CZECH UNIVERSITY OF LIFE SCIENCES PRAGUE

Faculty of Engineering

Department of Mechanical Engineering

DIPLOMA THESIS ASSIGNMENT

Yuk Ming Chui

Effects of compression speed and heat-treatment temperature on mechanical behaviour of sunflower and rape bulk oilseeds under compression loading

Supervisor

Ing. Abraham Kabutey, Ph.D.

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Objectives of thesis

(i) To describe the force-deformation curve characteristic of sunflower and rape bulk oilseeds in relation to speeds and heat treatment temperatures and forces.
(ii) To determine the response surface regression models for describing the deformation energy, percentage oil yield and oil expression efficiency of sunflower and rape bulk oilseeds in relation to speeds, temperatures and forces.
(iii) To determine the optimal speed, force and temperature of deformation energy, percentage oil yield and oil expression efficiency of sunflower and rape bulk oilseeds.

Methodology

The experiment will be conducted at the laboratory of the Mechanical Department of Faculty of Engineering. A universal compression testing machine (ZDM 50, Czech Republic) and a pressing vessel diameter of 60 mm with a plunger will be used for the compression test. The volume of the bulk samples will be measured at 60 mm pressing height. The varying speeds of 5, 10 and 15 mm/min; temperatures of 45, 60 and 75 °C and forces of 60, 80 and 100 kN will be examined using Box-Behnken Design. The data will be analysed statistically using the STATISTICA software (Statsoft, 2013).

Code for compiling the Diploma (MSc.) Thesis

1. Introduction

1.1 Research problem statement

1.2. Objectives

2. Literature review

2.1 A general overview of Sunflower and Rape oil-bearing crops.

2.2 Oil extraction methods of oil-bearing crops.

2.3 Optimization principles of oil extraction methods.

2.4 Biodiesel fuel production from oil-bearing crops

2.4.1. Fuel characteristics of biodiesel

2.4.2 Advantages

2.4.3 Disadvantages

2.5 Physical and mechanical properties of Sunflower and Rape bulk oilseeds.

3. Materials and Methods

4. Results and Discussion

5. Recommendations and Conclusions

6. References

7. Appendixes

The proposed extent of the thesis

60 – 70 pages

Keywords

Bulk oilseeds, compression test, mechanical properties, numerical estimation, percentage oil yield

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Recommended information sources

Demirel C, Kabutey A, Herak D, Gurdil G.A.K. 2017. Numerical estimation of deformation energy of selected bulk oilseeds in compression loading. IOP Conference Series: Materials Science and Engineering, 237(1), 1–5.

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Oworlarafe OK, Olabige MT, Faborade MO (2007). Physical and mechanical properties of two varieties of fresh oil palm fruit. Journal of Food Engineering, 78, 1228–1232.

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Abstract

Oil from the oil crops has been utilized by humans in daily life for many years. One of the most popular usages of oil crops nowadays is green energy or biodiesel production. And the most common materials to produce oil are rapeseeds and sunflower seeds. To be eco-friendly, energy consumption in the compression process of sunflower seeds and rapeseeds production should be minimized for a large amount of oil production. This study focuses on the effects of compression speed and heat treatment temperature on the mechanical behaviour of sunflower and rape bulk oilseeds under compression loading. To understand the background more clearly, a general overview of sunflower and rape-oil bearing crops was explored. Some common oil extraction methods of oil-bearing crops and optimization principles of those methods were described. Besides, some biodiesel fuel production from oil-bearing crops and their pros and cons were discussed. Moreover, the physical and mechanical properties of sunflower and rape bulk oilseed were reviewed. To study the mechanical behaviour of sunflower and rape bulk oilseeds under compression loading, response surface methodology (RSM) was employed. Three independent process variables namely compression speed, temperature and compression force were studied and optimized. A set of experimental data through the Box Behnken approach were analyzed using STATISTICA software. After carrying out the experiments, the optimum compression conditions for processing oil from sunflower seeds and rapeseeds were identified and then validated.

Keywords: Oil bearing crops, compression process, energy consumption, Box Behnken Design, response surface methodology

DECLARATION

I hereby declare that I have done this MSc. Thesis entitled *Effects of compression speed and heat-treatment temperature on mechanical behaviour of sunflower and rape bulk oilseeds under compression loading* independently and all texts in this Thesis are original. All literature sources used have been acknowledged by providing a list of references according to the Citation rules of Faculty of Engineering, Czech University of Life Sciences Prague.

In Prague Date:

16.4.2020

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LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDIXES

TABLE OF CONTENTS

1 INTRODUCTION

1.1 Background

In this thesis, a general overview of sunflower and rape-oil bearing crop was explored. Some common oil extraction methods of oil-bearing crops and optimization principles of those methods were described. Besides, some biodiesel fuel production from oil-bearing crops and their pros and cons were discussed. Also, the physical and mechanical properties of sunflower and rape bulk oilseed were reviewed.

For the experimental part, a Response Surface Methodology (RSM) was employed to study the mechanical behaviour of sunflower and rape bulk oilseeds under compression loading. RSM is a set of mathematical and statistical techniques which is used for studying the effects of several factors at a different level and their impacts on each other (Jana, Roy and Dey., 2018). For the reason that RSM is easy to be established, clear on parameter sensitivity, it is usually used in food process engineering domain (Jin, Chen and Simpson., 2001 as cited in Huang et al., 2019). Fakayode and Ajav., (2016) as cited in Huang et al., (2019) used RSM to study the relationship between the yield and the process parameters of mechanical pressing and obtained the maximum yield by optimizing the mechanical oil extraction process from Moringa oleifera seeds. Chapuis, Blin, Carré, and Lecomte., (2014), Karaj and Müller., (2011) as cited in Huang et al., (2019) used RSM to consider the energy consumption when optimizing the mechanical oil extraction process.

To optimize the response factors investigated is the main purpose of using RSM. To start using RSM, the main problem should be identified. Then, find out which response is to be measured, how it is to be measured, which variables are to be explored. And design the experiments afterwards. The advantages of RSM according to Jana, Roy and Dey., (2018) are: the number of experiments can be reduced instead of designing full experiments at the same level, the effects of a factor can be estimated at different levels of other factors and the surface contour can be obtained which provides a good way for indicating the interaction of the parameters.

For the statistical part, Box-Behnken design was applied for modelling the RSM. It is the most common and efficient method devised by George E. P. Box and Donald Behnken. The fundamentals, benefits and constraints of Box-Behnken design for different optimization of analytical were described by (Ferreir et al., 2007 as cited in Jana, Roy and Dey., 2018). Mourabet et al., (2017) showed that certain studies utilized the Box-Behnken design effectively such as the adsorption of methylene blue by kapok fiber treated by sodium chlorite Liu et al., (2012), the Cr (VI) adsorption onto activated carbons Ozdemir et al., (2011), the degradation of Acid Red 274 using H_2O_2 in subcritical water Kayan and Gozmen., (2012) and the removal of fluoride from aqueous solution by adsorption on Apatitic tricalcium phosphate (Mourabet et al., 2012).

All spherical designs are included and only three levels of required factors can be run in Box-Behnken design. The main benefits behind the application of this design according to Jana, Roy and Dey., (2018) are: each independent variable is placed at one of three equally spaced values which are generally coded as −1, 0 and +1, the design should be sufficient to fit a quadratic model, that is one containing squared terms, products of two factors, linear terms and an intercept. The ratio of the numbers of experimental points to the number of coefficients in the quadratic model should be reasonable (1.5 to 2.6).

In this thesis, three independent process variables: compression speed, temperature and compression force were studied through the Box-Behnken design of an experiment. Optimum compression conditions for both sunflower seed and rapeseed were identified and validated.

1.2 Research problem statement

One of the main intentions to cultivate sunflower seed and rapeseed is for energy use. Therefore, energy consumption in the compression process of sunflower seed and rapeseed conduction should be minimized for a large amount of oil production. Divišová et al., (2014) studied the deformation, deformation energy and energy density for the compression of rapeseed and sunflower seeds. Kabutey et al., (2017) studied the deformation, deformation energy and oil yield of rapeseed under different heat treatment temperature, force and speed. Kabutey, Herak, Sigalingging and Demirel., (2018) determined the deformation energy and oil yield of sunflower seed under different heat treatment temperature and heating time.

In this study, deformation energy, percentage oil yield and oil expression efficiency of sunflower seed and rapeseed will be determined under different compression speed, force and heating temperature. Therefore, the optimum speed, force and temperature for seeds compression can be found out to minimize energy during oil production.

1.3 Objectives

(i) To describe the force-deformation curve characteristics of sunflower and rape bulk oilseeds in relation to speeds, heat treatment temperatures and forces.

(ii) To determine the response surface regression models for describing the deformation energy, percentage oil yield and oil expression efficiency of sunflower and rape bulk oilseeds in relation to speeds, temperatures and forces.

(iii) To determine the optimal speed, force and temperature of deformation energy, percentage oil yield and oil expression efficiency of sunflower and rape bulk oilseeds.

2 LITERATURE REVIEW

2.1 A general overview of sunflower and rape-oil bearing crops

2.1.1 History of sunflower and rapeseeds

Helianthus, the genus name of sunflower (*Helianthus annuus* L.) is derived from the Greek words "helios" which mean sun and "anthus", meaning flower (Hu, Seiler and Kole., 2010). Sunflower is originated in North America. It was then migrated to eastward and southward of North America. Sunflower was first introduced to Europe through Spain. Then, it spread through Europe and adapted in Russia eventually. In 1860, Russia started selection for high oil which increases oil content from 28% to almost 50%. After World War II, high-oil lines were reintroduced into the U.S. The marketers in the Great Plains states found out the new usages for the sunflower seeds as an oil crop, a bird seed crop and as a human snack food. As a result, the production of sunflowers rises dramatically. In the 1980s, the production of sunflowers declined due to pests and diseases as well as problems of birds picking up seeds (Putnam et al., 1990).

The term "rape" is originated from the Latin word for trunip, rapa or rapum, cognate with the Greek word harpy's Etymonline.com., (2019). Rapeseed has been grown as an oilseed crop in Europe since the Middle Ages and it was used extensively as a stream engine lubricant during the Industrial Revolution due to the high containment of erucic acid oil. In 1970s, Canola was developed by Canadian breeders to reduce the concentration of erucic acid which is cardiotoxic agent for human beings (Hristov et al., 2011). The "industrial rapeseed" refers to any rapeseed with a high content (at least 45 %) of erucic acid in the oil. The "Canola" was registered in 1979 in Canada and refers to the edible oil crop which is characterized by low erucic acid (less than 2 %) and low levels of glucosinolates (Agmrc.org., 2020). Rapeseed is the most abundant oil plant in the European Union, and it is also the third most abundant oil plant in the world after palm and soya (Fetzer et al., 2018). The global annual production of rapeseed accounted for 73.8 Mt, with a share of 24.3 Mt in the EU (Food and Agriculture Organization of the United Nations (FAO), 2017 as cited in Fetzer et al., 2018).

2.1.1.1 Description

Sunflower is an annual herb. The stem of the sunflowers is rough hairy, around 1-4.5 m high and broad. It is also coarsely toothed, rough leaves 7.5–30 cm long arranged in spirals. The head of the flowers in wild specimens is usually 7.5–15 m while the head of cultivated types sunflowers are 30 cm or more. The disk flowers are brown, yellow or purple and the petal-like ray flowers are yellow (Britannica Academic., 2019).

Rapeseed plant is an annual herb which is harvested after one growing season. Rapeseed plant has several erect with a single base and the stem are purple towards the base. The leaves of the plant are smooth and bluish-green. The plant produces pale to bright yellow flowers with a diameter of 11–15 mm. After pollination, the plant develops pods containing 20–40 single row of dark brown to black seeds. The height of the plant can reach 1–2.5m (Plantvillage.psu.edu., 2019).

2.1.1.2 Environmental requirement

Sunflower seeds can germinate at 3.89 °C but temperatures of 7.78 °C to 10 °C are the best temperature range for germination. In the early germination stages, seeds are not affected by the low temperature and can survive in temperatures down to -5 °C. However, the freezing temperature may injure the crop at later stages. Sunflower plant can grow between 17.77 °C to 32.77 °C but the optimum temperature for sunflower plant to grow are 21.1 °C to 25.56 °C. Extremely high temperatures can lower oil percentage, seeds fill and germination (Putnam et al., 1990).

