CZECH UNIVERSITY OF LIFE SCIENCES PRAGUE

Department of Mechanical Engineering

MASTER'S THESIS

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Application of response surface methodology for optimizing the processing factors of sesame seeds oil extration in linear pressing

Supervisor

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DIPLOMA THESIS ASSIGNMENT

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Agricultural Engineering

Thesis title

Application of response surface methodology for optimizing the processing factors of sesame seeds oil extraction in linear pressing.

Objectives of thesis

(i) To determine the percentage oil content of the sesame seeds using the Soxhlet extraction procedure.
(ii) To describe the force-deformation curve characteristics of sesame seeds under different processing factors.
(iii) To determine the response surface regression models for estimating the percentage oil yield, oil expression efficiency, and energy demand of sesame seeds as a function of different processing factors.
(iv) To validate the optimal processing factors for estimating the percentage oil yield, oil expression efficiency, and energy demand of sesame seeds.
(v) To describe the spectral curves and/or to determine the physicochemical properties of sesame seeds oil under different pretreatment conditions.

Methodology

The experiment will be conducted at the laboratory of the Mechanical Department of the Faculty of Engineering. A universal compression testing machine (ZDM 50, Czech Republic) will be used for the compression tests of sesame seeds by applying the Box-Behnken Design of the experiment (response surface methodology). The initial pressing height of the sesame seeds will be measured at 60 mm using the vessel diameter of 60 mm. The processing factors: forces, speeds, and heating temperatures; with each factor set at three levels will be considered. The moisture content of the sesame seeds will be determined using the conventional oven method. The data will be analyzed statistically using STATISTICA software (version 13).

Code for compiling the Master Thesis

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The proposed extent of the thesis

60-70

Keywords

Oil-bearing crops, linear compression process, oil extraction methods, regression models

Recommended information sources

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ABSTRACT

The Master's Thesis examined bulk sesame seeds under linear pressing involving a universal compresson testing machine and pressing vessels of diameters 30, 60 and 80 mm with a plunger. The bulk sesame seeds was measured at a pressing height of 100 mm and compressed at pressing speeds of 4 and 5 mm/min. Three separate experiments were conducted: experiment one evaluated the effect of pressing force on oil output parameters using the pressing vessel of diameter 30 mm at a constant pressing speed of 4 mm/min; experiment two investigated the effect of pressing vessel diameters on oil output parameters at constant pressing speed of 5 mm/min and experiment three analyzed the optimal input factors (force, heating temperature and heating time) and their determined parameters (oil yield, oil expression efficiency and energy) using the pressing vessel diameter 30 mm at a constant speed of 4 mm/min. The input factors were set at three levels each. The design of the experiment three was based on the Box-Behnken and response surface approaches. The data obtained were statistically analyzed by employing the correlation, ANOVA and response surface regression analyses. The results show that oil yield and oil expression efficiency were not statistically significant ($p > 0.05$) with the increase in diameter of the pressing vessel whereas the energy and stress parameters were statistically significant ($p < 0.05$). The increase in pressing force significantly increased the oil output parameters. The regression models described for the determined parameters were statistically significant. The optimized input factors (force: 4.8 kN (+1), heating temperature: 40 (-1) and heating time: 45 min (0)) and desirability values between 0.999 and 1 were the found for the parameters (oil yield, O_{yd}(%) oil expression efficiency, O_{exp} (%) and energy, E_{df}(J). In future research, there is the need to validate the regression models established in this study using the compression machine of a maximum load of 500 kN and a pressing vessel of diameter 60 mm at a pressing speed of 4 mm/min.

KEYWORDS: Oil-bearing crops, linear compression process, oil extractoin methods, regression models.

DECLARATION

I hereby declare that this Master's Thesis '**Application of response surface methodology for optimizing the processing factors of sesame seeds oil extration in linear pressing**' is the result of my own work and that it has not been submitted to this University or any institution for a degree. All references, however, used in the development of the work have been dully acknowledged in the text and provided in the list of references.

In Prague   Date:

Rachid Mahroum

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I would like to also thank my parents and siblings for their love and prayers throughout my entire life.

And I would like to thank my friends for being there as always

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1 INTRODUCTION

Sesame (*Sesamum indicum* L.) belong to the family of Pedaliaceae (Lammari et al., 2023). It is a small, seeded plant which has been grown for more than 6000 years, and it is native to the Middle East and Africa (Adatia et al., 2022; Hussain, Ul-Allah and Farooq, 2023). The oilseed plant is used in various aspects of daily life such as food, feed and cosmetics, and it is an important medicinal and edible homologous food. Sesame seeds are rich in vitamin B-complex, dietary fibre, oil, protein, antioxidants, and minerals; especially, iron, calcium, phosphorus, zinc, and magnesium (Hussain, Ul-Allah and Farooq, 2023). The crop has the potential to sustainably support the livelihoods of poor farmers since it can be grown in different soil types and can as well meet the increasing oil demand (Pandey et al., 2020). Sesame contains 50–60% oil of high stability due to the presence of natural antioxidants such as sesamolin, sesamin and sesamol (Anilakumar et al., 2010; Pandey et al., 2020). The sesame meal, a by-product after oil extraction, contains 35–40% protein which is a major protein source used to feed animals (Onsaard, Pomsamud and Audtum, 2010; Sharma and Singh, 2016). The international demand and market for sesame were USD 6,559 million in 2018-19 which has been projected to reach beyond USD 7,244 million by the year 2024 (Pandey et al., 2020). The major sesame production and consumption countries are the United States, India and China (He et al., 2023). In Western countries sesame is widely consumed in the form of sesame oil, paste or whole sesame seed (Gou et al., 2022). Recently, it has been reported that sesame seeds exhibit a potential for protection from COVID-19 (Allam et al., 2021; Lammari et al., 2023).

The response surface methodology is a statistical technique for designing experiments, developing models, evaluating the effects of processing factors, reducing the number of experimental runs and identifying possible interactions (Sandhu et al., 2020; Asfaw, Tafa and Satheesh, 2023; Haridy et al., 2023; Kamal et al., 2023; Sirisangsawang and Phetyim, 2023). It is an easy and effective tool compared to other statistical techniques available for optimization (Sharma and Singh, 2016; Sandhu et al., 2020). In linear pressing, the response surface technique has been used to determine the optimal operating factors and responses for bulk pumpkin seeds and rapeseeds (Kabutey et al., 2020; Rachid, 2020; Demirel et al., 2021; Demirel et al., 2022).

Therefore, to obtain the optimal oil extraction conditions for processing sesame seeds, the response surface methodology was used to assess the interactive effect of the processing factors: compression force, heating temperature and heating time on the oil yield, oil expression efficiency and energy of sesame seeds under linear pressing.

1.1 Research problem statement

Sesame remains a neglected crop and is mostly grown in marginal lands despite its nutritional and medicinal value (Hussain, Ul-Allah and Farooq, 2023). In recent years, the health food applications of sesame are increasing (Wei et al., 2022). In the linear pressing process, there is a limited study conducted on bulk sesame seed oil extraction based on response surface methodology. The linear pressing process provides a better understanding or a visualization of the mechanical screw or expeller pressing of vegetable oil extraction; where the latter process needs to be improved for a higher percentage oil yield and lower percentage residual oil in the seedcake/meal with the corresponding efficient energy utilization which is both dependent not only on the screw press configuration but also the processing factors and the oil-bearing material (Herak et al., 2010; Karaj and Muller, 2011; Savoire and Lanoiselle, 2013; Romuli et al., 2017; Bogaert et al., 2018; Huang et al., 2019).

1.2 Objectives

The objectives of the Diploma Thesis are to:

(i) determine the percentage oil content of the sesame seeds using the Soxhlet extraction procedure.

(ii) describe the force-deformation curve characteristics of sesame seeds under different processing factors.

(iii) determine the response surface regression models for estimating the percentage oil yield, oil expression efficiency, and energy demand of sesame seeds as a function of different processing factors.

(iv) validate the optimal processing factors for estimating the percentage oil yield, oil expression efficiency, and energy demand of sesame seeds.

(v) describe the spectral curves and/or to determine the physicochemical properties of sesame seeds oil under different pretreatment conditions.

2 LITERATURE REVIEW

2.1 A general overview of oilseeds

In the food industry, oilseeds are important sources for extracting vegetable oils which are processed in large quantities and utilized in many applications ranging from food, feed and oleochemicals (Zhang et al., 2021; Dunford, 2022). Most vegetable oils can be used either for cooking or fuel and diesel production (Statista Research Department, 2023). Around the world, the major vegetable oils include palm, soybean, rapeseed, sunflower seed, palm kernel, peanut, cottonseed, coconut and olive oil (Zhang et al., 2021). Worldwide, in 2021/2022, vegetable oil production amounted to 208.81 million metric tons, and it is forecast to increase to more than 217 million metric tons in the 2022/2023 marketing year. In 2021/2022, the total consumption of the major vegetable oils mentioned above amounted to 204.17 million metric tons whereas the forecast for 2022/2023 is 213.08 million metric tons (Statista Research Department, 2023). The exports of oilseeds and oilseed products by region, world oilseed prices and the share of vegetable oil used for biodiesel production are shown in Figures 1, 2 and 3 respectively (OECD-FAO Agriculture Outlook 2020-2029).

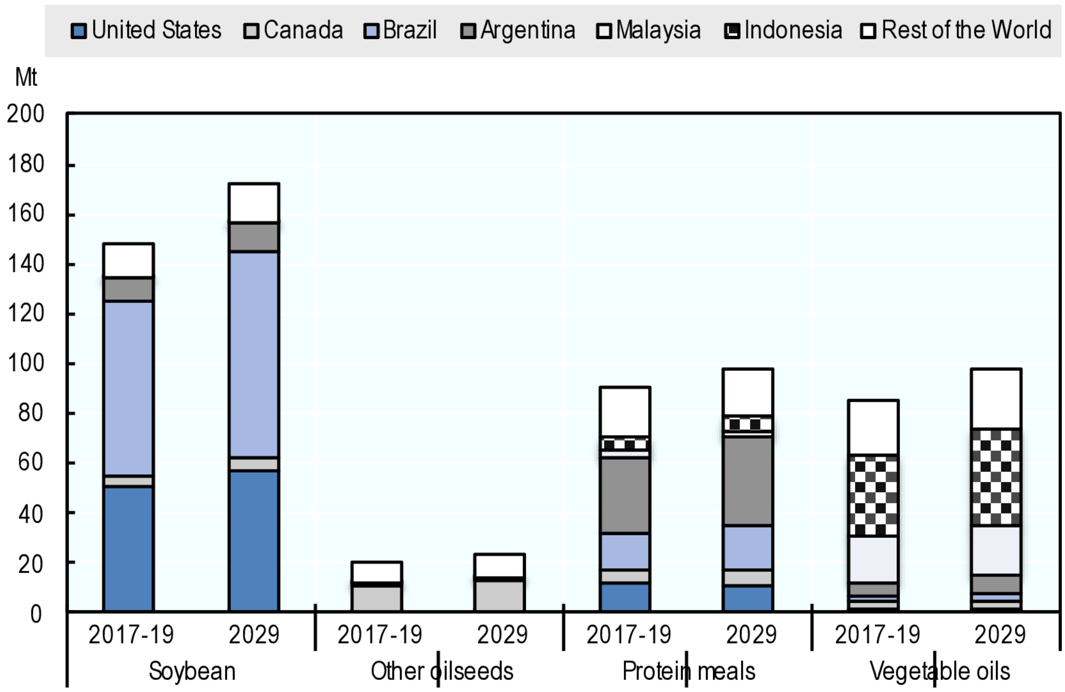


Figure 1. Exports of oilseeds and oilseed products by region (Source: OECD/FAO (2020), "OECD-FAO Agricultural Outlook", OECD Agriculture statistics (database), http://dx.doi.org/10.1787/agr-outl-data-en.

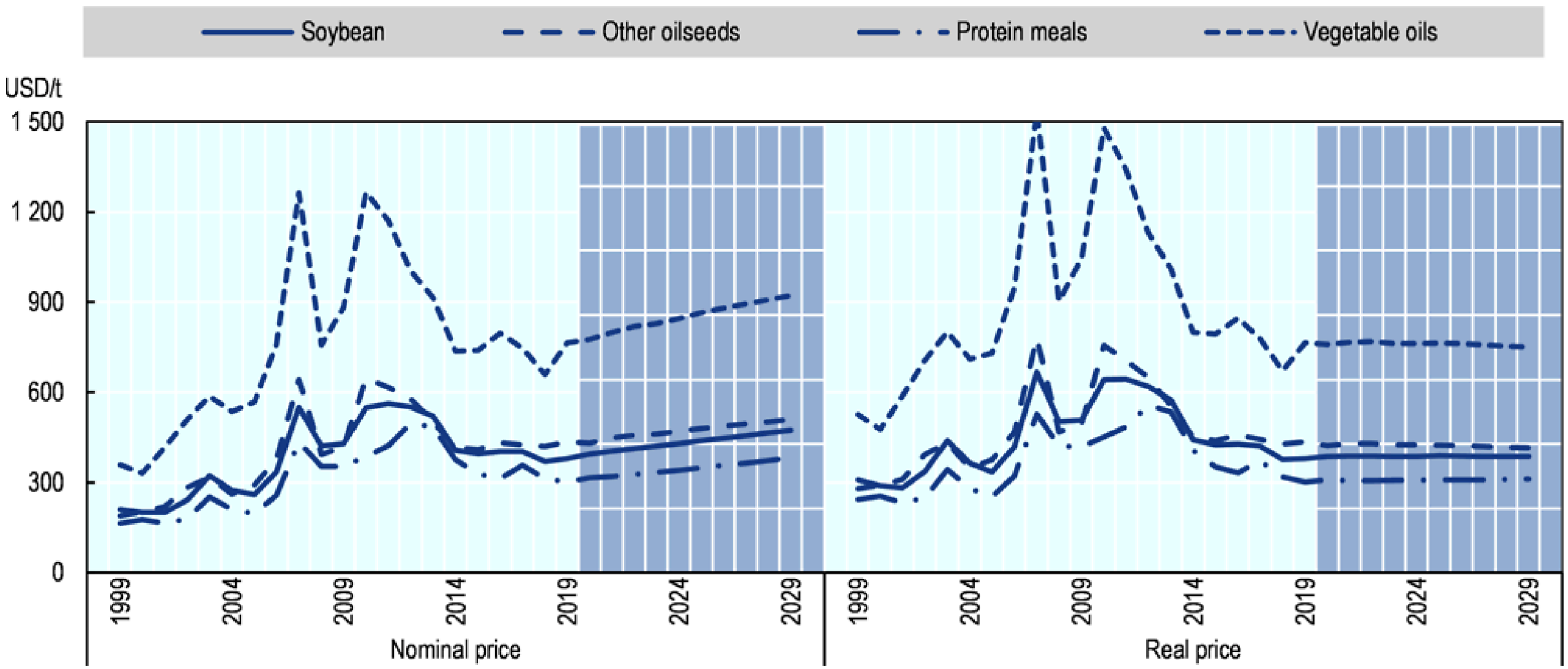


Figure 2. Evolution of world oilseed prices (Source: OECD/FAO (2020), "OECD-FAO Agricultural Outlook", OECD Agriculture statistics (database), http://dx.doi.org/10.1787/agr-outl-data-en).

