# **CZECH UNIVERSITY OF LIFE SCIENCES PRAGUE**



## **Faculty of Engineering**



## Department of Mechanical Engineering

## **DIPLOMA THESIS ASSIGNMENT**

Mayrina Andriani

Description of mechanical and relaxation behaviours of medium desiccated coconut under uniaxial compression using statistical response surface methodology

Supervisor

doc. Ing. Abraham Kabutey, Ph.D.

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# **DIPLOMA THESIS ASSIGNMENT**

Mayrina Andriani, BE (mechanical)

Agricultural Engineering

Thesis title

Description of mechanical and relaxation behaviours of medium desiccated coconut under uniaxial compression using statistical response surface methodology

## **Objectives of thesis**

The objectives of the Master Thesis are to:

(i) determine the percentage oil content of medium desiccated coconut using the Soxhlet extraction procedure.

(ii) describe the force-deformation curves of medium desiccated coconut under varying processing factors.

(iii) determine the response surface regression models for estimating the mass of oil,

oil yield, oil expression efficiency, and energy of medium desiccated coconut dependent on the processing factors.

(iv) validate the optimal processing factors for estimating the mass of oil, oil yield,

oil expression efficiency, and energy of medium desiccated coconut.

(v) describe the spectral curves and/or determine the chemical properties of medium desiccated coconut under pre-treatment temperatures and heating times.

## Methodology

The experiment will be conducted at the laboratory of the Mechanical Department of the Faculty of Engineering. The universal compression testing machine (ZDM 50, Czech Republic) of a load capacity of 500 kN will be used for the compression tests of medium desiccated coconut by applying the Box-Behnken Design (Response Surface Methodology) of the experiment. The compression speed will be set at 4 mm/min. The initial pressing height of the medium desiccated coconut will be measured at 100 mm using the vessel diameter of 60 mm with a plunger. The processing factors: forces, pre-treatment temperatures, and heating times at three levels each based on the Box-Behnken Design will be evaluated. The moisture content and oil content of the medium desiccated coconut will be determined using conventional methods. The data will be analyzed statistically using Statistica software (version 13). Code for compiling the Master Thesis

- 1. Introduction
- 1.1 Research problem statement
- 1.2. Objectives
- 2. Literature review
- 2.1 A general overview of the coconut tree crop
- 2.1.1 Origin, classification and production
- 2.1.2 Fruit structure
- 2.1.3 Intercropping in coconut farming
- 2.1.4 Fertilizers requirement for coconut farming
- 2.1.5. Common pests and diseases in coconut farming and management
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- 2.3 Mathematical models describing bulk oilseeds under axial loading
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- 3. Materials and Methods
- 4. Results and Discussion
- 5. Conclusions and Recommendations
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- 7. Appendixes

## The proposed extent of the thesis

60-70

## Keywords

Oil-bearing crops, linear pressing, processing factors, oil extraction, mathematical models.

## **Recommended information sources**

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#### ABSTRACT

The study investigated the mechanical and relaxation behaviours of medium desiccated coconut under uniaxial compression by using statistical response surface methodology with Box-Behnken design (BDD) to determine the optimal conditions for extracting the oil. Three independent factors were examined namely the force (kN), heating temperature (°C) and heating time (min) with each variable set at three levels. The BBD generated 17 experiments with twelve combinations of factors and five replicate at the centre point. The universal compression testing machine (MPTest 5.050) of a maximum load of 5 kN was used together with a pressing vessel of diameter 30 mm and a plunger to record the compression data (dependencies between force and deformation as well as the relaxation force and time) on the medium desiccated coconut sample measured at an initial pressing height of 100 mm (sample volume of 7.07 x  $10^{-5}$  m<sup>3</sup>). The compression experiments were done at a speed of 4 mm/min. The parameters calculated were the mass of oil (g), oil yield (%), oil expression efficiency (%) and energy (J). The data were statistically analyzed using the response surface regression technique at a 5% significance level to obtain the regression models and the optimized processing conditions. Based on the BBD results, the maximum mass of oil of 7.81 g, oil yield of 27.02 % and oil expression efficiency of 43.67 % were recorded for the combined factors of force: 4.8 kN, temperature: 40 °C and heating time: 45 min. The corresponding energy was 82.32 J. The P values of the lack of fit of the regression models were non-significant (P > 0.05) indicating the reliability of the models. The optimized combined factors were force: 4.8 kN, temperature: 40 °C and heating time: 30 min. The percentage error between the predicted and experimental validated values ranged between 0.04 and 3.12 % confirming the combination of the optimized factors. The oil output at a lower temperature of 40 °C increased at a relaxation time of 23 minutes but it decreased at a higher temperature of 80 °C.

**KEYWORDS**: Oil bearing crops, linear pressing, processing factors, oil extraction, mathematical models

#### DECLARATION

I hereby declare that I have independently done the Master Thesis entitled **Description of mechanical and relaxation behaviours of medium desiccated coconut under uniaxial compression using statistical response surface methodology**, and all texts are original. However, literature sources used have been acknowledged by providing the list of references according to citation rules of the Faculty of Engineering.

In Prague

Date:....

Mayrina Andriani

Signature: .....

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#### **INTRODUCTION**

The coconut tree crop is a unique plant, it has a high level of consistency and continuity in flowering and fruit production, every month and every year for a decade and has the capability of free nature from severe seasonal or episodic constraints on growth and enables the coconut to grow in some part of the island or areas without any human attendance or management (Nair 2010). Coconut is classified as an important versatile tropical tree that can give life to people by various benefits including the useful part of the whole coconut tree by the seed and the fruit (Jerard et al. 2008). Mostly grown in Asia and the Pacific region, coconut tree crops are becoming one of the lavish sources of nature in the world. Widely known as the "tree of life" among the community in different regions, the coconut tree provides a massive variety of products that can help the local economy of the communities. In particular, the coconut industry holds an important role in the exporting movement in some Southeast Asia countries, that's why the revitalization of the coconut industry needs to be done for the country that has coconut as an important role in their economy. It is very challenging for the coconut industry to compete amid global competition in the 21<sup>st</sup> century, the coconut industry needs to meet the standards of safety and quality in all aspects of production the processing as the international trade guide standards (Pham 2016).

Alouw and Wulandari (2020) reported an overview of the development of the coconut industry in Indonesia, which took concern about all the aspects that can affect the economic welfare of farmers. Indonesia and the Philippines have been contributed to almost 67% of crude coconut oil (CNO) export to the global market nowadays, which is now facing the fluctuating issues of the low price of CNO while the needs are increasing rapidly. There are 6.6 million farmers who depend on coconut and coconut-based products, mostly copra and CNO as the main source of living. The technology and innovations for smallholder farmers will be the right things to develop in the future to produce a good and high-quality product of coconut to solve the existing problems to ensure the sustainability of the coconut sector. Agu, et al. (2020) also mentioned the global concern against the safety of fossil fuels as well as the environmental concern about non – biodegradability sources, which leads to more interest in the source of plant-based oils. Edibles oils are preferable plant-based is rich in fatty acid and other lipophilic antioxidants for our body. coconut kernel (Cocos nucifera) many used in India and The Asia Pacific, which are copra oil (CO), Virgin coconut

oil (VCO), and refined, bleached and, deodorized (RBD) oil, divided by their preparation, their composition, and variation of their biological effects.

Narayanankutty, et al. (2018) found that there is no proof of the differences in the fatty acid profiles of CO, VCO, and RBD oils, however, they found out that the polyphenol contents were high in VCO, probably caused by less harsh treatment on the preparation stages. Nowadays the most common methods used to gain oil are mechanical pressing and solvent extraction. The mechanical pressing procedure can provide a view of the important advantages, this requires intensive study of the pressing procedure and specifically to do more study of the factors that can be affecting the efficiency of the press, characteristics of the recovered oil from the oleaginous material. There are many types of pressing machines that are usually used in the mechanical pressing method, mostly known are hydraulic press machines that are classified as batch mechanical pressing machines meanwhile for continuous pressing machines there is a screw press machine. Three main methods for oil extraction by using possible variations for the extraction procedure which are batch hydraulic pressing, pressing mechanical continues (expeller), and solvent extraction, hydraulic pressing is the most common method used by the small-scale industry because of the low-cost maintenance. The application of the hydraulic pressing generally by putting the oleaginous material in a cylindrical cage perforated laterally and will have results that consist of axial compaction and radial flow (Ionescu, et al. 2016). On the industrial scale, seed oil recovery can be reached by a sequential process of mechanical expression and hexane extraction. In general, mechanical expression becoming the most reliable technique to produce good quality virgin oil (Bogaert, et al. 2018).

In recent research, Kabutey, et all. (2021), studied pumpkin seed oil extraction by using the uniaxial loading process, by using the response surfaces methodology (RSM) as the statistical tool to analyze the effect of the independent variables or the processing factors' responses. The uniaxial compression process is used to foresee the mechanical and relaxation behaviours of the pumpkin seed. Process able to help for describing the mechanical behaviours in force-deformation curve characteristic forms, oil yield, oil expression efficiency, and energy demand. Adequate knowledge can help to maximize the mechanical screw press, especially in small-scale production.

#### **1.1.** Research problem statement

Now, the demand for coconut products is reviving following the increasing world population growth and global market needs, particularly for virgin coconut oil products and the young fresh coconut fruit, which is recognized as a healthier food option. To support the changing lifestyle for choosing coconut based as a healthier substitute product, the productivity and average national production need to be lifted especially in a country that has a big number of resources in coconut farming with less knowledge of recent technology. The Indonesian researchers have also emphasized the importance of the use of quality planting materials, replanting the unproductive palms, micro propagation of elite types of coconut, promotion of good agricultural practices and management of pests and diseases to provide high-value crops plantation to meet people's request on healthy oil, food and beverages (Alow and Wulandari, 2020). Various efforts can be made to optimize agricultural yields from coconut. The post-harvesting process is also important to consider for continuous development. Many ways can be used to produce products by considering the process being carried out to find the best results by optimizing the process and paying attention to its impact on the surrounding environment. This research will focus on coconut oil production from medium desiccated coconut under uniaxial compression by applying the Box-Behnken Design of the experiment (Response Surface Methodology). A recent study about uniaxial compression of pumpkin seeds oil extraction under the uniaxial loading method intended to predict the mechanical oil expression process by reducing the time-consuming nature of the classical experimental approach as well as minimizing the cost. The response surface methodology (RSM) has been identified as an efficient statistical tool for analyzing the effects of several independent variables or processing factors on the responses (Kabutey, et al. 2021).