Rapeseed requires a cool and moist environment to grow best. The best temperature for rapeseed to grow is 2–10 °C and temperatures closer to 10 °C promote the most rapid growth. In subtropical regions, rapeseed is grown as a cool-season crop and in temperate areas, rapeseed is grown as a winter crop. Rapeseed can be grown on different types of soils but medium textures, well-draining soil is the best for them. The pH range for the soil should between 5.5 to 8.3 (Plantvillage.psu.edu., 2019).

2.1.1.3 Utilization

Sunflower oil can be used to produce soaps, detergents, pesticide carrier. It can also be used in the production of agri-chemicals, surfactants, adhesives, plastics, fabric softeners, lubricants and coatings. Besides, because of its good semi-drying properties and without color modification, it is also widely used in certain paints, varnishes and plastics. In the US, sunflower oil contains 93 % of the energy of US Number 2 diesel fuel.

On the other hand, sunflower seed is used as a bird feed and human snack. Different usage for non-oilseed purposes depend on their size; 1) larger seed for in-shell roasting, 2) medium for dehulling and 3) small for birdseed. According to Putnam et al., (1990) sunflower seed has been used in 77 countries in the world. Ukraine is the biggest producer in the world which produced 13,626,890 tonnes of sunflower seeds in 2016 (Fao.org., 2018). In Brazil, sunflower seed is one of the main raw materials in the National Program of Biodiesel Production (NBPB) (Mourad et al., 2018). There are three types of sunflower seeds. They are the traditional sunflower seed, medium-oleic acid sunflower seed and high-oleic acid sunflower seed which contain 14–39 %, 42–72 % and 75–91 % oleic acid respectively (Zheljazkov et al., 2011). To classify different types of sunflower seeds we can use infrared spectroscopy and multivariate calibration method (Cantarelli et al., 2018) and air separation method (Munder, Argyropoulos and Müller., 2018).

Rapeseed press cake (RPC) is the residual material left after defatting rapeseed by mechanical-extraction methods such as screw pressing. RPC can be divided to full-pressed press cake (FPC) and the pre-pressed press cake (PPC). FPC is obtained by completed mechanical oil extraction to fat content of 5%–10% and PPC is obtained by mechanical extraction to fat content of 15%–18 % (Fetzer et al., 2018). Besides, cold press cake (CPC) is the residual material after cold-pressing. It is applied for the production of niche market native rapeseed oils and has a residual fat content of approximately 15%–18% (Leming and Lember., 2005 as cited in Fetzer et al., 2018). Rapeseed oil can be used for cooking, cosmetics and home cuisine. Cold-pressed rapeseed oil is beneficial for health. It is used for the production of mayonnaise, tartar sauce, salad dressing and cream cheese spread. Besides, it is used in the baking industry, canning industry and poultry industry for food production. Also, the usage of rapeseed oil is common in-home cuisine. It has a beneficial effect on people on diet aiming at reducing cholesterol level and improves the function of the human endocrinologic system and strengthens muscle cells and nervous system. Moreover, rapeseed oil can be used as the production of creams for the skin of the human body and helps to treat skin disease. Natural liquid and solid soap made from rapeseed oil has proven to have beneficial and desired effect on skin comparing to the normal toilet soap due to the traditional way of cold manufacture which preserves the contents of all useful components

(Bioenergo-komplex.cz., 2019). Besides, rapeseed oil can also be used in the production of biodiesel (Dworakowska, Bednarz and Bogdal., 2011). Canada is the biggest producer of rapeseed which produced 18,423,600 tonnes in 2016 (Fao.org., 2018). In China, rapeseed is the largest oilseed crop and accounts for about 20% of world production (Hu et al., 2018).

2.1.2 Physical and mechanical properties of sunflower and rape bulk oilseeds

For optimizing equipment design, oil extraction and handling, the physical and mechanical properties have to be known (Karaj and Muller, 2010; Sirisomboon et al., (2007).

2.1.2.1 Physical properties

2.1.2.1.1 Unit mass of the seed

Mirzabe, Khazaei and Chegini., (2012), measured the single seed mass of sunflower seeds using a digital balance with an accuracy of 0.01 g. The values ranged from 0.01–0.23 g, 0.01–0.14 g and 0.01–0.27 g at the central, middle and side locations of the sunflower seed varieties; Mikhi, Sirena and Songhoir of moisture contents of 21.55%, 18.43% and 30.6% (w.b.). Gupta and Das., (1997) used an electronic balance reading of 0.001 g to weigh the unit mass of the seed and kernel. The average unit mass of the seed was 0.049 g and for the kernel was 0.034 g.

Çalışır et al., (2005) also reported the unit mass of rapeseeds (a 10 seed group mass) between 0.0040 and 0.0065 g for moisture contents between 4.7% and 23.96% (d.b.).

2.1.2.1.2 1000 seed mass

Isik and Izli., (2007) reported the 1000 seed mass between 66 g and 70 g for moisture content between 10.06 % and 27.06 (% d.b.). Mirzabe, Khazaei and Chegini., (2012) described the measurement of 1000 sunflower seeds mass by selecting 300 sunflower seeds randomly. Then the seeds were divided into three bins so that in each bin 100 seeds were placed. The weight of the bin was multiplied by 10 to obtain mass of 1000 seeds.

Çalışır et al., (2005) mentioned that the 1000 seed mass values of rapeseeds at moisture contents of 4.7% and 23.96% varied between 5.1 g and 6.36 g. The authors also described an increasing relationship between 1000 seed mass and moisture content.

Baran et al., (2016) indicated 1000 seed mass of rapeseeds of 4.74 g at 8.4% moisture content. Izli, Unal and Sincik., (2009) also measured 1000 mass of rapeseeds varieties of Capitol, Jetneuf and Samurai. The mass of the seeds increased with increased moisture content between 7.3% and 27.4% (w.b.).

2.1.2.1.3 Geometric Mean and Arithmetic Mean Diameters

According to Mohsenin, (1970) as cited in Isik and Izli., (2007), the arithmetic mean diameter and geometric mean diameter of sunflower seeds were calculated using equations (1) and (2).

$$D_a = \frac{(L + W + T)}{3} \tag{1}$$

$$D_g = (LWT)^{\frac{1}{3}} \tag{2}$$

where D_a is arithmetic mean diameter (mm), D_g is geometric mean diameter (mm), L is length (mm), W is width (mm), T is thickness (mm). The authors indicated that arithmetic mean diameter increased from 6.37 to 8.05 mm and the geometric mean diameter also increased from 6.15 to 7.93 mm with increased moisture from 10.026% to 27.06 % (d.b). Mirzabe, Khazaei and Chegini., (2012), indicated the range of geometric diameter values of 4.98–9.79 mm, 3.94–8.51 mm and 5.97–10.83 at the central, middle and side locations of the sunflower seed varieties; Mikhi, Sirena and Songhoir of moisture contents of 21.55%, 18.43% and 30.6% (w.b.). Gupta and Das., (1997) also reported the geometric mean diameter values of the sunflower seed and the kernel of 5.39 mm and 4.32 mm respectively.

Izli, Unal and Sincik., (2009) indicated that the geometric mean diameter of rapeseeds increased with the increase in moisture contents. For Capitol, the geometric mean diameter increased from 2.11 to 2.23 mm with an increase in moisture content from 8.3% to 25.9% (d.b.). For Jetneuf, the geometric mean diameter increased from 1.98 to 2.12 mm with an increase in moisture content from 7.7% to 27.4% (d.b.). And for Samurai, the geometric mean diameter increased from 1.96 to 2.09 mm with an increase in moisture content from 7.3% to 26.4% (d.b.). According to Baran et al., (2016), the geometric mean diameter can be calculated using equation (3).

$$D_g = (LD^2)^{1/3} \tag{3}$$

where D is the diameter of the seed (mm). The authors reported geometric average diameter of canola seed (rapeseed) of 2.07 mm at a moisture content of 8.4%.

2.1.2.1.4 Sphericity

According to Mohsenin., (1970) as cited in Isik and Izli., (2007), the sphericity of sunflower seed was calculated using equation (4).

$$\phi = \frac{(LWT)^{\frac{1}{3}}}{L} \tag{4}$$

where ϕ is sphericity (%), L is length (mm), W is width (mm), T is thickness (mm). The authors indicated that the sphericity of sunflower seeds increased from 0.789 to 0.835 with the increase in moisture content. Mirzabe, Khazaei and Chegini., (2012), also reported the range of sphericity values of 36.09–56.51%, 34.28–64.05% and 30.45–53.98% at the central, middle and side locations of sunflower seeds varieties; Mikhi, Sirena and Songhoir of moisture contents 21.55%, 18.43% and 30.6% (w.b.).

Izli, Unal and Sincik., (2009) indicated that the sphericity of rapeseeds of different varieties (Jetneuf, Samurai and Capitol) increased with the increase in moisture content.

2.1.2.1.5 Surface area

The surface area of sunflower seeds was calculated using equation (5) (Tunde-Akintunde and Akintunde., 2004 as cited in Isik and Izli., 2007).

$$A_s = \pi D_g^2 \tag{5}$$

where $A_s$is surface (mm^2), D_g is geometric mean diameter (mm). The authors reported the surface area of sunflower seeds of 118.76 mm^2 at 10.06 (% d.b.) and 197.65 mm^2 at 27.06 (% d.b.). Mirzabe, Khazaei and Chegini., (2012), also reported the range of surface area values of 78.03–301.17 mm^2, 48.82–227.38 mm^2 and 112.121–368.41 mm^2 at the central, middle and side locations of sunflower seeds varieties; Mikhi, Sirena and Songhoir of moisture contents 21.55%, 18.43% and 30.6% (w.b.).

Izli, Unal and Sincik., (2009) reported that the surface area of rapeseeds varieties (Capitol, Jetneuf and Samurai) increased linearly with increasing moisture content (97.7% to 27.4% (d.b.) from 14.1 to 15.7 mm^2, 12.3 to 14.1 mm^2 and 12.1 to 13.8 mm^2.

2.1.2.1.6 Projected area

Mirzabe, Khazaei and Chegini., (2012) calculated the projected area of sunflower seeds using equation (6).

$$A_p = \left(\frac{\pi WL}{4}\right) \quad (6)$$

where A_p is projected area (mm^2), W is width (mm) and L is length (mm). The range of the projected area values was 44.58–151.38 mm^2, 27.38–86.05 mm^2 and 75.73–175.12 mm^2 at central, middle and side locations of sunflower seeds varieties; Mikhi, Sirena and Songhoir of moisture contents 21.55%, 18.43% and 30.6% (w.b.).

Çalışır et al., (2005) also measured the projected area of rapeseeds using a digital camera (Canon A200) and the (Sigma Scan Pro 5 Program). For moisture content between 4.7% and 23.96% (d.b.), the projected area varied from 3.71 to 4.67 mm^2. The authors also described the equation representing the relationship between projected and moisture content.

2.1.2.1.7 Volume of the seeds

Mirzabe, Khazaei and Chegini., (2012) calculated the volume of sunflower seeds using equation (7). The range of volume values was 64.82–491.47 mm^3, 32.11–322.42 mm^3 and 111.63–664.93 mm^3 at the central, middle and side locations of sunflower seeds varieties; Mikhi, Sirena and Songhoir of moisture contents 21.55%, 18.43% and 30.6% (w.b.).

$$V = \left(\frac{\pi D_g^3}{6}\right) \quad (7)$$

where V is volume of seeds (mm^3), D_g is geometric mean diameter (mm).

Izli, Unal and Sincik., (2009) indicated that the volume of rapeseeds varieties (Capitol, Jetneuf and Samurai) increased linearly with the increase in moisture content. For Capitol variety, the volume increased from 5.66 to 6.60 mm^3 at 8.3% to 25.9% (d.b.). The volume increased from 4.56 to 5.48 mm^3 at 7.7% to 27.4% (d.b.) for Jetneuf. And for Samurai variety, the volume increased from 4.45 to 5.28 mm^3 at 7.3% to 26.4% (d.b.). The authors also described the equation representing the relationship between volume and moisture content.