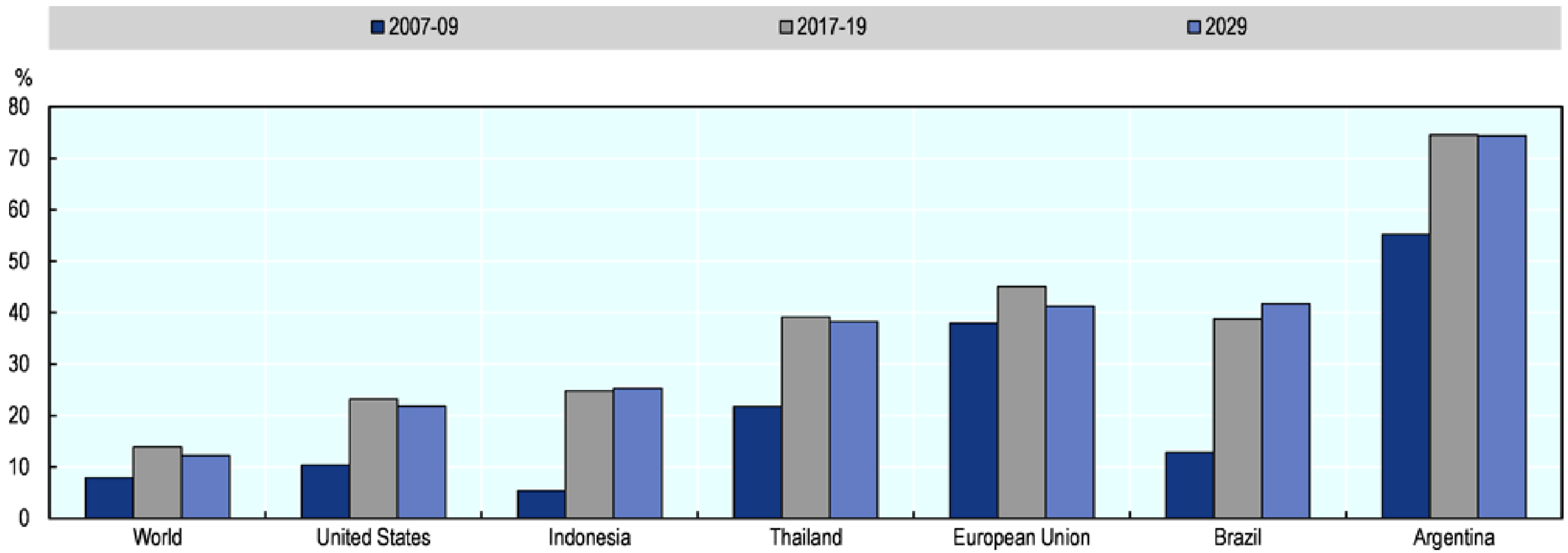


Figure 3. Share of vegetable oil used for biodiesel production (Source: OECD/FAO (2020), "OECD-FAO Agricultural Outlook", OECD Agriculture statistics (database), http://dx.doi.org/10.1787/agr-outl-data-en).

2.2 Physical, mechanical and chemical properties of oilseeds

To design and fabricate the equipment related to the process, the physical, mechanical and chemical properties of fruits, nuts and kernels/seeds are important design parameters and useful for the classification of seed quality (Garnayak et al., 2008; Karaj and Muller, 2010).

2.2.1 Physical properties

The physical properties reported on oilseeds/kernels include unit mass, arithmetic diameter, geometric diameter, volume sphericity, bulk density, solid/true density, porosity, surface area, specific surface area, coefficient of friction and angle of repose (Karaj and Muller, 2010). According to Karaj and Muller (2010), there was a positive correlation between fraction mass and terminal velocity of *Jatropha curcas* seeds. For separating seeds heavier than 0.35 g, the authors indicated that it was necessary to apply a terminal velocity of 8.1 m/s and that of bigger masses of jatropha seeds of 0.52g and 0.69 g, terminal velocities of 9.2 and 10.8 m/s were applied. The authors further mentioned that surface area increased with unit mass, but the specific surface area did not increase with unit mass. Again, the angle of repose of seeds and kernels decreased with increasing unit mass. The authors explained this trend that smaller seeds and kernels show higher cohesion than larger ones. Garnayak et al. (2008) also studied the moisture-dependent physical properties of jatropha seed. The authors reported that each principal dimension (length, width and thickness) and average diameters (arithmetic and geometric means) were linearly dependent on moisture content with high correlation. In addition, from their study; the sphericity, seed mass and surface area increased along with the moisture content increment. Whereas bulk density decreased with the moisture content of jatropha seeds, the true density and porosity values increased with moisture content increment. The authors explained that the increase in true density with moisture content might be due to the relatively lower true volume as compared to the corresponding mass of the seed attained due to the adsorption of water. The angle of repose and static coefficient of friction also increased with moisture content. For the static coefficient of friction, the authors explained that at higher moisture content, water is present in the seed offering a cohesive force on the surface of contact.

2.2.2 Mechanical properties and force-deformation curve characteristics

Mechanical properties such as rupture force, deformation at the rupture point, deformation ratio at the rupture point, hardness and energy are useful for designing deshelling equipment and oil extractor (Sirisomboon et al., 2007; Karaj and Muller, 2010). In these studies, the authors reported that the force needed to rupture a nut was higher than the kernel since the nut has a hard shell whereas the kernel has a soft texture. Deformation at the rupture point of a fruit was higher than the kernel. Deformation at the rupture point and deformation ratio at the rupture point of kernels showed higher values than seeds indicating that seeds need lower compression to rupture than kernels. The energy for rupture for seeds was higher than for the kernels. For the force-deformation curve characteristics, Gupta and Das (2000) described the force-deformation characteristics as a function of moisture content for sunflower seed deformed at 1 mm/min under different loading orientations. An increase in force increased the deformation but the force-deformation curve was no longer smooth rather it showed a high undulation or serration effect. Similar behaviour was reported by Divisova et al. (2014) for bulk rapeseeds and sunflower seeds using different pressing vessel diameters at a deformation rate of 60 mm/min and Kabutey et al. (2011) on bulk jatropha seeds with a pressing vessel diameter of 76 mm at a pressing rate of 60 mm/min.

2.2.3 Chemical properties/proximate compositions

Chemical properties namely oil content, moisture content, crude protein and ash content have been reported on some oilseeds/kernels/nuts such as jatropha seeds and kernels (Karaj and Muller, 2010). In their study, for instance, the authors indicated that the oil content of kernels was higher than that of seeds due to the fact the oil-free shells were removed from the kernels. The authors further said that the oil content of kernels and seeds correlated with unit mass indicating that seeds and kernels with low unit mass are immature and have not reached their potential oil content at harvest. Again, the authors reported that the moisture content of seeds and kernels decreased with unit mass indicating that heavy seeds or kernels have less water than light seeds or kernels. Regarding crude protein, the authors mentioned that crude protein in kernels was higher than that observed in the seeds. On the other hand, the ash content of kernels was observed to be higher than the seeds indicating that kernels contain more inorganic matter than the seeds. Musa, Endes and Er (2010) also reported the proximate compositions of some oil-bearing materials (flax, soybean, rice bran, peanut, grape, sesame, almond, sorghum and pistachio). Among these materials, the oil content ranged from 13.7 ± 1.6 to 53.7 ± 5.7%; crude protein ranged from 9.7 ± 1.3 to 22.3 ± 1.2%; crude ash ranged from 0.43 ± 0.13 to 16.7 ± 0.11% and crude fibre ranged from 2.3 ± 0.1 to 28.9 ± 1.7%.

2.2.4 Other studies

Several authors have also studied the physical, mechanical and chemical properties of oilseeds. To mention but a few include Sirisomboon and Kitchaiya (2009) on the physical properties of jatropha kernels after heat treatment; Kibar and Ozturk (2008) on the physical and mechanical properties of soybean; Coskuner, Ersan and Karababa (2007) on the physical properties of coriander seeds (*Coriandrum sativum* L.); Burubai et al. (2007) on the strength properties of African nutmeg (*Monodora myristica*) and the effect of temperature and moisture content; Lysiak (2007) on the fracture toughness of peas; Izli, Unal and Sincik (2009) on the physical and mechanical properties of rapeseed at different moisture content; Gely and Santall (2000) on the effect of some physical and chemical properties of oilseeds on drying kinetics parameters and Kaliniewicz et al. (2021) on selected physical and mechanical properties of hemp seeds.

2.3 Compression and relaxation processes of bulk oilseeds

In the literature, some of the studies on the compression and relaxation processes of bulk oilseeds include Akangbe and Herak (2017) on camelina, pumpkin and sesame bulk seeds; Akangbe and Herak (2018) on sesame bulk seeds; Gurdil et al. (2020) on bulk sunflower seeds. Kabutey et al. (2021) on bulk pumpkin seeds and Demirel et al. (2021) and (2022) on bulk rapeseeds. In these studies, the oil yield, oil point pressure, compressive stress, repetitive strain, optimum expression parameters (input factors and responses) and other mechanical properties were determined. According to the above-mentioned authors, in linear pressing, the compression process is where the maximum oil output can be achieved with the corresponding energy demand whereas the relaxation process is where the residual oil is obtained within a given time frame at a constant strain. In the other words, the compression process can be described as the dependency between the force and deformation of the bulk oilseeds whereas the relaxation process is described as the relaxation force and time. These two processes are useful for understanding the amount of oil that can be recovered from an initial mass of the bulk oilseeds and the residual oil remained in the seedcake.

2.4 Oil extraction methods of oilseeds

Oilseed extraction techniques affect oil yield and thus determine the concentration and types of extracted minor lipid components in the oil (Liu et al., 2020). In the Industry, oil extraction techniques used are solvent extraction, screw or expeller pressing, enzyme-assisted aqueous extraction and supercritical fluid extraction (Nde and Foncha, 2020; Cai et al., 2021). Enzyme-assisted aqueous extraction and supercritical fluid extraction are the most efficient and environmentally friendly; however, they have low capacity and higher equipment and reagent or enzyme costs (Rosenthal, Pyle and Niranjan, 1996; Cai et al., 2021). Therefore, solvent extraction and screw-pressing methods are most utilized in the industry (Cai et al., 2021). In the mechanical oil extraction method also known as cold pressing extraction, the raw material is placed between two barriers where the volume available to the raw material is reduced by pressing and expelling the oil (Sorita et al., 2023). The mechanical method has a low oil yield because the oil is physically forced out of the vegetable matrix, leaving a cake/meal which is a by-product with significant residual oil content between 15 and 20% (Sorita et al., 2023). For a better oil yield, the cake is passed through a solvent extraction step (Yang et al., 2021; Yang et al., 2021; Sorita et al., 2023). The solvent extraction uses hexane due to its high oil solubility which leads to a high oil yield and easy oil recovery (Sorita et al., 2023). It is more efficient than mechanical pressing because the solvent can penetrate the raw material through the vegetable matrix pores and walls and solubilizes the oil (Sorita et al., 2023). However, organic solvents are environmentally unfriendly, flammable and can evaporate, causing dizziness, nausea and headache to operators (Nde and Foncha, 2020; Punia et al., 2021; Sorita et al., 2023).

Emerging and green oil extraction technologies are ultrasound and microwave-assisted extractions (Ferreira et al., 2022). In ultrasound-assisted extraction, the application of sound waves results in compression and expansion cycles, and therefore, substantial amounts of energy are released leading to plant tissue disruption. In microwave-assisted extraction, the energy from microwaves is converted to heat leading to physical modification of the matrix sample and consequently improving the extraction efficiency (Azmir et al., 2013; Danlami et al., 2014;

Pereira et al., 2017; Satriana et al., 2019; Ferreira et al., 2022). Other emerging oil extraction technologies are high pressure, ohmic heating and pulse electric fields which are being studied and showing great potential to extract vegetable oils from nuts/oilseeds (Ferreira et al., 2022).

2.5 Effect of processing factors on the oil extraction process

2.5.1 Pressure

The pressure change in a press is designed by changing the opening between the throttling element and the barrel. The smaller the opening, the maximum pressure is recorded along the barrel. But an increase in pressure increases the temperature along the barrel which consequently affects the quality of the oil. Most importantly, the more pressure is applied the higher the oil extraction yield (Karaj and Muller, 2011; Savoire, Lanoiselle and Vorobiev, 2013).

2.5.2 Temperature

The rise in temperature causes a decrease in oil viscosity and can alter the cellular structure and plasticity of the raw material. The seed temperature increase results in an increase in the barrel temperature. The oil extraction yield increases with an increase in temperature (Deli et al., 2011; Karaj and Muller, 2011; Savoire, Lanoiselle and Vorobiev, 2013).

2.5.3 Screw rotation speed

The rotation speed acts with a change in pressure and temperature in the barrel. Increasing the screw rotation speed causes a pressure decrease and temperature increase. Screw rotation speed can be related to the oilseed deformation velocity depending on screw geometry. Screw rotation speed in continuous pressing corresponds to the speed with which the piston compresses the seeds in hydraulic pressing (Deli et al., 2011; Karaj and Muller, 2011; Savoire, Lanoiselle and Vorobiev, 2013).

2.5.4 Moisture content

The moisture content of oilseeds is a key factor controlling solid-liquid expression. An increase in moisture content leads to a decrease in the oil yield. Variations in the moisture content of seeds affect the capacity of presses. An optimum moisture content of 5% for rapeseed and between 9 and 11% for flaxseed has been reported. An increase in moisture content can also result in an increase in the maximum pressure and a decrease in barrel temperature. The moisture content of seeds during the pressing process also affects the quality of the oil. The decrease in water content causes an increase in chlorophyll content in oil as well as the content of phospholipids (Vadke and Sosulski, 1998; Zheng et al., 2003; Evangelista and Cermak, 2007; Deli et al., 2011; Karaj and Muller, 2011; Savoire, Lanoiselle and Vorobiev, 2013).