## 1.2. Objectives

The objectives of the Master Thesis are to:

- (i) determine the percentage oil content of medium desiccated coconut using Soxhlet extraction procedure.
- (ii) describe the force-deformation curves of medium desiccated coconut under varying processing factors.
- (iii) determine the response surface regression models for estimating the mass of the oil, oil yield, oil expression efficiency and energy of medium desiccated coconut dependent on the processing factors.
- (iv) validate the optimal processing factors for estimating the mass of oil, oil yield, oil expression efficiency and energy of medium desiccated coconut.
- (v) describe the spectral curves and/or to determine the chemical properties of medium desiccated coconut under pre-treatment temperatures and heating times.

#### LITERATURE REVIEW

#### 2.1. A general overview of the coconut tree crop

The coconut palm is grown in more than 98 countries in the world with an estimated area of over 12 million hectares and ranked in seventh widest oil crop cultivated around the world in 2013, in the same year coconut production averaged 62.45 million tons with Indonesia, Philippines, and India as the most producer. The versatility can be found in the coconut tree where almost every part of the tree can be utilized. In forms of productivity, Brazil become the most productive at 12.12 tonnes per hectare more than double the size of the average world productivity at 5.17 tons (Arulandoo et al. 2017). *Cocos nucifera L*. is the source of edible components such as coconut water, virgin coconut oil, copra, and coconut milk, as well as natural fibre (husk) and activated charcoal (nutshell) (Ignatio and Miguel 2021).

#### 2.1.1. Origin, classification and production

The origin of the coconut palm is still arguable, research on fossil and molecular data aimed the indications of the probability of coconut palm tree commenced and by the time dispersed from Southeast Asia. Coconut can be found around the world in many tropical and subtropical areas mostly in the coastal ecosystem, mostly the low-lying atolls of the pacific. The Coconut palm is categorized as a family of Arecaceae and subfamily of Coccoidea which cover around 27 genera and 600 species and is specified as a monocotyledon with the support from the adventitious root systems and unbranched trunk with a height that can reach over than 30m. Under ideal conditions, a palm can produce up to 17 fronds per year. Coconut palms are also divided into two broad categories, Dwarfs and Tall varieties. Dwarf varieties have a shorter economic life span averaging 30–40 years with average nut yields ranging from 100 to 151 nuts per palm per year. Unlike the Tall varieties, dwarfs are capable of self-pollination and thus evolved to be more homozygous palms (Arulandoo et al. 2017).

#### 2.1.2. Fruit structure



Figure 1. Structure of coconut fruit (Nwankwojike et al. 2012).

The nut varies from 147 to196 mm in diameter and 245 to 294 mm long. Three sunken holes of softer tissue called "eyes" are at one end of the nut. Inside the shell is a thin, white, fleshy layer, about 12.25 mm thick at maturity, known as the "coconut meat". The interior of the nut is hollow and partially filled with a watery liquid called "coconut milk". The meat is soft and jelly-like when immature and becomes firm at maturity. Coconut milk is abundant in unripe fruits, but it is gradually absorbed as ripening proceeds. The shell is used for fuel purposes, shell gasifier is as an alternate source of heat energy (Nwankwojike et al. 2012).

#### 2.1.3. Intercropping in coconut farming

The rationale for intercropping is that other crops can profitably be grown between or under coconuts. Adequate supplies of water and labour are the two major inputs needed for the success of the system (Liyanage et al. 1984). Inter/mixed crops can be selected based on the climatic requirement of the inter/mixed crop, irrigation facilities and soil type. The canopy size, age and spacing of the coconut are also to be considered. Market suitability should be taken into consideration before selecting an intercrop. For coconut trees below seven years of age, any suitable annual crop for soil type and climatic condition may be raised as intercrops up to five years after planting depending upon the canopy coverage. Groundnut, sesamum, sunflower, tapioca, turmeric and banana can be grown and better to avoid crops like paddy and sugarcane.

For coconut trees aged seven to twenty years, green manure crops and fodder crops also can be grown. The coconut tree above twenty years of age has to be adjusted based on the sunlight transmission of above 50% inside the canopy and the crops can be grown depending on the soil and climatic suitability (Agritech portal 2014).

Dauzat and Eroy (1997) from the Davao Research Centre of the Philippines Coconut Authority performed intercropping experiments on corn and mung beans at different densities under coconut stands. Yields obtained in intercropping experiments are linear functions of the photosynthetically active radiation measured under the trees. Results exhibit a nearly linear relationship between light transmission and tree density. Pruning also appears as an effective means of increasing the light permeability of coconut stands. These results are interpreted in terms of corn and mung bean yields by combining radiative simulations and field intercropping experiments.

Mapa (2012), evaluated the effect of intercropping coconut lands on soil water retention, water availability, porosity and their relations to organic matter contents. The results revealed that water holding capacity and available water increased significantly at both depths (0–20 and 20–40 cm), which can lead to better soil quality and stability related to higher organic matter and root activity in intercropping treatments. Intercropping increased the total porosity and macro-porosity. The coconut yields of intercropped treatments increased by 5% to 34% over monoculture. The intercropped did not show significantly higher results of organic matter, but the trend showed an increasing effect. The clay content of this soil was as low as 8% in the top soil and increased to 17% in the subsoil, which already gave a significant effect on soil aggregation and water retention.

#### 2.1.4. Fertilizers requirement for coconut farming

Malhotra et al. (2017), suggested the innate soil constraints should be ameliorated to improve the productivity of the coconut (Cocos nucifera L.) as an important perennial plantation crop. The red, lateritic and sandy coconut soils are suffering from soil acidity, low CEC and poor nutrient reserve. The potassium supply in the acid soils is poor, coconut being a huge feeder of potassium needs a constant supply of potassium. The soil management strategies needed to improve as the resolution for having the coconut yield in an eco-friendly way. The study of biomass recycling under coconut-based cropping systems gave promising results in achieving the nutrient supply with the availability

of biomass recycling through vermicompost. Optimizing the biomass inside the farm like the husk can help to improve the potassium in the palms by organic farming cycle, as on the microbiology side showed that biomass recycling improved the beneficial microorganism in the rhizosphere for the nutrient supplies. Solangi et al. (2016), studied the potential benefits of balanced fertilization using crop residues, organic manures and green manuring in maintaining the levels of soil organic matter have been increasingly emphasized. Some soil amendments have value as fertilizer and as systemic pesticides. Inorganic amendments include Urea for nitrogen (N); Di-ammonium phosphate (DAP) for phosphorus (P) and Muriate of potash for potassium (K). These (NPK) work well in combination with Neem seed powder (NSP) and Gliricidia sepium leaves (GSL) at different levels.

#### 2.1.5. Common pests and diseases in coconut farming and management

The coconut palm is also potentially vulnerable to acute insect and microbial bio-hazards specific to diverse locations worldwide, such as the Lethal Yellowing phytoplasma, Phytophthora bud-rot fungus and the Brontispa leaf beetle (Bila et al. 2015). Aceria guerreronis is the only species of *eriophyid* mite that is a serious pest of coconuts. Populations of the mite develop in the meristematic zone of the fruits, which is covered by the perianth. Feeding of mites in this zone causes physical damage so that as newly formed tissue expands, the surface becomes necrotic and suberized. Uneven growth results in the distortion and stunting of the coconut, leading to reductions in crop yield. Although being a serious pest currently, far worse losses would occur if Aceria guerreronis spreads to Asia and Oceania, where the coconut is of much greater importance to daily life. There are promising lines of research that could lead to the management of the pest using resistant cultivars, agronomic manipulation, and biological control (Moore and Howard 1996). The coconut mite, Aceria guerreronis Keifer (Prostigmata: Eriophyidae), is a major pest in several coconut production areas worldwide. Information on the region of origin and sources of recent introductions of this mite are important aspects to guide the evaluation of biological control agents and the adoption of quarantine measures. Geometric morphometric analyses allow us to quantify and visualize shape variation, eliminating the effect of size, position and orientation, within and among samples of organisms. Considerable morphometric variability was observed between American populations, which in turn were distinct from African and Asian populations. Navia (2006) reported the recent discovery of Bogia coconut syndrome in Papua New Guinea

(PNG) is the first report of a lethal yellowing disease (LYD) in Oceania. Numerous outbreaks of LYDs of coconut have been recorded in the Caribbean and Africa since the late nineteenth century. There is no economically viable treatment for LYD and management is hampered by the fact that vectors have been positively identified in very few cases. (Gurr et al. 2016).

#### 2.1.6. Harvesting and storage of coconut fruits

Siriphanich et al. (2011) mentioned in their study about mature coconut, which has a moisture content of the fresh kernel of nearly 90% at around 8 months, which decreased to 50-60% at maturity. The fat content at harvest is around 36–41% fresh weight, and after drying, to about 6% moisture, the fat content increases to 60-74%. Free fatty acid content decreases from around 6% at 7 months to 0.5% at full maturity. Burns et al. (2020) reported that coconut fruit takes on average 11-12 months to mature, during this period fruits can be divided into three categories based on the chemical composition of their water. Immature or Tender (6–8 months), Mature (9–11 months), and Overly-mature (12 months or older). The turbidity of the coconut water increased with maturity, the pH tended to rise, and the soluble solids (Brix, the total sugar content) tended to increase but fall off with maturities above 12 months. Most fruit sold in the international market is mature fruit used for processing and cooking in a bakery or hot dishes and desserts of Asian cuisine. Because of the advance in postharvest technology, young coconut is now exported across the continents. Mechanical tools are needed to replace labour, particularly in trimming the young fruit. Improvements are also needed in the procedure to prevent browning and mould growth. Its storage life is up to two months at 2°C. However, the handling processes to prepare the fruit are labour intensive. Two to three bunches of coconuts could be harvested from each palm if this cycle is followed. The methods of harvesting coconuts vary among countries or even among provinces within the same country. Producers from certain countries, especially in the Pacific, do not harvest their coconuts. There are two common methods of harvesting coconuts. These are the pole and the climbing method. A third method is only practised in Thailand, Malaysia and Indonesia. This procedure involves harvesting mature coconuts using trained monkeys. The pole method of harvesting is common in many countries in the region and it is a common practice to store harvested nuts in heaps under shade for a few days, known as seasoning before they are further processed (Punchihewa and Aracon 1999).

Studies have shown that storage of harvested nuts is beneficial only if the nuts are fully ripe and that good-quality copra can be obtained from nuts even immediately after harvest (Manikantan et al. 2018).