2.1.2.1.8 Bulk density

Isik and Izli., (2007) cited Singh and Goswarmi., (1996) and Gupta and Das., (1997) as references to determine the average bulk density of sunflower seeds by using the standard test weight procedure. In the test procedure, a container of 500 mL was filled with the grain from a height of 150 mm at a constant rate and then weighed the content. The results showed a linearly decreasing trend from 415.40 to 405.56 kg/m^3 in bulk density due to the reduction of moisture content. Mirzabe, Khazaei and Chegini., (2012) calculated the bulk density of sunflower seeds varieties; Mikhi, Sirena and Songhoir of moisture contents 21.55%, 18.43% and 30.6% (w.b.) using the standard method. The values were 331.027 kg/m^3, 422.015 kg/m^3 and 319.346 kg/m^3. Gupta and Das., (1997) measured the bulk density of sunflower seed and kernel for moisture content range from 8 to 19.8% (d.b.) by filling a 500ml container with grain from a height of 15 cm, striking the top level and then weighing. The bulk density of the rewetted seed decreased from 462 to 434 kg/m^3. And for the kernel, the value of the bulk density increased from 574 to 628 kg/m^3. The bulk density was expressed using equation (8).

$$\rho_b = \frac{m}{V} \tag{8}$$

where ρ_b is bulk density (kg/m^3), m is mass (kg) of the sample, V is volume (m^3) of the sample.

Çalışır et al., (2005) stated that the bulk density values of rapeseed at moisture contents of 4.7% and 23.96% varied between 612.1 kg/m^3 and 585.1 kg/m^3. Izli, Unal and Sincik., (2009) found that while the moisture content increased, the bulk density of Capitol rapeseed decreased from 676.3 to 617.4 kg/m^3, for Jetneuf rapeseed, it decreased from 635.0 to 593.6 kg/m^3 and of Samurai rapeseed variety it increased from 664.8 to 609.7 kg/m^3.

2.1.2.1.9 True density

Isik and Izli., (2007) used the toluene displacement method to determine the true density of sunflower seeds of moisture content range from 10.31–27.06 (% d.b). The true density varied from 885 to 902 kg/m^3 for the moisture content range mentioned above. Mirzabe, Khazaei and Chegini., (2012) calculated the true density of sunflower seeds varieties; Mikhi, Sirena and Songhoir of moisture content 21.55%, 18.43% and 30.6% (w.b.) using the water displacement method. The values were 497.5 kg/m^3, 580.368 kg/m^3 and 471.746 kg/m^3, respectively. Gupta and Das., (1997) measured the true density of the sunflower seeds and kernel by using an electronic balance and air comparison pycnometer which was the method mentioned in (Beckman, Model 930). The true density of the seeds was found to vary from 706–765 kg/m^3 and for the kernel from 1050–1250 kg/m^3. According to Karaj and Muller., (2010), the true/solid density can be expressed using equation (9).

$$\rho_{t/s} = \left(\frac{m}{n \cdot V_u}\right) \tag{9}$$

where $\rho_{t/s}$ is true/solid density (g/cm3), V_u is the unit volume of the seeds (cm^3), n is number of seeds/kernels in the sample.

Izli, Unal and Sincik., (2009) found that while the moisture content increased, the true density of Capitol rapeseed variety decreased from 14071.2 to 1015.8 kg/m^3, for Jetneuf variety, it decreased from 1091.3 to 1047.1 kg/m^3 and for Samurai type it decreased from 1083.1 to 1028.4kg/m^3.

2.1.2.1.10 Porosity

Mohsenin., (1970) calculated porosity using equation (10) as cited in Isik and Izli., (2007).

$$P_f = \left(1 - \frac{\rho_b}{\rho_t}\right) 100 \tag{10}$$

where P_f is porosity (%), ρ_b is bulk density (kg/m^3) and ρ_t is true density (kg/m^3). According to Isik and Izli., (2007), the porosity of sunflower seed increased from 53.06 to 54.93% for moisture content range between 10.06 and 27.06% (d.b.). Mirzabe, Khazaei and Chegini., (2012) indicated porosity values of 33.46%, 27.28% and 32.30% of sunflower seeds varieties; Mikhi, Sirena and Songhoir of moisture contents 21.55%, 18.43% and 30.6% (w.b.). Gupta and Das., (1997) results showed that porosity of sunflower seeds increased from 34.3% to 43.3% and that of kernels increased from 45.4 % to 50.2 % for the moisture content range between 4 and 20% (d.b.).

Çalışır et al., (2005) stated that the porosity values of rapeseeds at moisture contents of 4.7% and 23.96% varied between 48.2 and 60.6%. Izli, Unal and Sincik., (2009) also indicated that while the moisture content increased from 7.7% to 27.4% (w.b.), the porosity increased for all the rapeseed varieties. It increased for Capitol from 36.6 to 38.9%, for Jetneuf it increased from 41.5 to 43.2%, and for Samurai it increased from 38.4% to 40.6%.

2.1.2.1.11 Terminal velocity

Khodabakhshian et al., (2010) revealed that the terminal velocity values of sunflower seed and its kernel (large, medium and small) for moisture contents between 3% and 14% (d.b.) increased for all sizes as moisture content increased. Sunflower seed terminal velocity varied from 5.36 to 6.12 m/s whiles the kernel varied from 5 to 5.68 m/s with increasing moisture content. Isik and Izli., (2007) study showed that the terminal velocity increased linearly from 4.07 to 4.57 m/s as the moisture content increased from 10.06 to 27.06% (d.b.). Gupta and Das (1997) reported at any moisture content between 4% and 20% (d.b.), the terminal velocity of sunflower seed was higher than that of kernel and hull. The terminal velocity increased from 5.8 to 7.6m/s, 3.5 to 5.8 m/s and 1.7 to 2.8 m/s for seed, kernel and hull respectively.

Calisir et al., (2005), measured the terminal velocities of rapeseed at different moisture contents using an air column and an electronic anemometer having a least count of 0.1m/s. Terminal velocities values at moisture contents of 4.70% and 23.96% varied between 3.16 and 3.74 m/s. Izli, Unal and Sincik., (2009) also reported that the terminal velocity of rapeseed varieties increased linearly with the increase in moisture contents. It increased for Capitol variety from 3.61 to 3.77 m/s, for Jetneuf type it increased from 3.49 to 3.66 m/s and for Samurai it increased from 3.44 to 3.52 m/s.

2.1.2.1.12 Static and dynamic coefficient of friction

Isik and Izli., (2007) reported static coefficient of friction of sunflower seeds on six surfaces (rubber, stainless steel, aluminium, glass, medium density fiberboard and galvanized iron) against moisture content in the range between 10.06 to 27.06% (d.b.) using the tilting surface/sliding method. The authors indicated that the static coefficient of friction increased with increase in moisture content for all surfaces. However, the least static coefficient of friction was found on the glass as a result of a smoother and more polished surface. Similar results were reported by Khodabakhshian et al., (2010) for sunflower seeds of different sizes (large, medium and small). Gupta and Das., (1997) also mentioned that the static coefficient of friction of sunflower seed was lower than that of the kernel and varied from 0.40 to 0.58 for seed and from 0.43 to 0.81 for the kernel with the increase in moisture content between 4% and 20% (d.b.).

Calisir et al., (2005) described the method for the determination of coefficients of friction of rapeseed using a friction device which had three main components: stationary sample container with its support shaft, a driving unit with a rotating disc and a data acquisition system. The authors further explained that the samples were placed on the rotating surface and the torque necessary to restrain the sample was used to determine the static and dynamic coefficients of friction using equation (11).

$$\mu = \frac{T_m}{wq} \tag{11}$$

where μ is coefficient of friction, T_m is torque, w is length of the torque arm and q is sample weight on the rotating surface. The authors indicated that the maximum value of torque obtained as the disc started to rotate was used to calculate the static coefficient of friction and the average value of the torque during the rotation of the disc was used to calculate the dynamic coefficient of friction. Their results showed that static and dynamic coefficients of friction of rapeseeds for iron sheet, galvanized sheet and plywood materials increased with moisture content.

Izli, Unal and Sincik., (2009) also indicated that the static coefficient of friction for rapeseeds varieties (Capitol, Jetneuf and Samurai) on frictional surfaces of stainless steel, aluminium, glass, galvanized iron, plywood and rubber increased with moisture content (7.3% to 27.4% (d.b). The highest static coefficient of friction was obtained on the rubber surface whiles the minimum was found on stainless steel due to its smoothness and polished surface.

2.1.2.1.13 Angle of repose

Mirzabe, Khazaei and Chegini., (2012) indicated the values of angle of repose of 25.08º, 26.80º and 24.39º on the wood surface of sunflower seeds varieties; Mikhi, Sirena and Songhoir of moisture contents 21.55%, 18.43% and 30.6% (w.b.). The values on the galvanized surface were 22.23º, 23.86º and 24.39º. The authors stated that the value of angle of repose on the wood surface was higher than that of the galvanized surface as a result of higher friction on the wood surface than the galvanized surface. Gupta and Das., (1997) also reported that the angle of repose of sunflower seed increased from 34 to 41º for seed and 27 to 38º for kernel in the moisture content range between 4 and 20% (d.b.). Jafari et al., (2011) also mentioned the angle of repose values of sunflower seeds between 15.69 º and 38.83º at moisture content range from 8 to 20% (w.b.).

Izli, Unal and Sincik., (2009) reported that the angle of repose of rapeseeds varieties (Capitol, Jetneuf and Samurai) increased as the moisture content increased between 7.3% and 27.4% (d.b). The angle of repose for Capitol ranged from 21.37 to 26.81º, Jetneuf from 18.11 to 22.11º and Samurai from 18.91 to 24.56º.

2.1.2.2 Mechanical properties

2.1.2.2.1 Rupture Force

The rupture force indicates the minimum force required for extracting the oil from seeds or kernels. Khodabakhshian et al., (2010) measured the rupture force of the sunflower seed and its kernel by using an Instron Universal Testing Machine (Model QTS 25) equipped with a 25 kg load cell and integrator. Each seed or kernel was loaded between two parallel plates of the machine and compressed until rupture occurred. The results showed that rupture force for both sunflower seed and its kernel decreased as the moisture content increased. The force required to rupture the hull of seed and kernel increased as seed size increased. Izli et al., (2009) also determined the rupture strength of rapeseeds by using a biological material test device. The device was equipped with a load cell of 50N capacities (Sundoo 50SH Digital Push Pull Gauge). The result showed that the rupture force decreased with increased moisture content.

2.1.2.2.2 Deformation

The deformation at rupture point can be used for the determination of the gap size between the surfaces to compress the fruit or nut for dehulling or shelling. Deformation values of sunflower seeds and rapeseeds with the increasing function and serration effect on the force-deformation curve at different pressing vessels of diameters between 40 and 100 mm and seeds pressing heights between 20 and 80 mm were reported by Divišová et al., (2014). Increased vessel diameters decreased the deformation values at a maximum force of 100 kN and speed of 60 mm/min. Kabutey et al., (2017) also determined the deformation of rapeseed at varying pressing height (mm), forces (kN), speeds (mm/min) and heat treatment temperatures (°C). The results showed that by varying the pressing height (mm) at a constant speed of 10 mm/min and force 100 kN, the maximum deformation ranged from 10.95 ± 0.06 to 48.74 ± 0.09 (mm). For different forces, the maximum deformation ranged from 45.10 ± 0.03 to 50.43 ± 0.09 (mm). By varying the speeds, the maximum deformation ranged from 45.10 ± 0.03 to 50.43 ± 0.09 (mm). And by changing the heat treatment temperatures, the maximum deformation ranged from 30.05 ± 0.09 to 31.44 ± 1.63 (mm).

2.1.2.2.3 Deformation energy

Deformation energy is characterized by the area under the force-deformation curve (Gupta and Das., 2000). Deformation energy values of sunflower seeds and rapeseeds were reported by Divišová et al., (2014). Energy values under the force-deformation curve with serration effect were higher than the area without the serration effect at a maximum force of 100 kN and speed of 60 mm/min. Rapeseeds required higher energy for recovering the oil than sunflower seeds. Kabutey et al., (2017) determined the deformation energy of rapeseeds at varying pressing heights (mm), forces (kN), speeds (mm/min) and heat treatment temperatures (°C). The results showed that by varying the pressing height (mm) at a constant speed of 10 mm/min and force 100 kN, the maximum deformation energy ranged from 231.08 ± 1.91 to 757.72 ± 2.40 J. For different force, the maximum deformation energy ranged from 538.99 ± 1.19 to 1045.59 ± 2.33 J. By varying the speeds, the maximum deformation energy ranged from 482.79 ± 1.42 to 452.63 ± 9.43 J. Also, by changing the heat treatment temperatures, the maximum deformation energy ranged from 503.40 ± 5.57 to 580.45 ± 7.52 J.