2.5.5 Raw material and pre-treatments

The characteristics of the raw material (variety, moisture content, hull and dehulled seeds) and the pre-treatments (dehulling, flaking and cooking) also affect the oil extraction process. For instance, Zheng (2003) indicated hull is considered an uncompressible matter and it is responsible for the formation of drainage pores within the cake. Thinner hulls are more sensitive to compression and allow less pore formation. The effect of cooking temperature and time on oil yield depends on the raw material processed and the moisture content. Wiesenbom et al. (2001) reported that for crambe seeds, the change in cooking temperature led to differences in oil yield of 8% and 28% for 5 and 20 min of cooking. Shelling or dehulling according to seed type is used to separate the seed almond-rich oil, usually cotyledons of a woody shell (hull and shell) that surrounds it. Dehulling prevents oil absorption by the shell which would be responsible for losses during further processing. It may also be considered for seeds such as soybeans to adjust the protein concentration in the meal at the request of the feed chain (Lanoiselle and Vorobiev, 2013). On the other hand, flaking or flattening is used to partially break the seeds and increase their surface area to facilitate solvent extraction. Khan and Hanna (1984) and Singh et al. (1984) reported that the oil yield obtained by hydraulic pressing from the flattened soybean and sunflower seeds was higher than that obtained from whole seeds, while for the dehulled seeds, a lower oil yield was observed.

2.6 Effect of roasting on oil yield and quality of oils

Oven roasting or dry air roasting is the most common heat transfer method with low operating costs which is achieved by combining heat transfer mechanism namely radiation from the heat source, convection from hot air and conduction from surfaces in direct contact with oilseeds (Zou et al., 2018; Sruthi et al., 2021). Oilseed roasting facilitates the release of oil and thus increases the oil extraction efficiency. For mechanical oil extraction, it has been cited in Zhang et al. (2021) that the oil yield of rapeseed oil increased from 15% to 25% (Azadmard-Damirchi et al., 2010), hazelnut oil from 6.1% to 45.3% (Uquiche, Jerez and Ortiz, 2008), peanut oil from 41.17% to 46.28% and for solvent extraction from 47.75% to 55.35% (Suri et al., 2019), black cumin seed oil from 23.4% to 34.3% (for dry air roasting) and 35.1% (infrared roasting (Suri et al., 2019). Liu (2009) cited in Zhang et al. (2021) mentioned the roasting conditions for producing fragrant oils in the industry depending on the oilseed variety. The roasting conditions for rapeseed were at 150–180°C for 30–45 min, peanut at > 180°C for 30–40 min and sesame seed at 200–220°C for 20–40 min. Roasting also affects the nutritional value and oxidative stability of the extracted oil. The effects of roasting on the properties of oils have been reported for some oilseeds such as argan, pumpkin, walnut, rapeseed and chia seed (Durmaz et al., 2010; Vujasinovic et al., 2012; Wroniak et al., 2016; Gao et al., 2019; Ozcan et al., 2019; Zhang et al., 2021). Table 1 summarizes the selected oilseeds, roasting type, measurement indicators and key findings in the literature.

Table 1. Effect of oilseed roasting on the quality of selected oilseed oil.

Oilseeds	Roasting type	Roasting conditions	Measurement indicators	Key findings	References
Sesame	Automatic electric roaster	160, 180, 200, 220, 240 and 260 °C for 10, 20, 30, 40 and 50 min	AV, POV, FA composition, tocols, sesame lignans and PAHs.	- PV and colour increased but the total tocopherols and sesamolin decreased during roasting. - No obvious differences in the FA profile of oils prepared from unroasted and roasted seeds. - Increased roasting temperature or time facilitated the formation of sesamol. - PAHs increased with elevated roasting temperatures and extended times.	Ji et al., 2019 Zhang et al., 2021

Oilseeds	Roasting type	Roasting conditions	Measurement indicators	Key findings	References
Flaxseed	Oven roasting	160, 180, 200 and 220 °C for 8 min	FA composition, tocochromanols, sterols, MRP and OSI	- Phytosterol content increased after roasting. - FA profiles of oils extracted from raw seeds and roasted seeds did not differ. - γ-tocopherol degradation decreased, but the plastochromanol-8 increased at ≥ 200 °C. - Temperature ≥ 200 °C significantly accelerates in situ oil oxidation.	Waszkowiak et al., 2020 Zhang et al., 2021

Oilseeds	Roasting type	Roasting conditions	Measurement indicators	Key findings	References
Walnut kernel	Oven roasting	140, 160 and 180, °C for 5, 10 and 15 min	AV, POV, FA composition, tocopherols phytosterol, squalene, polyphenols, OSI and free radical scavenging capacity.	- Roasting increased the amounts of tocopherols, phytosterols and polyphenols, but decreased the squalene. - SFA decreased, MUFA slightly decreased and PUFA fluctuates after roasting. - OSI and antioxidant capacity of walnut oils increased with intensified roasting. - Roasting was found to be a suitable pre-processing method to produce walnut oil for application in healthcare.	Gao et al., 2019 Zhang et al., 2021

Oilseeds	Roasting type	Roasting conditions	Measurement indicators	Key findings	References
Sunflower seed	Microwave roasting	500 W for 5, 10 and 15 min	Moisture content, density, RI, FFA, POV, ρ-AV, IV, SV, colour, tocopherol contents and FA composition	- Contents of FFA, ρ-AV, SV conjugated diene, conjugated triene, density and colour increased in the oils from sunflower seeds roasted for 10 and 15 min. - IV and tocopherol contents decreased after roasting. - Oleic acid content increased 16-42%, but linoleic acid decreased 17-19%.	Anjum et al., 2006 Zhang et al., 2021

Oilseeds	Roasting type	Roasting conditions	Measurement indicators	Key findings	References
Tree peony seed	Oven roasting	120, 130, 140 and 150 °C for 15 min	Colour, AV, FA composition, phenols, antioxidant capacity, volatiles and benzoic acid	- Roasting resulted in increasing antioxidant capacity and AV. - FA compositions were not affected by different roasting temperatures. - Pyrazines, aldehydes and furans increased after roasting and contributed to a nutty aroma.	Jin et al., 2019 Zhang et al., 2021

FA, fatty acids; AV, acid value, POV, peroxide value; SV, saponification value; IV, iodine value; RI, refractive index; OSI, oxidative stability index; PAHs, polycyclic aromatic hydrocarbons; MRP, Maillard reaction product; *p*-AV; RSA, radical scavenging activity; SFA, Saturated fatty acids and MUFA, Monounsaturated fatty acid.

3 MATERIALS AND METHODS

3.1 Experiments

The experiments were done at the laboratory of the Department of Mechanical Engineering, Faculty of Engineering, Czech University of Life Sciences Prague.

3.2 Samples

Sesame oilseed (hereafter as a sample) was used for the experiment. The samples were purchased from Střední, Prague 6, Czech Republic.

3.3 Determination of sample moisture content

The sample moisture content was determined to be 2.50 ± 0.10 (% w.b.) and 2.56 ± 0.11 (% w.b.) according to Blahovec (2008) as described in equation (1).

$$MC = \left[\left(\frac{m_b - m_a}{m_b}\right) \cdot 100\right] \quad (1)$$

where MC is the percentage of moisture content of the sample (% w.b.), m_a and m_b are the masses of the sample before and after oven drying (g).

3.4 Determination of sample oil content

The sample percentage oil content was determined to be 38.73 ± 2.61 % using the Soxhlet extraction procedure (Niu et al., 2014; Danlami et al., 2015).

3.5 Compression tests of sample

3.5.1 Using MPTest 5.050 machine

The compression device the (MPTest 5.050, Czech Republic) (Figure 1) of a maximum force of 5 kN, was used for two separate experiments: the preliminary experiment and the Box-Behnken design of experiment of different processing factors combination (force, heating temperature and heating time). The preliminary experiment considered the varying forces from 1600 N to 4800 N at a constant speed of 4 mm/min, pressing height of 100 mm and vessel diameter of 30 mm. The Box-Behnken design of the experiment generated 15 experimental runs. The Box-Behnken design of the experiment was the focus of the study, however, additional experiments were done to obtain adequate information where necessary as described in Section 3.5.2.

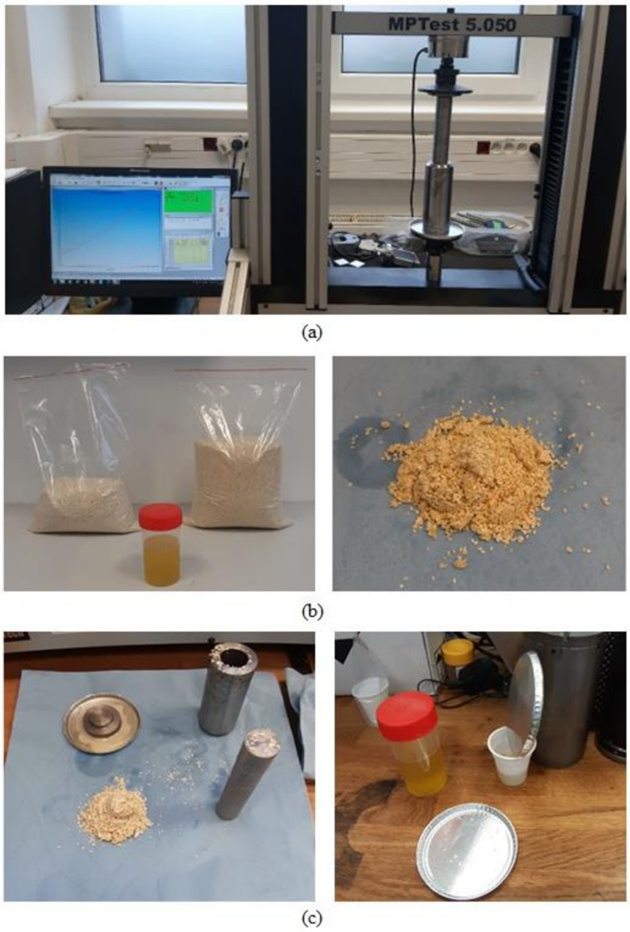


Figure 4. Sesame oil extraction process: (a) compression device; (b) sample after test and oil collected and (c) recovery of the oil after test.

3.5.2 Using Tempos, ZDM 50 machine

Additional experiments were done using the compression device (Tempos, ZDM 50, Czech Republic) (Figures 2 – 5) of a maximum load of 500 kN to examine the effect of different vessel diameters: 30, 60 and 80 mm (Appendix 1) at a constant speed of 5 mm, sample pressing height of 100 mm on the percentage oil yield, oil expression efficiency, energy and stress for processing sesame oilseeds.

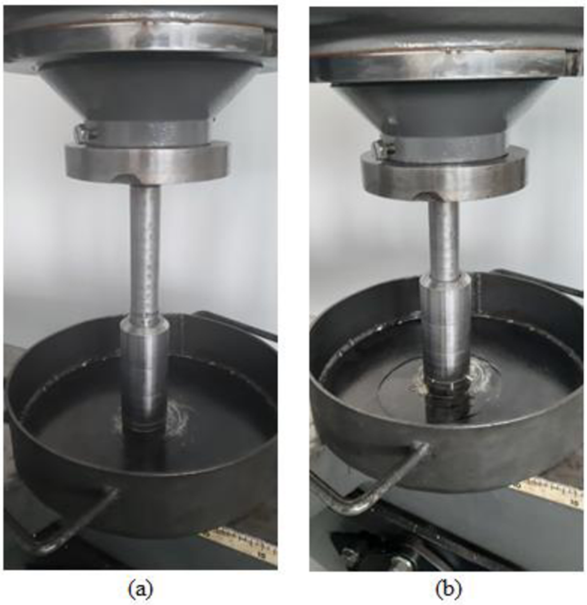


Figure 5. Compression test of bulk sesame seeds at vessel diameter 30 mm; (a) loaded sample before test and (b) unloaded sample after test with the extracted oil.

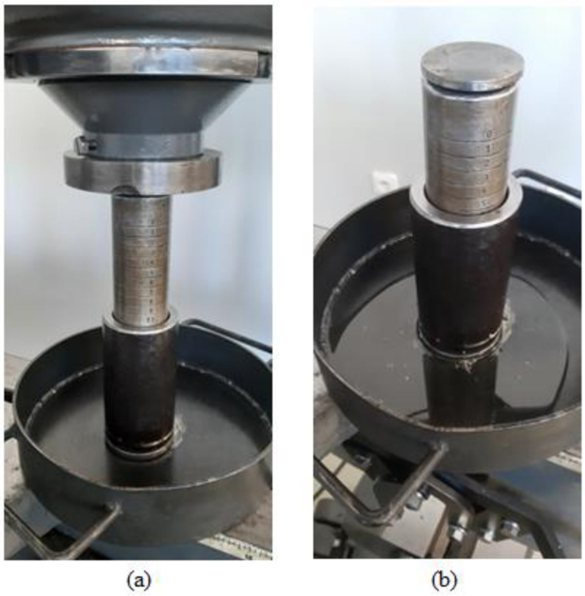


Figure 6. Compression test of bulk sesame seeds at vessel diameter 60 mm; (a) loaded sample before test and (b) unloaded sample after test with the extracted oil.

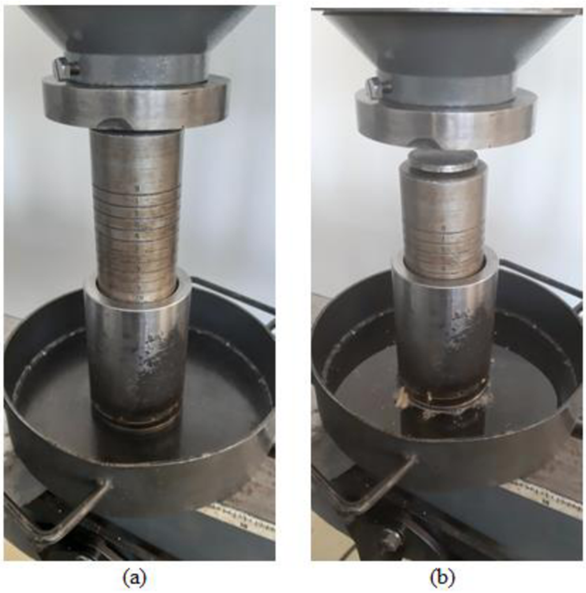


Figure 7. Compression test of bulk sesame seeds at vessel diameter 80 mm; (a) loaded sample before test and (b) unloaded sample after test with the extracted oil.

Figure 8. Sample of sesame seeds: (a) after test, (b) extracted oil in a beaker with the sample after test packed into a plastic bag.

3.6 Determined parameters from the compression tests

3.6.1 Oil yield

The oil yield was determined according to Deli et al., (2011) and Chanioti and Tzia, (2017) as described in equation (2).

$$O_{yd} = \left[\left(\frac{M_{ol}}{M_{is}}\right) \cdot 100\right] \tag{2}$$

where $O_{yd}$is oil yield (%), M_{ol} is the mass of oil determined as the difference between the mass of the seedcake and the initial mass of the sample M_{is} (g).