#### 2.2. Chemical composition, processing and utilization of coconut fruits/medium

Siriphanich et al. (2011) also explained the treatment process from harvesting the coconut to becoming desiccated coconut. It started from grated and dehydrated coconut meat, which is mainly used in the bakery and confectionery industries. The Codex Alimentarius Standard for various grades of desiccated coconut (CODEX STAN 177-1991) contains three size classifications: extrafine, fine and medium (Codex Alimentarius Commission, 1994). The process includes a selection of nuts, husking, shelling, removing the test (paring), washing, heating, disintegrating, drying, sieving and packaging. Before being transported to the factory, coconut fruits are de-husked in the field. The "testa" (outer brown part) is removed by using a special knife until it is clean then cut and the coconut water discarded. The kernel pieces are then pasteurized in live steam for 5 min at about 88°C or for 8 to 10 min at 70 to 80°C. The material is then immersed in sulphite solution for stabilization, followed by grinding or shredding and drying using a steam-heated dryer, in which the moisture content is adjusted to 2.5–3.0%. After that, the product is cooled, graded by size and packed. Desiccated coconut is rich in healthy saturated fats with no cholesterol and is also a good source of dietary fibre. Lauric acid, the medium-chain fatty acid from the fat of the coconut, is having antiviral, antibacterial, and antiprotozoal properties. Capric acid, another of coconut's fatty acids is also found to have antimicrobial properties. These fatty acids are found in large amounts only in traditional lauric fats, especially from coconut (Sebastian 2017).

#### 2.3. Mathematical models describing bulk oilseed under axial loading

#### 2.3.1. Mechanical behaviour of oil-bearing crops seeds

Herak et al. (2012) mentioned the importance to understand in detail the mechanical behaviour of seeds under compression loading for designing pressing technology with minimum energy efficiency with the maximum oil outcome. The study provides information about the comparison of mechanical behaviour of selected oil-bearing crops namely rapeseeds, sunflower seeds and jatropha seeds under compression loading.

From the compression test, the amounts of true deformation, maximal deformation energy and the compressive force of the pressed samples were calculated and mathematical equations describing the limit deformation, maximal deformation ratio, energy ratio and oil point deformation ratio were determined. Based on the measured amounts rapeseeds achieved the highest values followed by jatropha seed and then sunflower seed. The amount of deformation energy required for the seed deformation indicates the amount of energy needed for obtaining the oil from the seed.

Kabutey et al. (2017), described the oil point and mechanical properties of roasted and unroasted bulk oil palm kernels under compression loading by using a universal compression testing machine. The measured parameters were the deformation, deformation energy, oil yield, oil point strain and oil point pressure. Certainly, more energy is needed to obtain kernel oil from the unroasted kernels compared to the roasted bulk kernel. The reason could be related to the variation in the structural integrity of the oil-bearing materials, that is, the change in the physical (moisture content, initial bulk density, kernel density, porosity) and mechanical (contact and gradient pressure and compressibility) properties. It also showed a smaller amount of oil point pressure for roasted kernels compared to the unroasted ones.

Herak et al. (2013) and (2014) reported the mathematical models based on the tangent curve function and reciprocal slope transformation (RST) for describing the mechanical behaviour of bulk oilseeds under compression loading. The tangent curve function is dependent on the force coefficient of mechanical behaviour, deformation coefficient of mechanical behaviour and the value or exponent of the fitted curve. The tangent curve model has been described for different oil-bearing crops bulk seeds/kernels: jatropha, rape, sunflower, pumpkin and oil palm; as well as for different pressing vessel diameters and pressing heights. The tangent curve model can also describe the theoretical deformation energy. Regarding the RST, Herak et al. (2014), indicated that the RST describes two independent and dependent variables (Blahovec 2011). According to Herak et al. (2014), in the linear compression process, the deformation of the bulk oilseeds represents the independent variable whereas the compressive force represents the dependent variable. The dependency between compressive force and deformation can be transformed using the reciprocal slope transformation where the transformed compressive force is approximated by a third-order polynomial function.

The authors indicated that the coefficients of the polynomial function can be determined by the least-squares method using the MathCAD 14 software (Marquardt, 1963). The authors further indicated that the tangent curve function and the RST provide the background for the development of a generalized model for describing the mechanical behaviour of bulk oilseeds/kernels under axial loading.

#### 2.3.2. Relaxation behaviour of oil-bearing crops seeds

Herak et al. (2014) described the relaxation process of *Jatropha curcas* L. under compression loading at different bulk deformations by examining the rate of normalized force and second-time derivation of normalized force. The deformation at the oil point was identified and verified using the relaxation curves, normalized force, the rate of normalized force, and the second derivative of normalized force versus deformation of bulk seeds. The oil point can be determined from the relaxation process due to the minimum normalized force at the start of the relaxation process. The authors stated that the dependency of compressive force and relaxation time for different deformations showed a linear trend with 2 s of relaxation time, and after 10 s of the relaxation process, the rate of normalized force was constant, and the relaxation process showed no internal dynamic forces and stresses on the bulk seeds.

In a separate study by Herak et. (2015), the authors applied Wiechert models A and B as well as a Peleg model C to describe the relaxation behaviour of *Jatropha curcas* L. bulk seeds. Model A was assembled as three parallel linked branches with the first and second branches comprising a serially connected spring and dashpot whereas the third branch contained only a spring. Model B consisted of two parallel linked branches with the first branch comprising a serially connected spring and dashpot whereas the second branch contained a spring. On the other hand, the Peleg model (model C) is an empirical model which transforms the stress relaxation curve into a straight line (Peleg 1976). The authors indicated that the mechanical behaviour of relaxation involves compliance with the conditions of constant deformation and constant strain. The authors further mentioned that the coefficients (moduli of elasticity, normal viscosity, relaxation stresses, initial stresses, relaxation time and Peleg constants) were determined using the MathCAD 14 software (Pritchard 1998) which uses the Levenberg-Marquardt algorithm for data fitting (Marquardt 1963).

#### 2.4. Overview of response surface methodology

Response Surface Methodology (RSM) helps to determine the best experimental design to identify the relationship between variables. The RSM is used to optimize process parameters and to develop an experimental design that integrates independent variables by using the data from the experiment to reach a set of equations that can give theoretical value for the outcome (Said and Amin 2015). In a recent study by Elkelawy et al. (2022), the authors reviewed the importance of using response surface methodology in predicting the optimum performance and emission characteristics for diesel engines fueled with blends of diesel, alternative fuels, and nano-particle addictive. The study accomplished that the comparison between the experimental and the modelling by response surface methodology is similar that also can give accurate results and save money and time.

Orisaleye et al. (2022) developed the predictive models by using the response surface methodology and by adopting the Box-Behnken experimental design for producing briquettes under different process conditions from Abura sawdust. The variables considered were temperature, holding time and pressure. For the results, they found that temperature, holding time, pressure, square term of pressure, and interaction of pressure and temperature were statistically significant (p < 0.05) in determining the density of the sawdust briquettes. However, only linear terms of temperature and holding time were statistically significant (p < 0.05) for determining the water-resistant of the sawdust briquettes. The response surface model developed had close prediction to the experimental values and the plots showed the combination of the statistically significant terms within the model to determine the quality of the sawdust briquettes.

Adamu et al. (2021) studied the mechanical performance and optimization of high-volume fly ash concrete containing plastic wastes and graphene nanoplatelets by using response surface methodology for designing an experiment, modelling, and optimization. The variable was PW (plastic waste), HVFA (high-volume fly ash), and graphene nanoplatelets (GNP), and the responses were strengths and water absorption. The results of the experiment led to PW and HVFA reducing the strengths and absorption while GNP enhanced them. The proposed models developed were significant with a high level of correlation. The optimized mix was achieved by substituting 15.3% of coarse aggregate using PW, 6.07% of cement using HVFA, and adding GNP at 0.22%, and was experimentally validated with an error of less than 5%.

Mitraka, et al. (2022) conducted a study to assess whether the implementation of Supercritical Carbon dioxide Explosion (SCE) is an efficient approach for sewage sludge pre-treatment, by reaching the optimum SCE to develop a method attempting to increase the biodegradability of sewage sludge's organic matter content, and thus, to enhance the subsequent anaerobic digestion and methane production. Implementing response surface methodology as the statistical tool to evaluate the effects of the main pre-treatment parameters (i.e., temperature and time) and their interaction on methane yield, which was defined as the response. The authors found that temperature was the most significant variable and had the greatest effect on methane yield. The experiment was able to determine the optimum set of pre-treatment conditions corresponding to a temperature of 115°C and a time of 13 minutes. In a range of these optimum conditions, the predicted response value was 300 mL CH<sub>4</sub>/g of volatile solids. The corresponding experimental value obtained from the validation experiment fitted well with this value and demonstrates the effective use of response surface methodology in optimizing SCE.

#### 2.4.1. Box-Behnken design

Ferreira et al. (2007) described the fundamentals, advantages and limitations of the Box-Behnken design (BBD) for the optimization of analytical methods. A comparison between the Box-Behnken design and other response surface designs (central composite, Doehlert matrix and three-level full factorial design) has demonstrated that the Box-Behnken design and matrix are slightly more efficient than the central composite design but much more efficient than the three-level full factorial designs. The Box-Behnken is a good design for response surface methodology because it permits: (i) estimation of the parameters of the quadratic model, (ii) building of sequential designs, (iii) detection of lack of fit of the model, and (iv) use of blocks.

Muhammad et al. (2022) used three levels of Box-Behnken design surface methodology to optimize individual and interactive effects of parameter time (120–240 min), temperature (120–160°C), solvent-to-wet biomass ratio (2.0–4.67), and hydrochloric acid concentration (2–4 M). The temperature was the most significant factor for direct transesterification of wet microalgae (low *p*-value (0.0001) and high F value (53.89). The highest yield (19.00%) of fatty acid methyl ester was obtained on a dry biomass weight basis under the optimum conditions of 240 min, 146°C, 2.83 (vol/wt) and 3.86 M acid concentration.

Box-Behnken design data trained the artificial neural network and response surface methodology to predict responses and to develop and compare each model's predictive abilities. The accuracy of the results indicates that both models predict the experimental data for fatty acid methyl ester yields with high correlation coefficients ( $R^2$ ) of 0.94 and 0.92, respectively for artificial neural network and response surface methodology.

#### **MATERIALS AND METHODS**

#### 3.1. Sample and experimental conditions

The sample (coconut desiccated medium, Appendix 1) of the weight of 25 kg was obtained from the Farmet Company, Česká Skalice, Czech Republic). The sample was originally produced in Indonesia but was purchased from Poland by the Farmet Company. The experiment was conducted in a laboratory temperature of  $22.4 \pm 0.72$  °C and humidity of  $23.33 \pm 0.58$  %.

#### **3.2.** Determination of moisture content of the sample

The moisture content of the sample was determined using the conventional oven method of 105 °C and drying time of 17 hours (ISI 1996) as shown in Figure 2 and Appendix 2. The electronic balance (KERN & SOHN 440–35, Balingen, Germany) with an accuracy of 0.01 g was used for weighing the sample (Appendix 3). The sample moisture content of  $2.5 \pm 0.1$  (% w.b.) was calculated according to equation 1 (Blahovec 2008).

$$MC = \frac{m_{bf} - m_{af}}{m_{bf}} \tag{1}$$

where *MC* is the moisture content in wet basis (%),  $m_{bf}$  is the mass of the sample before drying and  $m_{af}$  is the mass of the sample after drying.