2.1.2.2.4 Energy density

The energy density (J/m^3) of seeds is the ratio of energy to the volume of seeds (Divisova et al., 2014; Chakespari et al., 2010; Gupta and Das, 2000). Divisova et al., (2014) determined the energy density for sunflower and rape bulk seeds at different pressing vessels of diameters between 40 and 100 mm and seeds pressing heights between 20 and 80 mm at a maximum force of 100 kN and speed of 60 mm/min for both the increasing function and serration effect on the force-deformation curve. According to Divisova et al., (2014), increased vessel diameters increased the energy density values of sunflower seeds and rapeseeds respectively.

2.2 Extraction methods of oil-bearing crops

Nowadays, there are several oil extraction methods of oil-bearing crops. The extraction set-up, procedure, advantages and disadvantages of the five most common extraction methods: Soxhlet extraction, microwave-assisted extraction, ultrasonication assisted extraction, pressurized liquid extraction and supercritical fluid extraction are discussed below.

2.2.1 Soxhlet Extraction

Soxhlet extraction is the most widely used leaching technique. It was developed by Franz Ritter von Soxhlet in 1879 and it has been a standard technique for over a century. The original intention of designing Soxhlet extraction is to determine fat in milk (Luque de Castro and Priego-Capote., 2010). The definition of Soxhlet extraction is the process of transferring the partially soluble components of a solid to the liquid phase using a Soxhlet extractor. The solid is placed in a filter paper thimble which is then placed into the main chamber of the Soxhlet extractor. The solvent (heated to reflux) travels into the main chamber and the partially soluble components are slowly transferred to the solvent (Rsc.org, 2019).

2.2.1.1 Extraction process of Soxhlet extraction

In the Soxhlet extraction, a Soxhlet extractor is used to extract the oil form the seed. In the beginning, heat is applied to the still pot (Figure 1). The solvent in the still pot then evaporates and travel up through the distillation path to the condenser. The condensed solvent then drops down to the extractor. When the extractor is filled up with the condensed solvent by reaching an overflow level (above the siphon), it returns to the distillation flask (Williams., 2019) (NileRed., 2019). The advantages and disadvantages of Soxhlet extraction according to Luque de Castro and García-Ayuso., (1998) are given below.

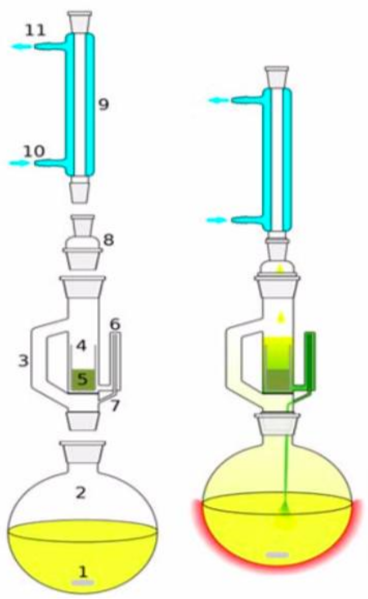

Figure 1. A schematic representation of a Soxhlet extractor (Kasiri, Masoud & Safapour, Siyamak., 2015).1: Stirrer bar; 2: Still pot (the still pot should not be overfilled and the volume of solvent in the still pot should be 3 to 4 times the volume of the Soxhlet chamber); 3: Distillation path; 4: Thimble; 5: Solid; 6: Siphon top;7: Siphon exit; 8: Expansion adapter; 9: Condenser; 10: Cooling water outlet; 11: Cooling water inlet

2.2.1.1.1 Advantages of Soxhlet extraction

i. No filtration is needed after the leaching.
ii. Equipment for Soxhlet is inexpensive so that number of samples can be increased by simultaneous extraction in parallel.
iii. The methodology of Soxhlet extraction is very simple. Therefore, specialized training is not required.
iv. Comparing to the latest method (microwave extraction, supercritical fluids, etc.), more sample mass can be extracted.
v. Non-matrix dependent.

2.2.1.1.2 Disadvantages of Soxhlet extraction

i. Long time is required for the extraction
ii. Large amount of solvent is used which is expensive to dispose off and can cause environmental problem.
iii. The conventional Soxhlet device is unable to provide stirring therefore the extraction cannot be accelerated.

iv. After extraction, evaporation/concentration step is mandatory due to a large amount of solvent used.
v. Solvent selectivity is restricted.
vi. Difficult to automate.

2.2.2 Microwave-Assisted Extraction (MAE)

The definition of microwave is nonionizing electromagnetic radiation which has a frequency of 300 MHz–300 GHz. There are two types of MAE systems, the closed vessel and the opened vessel. Closed vessel system is used when high temperature and pressure conditions are needed in the process of the extraction. And open vessel systems are used when atmospheric pressure conditions are required in the extraction process (Kaufmann and Christen, 2002 as cited in Danlami et al., 2014). MAE extraction technology has been used for both laboratory-scale level and full-scale commercialized extraction applications (Ying et al. 2013 as cited in Danlami et al., 2014). MAE technology is commonly used for isolating essentials oils, fats and oils (Deng et al., 2006, Bayramoglu et al., 2008 as cited in Danlami et al., 2014). MAE cannot be considered as a green technology due to the usage of organic solvents such as hexane (Danlami et al., 2014).

2.2.2.1 Extraction process of MAE

Bendahou et al., (2008), described the extraction of essential oil extract from plants by using MAE. The set-up of the experiment was according to (Craveiro et al., 1989). The experiment was performed at atmospheric pressure using an Arthur Martin multimode microwave oven operating at 2450 MHz and 850 W. The procedure was that a 25 g of plant material was inserted into an extraction vessel and 200 ml of hexane was added. The extraction time was 2 min and the extraction temperatures between 60 and 80 °C with a maximum power of microwave extractor. After cooling, the vessel was opened and the supernatant was filtered. Then, the filtrate was frozen for 12 hours to precipitate the fixed waxes and oil. After the second filtration, the extract was filtered through an activated carbon column to eliminate the pigments (Jean, Collin, & Lord., 1992 as cited in Bendahou et al., 2008). Lastly, the filtrate reduced by rotary evaporation, the extract was collected and dried under anhydrous sodium sulphate and stored at 4 °C until used. The extraction yield was 1.0 %.

2.2.2.1.1 Advantages of MAE

i. Microwave heating can reach a high temperature which reduces both extraction time and the amount of solvent required dramatically (Luque de Castro and García-Ayuso., 1998).

ii. The extraction efficiency improves when the polarity of the analytes increased due to the absorption of microwave energy which is proportional to the relative sample or solvent permittivity (Luque de Castro and García-Ayuso., 1998).

iii. There is no loss of heat to the environment (Luque de Castro and García-Ayuso., 1998).

2.2.2.1.2 Disadvantages of MAE

i. Microwave-assisted extraction can be very poor when the target analytes or the solvents are nonpolar or of low polarity as well as the solvent used to have low dielectric constants (Luque de Castro and García-Ayuso., 1998).

ii. Thermolabile sample cannot be extracted by microwave-assisted extraction due to degradation of solute (Mandal, Mandal and Das., 2015).

iii. Comparing with other conventional extraction approaches, the initial capital costs of MAE are high. Besides, solvent reduction step is required usually although solvent consumption is reduced significantly (Poole., 2020).

2.2.3 Ultrasonication Assisted Extraction (UAE)

The definition of ultrasound waves is the high-frequency sound waves above 20 kHz which is also above human hearing. Ultrasonic water bath and ultrasonic probe system fitted to horn transducers are two types of ultrasound equipment commonly used for extraction (Ibanez et al., 2012 as cited in Danlami et al., 2014). UAE extraction has been used for both laboratory-scale level, industrial-scale and full-scale commercialized extraction applications (Vilkhu et al., 2008, Chemat et al., 2011 as cited in Danlami et al., 2014). According to Danlami et al., (2014), UAE has been widely used for the extraction of nutritional material, like lipids Metherel et al., (2009), proteins Zhu et al., (2009), flavouring Chen et al,.(2007), Da Porto et al., (2009), essential oils, fats, and oils Kimbaris et al., (2006), and bioactive compounds (e.g., flavonoids) Ma et al., (2008), carotenoids Sun et al., (2006), Yue et al., (2006), and polysaccharides (Iida et al., 2008, Chen et al. 2010., Wei et al. 2010., Yan et al., 2011).

2.2.3.1 Extraction process of UAE

For instance, according to Bimakr et al., (2017), *M. sylvestris* leaves were put into a 150 mL Erlenmeyer flask and mixed with an appropriate solvent. The solvent ratio was 1:20 (w/v). The extraction was carried out by using 200W ultrasound equipment (UP200H/UP200S Hielscher, Germany) with a titanium ultrasonic probe (3 mm diameter). The probe delivered the ultrasonic energy to the leaves. The temperature was controlled at the desired levels. Ultrasound irradiations were performed for 40, 50 and 60 min. The applied ultrasonic power levels were 50, 100, and 150 W (Bimakr et al., 2017).

2.2.3.1.1 Advantages of UAE

i. Ultrasound-assisted extraction can shorten the extraction time (Luque de Castro and García-Ayuso., 1998).
ii. Using ultrasound-assisted extraction can increase the yield of extracted compounds (Vinatoru, Mason and Calinescu., 2017).
iii. Comparing with other current extraction techniques such as microwave-assisted extraction, the ultrasound apparatus is cheaper and have a more simple operation (Mandal, Mandal and Das., 2015).

2.2.3.1.2 Disadvantages of UAE

i. It is unable to renovate the solvent during the extraction process (Luque de Castro and García-Ayusom., 1998).
ii. It is mandatory to carry filtration and rinsing steps after extraction which take a longer time for the whole process (Luque de Castro and García-Ayusom, 1998).
iii. High solvent consumption (Luque de Castro and García-Ayuso., 1998).

2.2.4 Pressurized Liquid Extraction (PLE)

According to Guardia and Armenta., (2011), PLE is also called accelerated solvent extraction (ASE), Pressurized solvent extraction (PSE), high-pressure solvent extraction (HPSE), high-pressure, high-temperature solvent extraction (HPHTSE), pressurized hot solvent extraction (PHSE), and subcritical solvent extraction (SSE). PLE can be used for extraction form solid and semisolid sample matrices under elevated temperature (50–200 °C) and pressure (500–3000 psi) conditions for short periods (5–10 min).

2.2.4.1 Extraction process of PLE

PLE can be worked in both static and dynamic modes. In the static mode, the sample is placed in a stainless-steel vessel filled with an extraction solvent. After extraction, the remaining solvent is purged with N_2 into a collection vial. For the dynamic model, the solvent is pumped through the sample continuously (Guardia and Armenta., 2011). The advantages and disadvantages of PLE according to Guardia and Armenta., (2011) are stated below:

2.2.4.1.1 Advantages of PLE

i. Increasing the temperature can improve the extraction efficiency due to the increased solubility of target analytes which assist in breaking down analyte-matrix interactions and encourages the diffusion of the analyte to the matrix surface and mass transfer of organic compounds to the solvent.
ii. High pressure can maintain the solvent in a liquid state at high temperature and may increase the penetration of the solvent in the sample matrix.

2.2.4.1.2 Disadvantages of PLE

i. When using a hydrophobic organic solvent as water hinders contact between the solvent and analyte, the high-water percentages in the samples decreases analyte extraction efficiency when using hydrophobic organic solvents.
ii. The efficiency of the PLE can be influenced by the pressure and temperature conditions easily.

2.2.5 Supercritical Fluid Extraction (SFE)

Supercritical fluids have unique physicochemical properties which are useful as solvents for extraction. The average densities and solvating powers of supercritical fluids are similar to liquids which can be changed readily by adjusting the temperature and pressure. Also, supercritical fluids have gas-like transport properties of viscosity and intermediate diffusivity. Moreover, supercritical fluid has zero surface tension which allows efficient penetration into microporous materials (Rein, Cork and Furton., 1991).