3.6.2 Oil expression efficiency

The oil expression efficiency was determined according to Hernandez-Santos et al., (2016) as described in equation (3).

$$O_{exp} = \left[\left(\frac{O_{ld}}{O_{sc}}\right) \cdot 100\right] \tag{3}$$

where O_{exp} is the oil expression efficiency (%) and O_{sc} is the percentage of oil content (%) in the sesame seeds sample determined by soxhlet extraction.

3.6.3 Deformation energy

The deformation energy (hereafter as the energy) was determined according to Gupta and Das (2000) and Herak et al., (2015) as described in equation (4).

$$E_{df} = \sum_{n=0}^{n=i-1} \left[\left(\frac{F_{n+1} + F_n}{2} \right) \cdot (x_{n+1} - x_n) \right] \quad (4)$$

where E_{df} is the deformation energy (J), $F_{n+1} + F_n$ and $x_{n+1} - x_n$ are the compressive force (kN) and deformation (mm), n is the number of data points and i is the number of sections in which the axis deformation was divided.

3.6.4 Stress

The stress was determined as the ratio of the force to the cross-sectional area of the pressing vessel according to (Herak et al., 2015) as described in equation (5).

$$\delta_{ss} = \left(\frac{F_{cr}}{A_{er}} \right) \quad (3)$$

where δ_{ss} is the stress (MPa), F_{cr} is the force (N) and A_{er} is the cross-sectional area of the pressing vessel (mm^2). The cross-sectional area of the pressing vessel diameters of 30, 60 and 80 mm were calculated to be 706.86, 2827.43 and 5026.55 mm^2 respectively.

3.7 Experimental design with Box Behnken

3.7.1 Design of the experimental input factors

The experimental input factors were designed using STATISTICA 13 software (Statsoft, 2013). The input factors were the force, heating temperature and heating time with each set at three levels (force: 1600, 3200 and 4800 N; heating temperature: 40, 60 and 80 and heating time: 30, 45 and 60 min).

3.7.2 Coded values of the input factors

The input factors were coded as (-1, 0 and +1) representing the low, centre and high values according to Ocholi et al., (2018) and Witek-Krowiak et al., (2014) described in equation (6).

$$x_i = \frac{X_i - X_0}{\Delta X} \tag{6}$$

where x_i is the coded value of the i^{th} variable, X_i is the uncoded value of the i^{th} test variable and $X_0$is the uncoded value of the ith test variable at the centre point.

3.7.3 Regression models for the determined responses

The regression models describing the Box-Behnken Design of the experiment concerning the determined responses were established according to Huang et al. (2019) as described in equation (6).

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i_{1<j}}^{k} \sum_{j}^{k} \beta_{ij} X_i X_j \tag{7}$$

where Y is the response variable; *i* and *j* are linear and quadratic coefficients; β_0, β_i, β_{ii} and β_{ij} are the regression coefficients in the intercept, linear, quadratic and interaction terms respectively; X_i and X_j are the independent variables and k is the number of factors.

3.8 Statistical analysis

The experimental data were statistically analyzed by employing descriptive statistics, correlation and response surface regression techniques using STATISTICA 13 software (Statsoft 2013). Graphical illustrations were also done by the above-mentioned software and Microsoft Excel.

4 RESULTS AND DISCUSSION

In this study, several experiments were carried-out. The results obtained are presented and discussed.

4.1 Effect of pressing vessel diameters on determined parameters

4.1.1 Mass of oil, oil yield and oil expression efficiency

The results of the determined parameters (mass of oil, oil yield and oil expression efficiency) under the effect of the input factor (diameter of pressing vessel) are given in Table 2. The mass of oil values ranged from 10.38 ± 0.13 to 233.67 ± 1.20 %; the oil yield values ranged from 24.79 ± 0.30 to 21.60 ± 0.40 and the oil expression efficiency values ranged from 64.00 ± 0.78 to 55.77 ± 1.04 %. It was observed that the increase in the diameter of the pressing vessel increased the mass of oil, but oil yield and oil expression efficiency values decreased with the increase in pressing vessel diameter. The results follow the explanation given by Deli et al. (2011) where the authors compared two screw shafts of diameters 8 mm and 11 mm respectively. The authors indicated that the screw shaft with a diameter of 8 mm provided much smaller space for the oilseeds to be pressed in comparison with the screw shaft with a diameter of 11 mm which provided larger space during pressing. This means that the pressure towards the seeds in the smaller pressing vessel diameter was higher than the bigger pressing vessel diameters which received lower pressure during compression. The relationships between the determined parameters with the diameter of the pressing vessels are described in Figures 9 and 10 respectively with the fitting functions (linear and polynomial) indicating high coefficient of determination (R^2) values ranging from 0.752 to 1.

Table 2. Determination of the mass of oil, oil yield and oil expression efficiency of the sample under the effect of pressing vessel diameter.

Tests	Input factor	Sample mass		Determined parameters		
	D_{pv} (mm)	M_{sb} (g)	M_{sa} (g)	M_{ol} (g)	O_{yd} (%)	O_{exp} (%)
1	30	41.88	31.59	10.29	24.57	63.44
2	30	41.88	31.41	10.47	25.00	64.55
Mean ± SD			31.50 ± 0.13	10.38 ± 0.13	24.79 ± 0.30	64.00 ± 0.78
1	60	177.61	137.13	40.48	22.79	58.85
2	60	177.61	142.95	34.66	19.51	50.39
Mean ± SD			140.04 ± 4.12	37.57 ± 4.12	21.15 ± 2.32	54.62 ± 5.98
1	80	298.04	232.82	65.22	21.88	56.50
2	80	298.04	234.51	63.53	21.32	55.04
Mean ± SD			233.67 ± 1.20	64.38 ± 1.20	21.60 ± 0.40	55.77 ± 1.04

D_{pv}: Pressing vessel diameter; M_{sb}: Mass of sample before test; M_{sa}: Mass of sample after test; M_{ol}: Mass of oil; O_{yd}: Oil yield; O_{exp}: Oil expression efficiency and SD: Standard Deviation.

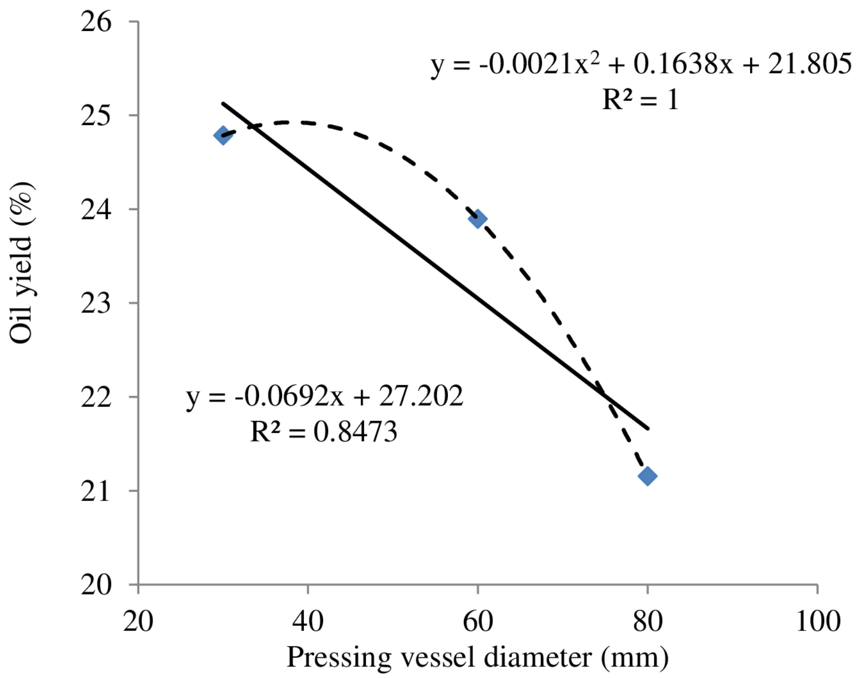
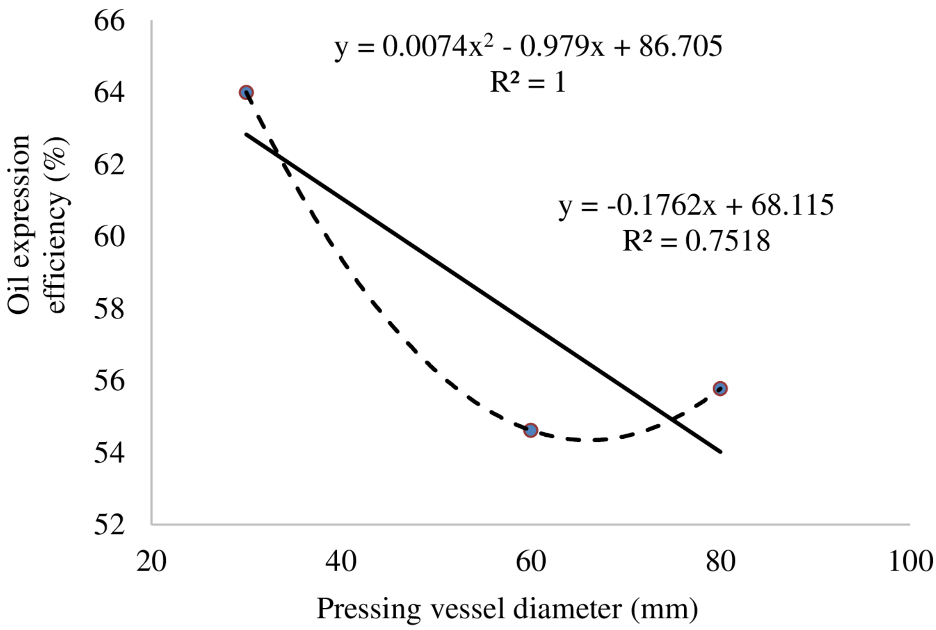


Figure 9. Dependency between oil yield and pressing vessel diameter.



10. Dependency between oil expression efficiency and pressing vessel diameter.

4.1.2 Force, deformation, energy and stress

The mechanical properties (force, deformation, energy and stress) under the effect of the varying pressing vessel diameters are given in Table 3. The increase in diameter of the pressing vessel at a constant sample pressing height of 100 mm increased the force required for obtaining the oil. The deformation values did not linearly correspond to the increase in the diameter of the pressing vessel. The energy and the stress values linearly corresponded to the pressing vessel diameters increment. The relationships between the energy and stress in relation to the diameter of the pressing vessels are described in Figures 11 and 12 respectively with the fitting functions (linear and polynomial) indicating high coefficient of determination (R^2) values ranging from 0.902 to 1. The oil yield, oil expression efficiency, stress and energy are graphically illustrated in Figure 13.

Table 3. Determination of force, deformation, energy and stress of sample under the effect of pressing vessel diameter.

Tests	Input factor	Determined parameters			
	D_{pv} (mm)	F_{cr} (N)	D_{fx} (mm)	E_{df} (J)	δ_{ss} (MPa)
1	30	21064.00	56.11	121.00	29.80
2	30	19899.00	57.91	109.15	28.15
Mean ± SD		20481.50 ± 823.78	57.01 ± 1.27	115.08 ± 8.38	28.98 ± 1.17
1	60	67293.00	59.55	369.05	23.80
2	60	60406.50	56.92	351.28	21.36
Mean ± SD		63849.75 ± 4869.49	58.24 ± 1.86	360.17 ± 12.56	22.58 ± 1.72
1	80	105600.00	56.50	588.51	21.01
2	80	114381.50	57.12	642.36	22.76
Mean ± SD		109990.75 ± 6209.40	56.81 ± 0.44	615.43 ± 38.08	21.88 ± 1.24

D_{pv}: Pressing vessel diameter; F_{cr}: Force; D_{fx}: Deformation; E_{df}: Energy; δ_{ss}: Stress and SD: Standard Deviation.

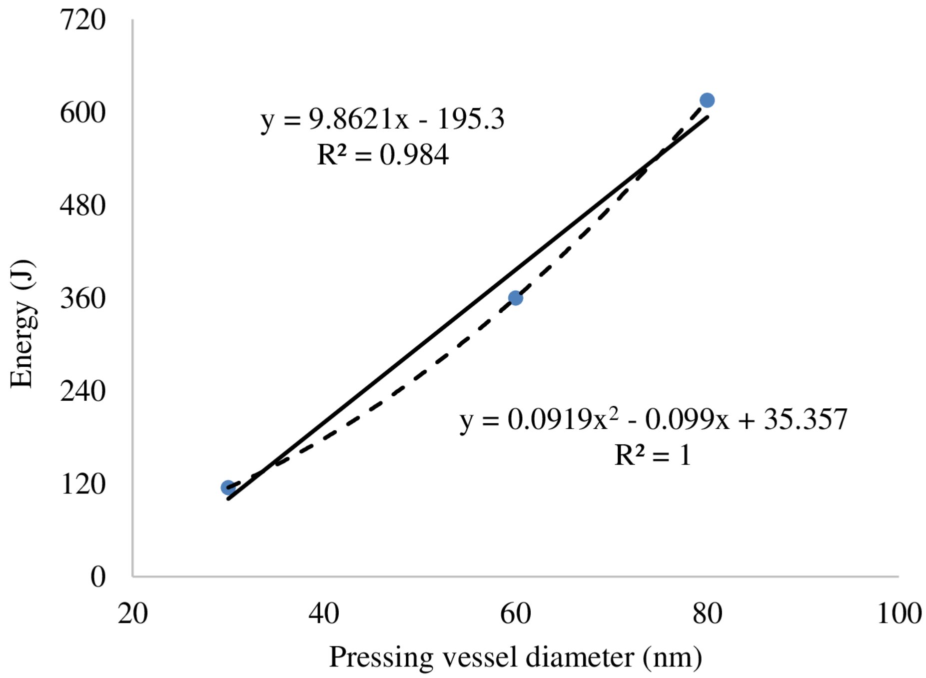


Figure 11. Dependency between energy and pressing vessel diameter.