Figure 2. Measured sample in triplicate (A: before drying; B: after drying).

#### **3.3.** Determination of oil content of the sample

The sample oil content was determined using the Soxhlet extraction procedure (Figure 3). According to the procedure (Mohammadpour et al. 2019; Gurkan et al. 2020), approximately

11 g of the sample was ground in a mini grinder. The ground sample was put into a thimble and cotton wool was placed atop. The thimble was inserted into the Soxhlet extractor which was then connected to a 500 mL round bottom flask containing 250 mL of petroleum ether. The setup was placed under a heating source at 60 °C and the solvent was heated to reflux for 24 h. The extracted oil was left in the oven at 50 ° C for 4 h to remove the residual solvent. The electronic balance (KERN & SOHN AEJ 200–4CM, Balingen, Germany) with an accuracy of 0.0001 g was used for the measurements. The experiment was repeated twice and averaged (Table 1). The sample oil content of  $61.88 \pm 0.42$  (%) was calculated according to equation 1 as stated above.



Figure 3. Soxhlet extraction procedure for the determination of sample oil content.

Test 1	Weight (g)	Test 2	Weight (g)
Timble ( <i>T</i> )	2.0993	Timble ( <i>T</i> )	2.2576
Sample $(S) + (T)$	12.9713	Sample $(S) + (T)$	13.1673
S	10.872	S	10.9097
Round-bottom flask (F)	192.9303	Round-bottom Flask ( <b>F</b> )	171.9367
$\mathbf{F} + \operatorname{Oil}(\boldsymbol{O})$	199.6256	$\mathbf{F} + \operatorname{Oil}(\boldsymbol{O})$	178.7199
0	6.6953	0	6.7832
Oil content (%)	61.5829	Oil content (%)	62.1759
Mean + SD		$61.88 \pm 0.42$	

Table 1. Measurements for the sample oil content determination at 24 h cycle.

SD: Standard Deviation

#### 3.4. Box-Behnken experimental design

A Box-Behnken design (BBD) was used to generate 17 experiments from the combination of the operating factors (force: 1.6, 3.2 and 4.8 kN; temperature: 40, 60 and 80 °C and heating time: 30, 45 and 60 min). Experimentally, the combined factors will produce 27 runs, and a total of 81 runs when replicated thrice. The BBD is useful for reducing the several experiments (Chanioti and Tzia 2017; Huang et al. 2019; Kabutey et al. 2021; Cimen et al. 2022). The mathematical equation defining the BBD is given in equation 2.

$$Y = \beta_0 + \sum_{i=1}^k \beta_i X_i + \sum_{i=1}^k \beta_{ii} X_i^2 + \sum_{i_{1 < j}}^k \sum_j^k \beta_{ij} X_i X_j$$
(2)

where Y is the response variable;  $\beta_0$ ,  $\beta_i$ ,  $\beta_{ii}$  and  $\beta_{ij}$  are the regression coefficients of the intercept, linear, quadratic and interaction terms respectively; X<sub>i</sub> and X<sub>j</sub> are the independent variables and k is the number of factors. The factors-levels stated above were coded from -1 (low value) to +1(high value) with 0 being the center value according to equation 3 (Ocholi et al. 2018; Kabutey et al. 2021; Cimen et al. 2022).

$$x_{i} = \frac{X_{i} - X_{0}}{\Delta X}$$
(3)

where  $x_i$  is the coded value of the i<sup>th</sup> variable,  $X_i$  is the uncoded value of the i<sup>th</sup> test variable,  $X_0$  is the uncoded value of the i<sup>th</sup> test variable at the centre point and  $\Delta X$  is the step-change in the real value of the variable *i* corresponding to the variation in a unit for the dimensionless value of the variable *i*.

#### 3.5. Sample pretreatment

The oven (MEMMERT GmbH + Co. KG, Germany) was used for the pretreatment of the sample at temperatures of 40, 60 and 80 °C before the compression test (Appendix 2).

#### **3.6.** Compression tests

The compression tests of the sample (control without pretreatment and pretreatments based on the Box Behnken design) as shown in Figure 4 were done using the universal compression testing machine (MPTest 5.050, Czech Republic) of a maximum load of 5 kN and a pressing vessel of diameter 30 mm with a plunger. The initial pressing height of the sample was measured at 100 mm (the volume of the sample was calculated to be  $7.07 \times 10^{-5} \text{ m}^3$ ). The control experiments were done at a speed of 4 mm/min and forces of 1.6, 3.2 and 4.8 kN (equivalent pressures of 2.26, 4.53 and 6.79 MPa in respect to the cross-sectional area of the pressing vessel). The experiments from the Box Behnken design were done also at a speed of 4 mm/min. Each compression test produced the force-deformation data which was further used to calculate the parameters mentioned in Section 3.8. The relaxation experiments at optimized conditions were done using the same equipment. The pressed sample cakes and extracted oil are shown in Appendix 4.





Figure 4. (A) Compression test setup (compression machine of load 5 kN, vessel diameter of 30 mm with a plunger and a computer monitor showing the display of the data), (B) Sample, cake and semi-solid coconut oil collected and (C) Sample test showing the oil leakage.

## 3.7. Spectral properties of sample oil extracted

Using the UV-VIS spectrophotometer (SpektrofotometrOnda VIS V-10 Plus, Czech Republic) (Appendix 5), the absorbance and transmittance values of the extracted oil samples at control and pretreatment temperatures (Appendixes 6 and 7) were determined at wavelengths between 325 and 600 nm (Gurkan et al. 2020; Cimen et al. 2021, Kabutey et al. 2021).

## **3.8.** Calculated parameters from the compression tests

#### 3.8.1. Mass of oil

The mass of oil  $M_{ol}$  was calculated as the initial mass of the sample  $M_{sp}$  minus the sample cake after compression (Kabutey et al. 2015).

#### 3.8.2. Oil yield

The oil yield was calculated as the ratio of the mass of oil to the mass of the sample multiplied by 100 according to equation 4 (Deli et al. 2011; Chanioti and Tzia, 2017).

$$O_{yd} = \left[ \left( \frac{M_{ol}}{M_{sp}} \right) \cdot 100 \right] \tag{4}$$

where  $O_{vd}$  is the oil yield (%),  $M_{ol}$  is the mass of oil (g) and  $M_{sp}$  is the initial mass of sample (g).

#### 3.8.3. Oil expression efficiency

The oil expression efficiency was calculated as the ratio of oil yield to that of percentage oil content according to equation 5 (Gurkan et al. 2020).

$$O_{ee} = \left[ \left( \frac{O_{yd}}{O_{cs}} \right) \cdot 100 \right]$$
(5)

where  $O_{ee}$  is the oil expression efficiency (%) and  $O_{cs}$  is the sample oil content (%) by Soxhlet extraction.

#### 3.8.4. Energy

The energy  $E_{nr}$  was calculated based on the trapezoidal rule as stated in equation 6 (Lysiak, 2007; Chakespari et al. 2010; Herak et al. 2012; Divisova et al. 2014).

$$E_{nr} = \sum_{n=0}^{n=t-1} \left[ \left( \frac{F_{n+1} + F_n}{2} \right) \cdot (x_{n+1} - x_n) \right]$$
(6)

where  $E_{nr}$  is the energy (J),  $F_{n+1} + F_n$  representing  $F_{cr}$  as the force (N) and  $x_{n+1} - x_n$  representing  $D_{fm}$  as the deformation (mm), *n* is the number of data points and *i* is the number of sections in which the axis deformation was divided.

#### 3.8.5. Deformation

The deformation  $D_{fm}$  (mm) values (Appendixes 8 and 9) were obtained directly from the compression data (Kabutey et al. 2021).

#### 3.8.6. Hardness

The hardness  $H_{dx}$  values were calculated as the ratio of force to that of deformation (Appendixes 8 and 9) as stated in equation 7 (Chakespari et al. 2010; Divisova et al. 2014).

$$H_{dx} = \frac{F_{cr}}{D_{fm}} \tag{7}$$

#### 3.8.7. Volume of sample

The volume  $V_{sp}$  (m<sup>3</sup>) of the sample was calculated based on the area of the pressing vessel multiplied by the initial pressing height of the sample as stated in equation 8 (Chakespari et al. 2010; Divisova et al. 2014).

$$V_{sp} = \frac{\pi \cdot D^2}{4} \cdot H \tag{8}$$

where D is the diameter of the pressing vessel and H is the initial pressing height of the sample.

#### 3.9. Statistical analysis of experimental data

The experimental data (Tables 2 to 4 and Appendixes 6 and 7) were analyzed by employing the General Linear Model technique (response surface regression) at a 5% significance level using STATISTICA 13 (Statsoft 2013).

#### **RESULTS AND DISCUSSION**

#### 4.1. Percentage oil content and control data

The sample oil content of  $61.88 \pm 0.42$  (%) was determined through the Soxhlet extraction procedure (Blahovec 2008; Mohammadpour et al. 2019; Gurkan et al. 2020). The measurements are shown in Table 1 (Section 3.3). The control data are given in Table 2. The control measurements of the sample were done at a laboratory temperature of 22 °C. The sample's initial pressing height was 100 mm and pressed at different forces between 1.6 and 4.8 kN at a speed of 4 mm/min. The mass of oil, oil yield, oil expression efficiency and energy were calculated. The results are displayed in Figure 5 where the increase in the forces increased all the calculated amounts. The deformation and hardness values are given in Appendix 8. The mean and standard deviation values for oil yield ranged from  $0.47 \pm 0.28$  to  $3.99 \pm 0.33$  g; for oil yield from  $1.63 \pm 0.97$  to  $13.82 \pm 1.15$  %; for oil expression efficiency from  $2.63 \pm 1.57$  to  $22.33 \pm 1.86$  %; for energy from  $25.73 \pm 1.03$  to  $51.82 \pm 4.17$  J; for deformation from  $56.54 \pm 2.70$  to  $68.08 \pm 3.74$  mm and hardness from  $28.34 \pm 1.32$  to  $70.65 \pm 3.85$  N/mm. Three levels of the forces were combined with three levels each for the pretreatment temperatures and heating times based on the Box-Behnken Design to determine the optimal processing factors (Section 4.3).