2.2.5.1 Extraction process of SFE

In Sodeifian, Sajadian and Saadati Ardestani., (2017), a bench-scale SC-CO2 apparatus was used to carry out the extraction experiments. An extraction vessel with 74 mL capacity, 0.025 m internal diameter and 0.15 m height was used to load the kotschyi seeds. CO_2 was liquefied by a gas tank equipped with a condenser. To meet the pressure requirement, a pump was used to put the liquefied CO_2 under pressure. Then, the highly pressurized carbon dioxide was fed into the packed bed vessel after passing through a surge tank. The warm water was circulated within the shell-and tube surge tank and vessel at a constant temperature. The SC-CO^2 was fed into the vessels with the outlet back pressure-valve closed for 30 mins static time. After that, the back-pressure valve was opened to initialize the dynamic conditions. Finally, the sample was extracted using an ice and salt bath with a temperature below 0 °C during the dynamic extraction time. The schematic diagram of supercritical carbon dioxide (SC-CO2) extraction is shown in Figure 2.

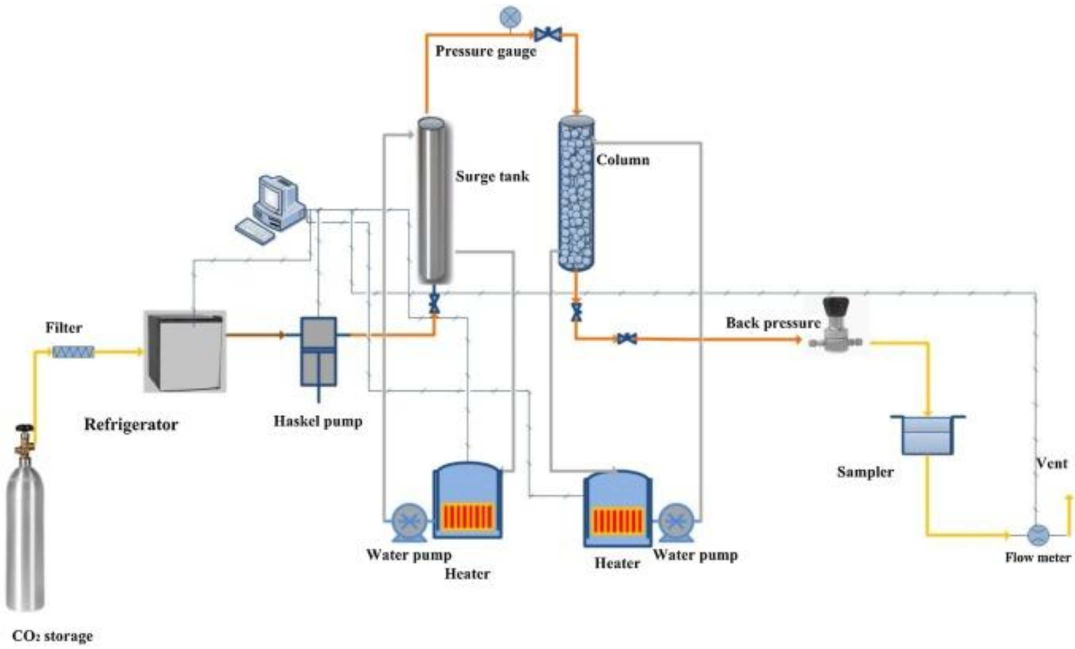

Figure 2. Schematic diagram of supercritical carbon dioxide (SC-CO2) extraction set-up. (Sodeifian, Sajadian and Saadati Ardestani., 2017).

2.2.5.1.1 Advantages of SFE

i. SFE can reduce a lot of time.

ii. It is unnecessary to clean up due to high selectivity achieved by manipulating pressure and temperature.

iii. Suppression of solvent removal steps because the extractant is released from the leached species after depressurization.

2.2.5.1.2 Disadvantages of SFE

i. There is a big difference in efficiency between spiked and natural samples.
ii. The number of methods reported in the literature in which the efficiency is lower than that provided by Soxhlet methods.
iii. The poor ruggedness of SF extractors, especially the restriction and trapping units.

2.2.6 Mechanical Screw Press

A mechanical screw press can separate the liquid from a solid-liquid mixture in a unit operation called solid-liquid expression (Bogaert et al., 2018). It is the most popular method of oil separation from vegetable oilseeds in the world (Mrema& McNulty., 1985 as cited in Singh and Bargale., 2000). Nearly 90% of the total 24 million tonnes of produced oilseeds are crushed by mechanical pressing in India. It is often used in developing countries due to many advantages including simple and sturdy construction, can be maintained and operated by semi-skilled supervisors easily, can be adapted quickly for processing of different kinds of oilseeds and the oil can be obtained in a short time (Singh and Bargale., 2000). Hydraulic presses are commonly used on laboratory and pilot scale to ensure discontinuous and unidirectional compression. The screw press is used at the industrial scale for continuous pressing of oilseeds (Beach., 1983; Hoffman., 1989; Homann et al., 1978; Laisney., 1983; Tindale and Hill Haas., 1976; Ward., 1976 as cited in Bogaert et al., 2018).

2.2.6.1 Extraction process of Mechanical Screw Press

Oil is present in the cells in the form of oil globules with other constituents such as protein, globoids and nucleus. All the constituents are surrounded by a tough membrane called the wall. During the compression process, oilseed medium is fed into the screw press continuously and compressed under high pressure (4 ± 35 MPa) which ruptures the cell walls so that the oil globules escape and flow through the slits provided along the barrel length. The compressed solids are then discharged through a choke provided at the end of the barrel. The detail of the components of screw presses, their functions and some design criteria are discussed by (Singh and Bargale., 2000; Veljković et al., 2018).

The advantages and disadvantages of Mechanical Screw Press according to Veljković et al., (2018) are stated below:

2.2.6.1.1 Advantage of Mechanical screw press

i. Safe and easy to control.
ii. Special knowledge is not required for the operator.
iii. Good for high capacity.
iv. Useful for feedstocks with low oil content.

2.2.6.1.2 Disadvantages of Mechanical screw press

i. Oil may remain in the oil press cake.
ii. Low oil production.
iii. Consume a huge amount of energy.
iv. Maintenance of screw press is required frequently.

2.3 Optimization principles of oil extraction methods

2.3.1 Soxhlet Extraction

To optimize oil extraction methods, solvents selection are very important. In Zarnowski and Suzuki., (2004), Soxhlet extraction and six different types of organic solvents (acetone, chloroform, cyclohexane, diethyl ether, ethyl acetate and hexane) were used to extract lipids from whole grains. The result showed that cyclohexane was the best solvent for the extraction of resorcinolic lipid in whole grain. Therefore, we can see that selection of solvent is important for the extraction. The extraction procedure is also important to get the best result. To meet the requirements for routine usage, a new extraction procedure should be tested for its applicability to laboratory use, efficacy and safety for personnel. Luque de Castro and Priego-Capote, (2010) also mentioned some extraction methods such as ultrasound-assisted Soxhlet extraction and microwave-assisted Soxhlet extraction which can combine with Soxhlet extraction to achieve better performance. Besides, some modified Soxhlet extraction such as high-pressure Soxhlet extraction and automated Soxhlet extraction can also be used to get the best result of extraction (Luque de Castro and Priego-Capote., 2010).

2.3.2 Microwave Assisted Extraction

According to Luque de Castro and García-Ayuso., (1998), the performance of microwave-assisted extraction can be poor if the target analytes or the solvents are not polar or of low-polarity. It is also mentioned that the extraction efficiency can be improved when the polarity of the analytes increased. Therefore, it can see that the selection of solvents is important to get good results. The study of Farhat et al., (2011) showed that by using a microwave, the extraction temperature was increased rapidly to the desired temperature which increased the extraction rates. Therefore, the control of temperature is important for the optimization of MAE.

2.3.3 Ultrasonication Assisted Extraction

According to Mansur et al., (2019), UAE can be optimized by adjusting the temperature and extraction. It is also found out that extraction efficiency can be increased by adding more water.

2.3.4 Pressurized Liquid Extraction

Nieto et al., (2010) mentioned the parameters to optimize PLE and the most important parameters are the extraction solvent, temperature, pressure, static time and the number of cycles. Extraction solvent or a mixture of solvent can be used to ensure that the polarity of the solvent is similar to the target compounds. Temperature and pressure are important parameters because higher temperature decreases the viscosity of liquid solvents which enhance extraction. High temperature also decreases surface tension which allows the solvent to "wet" the sample matrix completely. Also, when the temperature above the solvent boiling point at atmospheric pressure is used, the pressure must be high enough to keep the solvent in the liquid state. The selection of temperature and pressure selection should be high enough to extract the target analytes but low enough not to extract interferences from the sewage sludge. Besides, the static extraction time should be long enough to ensure contact between the analytes and the solvent. The long exposure to the solvent allows the matrix to swell which enhancing penetration of solvent into the sample interstices and contact between solvent analyte. And extending the extraction time from 1 cycle to more cycles maintains favourable solvent/sample equilibrium and thus improves partitioning into the liquid phase. Moreover, increasing the continuous exposure to fresh solvent the extraction efficiency improved substantially.

2.3.5 Supercritical Fluid Extraction

Sahena et al., (2009) mentioned that optimal temperature and pressure are important to the SFE because they affect the extraction yield and the extract composition. Besides, methanol, ethanol and water can be used as co-solvents to change the polarity of SC-CO2 and thus to improve the extraction of polar lipids (Montarini et al., 1996 as cited in Sahena et al., 2009). The presence of water dissolved in the supercritical fluid is also important which can increase the solubility of polar compounds and it has been used successfully to analyze serval dairy products (Dionisi et al., as cited in Sahena et al., 2009). The extraction efficiency can be influenced by the moisture content of the samples since it affects the content structure (Stahl et al., 1988 as cited in Sahena et al., 2009). The lipid recovery can be increased by decreasing the moisture content and this method has been demonstrated in wet samples such as meat (King et al., 1989 as cited in Sahena et al., 2009). To improve efficiency, the sample should be frozen and dried before SC-CO2 extraction (Yamaguchi et al., 1986 as cited in Sahena et al., 2009).

2.3.6 Mechanical Screw Press

In the 1980s, the mechanical screw presses are inefficient, leaving about 8-14% of the available oil in the cake (Srikantha., 1980 as cited in Singh and Bargale., 2000). A large amount of edible oil (about 0.6 million tonnes) which worth US$57 million remains in the de-oiled cake annually (Singh and Bargale., 2000). Pathak, Singh, Singh & Verma., (1988) as cited in Singh and Bargale., (2000) raised oil recovery from 73% to 80% for rapeseed and peanut and from 60 % to 65 % for cotton seeds by improving the equipment of mechanical extraction and techniques. Tindale & Hass., (1976); Ward., (1976); Bredeson., (1983); Khan & Hanna., (1983); Nelson, Wijeratne, Yeh, Wei & Wei., (1987); Ohlson., (1992); Williams., (1995); Bargale, Ford, Sosulski, Wulfson& Irudayaraj., (1999) as cited in Singh and Bargale., (2000) improved the oil extraction efficiency of screw press by considering the physical (e.g. dehulling, cracking, size reduction), thermal (e.g. preheating, dry extrusion), hydrothermal (e.g. hot water soaking, steaming, blanching, flaking) and chemical (enzymatic hydrolysis) pre-treatments. Also, Ohlson., (1992) as cited in Singh and Bargale., (2000) increased the oil recovery levels by optimizing the process variables such as applied pressure, pressing temperature and moisture conditioning of the fed samples.