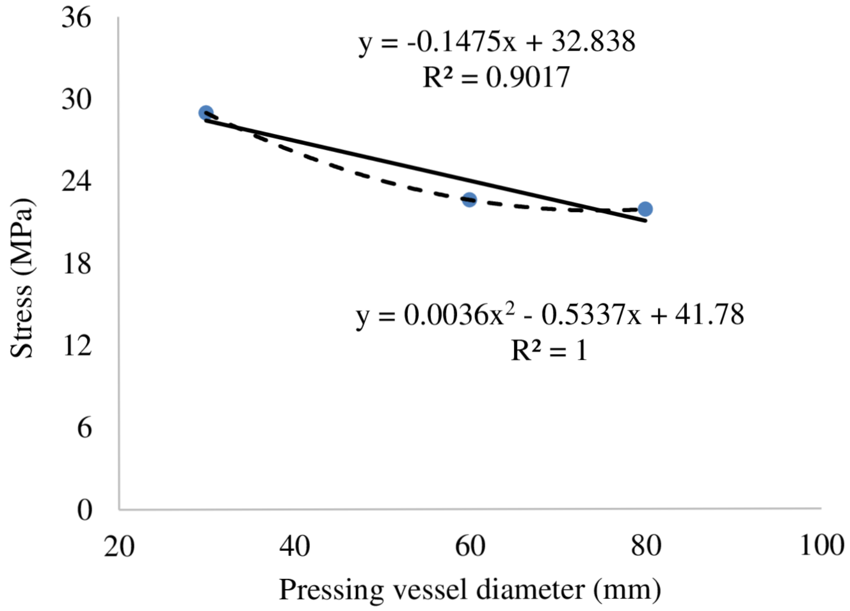


Figure 12. Dependency between stress and pressing vessel diameter.

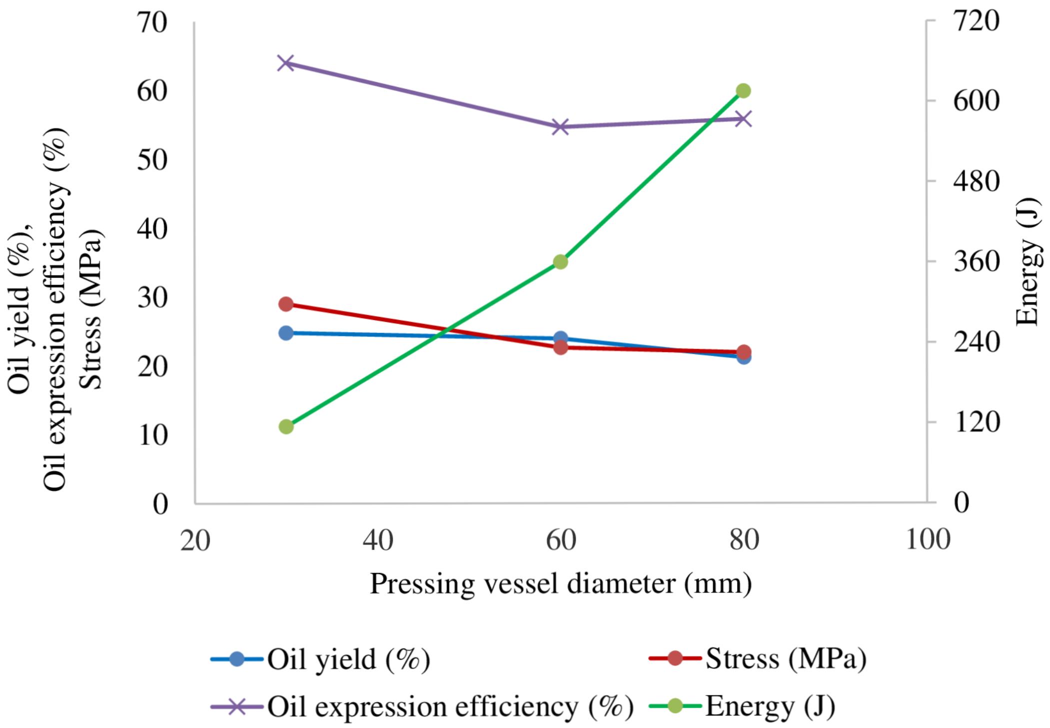


Figure 13. Relationship between determined parameters against pressing vessel diameter.

4.1.3 Statistical evaluation of the determined parameters

The results of the correlation and One-Way ANOVA analyses of the effect of the different pressing vessel diameters on the determined parameters are given in Table 4 and Table 5 respectively. Oil yield and oil expression efficiency were found to be not statistically significant ($p > 0.05$). However, both parameters correlated negatively with the diameter of the pressing vessel with a correlation value of (r = 0.744). The deformation values did not significantly correlate with the increase in pressing vessel diameter. On the other hand, the mass of oil, pressing force, energy and stress were found to be statistically significant ($p < 0.05$) with high correlation coefficient values between 0.991 and 0.989.

Table 4. Results of correlation between the input factor (D_{pv}) and the determined parameters.

Input factor	Determined parameters						
	M_{ol} (g)	O_{yd} (%)	O_{exp} (%)	F_{cr} (N)	D_{fx} (mm)	E_{df} (J)	δ_{ss} (MPa)
D_{pv} (mm)	r: 0.991	-0.744	-0.744	0.987	-0.008	0.989	-0.907
	$p <$ 0.05	$p >$ 0.05	$p >$ 0.05	$p <$ 0.05	$p <$ 0.05	$p <$ 0.05	$p <$ 0.05

D_{pv}: Pressing vessel diameter; M_{ol}: Mass of oil; O_{yd}: Oil yield; O_{exp}: Oil expression efficiency; F_{cr}: Force; D_{fx}: Deformation; E_{df}: Energy; δ_{ss}: Stress; r: Correlation coefficient; $p < 0.05$ is significant and $p > 0.05$ is non-significant.

Table 5. One-Way ANOVA results of the effect of input factor (D_{pv}) on the determined parameters.

Determined parameters	R^2	F-value	p-value
M_{ol} (g)	0.99	237.93	$p < 0.05$
O_{yd} (%)	0.74	4.19	$p > 0.05$
O_{exp} (%)	0.74	4.19	$p > 0.05$
F_{cr} (N)	0.99	190.98	$p < 0.05$
D_{fx} (mm)	0.31	0.68	$p > 0.05$
E_{df} (J)	0.99	223.77	$p < 0.05$
δ_{ss} (MPa)	0.91	15.67	$p < 0.05$

D_{pv}: Pressing vessel diameter; M_{ol}: Mass of oil; O_{yd}: Oil yield; O_{exp}: Oil expression efficiency; F_{cr}: Force; D_{fx}: Deformation; E_{df}: Energy; δ_{ss}: Stress; R^2: Coefficient of determination; $p < 0.05$ is significant and $p > 0.05$ is non-significant.

4.2 Effect of pressing forces on determined parameters

4.2.1 Mass of oil, oil yield and oil expression efficiency

The determined parameters from the effect of the pressing force are given in Table 6. The mass of oil, oil yield and oil expression efficiency linearly increased along with the increase in pressing force. The deformation and energy values positively corresponded to the increment of the pressing force. The stress values were calculated to be 2.26, 4.53 and 6.79 MPa as given in Table 7. The correlation values ranged from 0.968 to 1 as given in Table 8. The coefficient of the determination (R2) for the mass of oil, oil yield, oil expression efficiency, deformation and energy based on the One-Way ANOVA analysis was found to be 0.999 as given in Table 9.

Table 6. Determination of the mass of oil, oil yield and oil expression efficiency of the sample under the effect of force.

Tests	Input factor	Sample mass		Determined parameters		
	F_{cr} (kN)	M_{sb} (g)	M_{sa} (g)	M_{ol} (g)	O_{yd} (%)	O_{exp} (%)
1	1.6	41.88	41.37	0.51	1.22	3.14
2	1.6	41.88	41.36	0.52	1.24	3.21
Mean ± SD			41.37 ± 0.01	0.52 ± 0.01	1.23 ± 0.02	3.18 ± 0.04
1	3.2	41.88	36.47	5.41	12.92	33.36
2	3.2	41.88	36.45	5.43	12.97	33.48
Mean ± SD			35.46 ± 0.01	5.42 ± 0.01	12.94 ± 0.03	33.42 ± 0.09
1	4.8	41.88	33.64	8.24	19.68	50.80
2	4.8	41.88	33.62	8.26	19.72	50.93
Mean ± SD			33.36 ± 0.01	8.25 ± 0.01	19.70 ± 0.03	50.87 ± 0.09

F_{cr}: Force; M_{sb}: Mass of sample before test; M_{sa}: Mass of sample after test; M_{ol}: Mass of oil; O_{yd}: Oil yield; O_{exp}: Oil expression efficiency and SD: Standard Deviation.

Table 7. Determination of deformation, energy and stress of sample under the effect of force.

Tests	Input factor	Determined parameters		
	F_{cr} (kN)	D_{fx} (mm)	E_{df} (J)	δ_{ss} (MPa)
1	1.6	39.35	25.72	2.26
2	1.6	41.86	25.13	2.26
Mean ± SD		40.61 ± 1.77	25.43 ± 0.41	2.26 ± 0.00
1	3.2	54.65	60.23	4.53
2	3.2	55.75	60.91	4.53
Mean ± SD		55.20 ± 0.78	60.57 ± 0.48	4.53 ± 0.00
1	4.8	61.14	77.83	6.79
2	4.8	61.18	81.88	6.79
Mean ± SD		61.16 ± 0.03	79.86 ± 2.87	6.79 ± 0.00

F_{cr}: Force; D_{fx}: Deformation; E_{df}: Energy; δ_{ss}: Stress and SD: Standard Deviation.

Table 8. Results of correlation between the input factor (F_{cr}) and the determined parameters.

Input factor	Determined parameters					
	M_{ol} (g)	O_{yd} (%)	O_{exp} (%)	D_{fx} (mm)	E_{df} (J)	δ_{ss} (MPa)
F_{cr} (mm)	r: 0.988	0.988	0.988	0.968	0.985	1.000
	$p <$ 0.05	$p <$ 0.05	$p <$ 0.05	$p <$ 0.05	$p <$ 0.05	$p <$ 0.05

F_{cr}: Force; M_{ol}: Mass of oil; O_{yd}: Oil yield; O_{exp}: Oil expression efficiency; D_{fx}: Deformation; E_{df}: Energy; δ_{ss}: Stress; R: Correlation coefficient; $p < 0.05$ is significant and $p > 0.05$ is non-significant.

Table 9. One-Way ANOVA results of the effect of input factor (F_{cr}) on the determined parameters.

Determined parameters	R^2	F-value	p-value
M_{ol} (g)	0.999	204218.1	< 0.05
O_{yd} (%)	0.999	204218.1	< 0.05
O_{exp} (%)	0.999	204218.1	< 0.05
D_{fx} (mm)	0.999	178.7	< 0.05
E_{df} (J)	0.999	529.8	< 0.05

M_{ol}: Mass of oil; O_{yd}: Oil yield; O_{exp}: Oil expression efficiency; D_{fx}: Deformation; E_{df}: Energy; R^2: Coefficient of determination; and $p < 0.05$ is significant.

4.3 Effect of input factors combination on determined parameters

4.3.1 Mass of oil, oil yield, oil expression efficiency and energy

To better understand the input factors for processing sesame oilseeds under linear pressing, the Box-Behnken design of the experiment with the response surface methodology or regression analysis was applied. The input factors (pressing force, heating temperature and heating time) were set at three levels each. In all 15 experimental runs carried-out where 12 different factor-combinations were produced with 3 repetitions of the factors. The determined parameters were the mass of oil, oil yield, oil expression efficiency and energy were calculated as presented in Table 10 and Table 11 respectively. The determined parameters are graphically illustrated in Figure 14. The experimental runs 2, 4, 6 and 8 produced the maximum oil output parameters.

Table 10. Mass of oil from the input factors combination.

Run	F_{cr} (kN)	H_{pt} (°C)	H_{mt} (min)	M_{sb} (g)	M_{sa} (g)	M_{ol} (g)
1	1.6	40	45	41.88	41.37	0.51
2	4.8	40	45	41.88	32.91	8.97
3	1.6	80	45	41.88	40.14	1.74
4	4.8	80	45	41.88	33.32	8.56
5	1.6	60	30	41.88	41.29	0.59
6	4.8	60	30	41.88	32.93	8.95
7	1.6	60	60	41.88	40.81	1.07
8	4.8	60	60	41.88	33	8.88
9	3.2	40	30	41.88	35.14	6.74
10	3.2	80	30	41.88	35.46	6.42
11	3.2	40	60	41.88	36.13	5.75
12	3.2	80	60	41.88	35.11	6.77
13	3.2	60	45	41.88	35.21	6.67
14	3.2	60	45	41.88	35.77	6.11
15	3.2	60	45	41.88	35.56	6.32

F_{cr}: Force; H_{pt}: Heating temperature; H_{mt}: Heating time; M_{sb}: Mass of sample before test; M_{sa}: Mass of sample after test; M_{ol}: Mass of oil.

Table 11. Oil yield, oil expression efficiency and energy from the input factors combination.

Run	F_{cr} (kN)	H_{pt} (°C)	H_{mt} (min)	O_{yd} (%)	O_{exp} (%)	E_{df} (J)
1	1.6	40	45	1.22	3.14	23.62
2	4.8	40	45	21.42	55.30	76.32
3	1.6	80	45	4.15	10.73	24.67
4	4.8	80	45	20.44	52.78	73.07
5	1.6	60	30	1.41	3.64	25.07
6	4.8	60	30	21.37	55.18	71.02
7	1.6	60	60	2.55	6.60	24.03
8	4.8	60	60	21.20	54.75	71.57
9	3.2	40	30	16.09	41.56	58.52
10	3.2	80	30	15.33	39.58	55.43
11	3.2	40	60	13.73	35.45	57.60
12	3.2	80	60	16.17	41.74	54.82
13	3.2	60	45	15.93	41.12	53.90
14	3.2	60	45	14.59	37.67	56.41
15	3.2	60	45	15.09	38.97	56.11

F_{cr}: Force; H_{pt}: Heating temperature; H_{mt}: Heating time; O_{yd}: Oil yield; O_{exp}: Oil expression efficiency and E_{df}: Energy.

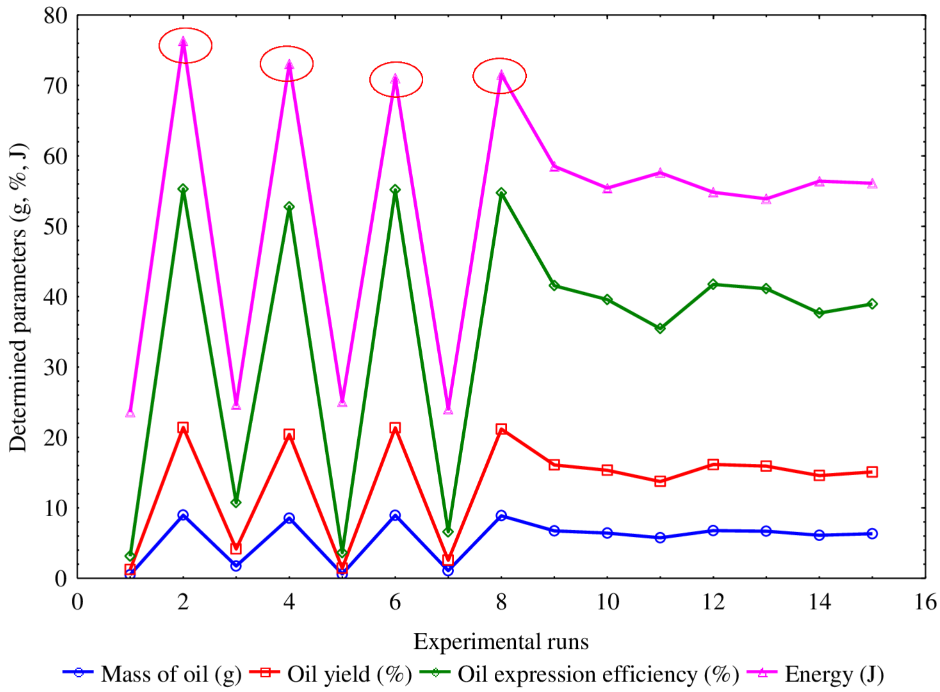


Figure 14. Scatterplot of the determined parameters against the experimental runs (circled portions at runs 2, 4, 6 and 8 showed the maximum output).