		Calculated parameters						
Replications	$F_{rc}$ (N)	$M_{ol}$ (g)	$O_{yd}$ (%)	<b>O</b> <sub>ee</sub> (%)	$E_{nr}$ (J)			
1		0.26	0.90	1.45	24.57			
2	1600	0.79	2.73	4.42	26.07			
3	1000	0.36	1.25	2.01	26.54			
Mean ± SD		$0.47 \pm 0.28$	$1.63 \pm 0.97$	$2.63 \pm 1.57$	$25.73 \pm 1.03$			
1		2.29	7.92	12.81	39.92			
2	2200	2.91	10.07	16.27	41.49			
3	5200	2.86	9.90	15.99	43.41			
Mean ± SD		$2.69 \pm 0.34$	9.30 ± 1.19	$15.02 \pm 1.93$	41.61 ± 1.75			
1		3.94	13.63	22.03	47.11			
2	1800	3.69	12.77	20.63	53.31			
3	4000	4.35	15.05	24.32	55.03			
Mean ± SD		3.99±0.33	$13.82 \pm 1.15$	$22.33 \pm 1.86$	$51.82 \pm 4.17$			

Table 2. Experimental data of desiccated coconut medium at a control temperature of 22 °C.

SD: Standard Deviation;  $F_{rc}$ : Force; Mass of oil,  $O_{yd}$ : Oil yield;  $O_{ee}$ : Oil expression efficiency (%), and  $E_{nr}$ : Energy (J).



Figure 5. Box plot of the calculated parameters grouped by the force.

#### 4.2. Compression and relaxation curve behaviours

In the uniaxial compression process, the dependencies between the force and deformation as well as the relaxation force and time can be described (Figures 6 and 7). The compression curve (Figure 6) is where the deformation energy is calculated for the oil output as the area under the curve (Divisova et al. 2014). The force-deformation curves did not show any serration pattern indicating that maximum oil output was recovered (Divisova et al. 2014). The relaxation area (Figure 7) allows for the residual oil in the seedcake to be recovered within a specific time interval (here for 23 minutes). It can be seen in Figure 7 that at a maximum force of 4.8 kN, the speed of 4 mm/min and pressing height of the sample at 100 mm, both the compression and relaxation processes elapsed for 40 minutes indicating that considerable time is needed during the uniaxial oil extraction of bulk oilseeds/kernels or medium desiccated coconut. According to Herak et al. (2015), the compression and relaxation forces can be transformed into stresses whereas the deformation

values into strains. The authors indicated that the mechanical behaviour of relaxation occurs at constant deformation and constant strain. In this present study, the oil output increased at a lower temperature of 40 °C after a relaxation time of 23 min but it decreased at a higher temperature of 80 °C (Table 3) indicating that the relaxation process is not required for the pretreatment temperature of the sample (medium desiccated coconut) above 40 °C.



Figure 6. Compression force and deformation curve of the sample 22 °C of representing other tests performed.



Figure 7. Compression and relaxation force and time curves of the sample of 22 °C represent other tests performed.

	F <sub>rc</sub>	M <sub>ol</sub>	O <sub>vd</sub>	0 <sub>ee</sub>
Replications	(kN)	(g)	(%)	(%)
1		4.44	15.36	24.83
2	1 9*	3.34	11.56	18.68
3	4.0	3.95	13.67	22.09
Mean ± SD		$3.91 \pm 0.55$	$13.53 \pm 1.91$	$21.86 \pm 3.08$
1		8.22	28.44	45.97
2	1 0**	8.34	28.86	46.64
3	4.0	8.43	29.17	47.14
Mean ± SD		$8.33 \pm 0.11$	$28.82 \pm 0.36$	$46.58 \pm 0.59$
1		6.75	23.36	37.75
2	1 0***	6.44	22.28	36.01
3	4.0	6.68	23.11	37.35
Mean ± SD		$6.62 \pm 0.16$	$22.92 \pm 0.56$	$37.04 \pm 0.91$

Table 3. Relaxation experimental data for the control and optimized factors combination.

SD: Standard Deviation; \* Control temperature of 22 °C; \*\* Optimized factors  $(F_{rc}: +1(4.8 \text{kN}); H_{tp}: +1(40 \text{ °C}) \text{ and } H_{tm}: +1(30 \text{ min}));$  \*\*\* Optimized factors  $(F_{rc}: +1(4.8 \text{kN}); H_{tp}: +1(60 \text{ °C}) \text{ and } H_{tm}: +1(60 \text{ min}));$   $F_{rc}:$  Force;  $M_{ol}$ : Mass of oil,  $O_{yd}$ : Oil yield and  $O_{ee}$ : Oil expression efficiency (%).

#### 4.3. Box-Behnken experimental data and regression models

Based on the Box-Behnken design (Table 4), 17 experiments were conducted for each factor combination. The calculated parameters were the mass of oil, oil yield, oil expression efficiency and energy. The factors combination:  $F_{rc}$ : +1(4.8kN);  $H_{tp}$ : +1(40 °C) and  $H_{tm}$ : +1(45 min) recorded the highest mass of oil of 7.81 g. The corresponding amounts of oil yield, oil expression efficiency and energy were 27.02%, 43.67% and 82.32 J respectively. The factors combination:  $F_{rc}$ : +1(4.8kN);  $H_{tp}$ : +1(60 °C) and  $H_{tm}$ : +1(30 min) achieved the next highest amounts of 6.94 g, 24.01%, 38.81% and 83.49 J. The factors combination:  $F_{rc}$ : +1(4.8kN);  $H_{tp}$ : +1(60 °C) and  $H_{tm}$ : +1(60 min) also obtained the amounts of 5.63 g, 19.48%, 31.48% and 85.89 J. The rest of the factors' combinations followed in that order of magnitude (Table 4). The data were analyzed statistically based on the response surface regression technique (ANOVA). The results are given in Tables 5 to 8. The regression models for predicting the calculated parameters (mass of oil, oil yield, oil expression efficiency and energy) with the factors (force, temperature and heating time) are described in equations 9 to 12. In the equations, the intercept and the coefficients of the factors were significant (P < 0.05). This means that the

non-significant coefficients (P > 0.05) were not included in the equations. The lack of fit P values for all the regression models for the calculated parameters were non-significant (P > 0.05) indicating that the determined models are adequate for predicting the calculated parameters. This statement is supported by Chanioti and Tzia (2017) in their study on the optimization of ultrasound-assisted extraction of oil from olive pomace using response surface technology: Oil recovery, unsaponifiable matter, total phenol content and antioxidant activity. It is vital to state that the regression models for the mass of oil, oil yield and oil expression efficiency are interrelated indicating that either parameter can represent the main parameter for understanding coconut oil extraction using the response surface methodology with the Box-Behnken design.

	F <sub>rc</sub>	$H_{tp}$	H <sub>tm</sub>	M <sub>ol</sub>	O <sub>yd</sub>	<b>O</b> <sub>ee</sub>	E <sub>nr</sub>
Run	(kN)	(°Ċ)	(min)	(g)	(%)	(%)	( <b>J</b> )
1	-1(1.6)	-1(40)	0(45)	0.42	1.45	2.35	26.70
2	1(4.8)	-1(40)	0(45)	7.81	27.02	43.67	82.32
3	-1(1.6)	1(80)	0(45)	0.71	2.46	3.97	24.35
4	1(4.8)	1(80)	0(45)	5.71	19.76	31.93	82.52
5	-1(1.6)	0(60)	-1(30)	0.48	1.66	2.68	26.29
6	1(4.8)	0(60)	-1(30)	6.94	24.01	38.81	83.49
7	-1(1.6)	0(60)	1(60)	0.56	1.94	3.13	27.18
8	1(4.8)	0(60)	1(60)	5.63	19.48	31.48	85.89
9	0(3.2)	-1(40)	-1(30)	3.85	13.32	21.53	57.29
10	0(3.2)	1(80)	-1(30)	4.04	13.98	22.59	53.12
11	0(3.2)	-1(40)	1(60)	3.21	11.11	17.95	55.69
12	0(3.2)	1(80)	1(60)	2.99	10.35	16.72	52.63
13	0(3.2)	0(60)	0(45)	3.62	12.53	20.24	56.25
14	0(3.2)	0(60)	0(45)	3.32	11.49	18.56	55.93
15	0(3.2)	0(60)	0(45)	3.41	11.80	19.07	55.41
16*	0(3.2)	0(60)	0(45)	3.65	12.63	20.41	54.03
17*	0(3.2)	0(60)	0(45)	3.48	12.04	19.46	54.18

Table 4. Experimental data of operating factors and calculated parameters.

 $F_{rc}$ : Force;  $H_{tp}$ : Heating temperature;  $H_{tm}$ : Heating time;  $M_{ol}$ : Mass of oil,  $O_{yd}$ : Oil yield;  $O_{ee}$ : Oil expression efficiency (%),  $E_{nr}$ : Energy (J) and  $D_{fm}$ : Deformation; \* Removed from the regression analysis due to the significance of the lack of fit value.

	Model	Standard	Sum of	df	Mean	F-value	
Effect	$M_{ol}$ (g) <sup>a</sup>	Error	Squares	ui	Square	I -value	P-value
Intercept	3.4500	0.1936	75.113	9	8.346	74.2058	0.0000*
F <sub>rc</sub>	2.9900	0.1186	71.5208	1	71.5208	3017.7553	0.0003*
$F_{rc}^2$	0.0462	0.1745	0.0079	1	0.0079	0.3333	0.6221**
H <sub>tp</sub>	-0.2300	0.1186	0.4232	1	0.4232	17.8565	0.0517**
$H_{tp}^2$	0.1662	0.1745	0.1021	1	0.1021	4.3060	0.1737**
$H_{tm}$	-0.3650	0.1186	1.0658	1	1.0658	44.9705	0.0215*
$H_{tm}^2$	-0.0938	0.1745	0.0325	1	0.0325	1.3693	0.3625**
$F_{rc} \ge H_{tp}$	-0.5975	0.1677	1.4280	1	1.4280	60.2542	0.0162*
$F_{rc} \ge H_{tm}$	-0.3475	0.1677	0.4830	1	0.4830	20.3808	0.0457*
$H_{tp} \ge H_{tm}$	-0.1025	0.1677	0.0420	1	0.0420	1.7732	0.3145**
Residual			0.5624	5	0.1125		
Lack of Fit			0.5149	3	0.1717	7.243	0.1237**
Total			75.6757	14			

Table 5. Regression estimates of mass of oil, Mol model.