2.4 Biodiesel fuel production from oil-bearing crops

Biodiesel is an alternative fuel for a diesel engine which is produced by animal fats or vegetable oil. In this chapter of the thesis, consideration is also given to biodiesel production from oilseeds. According to the American Society for Testing and Materials International, biodiesel is a fuel composed of monoalkyl esters of long-chain fatty acids derived from renewable vegetable oils or animal fats meeting the requirements of ASTM D6751 (ASTM2008a). The main constituent of vegetable oils and animal fats are triacylglycerols (TAG) which are consisted of glycerol (1,2,3-propanetriol) backbone bonding to long-chain fatty acids (Moser., 2009). The name ‘‘biodiesel’’ has been given to transesterified oil to describe its use as a Diesel fuel (Demirbaş., 2002). The earliest application of biodiesel, an original Diesel engine to run on vegetable oil was invented by Rudolph Diesel around 120 years ago. He also illustrated the method of using peanut oil to run the engine at the Paris Exposition in 1900 (Nitske., 1965). On the other hand, Scientists E. Duffy and J. Patrick conducted transesterification in 1853 and transesterified vegetable oil was powering heavy-duty vehicles in South Africa before World War II (Demirbaş., 2002). The most common international standards for biodiesel are the European EN14214 and the American Society for Testing and Materials (ASTM) D-6751. The requirement of the quality of different types of biodiesels is based on different factors and regions such as the availability of feedstock, the characteristics of the diesel fuel standard existing in each region, the predominance of diesel engine type in the region and the emission regulations governing those engines. In Europe, the diesel passenger car market is so much larger compared to the US, while the markets are mainly focused on the heavier duty diesel engines in the US. Therefore, there are some significant differences between the two standards (Sarin., 2012; Hassan and Kalam., 2013). The advantages and disadvantages of biodiesel according to Hassan and Kalam., (2013) are listed below:

2.4.1 Advantages

i. Biodiesel is portable, available and renewable.

ii. Comparing to diesel, fewer air pollutants such as CO_2, CO, SO_2, PM and HC are produced when using biodiesel.

iii. Biodiesel can be produced in a shorter time and more easily compare to diesel.

iv. For the reason that the cetane number of biodiesels is over 100, it can make the vehicles perform in a better way. The engine life can be prolonged, and the need for maintenance can be reduced.
v. Different to a diesel engine, an additional lubricant is dispensable for biodiesel due to its purity and clarity.
vi. Unlike diesel, drilling and refining are not required for biodiesel.
vii. Biodiesel can be produced locally so that it is more cost-efficient than diesel.
viii. Properties of biodiesel such as sulphur content, flash point, aromatic content and biodegradability are better than diesel fuel.
ix. Biodiesel is safer to handle, less toxic, more biodegradable and have a higher flashpoint.
x. Biodiesel is non-flammable and non-toxic, reduces tailpipe emissions, visible smoke and noxious fumes and odours.
xi. Biodiesel has a higher combustion efficiency.

2.4.2 Disadvantages

i. Comparing to diesel, the higher amount of NOx is produced by biodiesel.
ii. In cold weather, biodiesel has a higher pour and cloud point.
iii. The corrosive nature of biodiesel can damage copper and brass.
iv. For the reason that vegetable oil has a large molecule mass and chemical structure, the viscosity of biodiesel is about 11–17 times higher than diesel.
v. It can cause pumping, combustion and atomization in the injector system of a diesel engine.
vi. Engine speed and power can be reduced by biodiesel.
vii. Degrading of biodiesel occur due to long time storage.
viii. Injectors on piston and head of engine can be coked by biodiesel.
ix. Carbon deposits are produced on piston and head of the engine.

2.4.3 Transesterification process for biodiesel production

Hydrolyzing triglycerides (organic fats and oils) can form free fatty acids which then react with alcohol and formed ester or biodiesel (methyl or ethyl fatty ester) as well as glycerol (Figure 3). For the reason that free fatty acid reacts with alcohol, transesterification is also called alcoholysis. Because of the weight difference, the end products of the transesterification process are separated. Biodiesel settles on the top and glycerol settles on the bottom. The most common chemicals for transesterification are methanol and ethanol. Transesterification is also known as methanolysis when methanol is used to react with free fatty acids in the process.

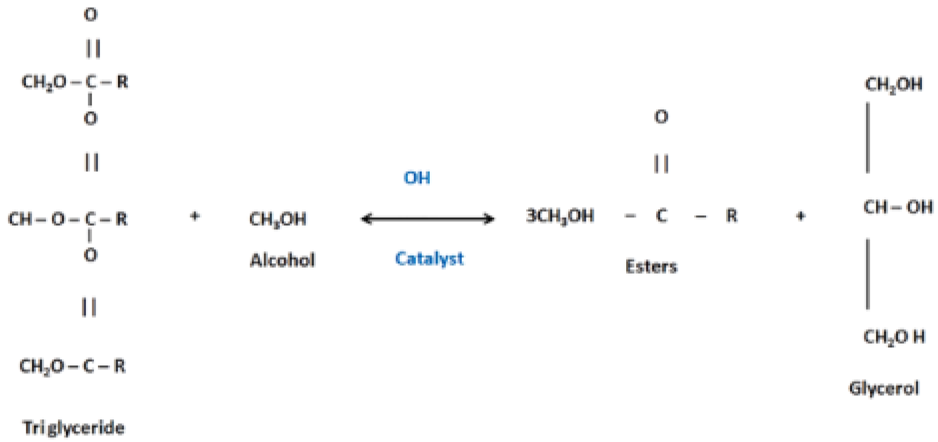

Figure 3. Transesterification reaction for biodiesel production (Singh et al., 2020).

2.4.4 Fuel characteristics of biodiesel

Properties of various types of biodiesels are distinct due to the unique chemical composition of different types of feedstock. To have better utilization of various types of biodiesel, it is important to analyze the properties of biodiesel such as oxidation stability, low-temperature operability, kinematic viscosity, sulfated ash, sulfur, alkali and alkaline earth metals, flash point, cetane number, methanol or ethanol content, copper strip content, copper strip corrosion, phosphorus content, ester content, distillation temperature, total contamination, water and sediments, acid number, free glycerin, mono-, di- and triglycerides, density, iodine value, linolenic acid methyl ester and polyunsaturation content. A brief description of these properties is described below.

2.4.4.1 Oxidation Stability

Oxidation stability is one of the most important properties of biodiesel. It primarily affects the stability of biodiesel during extended storage. Degradation by oxidation yields products may damage fuel properties, reduce fuel quality and engine performance (Pullen and Saeed., 2012).

2.4.4.2 Low-Temperature Operability

Fuel starvation and operability problems can cause crystallization of the saturated fatty acid easily in winters due to solidified material clogs to fuel lines and filters. The lower the temperature, the more solid is formed in the biodiesel. Once the material approaches the pour point, the biodiesel stops flowing (Dwivedi and Sharma., 2014).

2.4.4.3 Kinematic Viscosity

Using vegetable oils or fats with high viscosity as diesel fuels can cause operational problems such as poor atomization upon injection into the combustion chamber and engine deposit. Also, high viscosity at low temperature can interfere with the transport of the fuel from the tank to the engine. Transesterifying oils or fats to the corresponding alkyl esters can reduce the viscosity (Knothe and Steidley., 2007).

2.4.4.4 Sulfated Ash

Sulfated ash is a measure of ash formed from inorganic metallic compounds. When biodiesel is burned, it leaves some metallic ash and unburned hydrocarbon. Metallic ash can cause serious damage to the interface between the piston ring and the cylinder wall. To measure sulfated ash, the ash (metallic ash and unburned hydrocarbon) is collected after biodiesel is burned. The ash is then treated with sulfuric acid and heated to 775°C. This completely oxidizes the carbon residue, which evaporates as CO_2. All the metallic ash is transformed into metallic sulfates, such as calcium sulfate which is reported as sulfated ash after completely drying (Shrestha., 2019).

2.4.4.5 Sulfur

Sulfur is harmful to humans. Also, ultra-low sulfur diesel is hard to lubricate diesel engine fuel pumps due to the insufficient lubricant characteristics. Therefore, it is necessary to monitor the amount of sulfur when using biodiesel. Biodiesel made from virgin soybean oil does not contain any sulfur because soybean oil does not contain sulfur. However, Canola, rapeseed and mustard contain glucosinolates which are sulfur-containing compounds. Besides, some used vegetable oil may also contain sulfur. Ultraviolet fluorescence can be used to perform total sulfur analysis (Shrestha., 2019).

2.4.4.6 Flash Point

The flashpoint of a fuel is the lowest temperature at which its vapour can be ignited. The flashpoint is not related to engine performance directly, however, it is one of the most important considerations when storing and handling the fuel. The flashpoint for biodiesel is set at 93°C (200°F) minimum. Therefore, biodiesel falls under the non-hazardous category of the National Fire Protection Association codes (Shrestha., 2019).

2.4.4.7 Cetane Number

Ignition delay time and combustion quality are related to cetane number. The higher the cetane number the better the ignition properties of the fuel. Formation of white smoke can be minimized, and good cold-start properties can be ensured with high cetane numbers. Normally, biodiesel has slightly higher cetane numbers than fossil diesel (Sarin., 2012).

2.4.4.8 Phosphorus Content

Phosphorus level must be kept low because it can damage catalytic converters in the emissions control systems. Biodiesel produced from the US has been shown to have a low phosphorus content (below 1 ppm) and the specification maximum value is 10 ppm (ASTM, 2009 as cited in Shrestha., 2019).

2.4.4.9 Ester Content

Low ester content value may cause by inappropriate reaction conditions or various minor components within the original oil source. Ester content is measured according to EN 14103 where it is limited to a minimum of 96.5% m/m. There is no ASTM method or limit for ester content (Sarin., 2012).

2.4.4.10 Distillation Temperature

Distillation temperature can be used to determining the presence of different substances in the fuel such as ester content. The distillation temperature (90% recovered) is measured according to ASTM D-1160 where it is limited to 360 UC. There is no European method or limit for distillation temperature (90% recovered) (Sarin., 2012).

3 MATERIALS AND METHODS

In this chapter, Response Surface Methodology (RSM) was employed to study the mechanical behaviour of sunflower and rape bulk oilseeds under compression loading. Three independent process variables: compression speed (5, 10 and 15 mm/min), heat-treatment temperature (45, 60 and 75 °C) and compression force (60, 80 and 100 kN) were studied and optimized. Using the Box-Behnken approach, a set of experimental data was analyzed by STATISTICA software (Statsoft, 2010).

3.1 Experimental procedure

The experiment was conducted at the laboratory of the Mechanical Department of Faculty of Engineering.

3.1.1 Box-Behnken Design

The mathematical model describing the Box-Behnken Design (BBD) according to Huang et al., (2019) is given in equation (12).

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i_{1<j}}^{k} \sum_{j}^{k} \beta_{ij} X_i X_j \tag{12}$$

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. The coded values (-1, 0 and +1) of the independent variables (factors) were determined according to Ocholi et al., (2018) and Witek-Krowiak et al., (2014) as given in equation (13).

$$x_i = \frac{X_i - X_0}{\Delta X} \tag{13}$$

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.

3.1.2 Moisture content

The moisture content of the sunflower seeds and rapeseeds was determined as 5.16 ± 0.01% (w.b.) and 5.30 ± 0.04% (w.b.) was calculated based on the relation given by Blahovec (2008) as given in equation (14).

$$MC = \left[\left(\frac{m_b - m_a}{m_b}\right) \cdot 100\right] \qquad (14)$$

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

3.1.3 Determination of oil content in seeds

The percentage oil content contained in the seeds of sunflower and rape was determined using the Soxhlet extraction procedure described by Niu et al., (2014), Danlami et al., (2015) and Gurkan et al., (2020). The oil content of sunflower seeds was calculated to be 33.12 ± 0.87% and that of rapeseeds was 31.87 ± 2.40%.

3.1.4 Preparation of samples for heat-treatment

Before the compression test, impurities such as stones, dust, leaves among others were removed manually removed from the seeds. The samples were pre-treated at 45, 60 and 75 °C at the constant heating time of 1 hour using the Memmert oven, type UF110, Germany (Appendix 1) (Sirisoomboon and Kitchaiya, 2009)

3.1.5 Compression test of pre-heated samples

The compression test of the pre-heated samples was done universal compression testing machine (ZDM 50, Czech Republic) and a pressing vessel of diameter 60 mm with a plunger and a pan for collecting the pressed oil (Figure 11). After that, an electronic balance with an accuracy of 0.01 g was used to measure the mass of seedcake.