4.3.2 Statistical analysis by response surface regression

Table 12 is shown the transformed data from Table 11 by coding the input factors from -1 to +1. The response surface regression technique was used to generate the regression coefficients of the input factors about the determined parameters (oil yield, oil expression efficiency and energy). The estimates of the regression coefficients for the determined parameters are given in Tables 13, 14 and 15 respectively. For both the oil yield and oil expression efficiency; the coefficients of the intercept, linear and quadratic terms of the pressing force as well as the interaction between the pressing force and heating temperature were statistically significant ($p < 0.05$). The rest of the terms of the input factors and their interactions were not statistically significant ($p > 0.05$). The significant coefficients for the energy were the linear and quadratic terms of the pressing force and the intercept.

Table 12. Coded input factors and the determined parameters.

Run	F_{cr} (kN)	H_{pt} (kN)	H_{mt} (kN)	O_{yd} (%)	O_{exp} (%)	E_{df} (J)
1	-1	-1	0	1.22	3.14	23.62
2	1	-1	0	21.42	55.30	76.32
3	-1	1	0	4.15	10.73	24.67
4	1	1	0	20.44	52.78	73.07
5	-1	0	-1	1.41	3.64	25.07
6	1	0	-1	21.37	55.18	71.02
7	-1	0	1	2.55	6.60	24.03
8	1	0	1	21.20	54.75	71.57
9	0	-1	-1	16.09	41.56	58.52
10	0	1	-1	15.33	39.58	55.43
11	0	-1	1	13.73	35.45	57.60
12	0	1	1	16.17	41.74	54.82
13	0	0	0	15.93	41.12	53.90
14	0	0	0	14.59	37.67	56.41
15	0	0	0	15.09	38.97	56.11

F_{cr}: Force (-1: Low value – 1.6; 0: Centre value: 3.2 and +1: High value 4.8); H_{pt}: Heating temperature (-1: Low value – 40; 0: Centre value: 60 and +1: High value 80); H_{mt}: Heating time (-1: Low value – 30; 0: Centre value: 45 and +1: High value 60; O_{yd}: Oil yield; O_{exp}: Oil expression efficiency and E_{df}: Energy.

Table 13. Estimates of the regression coefficients of the input factors for oil yield.

Input factor /Effect	Oil yield, O_{yd} (%)			
	Regression coefficients	Standard error	t-value	p-value
Intercept	15.20	0.39	39.05	< 0.05
F_{cr}	9.39	0.24	39.38	< 0.05
F_{cr}^2	-3.54	0.35	-10.10	< 0.05
H_{pt}	0.45	0.24	1.90	> 0.05
H_{pt}^2	0.15	0.35	0.43	> 0.05
H_{mt}	-0.07	0.24	-0.29	> 0.05
H_{mt}^2	-0.02	0.35	-0.07	> 0.05
$F_{cr}{}^{x}H_{pt}$	-0.98	0.34	-2.90	< 0.05
$F_{cr}{}^{x}H_{mt}$	-0.33	0.34	-0.97	> 0.05
$H_{pt}{}^{x}H_{mt}$	0.80	0.34	2.37	> 0.05

F_{cr}: Force; H_{pt}: Heating temperature; H_{mt}: Heating time; $p < 0.05$ is significant and $p > 0.05$ is non-significant.

Table 14. Estimates of the regression coefficients of the input factors for oil expression efficiency.

Input factor /Effect	Oil expression efficiency, O_{exp} (%)			
	Regression coefficients	Standard error	t-value	p-value
Intercept	39.25	1.01	39.05	< 0.05
F_{cr}	24.24	0.62	39.38	< 0.05
F_{cr}^2	-9.15	0.91	-10.10	< 0.05
H_{pt}	1.17	0.62	1.90	> 0.05
H_{pt}^2	0.39	0.91	0.43	> 0.05
H_{mt}	-0.18	0.62	-0.29	> 0.05
H_{mt}^2	-0.06	0.91	-0.07	> 0.05
$F_{cr}{}^{x}H_{pt}$	-2.53	0.87	-2.90	< 0.05
$F_{cr}{}^{x}H_{mt}$	-0.85	0.87	-0.97	> 0.05
$H_{pt}{}^{x}H_{mt}$	2.07	0.87	2.37	> 0.05

F_{cr}: Force; H_{pt}: Heating temperature; H_{mt}: Heating time; $p < 0.05$ is significant and $p > 0.05$ is non-significant.

Table 15. Estimates of the regression coefficients of the input factors for energy.

Input factor /Effect	Energy, E_{df} (J)			
	Regression coefficients	Standard error	t-value	*p*-value
Intercept	55.48	0.92	59.99	< 0.05
F_{cr}	24.32	0.57	42.95	< 0.05
${F_{cr}}^2$	-7.36	0.83	-8.84	< 0.05
H_{pt}	-1.01	0.57	-1.78	> 0.05
${H_{pt}}^2$	1.30	0.83	1.56	> 0.05
H_{mt}	-0.25	0.57	-0.45	> 0.05
${H_{mt}}^2$	-0.19	0.83	-0.23	> 0.05
$F_{cr} {}^{x} H_{pt}$	-1.08	0.80	-1.34	> 0.05
$F_{cr} {}^{x} H_{mt}$	0.40	0.80	0.50	> 0.05
$H_{pt} {}^{x} H_{mt}$	0.08	0.80	0.10	> 0.05

F_{cr}: Force; H_{pt}: Heating temperature; H_{mt}: Heating time; $p < 0.05$ is significant and $p > 0.05$ is non-significant.

The significant terms explained above were confirmed by the coefficient of determination (R^2) values at 0.997 as given in Table 16. Figure 15 is illustrated the determined optimal input factors about the determined parameters (oil yield, oil expression efficiency and energy). The determined parameters are indicated on the right-side, the predicted values are indicated on the left-side, the input factors are on the top and their coded values are at the bottom. The predicted values of the determined parameters showed a high desirability value of 0.997 (Figure 15).

Table 16. Statistical evaluation of the determined parameters against the input factors.

Determined parameters	R^2	F-value	*p*-value
O_{yd} (%)	0.997	185.908	< 0.05
O_{exp} (%)	0.997	185.908	< 0.05
E_{df} (J)	0.997	214.850	< 0.05

O_{yd}: Oil yield; O_{exp}: Oil expression efficiency; E_{df}: Energy and $p < 0.05$ is significant.

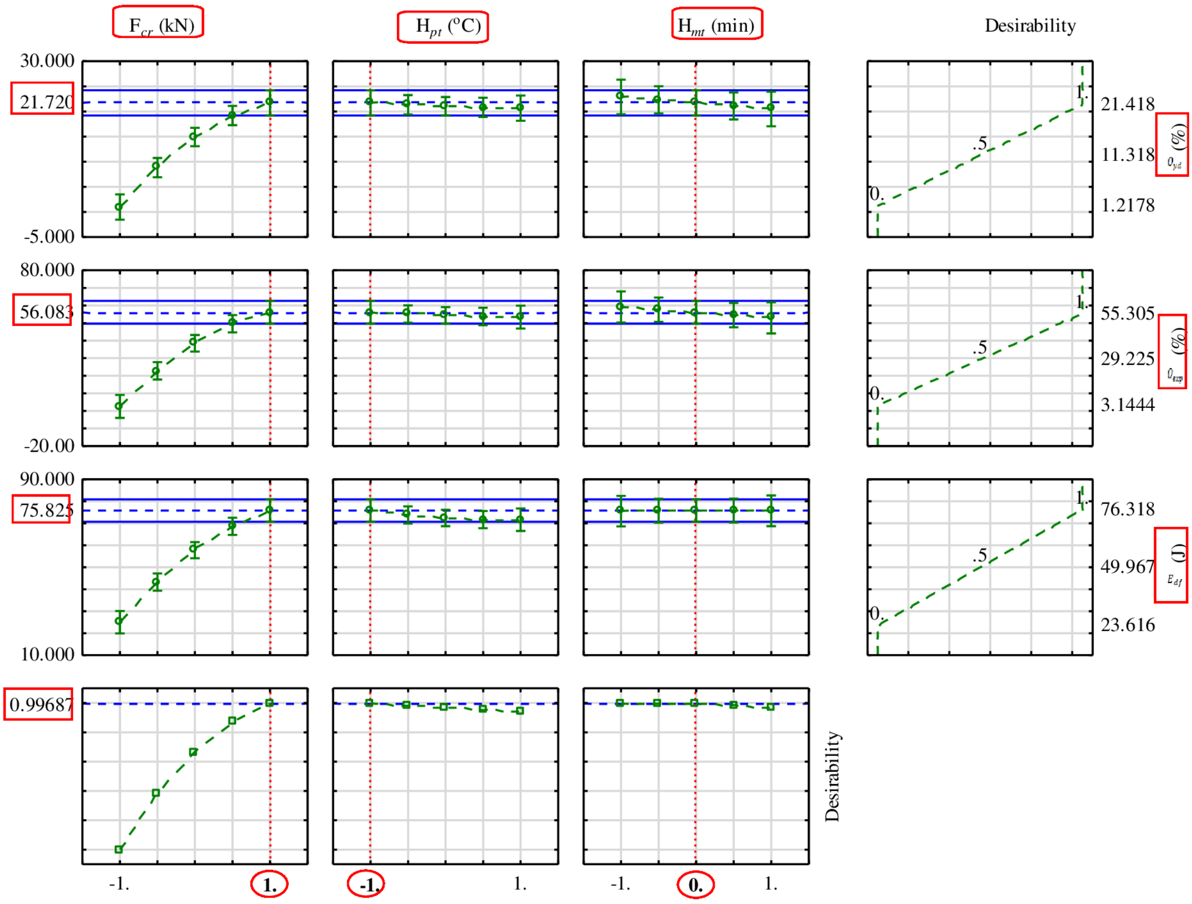


Figure 15. Predicted input factors (force: 4.8 kN (+1), heating temperature: 40 (-1) and heating time: 45 min (0)) and desirability values for the determined parameters (oil yield, $O_{yd}(\%)$ oil expression efficiency, O_{exp} (%) and energy, E_{df}(J).

4.3.3 Regression models of the determined parameters

The determined regression models and their statistical evaluation for predicting the oil yield, oil expression efficiency and energy are presented in Table 17. For the oil yield and oil expression efficiency; the coefficients of the interaction effect between the pressing force and heating temperature were not statistically significant ($p > 0.05$) (Table 17). This means that for predicting the determined parameters (oil yield, oil expression efficiency and energy), only the coefficients of the intercept as well as that of the linear and quadratic terms of the pressing force are suitable (Table 17) and (Appendixes 2 and 3). The suitability of the regression models was confirmed by the lack of fit which showed non-significant (Chanioti and Tzia, 2017).

Table 17. Determined models against the input factors.

Input factor /Effect	Model coefficients	Model sum of squares	Model df	Model Mean square	F-value
Oil yield, O_{yd} (%)					
Intercept	*15.20*	760.67	9	84.52	185.91*
F_{cr}	*9.39*	704.92	1	704.92	1544.83*
F_{cr}^2	*-3.54*	46.39	1	46.39	101.68*
$F_{cr}{}^{x}H_{pt}$	-0.98	3.83	1	3.83	8.40**
Residual		2.27	5	0.45	
** Lack of fit			3	0.45	0.99**
Oil expression efficiency, O_{exp} (%)					
Intercept	*39.25*	5071.62	9	563.51	185.91*
F_{cr}	*24.24*	4699.91	1	4699.91	1544.83*
F_{cr}^2	*-9.15*	309.35	1	309.35	101.68*
$F_{cr}{}^{x}H_{pt}$	-2.53	25.56	1	25.56	8.40**
Residual		15.16	5	3.03	
** Lack of fit			3	3.02	0.99**
Energy, E_{df} (J)					
Intercept	*55.48*	4960.24	9	551.14	214.85*
F_{cr}	*24.32*	4732.83	1	4732.83	2521.53*
F_{cr}^2	*-7.36*	200.25	1	200.25	106.69*
Residual		12.83	5	2.56	
** Lack of fit			3	3.02	1.6**

F_{cr}: Force; H_{pt}: Heating temperature; H_{mt}: Heating time; df: degrees of freedom; * $p < 0.05$ is significant and ** $p > 0.05$ is non-significant.

4.4 Force-deformation curves at different input factors

(i) The force-deformation curves obtained from all the experiments are illustrated in Figure 16 to Figure 19 and Appendixes 4 to 10 respectively. The area under the curve is the deformation energy (Figure 16) (Gupta and Das, 2000; Lysiak, 2007; Divisova et al., 2014; Herak et al., 2015). It was observed that under the compression machine with a maximum load of 5 kN (Figure 17), there was no serration effect on the force-deformation curves. However, the under the compression machine of a maximum load of 500 kN, the serration-effect was detected (Figure 18), hence compression of the sample ceased at the maximum force without the serration behaviour. The serration behaviour results in the ejection of the seedcake through the pressing vessel which could be due to the high pressure among other factors such as the seed moisture content, diameter of the pressing vessel and pressing speed (Gupta and Das, 2000; Lysiak, 2007;

Divisova et al., 2014). The maximum force for a sample of bulk sesame seeds at a pressing height of 100 mm with pressing vessel diameters 30, 60 and 80mm at a pressing speed of 5 mm/min without the serration effect on the force deformation curve ranged from 21 kN to 105 kN (Appendixes 4 to 6).