 $F_{rc}$ : Force;  $H_{tp}$ : Heating temperature;  $H_{tm}$ : Heating time; <sup>a</sup>: Coefficient of determination (R<sup>2</sup>) = 0.993; df: degrees of freedom \*: Significant (P < 0.05); \*\*: Non-significant (P > 0.05).

$$\boldsymbol{M_{ol}} = 3.45 + 2.99 \cdot F_{rc} - 0.365 \cdot H_{tm} - 0.598 \cdot F_{rc} \times H_{tp} - 0.348 \cdot F_{rc} \times H_{tm}$$
(9)

Effect	Model <i>O<sub>yd</sub></i> (%) <sup>b</sup>	Standard Error	Sum of Squares	df	Mean Square	F-value	P-value
Intercept	11.9377	0.6700	899.335	9	99.9261	74.2058	0.0000*
F <sub>rc</sub>	10.3460	0.4103	856.3212	1	856.3212	3017.755	0.0003*
$F_{rc}^2$	0.1600	0.6039	0.0946	1	0.0946	0.333	0.6221**
$H_{tp}$	-0.7958	0.4103	5.0670	1	5.0670	17.857	0.0517**
$H_{tp}^2$	0.5753	0.6039	1.2219	1	1.2219	4.306	0.1737**
H <sub>tm</sub>	-1.2630	0.4103	12.7609	1	12.7609	44.970	0.0215*
$H_{tm}^2$	-0.3244	0.6039	0.3885	1	0.3885	1.369	0.3625**
$F_{rc} \ge H_{tp}$	-2.0675	0.5802	17.0978	1	17.0978	60.254	0.0162*
$F_{rc} \ge H_{tm}$	-1.2024	0.5802	5.7833	1	5.7833	20.381	0.0457*
$H_{tp} \ge H_{tm}$	-0.3547	0.5802	0.5032	1	0.5032	1.773	0.3145**
Residual			6.7330	5	1.3466		
Lack of Fit			6.1655	3	2.0552	7.243	0.1237**
Total			906.0683	14			

Table 6. Regression estimates of oil yield  $O_{yd}$  model.

 $F_{rc}$ : Force;  $H_{tp}$ : Heating temperature;  $H_{tm}$ : Heating time; <sup>b</sup>: Coefficient of determination (R<sup>2</sup>) = 0.993; df: degrees of freedom \*: Significant (P < 0.05); \*\*: Non-significant (P > 0.05).

$$\boldsymbol{O}_{yd} = 11.938 + 10.346 \cdot F_{rc} - 1.263 \cdot H_{tm} - 2.068 \cdot F_{rc} \times H_{tp} - 1.202 \cdot F_{rc} \times H_{tm}$$
(10)

Effect	Model <i>O<sub>ee</sub></i> (%) <sup>c</sup>	Standard Error	Sum of Squares	df	Mean Square	F-value	P-value
Intercept	19.2919	1.0827	2348.709	9	260.9677	74.2058	0.0000*
F <sub>rc</sub>	16.7196	0.6630	2236.373	1	2236.373	3017.755	0.0003**
$F_{rc}^2$	0.2586	0.9759	0.247	1	0.247	0.333	0.6221**
$H_{tp}$	-1.2861	0.6630	13.233	1	13.233	17.857	0.0517**
$H_{tp}^2$	0.9296	0.9759	3.191	1	3.191	4.306	0.1737**
$H_{tm}$	-2.0410	0.6630	33.326	1	33.326	44.970	0.0215*
$H_{tm}^2$	-0.5242	0.9759	1.015	1	1.015	1.369	0.3625**
$F_{rc} \ge H_{tp}$	-3.3411	0.9377	44.653	1	44.653	60.254	0.0162*
$F_{rc} \ge H_{tm}$	-1.9432	0.9377	15.104	1	15.104	20.381	0.0457*
$H_{tp} \ge H_{tm}$	-0.5732	0.9377	1.314	1	1.314	1.773	0.3145**
Residual			17.5840	5	3.5168		
Lack of Fit			16.102	3	5.367	7.243	0.12373**
Total			2366.293	14			

Table 7. Regression estimates of oil expression efficiency,  $O_{ee}$  model.

 $F_{rc}$ : Force;  $H_{tp}$ : Heating temperature;  $H_{tm}$ : Heating time; <sup>c</sup>: Coefficient of determination (R<sup>2</sup>) = 0.993; df: degrees of freedom \*: Significant (P < 0.05); \*\*: Non-significant (P > 0.05).

$$\boldsymbol{O}_{ee} = 19.292 + 16.721 \cdot F_{rc} - 2.041 \cdot H_{tm} - 3.341 \cdot F_{rc} \times H_{tp}$$

$$-1.943 \cdot F_{rc} \times H_{tm}$$
(11)

	Model	Standard	Sum of		Mean		
Effect	$E_{nr}$ (J) <sup>d</sup>	Error	Squares	df	Square	<b>F-value</b>	<b>P-value</b>
Intercept	55.8633	0.7195	6617.836	9	735.3151	473.4733	0.0000*
$F_{rc}$	28.7125	0.4406	6595.261	1	6595.2613	36694.7028	0.0000*
$F_{rc}^2$	-0.4304	0.6485	0.684	1	0.6840	3.8058	0.1904*
$H_{tp}$	-1.1725	0.4406	10.998	1	10.9981	61.1909	0.0160*
$H_{tp}^2$	-1.4604	0.6485	7.875	1	7.8750	43.8150	0.0221*
H <sub>tm</sub>	0.1500	0.4406	0.180	1	0.1800	1.0015	0.4224**
$H_{tm}^2$	0.2796	0.6485	0.289	1	0.2886	1.6058	0.3327**
$F_{rc} \ge H_{tp}$	0.6375	0.6231	1.626	1	1.6256	9.0446	0.0951**
$F_{rc} \ge H_{tm}$	0.3775	0.6231	0.570	1	0.5700	3.1715	0.2169**
$H_{tp} \ge H_{tm}$	0.2775	0.6231	0.308	1	0.3080	1.7138	0.3207**
Residual			7.7651	5	1.5530		
Lack of Fit			7.406	3	2.469	13.73	0.0686**
Total			6625.601	14			

Table 8. Regression estimates of energy,  $E_{nr}$  model.

 $F_{rc}$ : Force;  $H_{tp}$ : Heating temperature;  $H_{tm}$ : Heating time; <sup>d</sup>: Coefficient of determination (R<sup>2</sup>) = 0.998; df: degrees of freedom \*: Significant (P < 0.05); \*\*: Non-significant (P > 0.05).

$$\boldsymbol{E_{nr}} = 55.863 + 28.713 \cdot F_{rc} - 1.173 \cdot H_{tp} - 1.460 \cdot H_{tp}^2 \tag{12}$$

#### 4.4. Optimal processing conditions of the calculated parameters

The optimized factors for predicting the calculated parameters are graphically shown and indicated in Figures 8 to 11 respectively. The predicted optimum combination of factors for the mass of oil, oil yield and oil expression efficiency were the force of 4.8 kN, the temperature of 40 °C and heating time of 30 min. The predicted optimum combination of factors for energy was the force of 4.8 kN, the temperature of 60 °C and the heating time of 60 min. The corresponding desirability values were between 0.98 and 1 indicating the adequacy of the predicted optimal factors (Kandar and Akil 2016).



Figure 8. Predicted and desirability values for mass of oil,  $M_{ol}$  (g) based on optimum operating factors.



Figure 9. Predicted and desirability values for oil yield,  $O_{yd}$  (%) based on optimum operating factors.



Figure 10. Predicted and desirability values for oil expression efficiency,  $O_{ee}$  (%) based on optimum operating factors.



Figure 11. Predicted and desirability values for oil expression efficiency,  $E_{nr}$  (J) based on optimum operating factors.

#### 4.5. Validation of optimal conditions

The optimized factors for the calculated parameters were validated according to Chanioti and Tzia, (2017). The results are presented in Tables 9 and 10. The validated experimental values were almost like the predicted values by the determined regression models. The percentage error values between the predicted and experimental validated ranged between 0.04 and 3.12 % explaining that the developed modes are valid and adequate for optimizing the oil extraction process of medium desiccated coconut. It is important to mention that the predicted and validated energies were 84.58 and 83.66 J indicating that the regression model may not be required since the energy can be equally determined from the optimized factors for either the mass of oil, oil yield or oil expression efficiency. Most importantly, the oil output at these optimal conditions was lower with higher energy instead of higher oil yield with minimum energy requirement (Herak et al. 2013).

Table 9. Validated values from the regression model at optimal conditions.

Calculated	Predicted values	Experimental values	Percentage
parameters	(Regression model)	(Validated)	Error (%)
$M_{ol}(g)^*$	7.751	$7.747 \pm 0.666$	0.05
0 <sub>yd</sub> (%)*	26.817	$26.805 \pm 2.305$	0.04
0 <sub>ee</sub> (%)*	43.338	$43.318 \pm 3.725$	0.05
$E_{nr}$ (J)**	84.576	83.663 ± 1.94	1.08

\*Optimized factors ( $F_{rc}$ : +1(4.8kN);  $H_{tp}$ : +1(40 °C) and  $H_{tm}$ : +1(30 min)); \*\* Optimized factors ( $F_{rc}$ : +1(4.8kN);  $H_{tp}$ : +1(60 °C) and  $H_{tm}$ : +1(60 min));  $M_{ol}$ : Mass of oil,  $O_{yd}$ : Oil yield;  $O_{ee}$ : Oil expression efficiency (%) and  $E_{nr}$ : Energy (J).

Table 10. Validated values from the profiles of predicted at optimal conditions.

Calculated	Predicted values	Experimental values	Percentage
parameters	(Predicted profile)	(Validated)	Error (%)
$M_{ol}(g)^*$	7.996	$7.747 \pm 0.666$	3.11
0 <sub>yd</sub> (%)*	27.669	$26.805 \pm 2.305$	3.12
0 <sub>ee</sub> (%)*	44.714	$43.318 \pm 3.725$	3.12
$E_{nr}$ (J)**	84.952	83.663 ± 1.94	1.52

\*Optimized factors ( $F_{rc}$ : +1(4.8kN);  $H_{tp}$ : +1(40 °C) and  $H_{tm}$ : +1(30 min)); \*\* Optimized factors ( $F_{rc}$ : +1(4.8kN);  $H_{tp}$ : +1(60 °C) and  $H_{tm}$ : +1(60 min));  $M_{ol}$ : Mass of oil,  $O_{yd}$ : Oil yield;  $O_{ee}$ : Oil expression efficiency (%) and  $E_{nr}$ : Energy (J).

#### 4.6. Spectral properties of coconut oil

The spectral profiles (absorbance and transmittance versus wavelength) of the extracted oil at different pretreatment temperatures are graphically described in Figures 12 and 13, and the data are provided in Appendixes 6 and 7. The pretreatment heating temperatures increased the absorbance values from 0.4 to 1.4 and the transmittance values from 3.3 to 40.3% along with the wavelength between 350 and 600 nm. The absorbance increase occurred at the wavelength between 330 and 340 nm and then decreased up to 600 nm. The transmittance values decreased steeply at 340 nm and then increased until 600 nm. Figures 12 and 13 reveal that absorbance and transmittance are inversely related. The inverse relationship between absorbance and transmittance is not linear but logarithmic (easierwithpractice.com). The high absorption and low transmission rates of extracted coconut oils under the various pretreatment conditions can be used for the prevention of ultraviolet radiation problems on human skin (Demirel et al. 2021; Kumar and Viswanathan 2013).