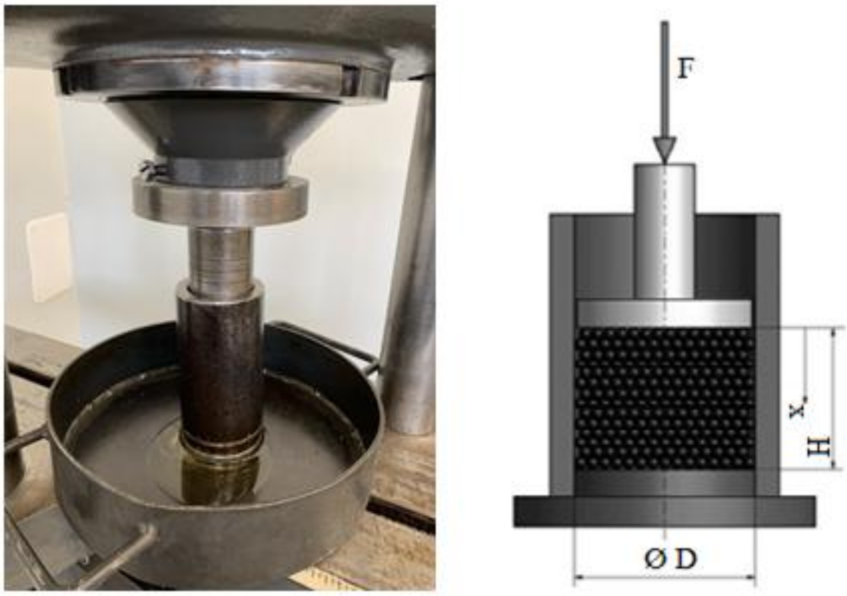

Figure 4. Schematic of pressing vessel diameter D (mm) with a plunger; F: force (kN); H: initial pressing height of sample (mm) and x: sample deformation (mm) (Kabutey et al., 2015).

3.2 Calculated parameters from the compression test

3.2.1 Oil yield (%)

The oil yield was calculated based on the relation reported by Deli et al., (2011) as given in equation (15).

$$OY = \left[\left(\frac{m_o}{m_s}\right) \cdot 100\right] \tag{15}$$

where OY is percentage oil yield (%), m_o is the mass of oil obtained as the difference of mass of seed cake and initial mass of the sample m_s (g).

3.2.2 Oil expression efficiency (%)

The oil expression efficiency was calculated according to the relation given by Hernandez-Santos et al., (2016) as given in equation (16).

$$OEE = \left[\left(\frac{OY}{O_s}\right) \cdot 100\right] \tag{16}$$

where O_s is percentage of oil content (%).

3.2.3 Deformation energy (J)

The deformation energy (oil expression energy) (Appendix 2) was calculated according to the relation given by Demirel et al., (2018), Divišová et al., (2014) and Gupta and Das, (2000) as given in equation (17).

$$DE = \sum_{n=0}^{n=i-1} \left[\left(\frac{F_{n+1} + F_n}{2} \right) \cdot (x_{n+1} - x_n) \right] \quad (17)$$

where DE 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.

4 RESULTS AND DISCUSSION

4.1 Calculated amounts of sunflower seeds and rapeseeds

In the control experiment, the mass of sunflower seeds and rapeseeds oil (g) was determined for varying compression speed (mm min^{-1}) (Table 1) and (Table 2). For sunflower seeds (Table 1), the highest mass of oil was 13.9 ± 0.42 g which was obtained at a speed of 5 mm min^{-1}. And the lowest mass of oil was 8.6 ± 0.00 g which was obtained at a speed of 15 mm min^{-1}. For rapeseeds (Table 2), the highest mass of oil was 14.96 ± 2.69 g which was obtained at a speed of 5 mm min^{-1}. And the lowest mass of oil was 8.36 ± 0.78 g which was obtained at the speed of 15 mm min^{-1}. The lower the compression speed the higher the mass of oil which confirms the results reported by Deli et al., 2011. Based on the Box-Behnken Design, 17 experiments for both sunflower seeds and rapeseeds where run for the combination of speeds (5, 10, 15) mm min^{-1}, heating temperatures (45, 60, 75) ^{o}C and forces (60, 80, 100) kN. For sunflower seeds (Table 1), the highest mass of oil was 18.5 g where the compression speed, heating temperature and compression force were at 5 mm min^{-1}, 60 ^{o}C and 100 kN. And the lowest mass of sunflower seeds oil was 11 g where the compressions peed, heating temperature and compression force were 15 mm min^{-1}, 45 ^{o}C and 80 kN. For rapeseeds (Table 2), the highest mass of oil was 30.23 g with the corresponding speed, heating temperature and force of 5 mm min^{-1}, 60 ^{o}C and 100 kN. And the lowest mass of oil was 12.09 g at compression speed, heating temperature and compression force of 15 mm min^{-1}, 45 ^{o}C and 80 kN respectively.

For the control and actual experiments, the deformation, oil yield, oil expression efficiency and deformation energy were determined/calculated as given in (Table 3) and (Table 4) respectively.

Table 1. Determination of the mass of oil for different factors of sunflower seeds compression

Run (-)	Speed (mm min^{-1}) q_1	Temperature (^oC) q_2	Force (kN) q_3	M_b (g)	M_a (g)	M_o (g)
CTR*	5	25	100	79.3	65.4 ±0.42	13.9 ±0.42
	10	25	100	79.3	69.45 ±0.07	9.95 ±0.07
	15	25	100	79.3	70.7 ±0.00	8.6 ±0.00
1	5	45	80	79.3	64.2	15.1
2	15	45	80	79.3	68.3	11
3	5	75	80	79.3	62.4	16.9
4	15	75	80	79.3	64.4	14.9
5	5	60	60	79.3	64.9	14.4
6	15	60	60	79.3	65.9	13.4
7	5	60	100	79.3	60.8	18.5
8	15	60	100	79.3	64.6	14.7
9	10	45	60	79.3	66.5	12.8
10	10	75	60	79.3	64.1	15.2
11	10	45	100	79.3	65.2	14.1
12	10	75	100	79.3	63	16.3
13	10	60	80	79.3	64.2	15.1
14	10	60	80	79.3	63.8	15.5
15	10	60	80	79.3	64.3	15
16	10	60	80	79.3	63.9	15.4
17	10	60	80	79.3	64.4	14.9

CTR*: Control of samples at laboratory temperature of 25 ^oC; M_b: Mass of sample before heat treatment and compression; M_a: Mass of sample after compression; M_o: Mass of oil after compression

Table 2. Determination of the mass of oil for different factors of rapeseeds compression

Run (-)	Speed (mm min^{-1}) Q_1	Temperature (^oC) Q_2	Force (kN) Q_3	M_b (g)	M_a (g)	M_o (g)
CTR	5	25	100	119.16	104.2 ±2.68	14.96 ±2.69
	10	25	100	119.16	109.94 ±1.07	9.23 ±1.07
	15	25	100	119.16	110.38 ±0.78	8.36 ±0.78
1	5	45	80	119.16	101.98	17.18
2	15	45	80	119.16	107.07	12.09
3	5	75	80	119.16	97.38	21.78
4	15	75	80	119.16	102.5	16.66
5	5	60	60	119.16	96.63	22.53
6	15	60	60	119.16	104.84	14.32
7	5	60	100	119.16	88.93	30.23
8	15	60	100	119.16	94.10	25.06
9	10	45	60	119.16	104.19	14.97
10	10	75	60	119.16	104.65	14.51
11	10	45	100	119.16	103.63	15.53
12	10	75	100	119.16	99.45	19.71
13	10	60	80	119.16	101.74	17.42
14	10	60	80	119.16	100.77	18.39
15	10	60	80	119.16	104.8	14.36
16	10	60	80	119.16	101.47	17.69
17	10	60	80	119.16	102.32	16.84

CTR*: Control of samples at laboratory temperature 25 ^oC; M_b: Mass of sample before heat treatment and compression; M_a: Mass of sample after compression; M_o: Mass of oil after compression.

Table 3. Box-Behnken design with observed responses for different factors of sunflower seeds compression

Run (-)	Factors			Observed responses			
	Speed (mm min^{-1}) q_1	Temperature (oC) q_2	Force (kN) q_3	DX (mm)	OY (%)	OEE (%)	DE (J)
CTR*	5	25	100	42.73 ±0.73	17.53 ±0.54	52.92 ±1.62	449.12 ±3.59
	10	25	100	41.01 ±0.74	12.42 ±0.08	37.50 ±0.27	434.91 ±0.36
	15	25	100	40.9 ±1.40	10.84 ±0.00	32.74 ±0.00	433.30 ±17.19
1	5 (-1)	45 (-1)	80 (0)	39.11	19.04	57.49	426.62
2	15 (1)	45 (-1)	80 (0)	41.25	13.87	41.88	427.09
3	5 (-1)	75 (1)	80 (0)	40.21	21.31	64.34	447.56
4	15 (1)	75 (1)	80 (0)	38.37	18.79	56.73	410.72
5	5 (-1)	60 (0)	60 (-1)	39.39	18.16	54.83	382.09
6	15 (1)	60 (0)	60 (-1)	37.27	16.90	51.03	379.56
7	5 (-1)	60 (0)	100 (1)	40.50	23.33	70.44	490.20
8	15 (1)	60 (0)	100 (1)	37.85	18.54	55.98	461.99
9	10 (0)	45 (-1)	60 (-1)	38.93	16.14	48.73	376.91
10	10 (0)	75 (1)	60 (-1)	39.34	19.17	57.88	375.99
11	10 (0)	45 (-1)	100 (1)	39.59	17.78	53.68	468.56
12	10 (0)	75 (1)	100 (1)	38.29	20.55	62.05	493.05
13	10 (0)	60 (0)	80 (0)	40.07	19.04	57.49	425.01
14	10 (0)	60 (0)	80 (0)	38.82	19.55	59.03	426.85
15	10 (0)	60 (0)	80 (0)	38.67	18.92	57.13	410.81
16	10 (0)	60 (0)	80 (0)	40.81	19.42	58.64	430.01
17	10 (0)	60 (0)	80 (0)	40.56	18.79	56.73	445.61

CTR*: Control of samples at laboratory temperature 25 ^{o}C; DX: Deformation of bulk sample; OY: Oil Yield; OEE: Oil Expression Efficiency; DE: Deformation Energy

Table 4. Box-Behnken design with observed responses for different factors of rapeseeds compression

Run (-)	Factors			Observed responses			
	Speed (mm min^{-1}) Q_1	Temperature (^{o}C) Q_2	Force (kN) Q_3	DX (mm)	OY (%)	OEE (%)	DE (J)
CTR*	5	25	100	29.2 ±1.00	12.55 ±2.26	39.4 ±7.08	517.3 ±19.11
	10	25	100	29.19 ±1.74	7.75 ±0.90	24.29 ±2.82	460.48 ±5.95
	15	25	100	27.48 ±1.13	7.02 ±0.66	22.02 ±2.04	432.91 ±7.33
1	5 (-1)	45 (-1)	80 (0)	29.79	14.42	45.24	501.09
2	15 (1)	45 (-1)	80 (0)	27.07	10.15	31.84	441.74
3	5 (-1)	75 (1)	80 (0)	32.07	18.28	57.35	570.81
4	15 (1)	75 (1)	80 (0)	29.53	13.98	43.87	490.06
5	5 (-1)	60 (0)	60 (-1)	26.21	18.91	59.33	430.59
6	15 (1)	60 (0)	60 (-1)	25.64	12.02	37.71	397.16
7	5 (-1)	60 (0)	100 (1)	26.97	25.37	79.60	521.68
8	15 (1)	60 (0)	100 (1)	26.26	21.03	65.99	469.59
9	10 (0)	45 (-1)	60 (-1)	28.49	12.56	39.42	430.19
10	10 (0)	75 (1)	60 (-1)	29.16	12.18	38.21	462.97
11	10 (0)	45 (-1)	100 (1)	27.82	13.03	40.89	524.28
12	10 (0)	75 (1)	100 (1)	30.79	16.54	51.90	553.72
13	10 (0)	60 (0)	80 (0)	28.36	14.62	45.87	526.57
14	10 (0)	60 (0)	80 (0)	29.97	15.43	48.42	531.65
15	10 (0)	60 (0)	80 (0)	30.22	12.05	37.81	523.44
16	10 (0)	60 (0)	80 (0)	27.67	14.85	46.58	493.38
17	10 (0)	60 (0)	80 (0)	28.62	14.13	44.34	487.77

CTR*: Control of samples at laboratory temperature 25 ^{o}C; DX: Deformation of bulk sample; OY: Oil Yield; OEE: Oil Expression Efficiency; DE: Deformation Energy

4.2 Force-deformation curve characteristics of sunflower seeds and rapeseeds

The compressive force and deformation curves of sunflower seeds and rapeseeds with compression speed are illustrated in (Figure 5) and (Figure 6). The area under the curve is the deformation energy (Demirel et al., 2017; Divisova et al., 2014; Gupta and Das., 2000). For sunflower seeds (Figure 5), the highest and lowest amounts of deformation energy were observed at speed 10 mm min^{-1} and 5 mm min^{-1} respectively. For rapeseeds (Figure 6), the highest and lowest amounts of deformation energy were noticed at speed 5 mm min^{-1} and 10 mm min^{-1} correspondingly. Besides, the force-deformation curves of sunflower seeds and rapeseeds in relation to compression speed, heating temperature and compression force are illustrated in (Figure 7) and (Figure 8). For sunflower seeds (Figure 7), the highest deformation energy was observed at a speed of 15 mm min^{-1}, the heating temperature of 60 ^{o}C and a compression force of 100 kN. And the lowest deformation energy for compression of sunflower seeds was found at speed of 5 mm min^{-1}, the heating temperature of 60 ^{o}C and compression force of 60 kN. For rapeseeds (Figure 8), the highest deformation energy was seen at the speed of 15 mm min^{-1}, the heating temperature of 60 ^{o}C and a compression force of 100 kN. And the lowest deformation energy at speed of 10 mm min^{-1}, the heating temperature of 75 ^{o}C and compression force of 100 kN.