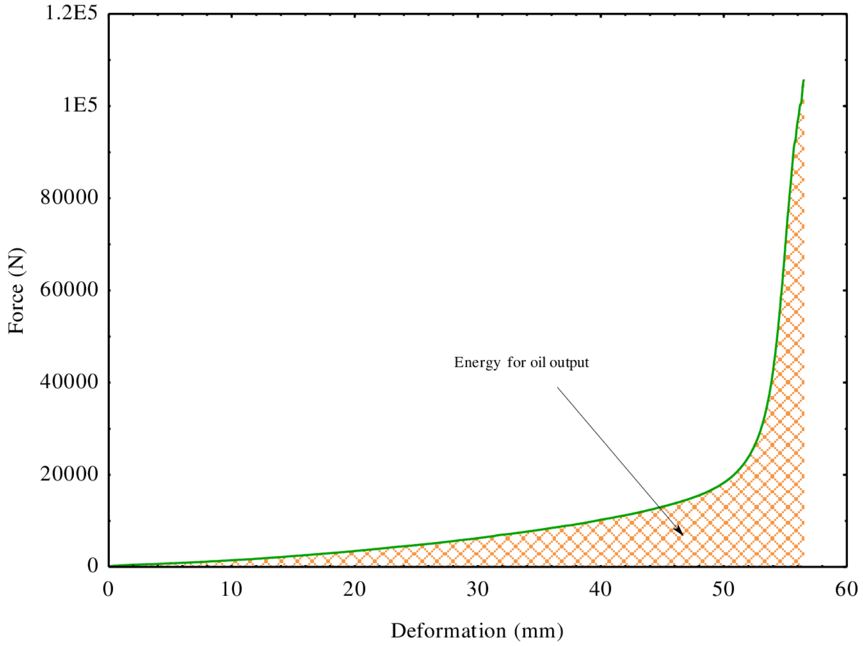


Figure 16. Force-deformation curve showing the energy for obtaining the oil.

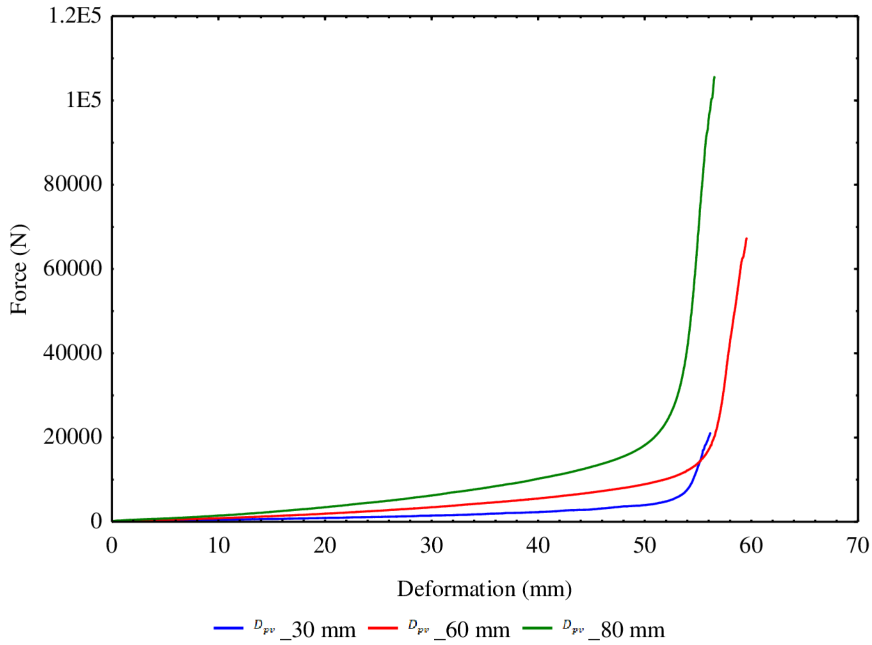


Figure 17. Force-deformation curves of the sample at different pressing vessel diameters, D_{pv}.

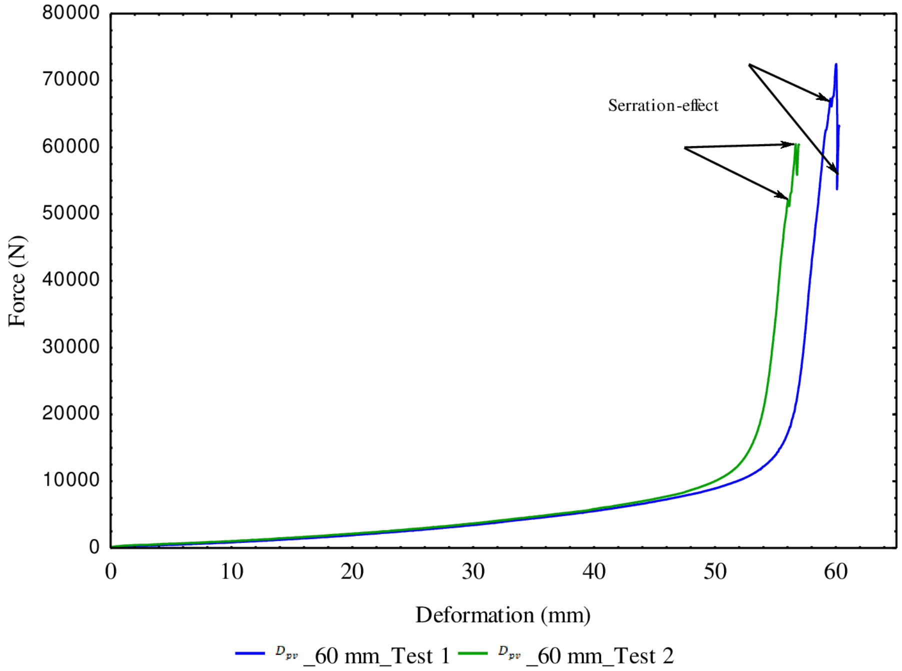


Figure 18. Force-deformation curve showing the serration-effect of the sample pressing.

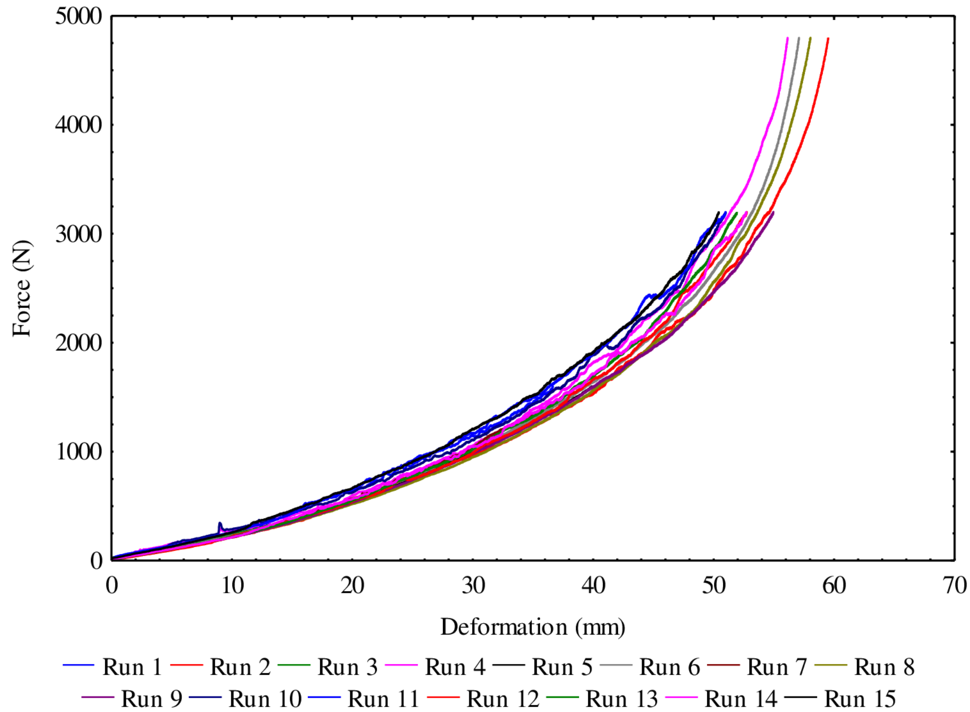


Figure 19. Force-deformation curves of the 15 experimental runs of the input factors combination.

5 CONCLUSIONS

i. It was found that the increase in the diameter of the pressing vessel increased the mass of oil whereas oil yield and oil expression efficiency values decreased with the increase in pressing vessel diameter.

ii. The increase in diameter of the pressing vessel at the constant sample pressing height of 100 mm increased the force required for obtaining the sesame seed oil.

iii. The deformation values did not linearly correspond to the increase in the diameter of the pressing vessel. The energy and the stress values linearly corresponded to the pressing vessel diameters increment.

iv. Oil yield and oil expression efficiency were found to be not statistically significant ($p > 0.05$). However, both parameters correlated negatively with the diameter of the pressing vessel with a correlation value of ($r = 0.744$). The deformation values did not significantly correlate with the increase in pressing vessel diameter.

v. The mass of oil, pressing force, energy and stress were found to be statistically significant ($p < 0.05$) with high correlation coefficient values between 0.991 and 0.989.

vi. The mass of oil, oil yield and oil expression efficiency linearly increased along with the increase in pressing force. The deformation and energy values positively corresponded to the increment of the pressing force.

vii. For both the oil yield and oil expression efficiency; the coefficients of the intercept, linear and quadratic terms of the pressing force as well as the interaction between the pressing force and heating temperature were statistically significant ($p < 0.05$). The rest of the terms of the input factors and their interactions were not statistically significant ($p > 0.05$). The significant coefficients for the energy were the linear and quadratic terms of the pressing force and the intercept.

viii. For the oil yield and oil expression efficiency; the coefficients of the interaction effect between the pressing force and heating temperature were not statistically significant ($p > 0.05$).

ix. For predicting the determined parameters (oil yield, oil expression efficiency and energy), only the coefficients of the intercept as well as that of the linear and quadratic terms of the pressing force are suitable. The suitability of the regression models was confirmed by the lack of fit which proved to be non-significant ($p > 0.05$).

x. The maximum force without the serration effect on the force deformation curve ranged from 21 kN to 105 kN for the sample of bulk sesame seeds at a pressing height of 100 mm with pressing vessel diameters 30, 60 and 80mm at a pressing speed of 5 mm/min.

6 RECOMMENDATIONS

In this Master's Thesis, the percentage oil yield, oil expression efficiency and energy demand for processing bulk sesame oil under linear pressing were determined and statistically evaluated. However, in future research, there is the need to:

(i) validate the optimal processing factors based on the regression models established herein for estimating the percentage oil yield, oil expression efficiency, and energy demand of bulk sesame seeds under linear pressing.

(ii) describe the spectral curves and/or determine the chemical properties of sesame seeds oil extracted under different pretreatment conditions.

(iii) process sesame seeds using small-medium mechanical screw presses/expellers to calculate the oil recovery efficiency, specific energy and residual oil in the press cake.

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8 INTERNET SOURCES

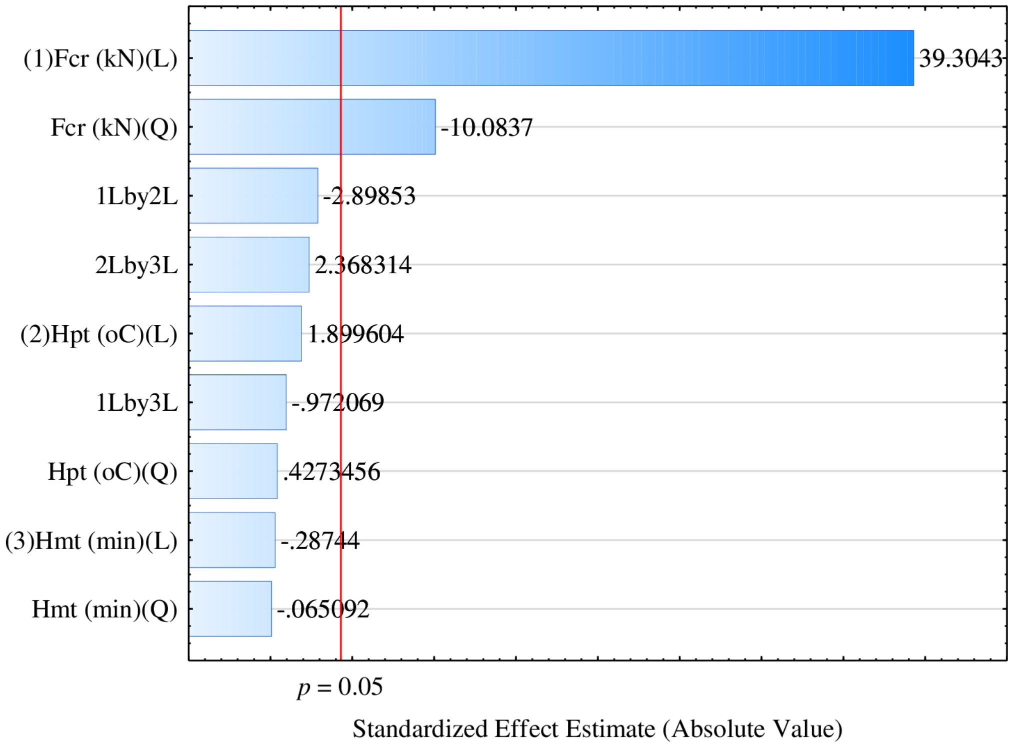
1. Vegetable oils: global consumption 2022/23 | Statista
2. Global vegetable oil production 2022/23 | Statista
3. OECD/FAO (2020), “OECD-FAO Agricultural Outlook”, OECD Agriculture statistics (database), http://dx.doi.org/10.1787/agr-outl-data-en.
4. Oilseeds and oilseed products | OECD-FAO Agricultural Outlook 2020-2029 | OECD library (oecd-ilibrary.org)

9 APPENDIXES

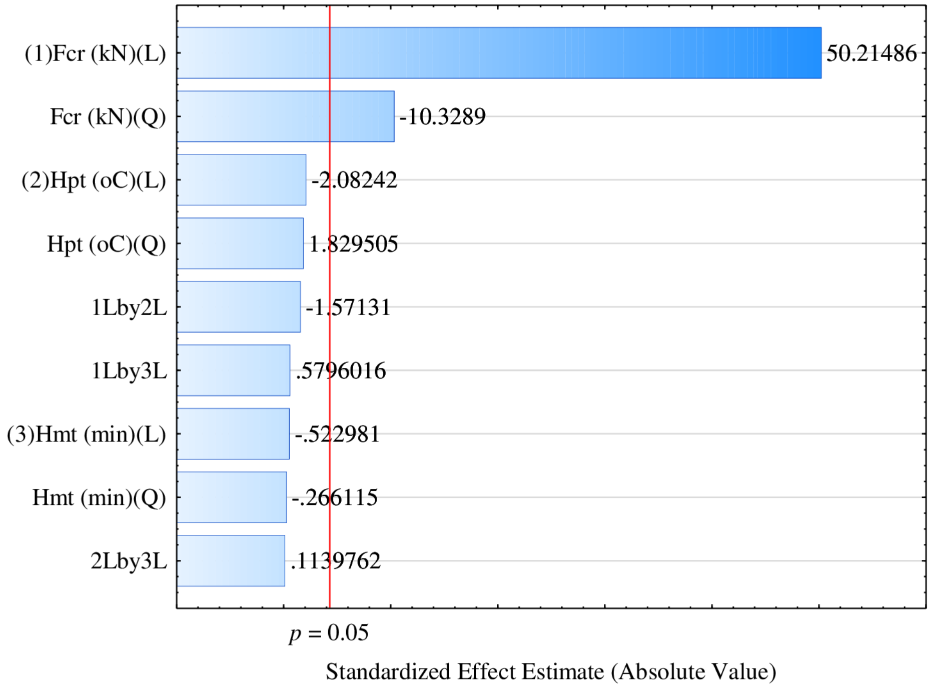
(a)