Figure 12. Relationship between absorbance and wavelength of coconut oil at different pretreatment temperatures.



Figure 13. Relationship between transmittance and wavelength of coconut oil at different pretreatment temperatures.

#### CONCLUSIONS

- (i) The oil content of  $61.88 \pm 0.42$  (%) of the medium desiccated coconut sample was determined by the Soxhlet extraction procedure.
- (ii) The force-deformation and the relaxation force-time curves of the medium desiccated coconut sample were described. The force-deformation curves did not show any serration pattern indicating that maximum oil output was recovered.
- (iii) From the experimental data of the Box-Behnken design of the processing factors, the maximum mass of oil of 7.81 g, the oil yield of 27.02 % and oil expression efficiency of 43.67 % was recorded for the combined factors of force: 4.8 kN; temperature: 40 °C and heating time: 45 min. The corresponding energy was 82.32 J.
- (iv) The regression models for estimating the mass of oil, oil yield, oil expression efficiency and energy were determined, and they were adequate for prediction based on the lack of fit of the *P* values being non-significant (P > 0.05).
- (v) The regression models for the mass of oil, oil yield and oil expression efficiency are interrelated indicating that either parameter can represent the main parameter for understanding coconut oil extraction using the response surface methodology/ Box-Behnken design.
- (vi) The optimized combined factors for the mass of oil of 7.75 g, the oil yield of 26.82 % and oil expression efficiency of 43.34 % were the force of 4.8 kN, temperature of 40 °C and heating time of 30 min.
- (vii) The optimized combined factors for the energy were the force of 4.8 kN, temperature of 60 °C and heating time of 60 min. The predicted and validated energies were 84.58 and 83.66 J indicating that the regression model may not be required since the energy can be determined from the optimized factors for either the mass of oil, oil yield or oil expression efficiency. In addition, the oil output at these optimal conditions was lower with higher energy.
- (viii) The validated values at the optimized combined factors for the mass of oil, oil yield and oil expression efficiency were 7.75 g, 26.81% and 43.32%. The percentage error values between the predicted (regression models and profiles and desirability) and

experimental validated ranged between 0.04 and 3.12 % indicating the reliability of the results achieved in this present study.

- (ix) The oil output increased at a lower temperature of 40 °C after a relaxation time of 23 min but it decreased at a higher temperature of 80 °C indicating that the relaxation process is not required for the pretreatment temperature of the sample (medium desiccated coconut) above 40 °C.
- (x) Pretreatment temperatures did not greatly increase the absorbance and transmittance values of the extracted coconut oil at a wavelength between 325 and 600 nm.
- (xi) The values of the spectral profiles (absorbance and transmittance versus wavelength between 325 and 600 nm) suggest that coconut oil can be utilized for the prevention of ultraviolet radiation problems on human skin (Kumar and Viswanathan 2013).

#### RECOMMENDATIONS

- (i) The different pressing vessel diameters among other factors should be examined for the medium desiccated coconut under a higher compression load with the response surface methodology/Box-Behnken design.
- (ii) The combinations of the pretreatment temperatures of the medium desiccated coconut along with the constant heating of the pressing vessel at a specific temperature during the compression process should be studied to estimate the oil yield or oil extraction efficiency.
- (iii) The residual oil in the seedcake after the compression process should be determined with the Soxhlet extraction procedure to fully understand the uniaxial oil extraction process.
- (iv) The mathematical and relaxation models should be described for the medium desiccated coconut under axial loading (Herak et al. 2013, 2014 and 2015).
- (v) The chemical properties of coconut oil in terms of quality under laboratory and pretreatment temperatures should be determined and studied extensively in future studies.
- (vi) The spectral profiles or peaks intensity (absorbance and transmittance versus wavelength) of different edible oils should be studied extensively using advanced spectroscopic techniques.
- (xii) The mechanical pressing (screw press) should be used to process the medium desiccated coconut at different processing conditions based on the response surface methodology/
   Box-Behnken design towards achieving or designing cost-efficient processing technology for application in developing countries such as Indonesia.

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## **APPENDIXES**



(A)

Appendix 1. (A) Coconut desiccated medium in a brown paper sack showing two prepared samples in a plane rubber for the experiment, (B) A paper showing the information of the coconut desiccated medium.



(A)



Appendix 2. (A) Sample moisture content determination, (B) pretreatment at 40  $^{\circ}$ C like 60  $^{\circ}$ C and 80  $^{\circ}$ C before the compression tests.



Appendix 3. (A) Initial mass of sample before compression test, (B) mass of sample cake after compression test, (C) Mass of sample before oven drying and (D) Mass of sample after 17 hr drying in the oven at 105 °C.



Appendix 4. (A) Extracted semi-solid coconut oil and packed sample cake (B) Compressed sample cake at different forces (1.6, 3.2 and 4.8 kN).



Appendix 5. A spectrophotometer for determining the absorbance and transmittance curves of the coconut oil.

		A 20	) °C	A 40	) °C	A 60	) °C	A 80	) °C
WL	Ν	Mean	± SD						
325	3	0.424	0.033	0.444	0.008	0.406	0.003	0.394	0.005
330	3	0.415	0.025	0.445	0.008	0.410	0.002	0.403	0.001
335	3	0.404	0.014	0.452	0.007	0.422	0.001	0.406	0.004
340	3	0.794	0.041	1.377	0.152	0.907	0.007	0.975	0.053
345	3	0.851	0.026	1.112	0.024	0.952	0.015	0.972	0.011
350	3	0.867	0.018	1.019	0.016	0.969	0.005	0.956	0.013
355	3	0.853	0.014	0.987	0.009	0.960	0.002	0.925	0.005
360	3	0.828	0.012	0.958	0.007	0.942	0.003	0.902	0.005
365	3	0.805	0.012	0.937	0.007	0.926	0.008	0.881	0.006
370	3	0.779	0.010	0.911	0.008	0.903	0.010	0.859	0.009
375	3	0.763	0.010	0.894	0.007	0.888	0.009	0.844	0.008
380	3	0.746	0.009	0.879	0.008	0.873	0.009	0.829	0.008
385	3	0.730	0.009	0.865	0.007	0.858	0.009	0.811	0.003
390	3	0.711	0.010	0.849	0.007	0.842	0.009	0.799	0.006
395	3	0.700	0.011	0.837	0.007	0.830	0.009	0.787	0.006
400	3	0.689	0.012	0.825	0.006	0.818	0.009	0.775	0.005
405	3	0.679	0.012	0.813	0.007	0.805	0.009	0.766	0.007
410	3	0.670	0.012	0.803	0.006	0.794	0.008	0.758	0.006
415	3	0.664	0.013	0.790	0.006	0.784	0.008	0.748	0.006
420	3	0.660	0.013	0.784	0.005	0.774	0.007	0.740	0.005
425	3	0.653	0.012	0.775	0.006	0.765	0.007	0.730	0.003
430	3	0.645	0.012	0.765	0.005	0.754	0.007	0.721	0.005
435	3	0.635	0.011	0.757	0.006	0.748	0.010	0.713	0.004
440	3	0.624	0.010	0.748	0.006	0.738	0.010	0.699	0.009
445	3	0.614	0.010	0.739	0.007	0.730	0.008	0.695	0.005
450	3	0.608	0.010	0.737	0.005	0.725	0.008	0.691	0.005
455	3	0.601	0.009	0.730	0.006	0.719	0.008	0.685	0.004
460	3	0.594	0.010	0.721	0.006	0.713	0.009	0.678	0.004
465	3	0.587	0.008	0.716	0.006	0.707	0.009	0.670	0.004
470	3	0.581	0.009	0.709	0.007	0.698	0.008	0.664	0.003
475	3	0.571	0.015	0.702	0.006	0.691	0.009	0.658	0.003
480	3	0.566	0.014	0.696	0.006	0.685	0.009	0.652	0.003
485	3	0.563	0.013	0.690	0.007	0.679	0.009	0.647	0.004
490	3	0.560	0.008	0.684	0.007	0.673	0.009	0.640	0.004
495	3	0.557	0.006	0.678	0.007	0.667	0.009	0.635	0.005
500	3	0.554	0.006	0.672	0.007	0.661	0.010	0.629	0.005
505	3	0.548	0.005	0.667	0.008	0.656	0.011	0.623	0.006
510	3	0.546	0.004	0.661	0.007	0.650	0.011	0.617	0.006
515	3	0.543	0.004	0.653	0.010	0.645	0.011	0.612	0.007
520	3	0.540	0.004	0.644	0.017	0.639	0.011	0.607	0.007
525	3	0.537	0.004	0.645	0.007	0.633	0.011	0.603	0.008

Appendix 6. Experimental data of absorbance against wavelength of the oil samples.

530	3	0.533	0.004	0.638	0.009	0.626	0.008	0.598	0.008
535	3	0.530	0.004	0.631	0.007	0.621	0.008	0.593	0.009
540	3	0.528	0.005	0.630	0.006	0.617	0.009	0.590	0.010
545	3	0.528	0.005	0.629	0.006	0.616	0.009	0.589	0.010
550	3	0.525	0.004	0.624	0.007	0.611	0.009	0.586	0.011
555	3	0.520	0.005	0.618	0.007	0.605	0.009	0.580	0.012
560	3	0.517	0.005	0.614	0.007	0.600	0.009	0.576	0.012
565	3	0.514	0.006	0.609	0.007	0.595	0.009	0.571	0.011
570	3	0.512	0.005	0.604	0.008	0.591	0.009	0.566	0.011
575	3	0.508	0.005	0.595	0.007	0.585	0.009	0.563	0.012
580	3	0.505	0.004	0.593	0.006	0.580	0.009	0.555	0.008
585	3	0.497	0.005	0.585	0.006	0.571	0.009	0.548	0.010
590	3	0.497	0.004	0.580	0.005	0.568	0.009	0.546	0.010
595	3	0.496	0.004	0.580	0.006	0.567	0.009	0.545	0.011
600	3	0.494	0.004	0.575	0.006	0.563	0.009	0.539	0.009

WL: Wavelength (nm); N: Number of replications; A: Absorbance (-) and SD: Standard Deviation.

