neutral	Like Slightly

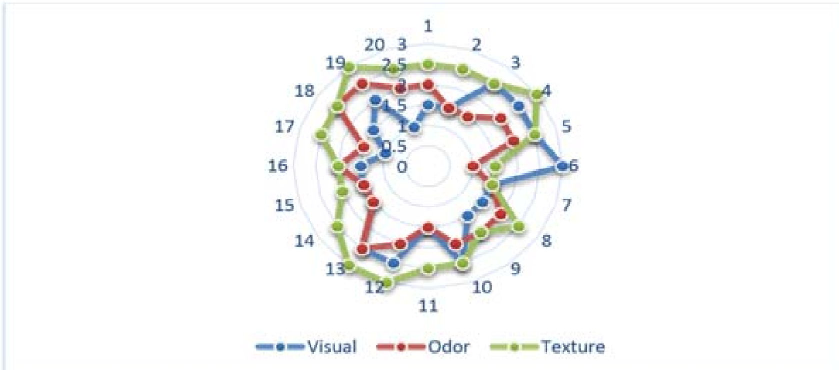


Figure 8. Radar graph of the criteria scores per category

SUMMARY AND CONCLUSION

The optimal pretreatment conditions for crude mango butter microwave-assisted extraction was studied using a Box and Behnken design of experiments with the following serving as the independent parameters of the experimental design: microwave power in W (160, 500, and 850), microwave exposure time in min (2.0, 3.5, and 5.0), and size reduction level through grating of the fresh sample in mm (1.5, 3, and 4.5). The pretreatment comprised a microwave set-up with varying power levels, different sizes of kitchen graters, and a mechanical extraction unit for the actual extraction process. Results show that varying levels of microwave power affect the physical and chemical characteristics of the crude butter as observed in the differences in color and the odor it emitted. The ANOVA of the data identified sample size and microwave power as the significant factors in the crude butter extraction and calculated an R^2 value of 0.88. The variation in the data can be attributed to the small sample size used in the experiment, which is susceptible to significant losses along the execution of the trials. The optimum extraction conditions determined by polynomial regression via desirability analysis were 160 W, 4.25-min exposure time and 1.5-mm size reduction level. The predicted and average actual yield values had a 6.5% difference based on the verification runs and had no significant difference based on the DMRT at 95% confidence interval. Evaluation through acceptability test of the crude mango butter processed with the optimum pretreatment conditions was conducted using the DMRT. The scores for all criteria indicated significant differences. The mean score for texture was significantly different and below than the visual appearance and odor mean scores, indicating the acceptability and feasibility of mango butter for inclusion in cosmetic products.

RECOMMENDATIONS

In view of the obtained results, future studies must consider the following: (1) increasing the sample sizes to 1 kg achieve lesser variations among the response variables, (2) consider the use of other local mango varieties and feasibility of extracting butter from the their waste seed kernel, (3) performing descriptive sensory evaluation on the crude mango butter and other derived products using trained panelists, and (4) performing an extensive physical and chemical analysis on the extracted butter from *Carabao* mangoes to further understand the effects brought by microwave pretreatment on the crude mango butter and the other relevant properties if it is to be used as a cosmetic ingredient or additive, or as a cocoa butter substitute.

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