(b)

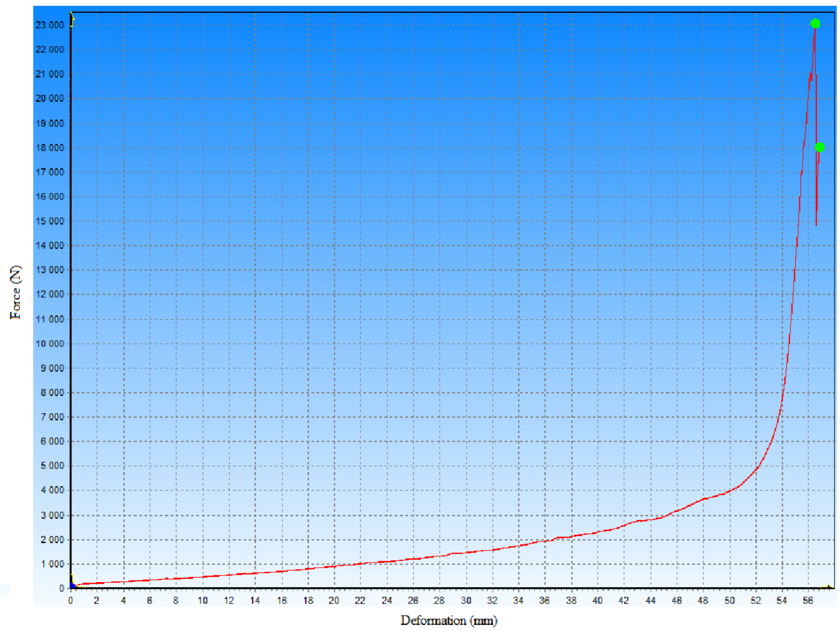
Appendix 1. Pressing vessel diameters (a) 30, 60 and 80 mm and (b) sesame samples.



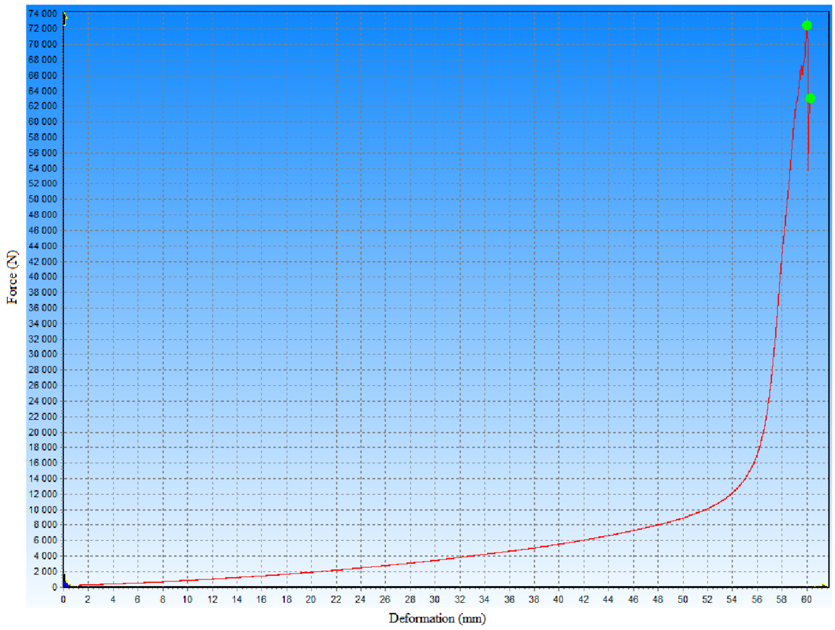
Appendix 2. Pareto chart of standardized effects for oil yield, O_{yd} (%) like oil expression efficiency, O_{exp} (%) (the red line determines the significant input factors)



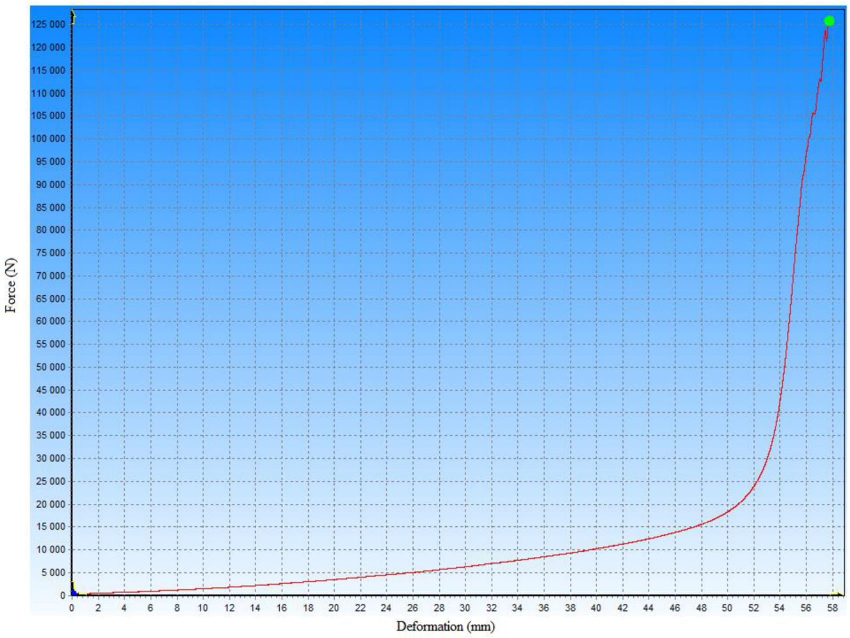
Appendix 3. Pareto chart of standardized effects for energy, E_{df}(J) (the red line determines the significant input factors).



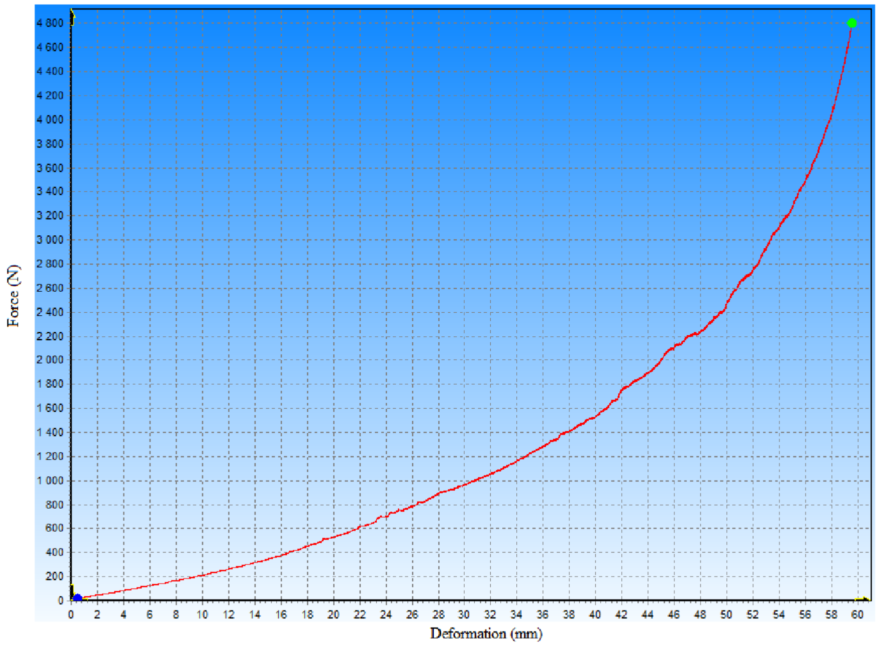
Appendix 4. Experimental force-deformation curve for vessel diameter 30 mm (Table 1 – Test 1) under Tempos, ZDM 50 machine.



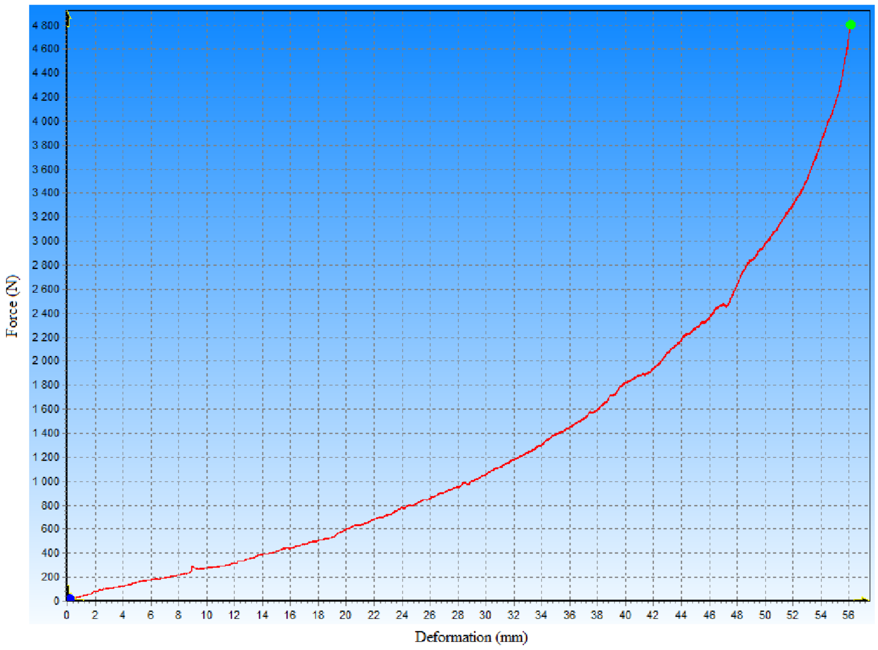
Appendix 5. Experimental force-deformation curve for vessel diameter 60 mm (Table 1 – Test 1) under Tempos, ZDM 50 machine.



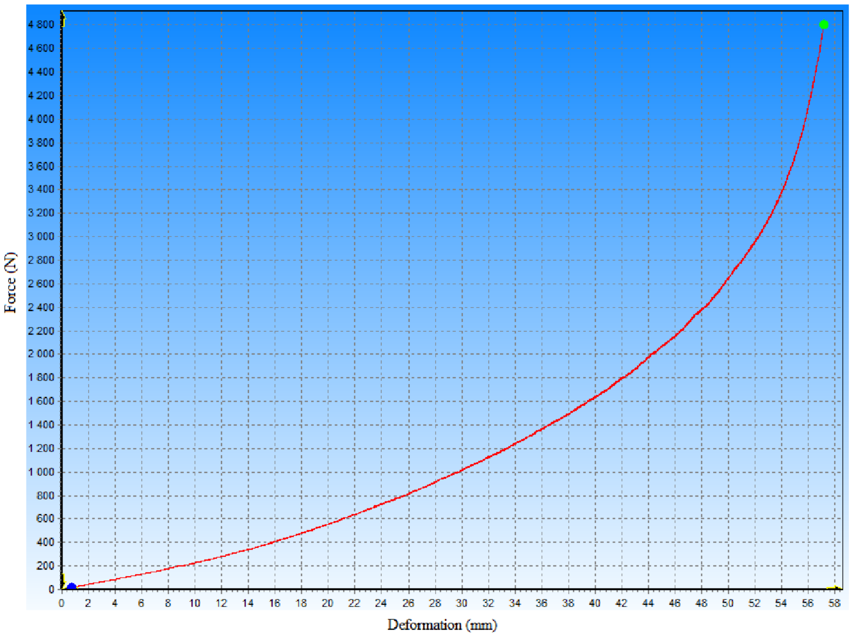
Appendix 6. Experimental force-deformation curve for vessel diameter 80 mm (Table 1 – Test 1) under Tempos, ZDM 50 machine.



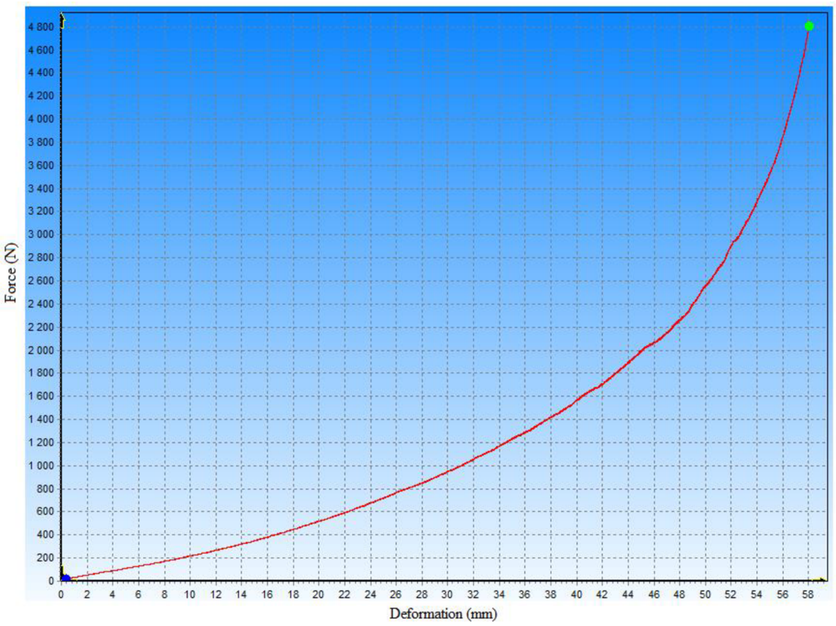
Appendix 7. Experimental force-deformation curve for run 2 – Box-Behnken design (Table 11) using vessel diameter 30 mm under MPTest 5.050 machine.



Appendix 8. Experimental force-deformation curve for run 4 – Box-Behnken design (Table 11) using vessel diameter 30 mm under MPTest 5.050 machine.



Appendix 9. Experimental force-deformation curve for run 6 – Box-Behnken design (Table 11) using vessel diameter 30 mm under MPTest 5.050 machine.



Appendix 10. Experimental force-deformation curve for run 8 – Box-Behnken design (Table 11) using vessel diameter 30 mm under MPTest 5.050 machine.