		T 20	) °C	Т 40 °С		T 60 °C		Т 80 °С	
WL	N	Mean	± SD	Mean	± SD	Mean	± SD	Mean	± SD
325	3	37.833	2.871	36.033	0.611	39.233	0.153	40.333	0.577
330	3	38.500	2.261	35.933	0.611	38.900	0.173	39.533	0.153
335	3	39.500	1.300	35.300	0.600	37.867	0.153	39.200	0.300
340	3	16.500	0.000	3.333	0.577	12.333	1.026	10.400	0.900
345	3	13.933	0.404	7.700	0.173	11.533	0.058	10.600	0.361
350	3	13.600	0.500	9.467	0.058	10.700	0.265	11.200	0.346
355	3	14.100	0.436	10.333	0.153	11.033	0.058	11.867	0.252
360	3	14.933	0.404	11.000	0.200	11.433	0.115	12.533	0.153
365	3	15.667	0.473	11.600	0.200	11.867	0.252	13.100	0.173
370	3	16.633	0.351	12.267	0.208	12.533	0.306	13.833	0.252
375	3	17.300	0.400	12.767	0.208	12.967	0.252	14.300	0.300
380	3	17.933	0.404	13.200	0.200	13.400	0.265	14.833	0.252
385	3	18.600	0.361	13.667	0.208	13.867	0.252	15.467	0.115
390	3	19.433	0.473	14.167	0.208	14.367	0.252	15.833	0.208
395	3	19.967	0.493	14.567	0.208	14.800	0.300	16.300	0.200
400	3	20.500	0.529	14.967	0.208	15.200	0.300	16.800	0.173
405	3	20.967	0.586	15.367	0.208	15.700	0.300	17.133	0.252
410	3	21.400	0.624	15.767	0.208	16.067	0.252	17.433	0.252
415	3	21.700	0.624	16.200	0.200	16.433	0.252	17.833	0.252
420	3	21.900	0.624	16.467	0.208	16.833	0.252	18.167	0.208
425	3	22.300	0.624	16.800	0.200	17.200	0.300	18.600	0.173
430	3	22.633	0.569	17.200	0.200	17.600	0.300	18.967	0.208
435	3	23.200	0.557	17.533	0.252	17.933	0.306	19.367	0.208

Appendix 7. Experimental data of transmittance against wavelength of the oil samples.

440	3	23.800	0.557	17.900	0.200	18.233	0.404	19.867	0.153
445	3	24.300	0.557	18.233	0.252	18.633	0.351	20.167	0.208
450	3	24.667	0.551	18.333	0.252	18.800	0.361	20.367	0.208
455	3	25.100	0.500	18.633	0.252	19.100	0.361	20.667	0.208
460	3	25.433	0.603	19.000	0.265	19.367	0.351	20.967	0.208
465	3	25.867	0.503	19.233	0.252	19.633	0.351	21.367	0.208
470	3	26.233	0.551	19.533	0.252	20.033	0.404	21.700	0.173
475	3	26.900	0.985	19.833	0.252	20.333	0.404	22.033	0.153
480	3	27.167	0.862	20.133	0.252	20.633	0.404	22.300	0.200
485	3	27.367	0.802	20.433	0.321	20.967	0.451	22.567	0.208
490	3	27.533	0.451	20.700	0.361	21.233	0.404	22.900	0.200
495	3	27.700	0.361	21.000	0.361	21.533	0.404	23.167	0.208
500	3	27.933	0.351	21.267	0.379	22.033	0.808	23.467	0.252
505	3	28.300	0.300	21.567	0.379	22.067	0.551	23.833	0.306
510	3	28.400	0.265	21.833	0.321	22.400	0.600	24.167	0.351
515	3	28.633	0.208	22.133	0.321	22.667	0.503	24.433	0.404
520	3	28.800	0.265	22.400	0.361	22.967	0.603	24.700	0.361
525	3	29.033	0.289	22.633	0.321	23.300	0.557	24.933	0.404
530	3	29.300	0.265	23.033	0.493	23.667	0.451	25.267	0.416
535	3	29.467	0.231	23.200	0.361	23.933	0.451	25.500	0.529
540	3	29.700	0.346	23.467	0.306	24.167	0.451	25.700	0.529
545	3	29.667	0.321	23.500	0.361	24.233	0.503	25.767	0.586
550	3	29.867	0.321	23.767	0.306	24.467	0.451	25.967	0.666
555	3	30.167	0.321	24.100	0.361	24.833	0.503	26.300	0.693
560	3	30.400	0.361	24.367	0.379	25.100	0.500	26.600	0.693
565	3	30.600	0.361	24.600	0.361	25.400	0.500	26.867	0.666
570	3	30.767	0.321	24.867	0.379	25.667	0.551	27.133	0.723
575	3	31.067	0.321	25.433	0.404	26.000	0.500	27.467	0.666
580	3	31.267	0.321	25.533	0.351	26.300	0.500	27.733	0.643
585	3	31.800	0.346	26.033	0.404	26.867	0.551	28.300	0.700
590	3	31.833	0.289	26.233	0.404	27.033	0.503	28.467	0.666
595	3	31.867	0.321	26.267	0.379	27.100	0.557	28.567	0.666
600	3	32.067	0.321	26.533	0.321	27.367	0.551	28.867	0.577

WL: Wavelength (nm); N: Number of replications; T: Transmittance (%) and SD: Standard Deviation.

	F <sub>rc</sub>	M <sub>bf</sub>	M <sub>af</sub>	M <sub>ol</sub>	D <sub>fm</sub>	H <sub>dx</sub>
Replications	(N)	(g)	(g)	(g)	(mm)	(N/mm)
1		28.9	28.64	0.26	55.07	29.05
2	1600	28.9	28.11	0.79	59.66	26.82
3		28.9	28.54	0.36	54.89	29.15
Mean	±	<b>28.9</b> ±	28.43 ±	0.47 ±	56.54 ±	28.34 ±
SD		0.00	0.28	0.28	2.70	1.32
1		28.9	26.61	2.29	66.8	47.9
2	3200	28.9	25.99	2.91	67.36	47.51
3		28.9	26.04	2.86	62.62	51.1
Mean	±	<b>28.9</b> ±	26.21 ±	<b>2.69</b> ±	65.59 ±	<b>48.84</b> ±
SD		0.00	0.34	0.34	2.59	1.97
1		28.9	24.96	3.94	64.55	74.36
2	4800	28.9	25.21	3.69	67.68	70.92
3		28.9	24.55	4.35	72	66.67
Mean ±		<b>28.9</b> ±	24.91 ±	<b>3.99</b> ±	68.08 ±	70.65 ±
SD	SD		0.33	0.33	3.74	3.85

Appendix 8. Control measurements repeated thrice for each force.

SD: Standard Deviation;  $F_{rc}$ : Force;  $M_{bf}$ : Mass of sample before compression test, and  $M_{af}$ : Mass of sample (cake) after compression test,  $M_{ol}$ : Mass of oil;  $D_{fm}$ : Deformation and  $H_{dx}$ : Hardness.

Appendix 9. Relaxation measurements repeated thrice for the control temperature of 22 °C for 23 minutes at a maximum force and optimal factors.

	F <sub>rc</sub>	$M_{bf}$	M <sub>af</sub>	M <sub>ol</sub>
Replications	(N)	(g)	(g)	(g)
1		28.9	24.46	4.44
2	4.8*	28.9	25.56	3.34
3		28.9	24.95	3.95
Mean	± SD	$28.9 \pm 0.00$	$24.99 \pm 0.55$	$3.95 \pm 0.55$
1		28.9	20.68	8.22
2	1 0**	28.9	20.56	8.34
3	4.0	28.9	20.47	8.43
Mean	± SD	$28.9 \pm 0.00$	$20.57 \pm 0.11$	8.33 ± 0.11
1		28.9	22.15	6.75
2	4.8***	28.9	22.46	6.44
3		28.9 22.22		6.68
Mean	± SD	$\textbf{28.9} \pm \textbf{0.00}$	$22.28 \pm 0.16$	$6.62 \pm 0.16$

SD: Standard Deviation; \* Control temperature of 22 °C; \*\* Optimized factors  $(F_{rc}: +1(4.8\text{kN}); H_{tp}: +1(40 \text{ °C}) \text{ and } H_{tm}: +1(30 \text{ min}));$  \*\*\* Optimized factors

( $F_{rc}$ : +1(4.8kN);  $H_{tp}$ : +1(60 °C) and  $H_{tm}$ : +1(60 min);  $F_{rc}$ : Force;  $M_{bf}$ : Mass of sample before compression test, and  $M_{af}$ : Mass of sample (cake) after compression test, and  $M_{ol}$ : Mass of oil.

	<b>F</b> <sub>rc</sub>	H <sub>tv</sub>	H <sub>tm</sub>	M <sub>bf</sub>	M <sub>af</sub>	M <sub>ol</sub>	$D_{fm}$	H <sub>dx</sub>
Run	(kN)	(°C)	(min)	(g)	(g)	(g)	(mm)	(N/mm)
1	-1(1.6)	-1(40)	0(45)	28.9	28.48	0.42	48.01	33.33
2	1(4.8)	-1(40)	0(45)	28.9	21.09	7.81	66.65	72.02
3	-1(1.6)	1(80)	0(45)	28.9	28.19	0.71	50.00	32.00
4	1(4.8)	1(80)	0(45)	28.9	23.19	5.71	70.74	67.85
5	-1(1.6)	0(60)	-1(30)	28.9	28.42	0.48	53.95	29.66
6	1(4.8)	0(60)	-1(30)	28.9	21.96	6.94	74.81	64.16
7	-1(1.6)	0(60)	1(60)	28.9	28.34	0.56	53.87	29.70
8	1(4.8)	0(60)	1(60)	28.9	23.27	5.63	75.67	63.43
9	0(3.2)	-1(40)	-1(30)	28.9	25.05	3.85	64.88	49.32
10	0(3.2)	1(80)	-1(30)	28.9	24.86	4.04	64.81	49.37
11	0(3.2)	-1(40)	1(60)	28.9	25.69	3.21	66.84	47.88
12	0(3.2)	1(80)	1(60)	28.9	25.91	2.99	60.06	53.31
13	0(3.2)	0(60)	0(45)	28.9	25.28	3.62	68.55	46.68
14	0(3.2)	0(60)	0(45)	28.9	25.58	3.32	68.90	46.44
15	0(3.2)	0(60)	0(45)	28.9	25.49	3.41	68.01	47.05
16*	0(3.2)	0(60)	0(45)	28.9	25.25	3.65	63.18	50.65
17*	0(3.2)	0(60)	0(45)	28.9	25.42	3.48	58.40	54.79

Appendix 10. Compression data of the operating factors combination.

 $F_{rc}$ : Force;  $H_{tp}$ : Heating temperature;  $H_{tm}$ : Heating time;  $M_{bf}$ : Mass of sample before compression test,  $M_{af}$ : Mass of sample (cake) after compression test,  $D_{fm}$ : Deformation and  $H_{dx}$ : Hardness and \* Removed from the regression analysis due to the significance of the lack of fit value.