All the force-deformation curves below showed a smooth curve behaviour which meant the maximum oil output was obtained based on the input factors. There was no serration effect on the curves which has been reported as the ejection of the seed cake through the holes of the pressing vessel as a result of higher compressive force, moisture content, speed and smaller pressing vessel (Divisova et al., 2014; Kabutey et al., 2014; Gupta and Das, 2000).

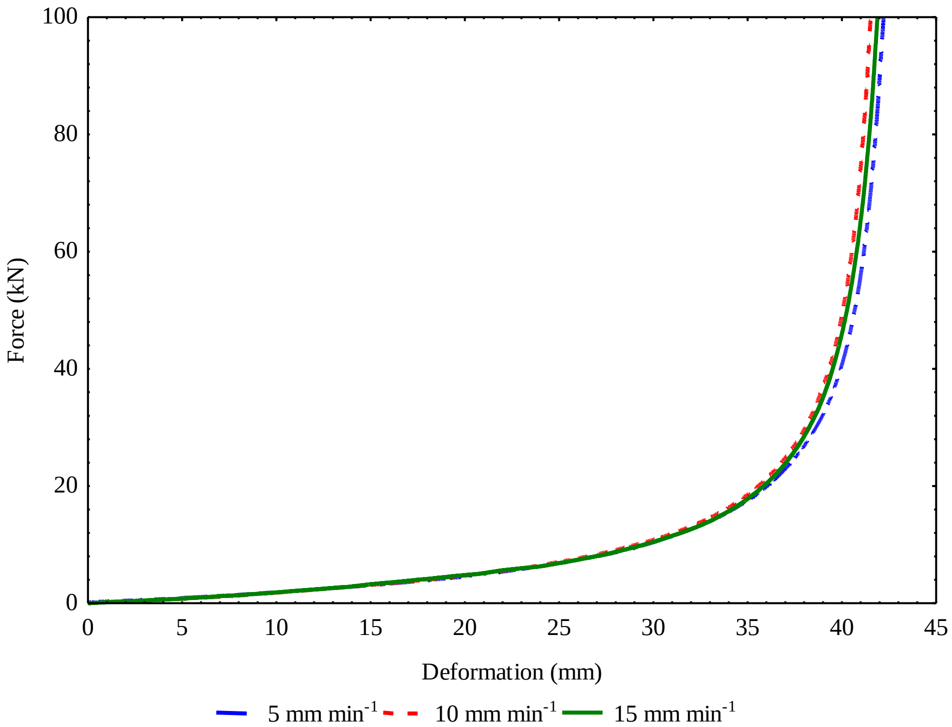

Figure 5. Force and deformation curve characteristics of sunflower seeds (control) in relation to speed.

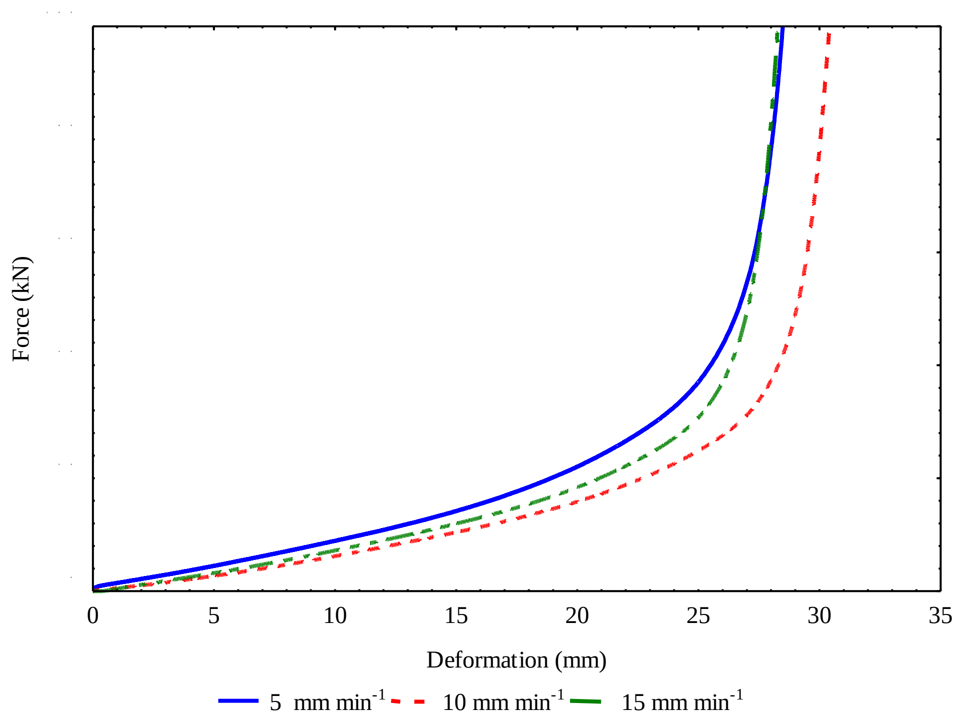

Figure 6. Force and deformation curve characteristics of rapeseeds (control) in relation to speed.

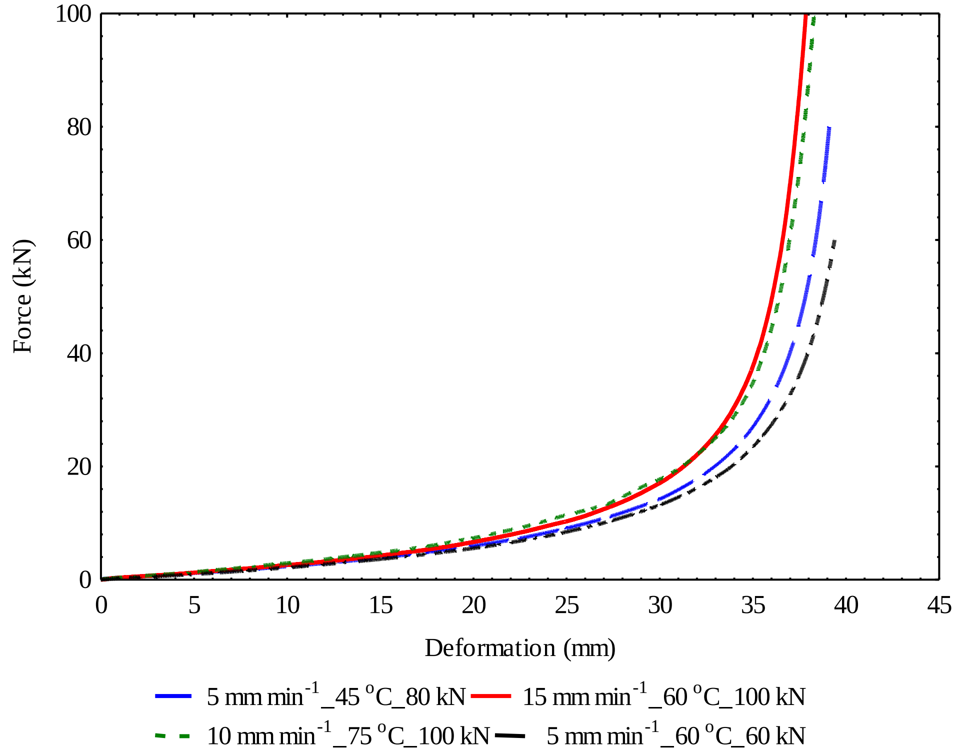

Figure 7. Force and deformation curve characteristics of sunflower seeds in relation to pressing factors.

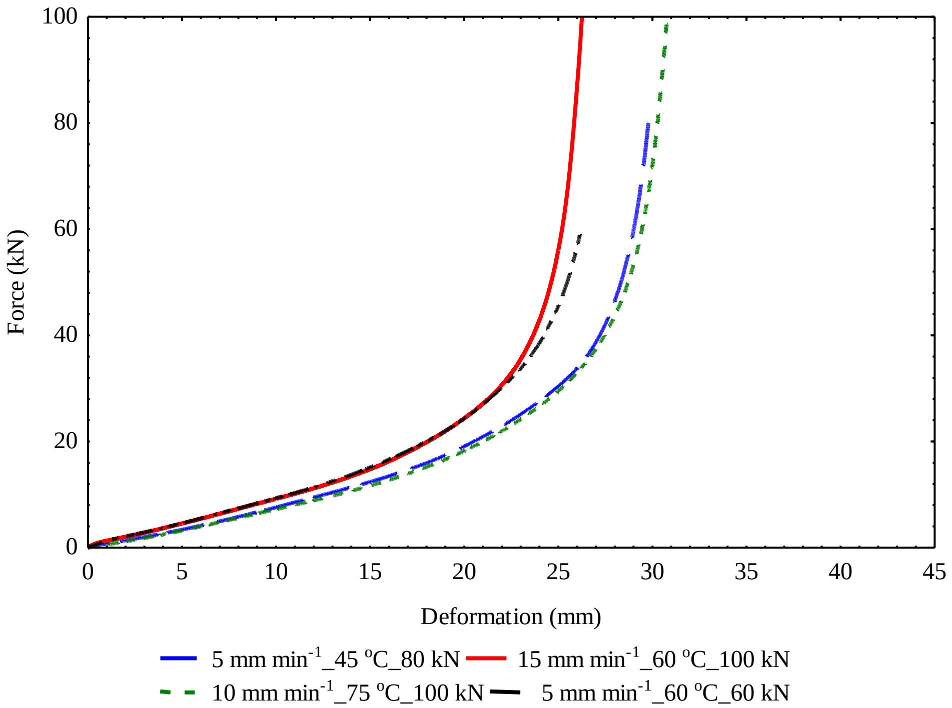

Figure 8. Force and deformation curve characteristics of rapeseeds in relation to pressing factors.

4.3 Response regression models describing calculated parameters of sunflower seeds and rapeseeds.

The response surface methodology was applied to study how the response variables: oil yield (%), oil expression efficiency (%) and deformation energy (J) were affected by the independent variables: speeds (5, 10, 15) mm min^{-1}, heating temperatures (45, 60, 75) °C and forces (60, 80, 100) kN of sunflower seeds and rapeseeds oil processing under linear compression. The regression models of the response variables based on the ANOVA results are presented in Tables 9 to 16 respectively. The significance of the models was evaluated based on F-values > P-values or P-values < 0.05.

For sunflower seeds, (Table 5 and 6), the quadratic terms of speed, force as well as the interaction terms of speed and temperature, and then temperature and force for oil yield (%) were not significant (F-values < P-values or P-values > 0.05. The significant terms of oil yield (%) model including the intercept (P-values < 0.05) are given in equation (18).

For rapeseeds, (Table 7 and 8), the quadratic term of temperature, as well as interaction terms of speed and temperature, speed and force and temperature and force, were not significant (P-values > 0.05) or smaller F-values. The significant terms of the model including the intercept (P-values < 0.05) for oil yield are given in equation (19).

Table 5. Analysis of variance for the response surface of oil yield, OY (%) of sunflower seeds

Model	Model coefficients	Standard Error	t-value	p-value
Intercept	19.14	0.28	67.68	0.00
q_1	-1.72	0.22	-7.68	0.00
q_1^2	-0.03	0.31	-0.11	0.91
q_2	1.62	0.22	7.26	0.00
q_2^2	-0.86	0.31	-2.78	0.03
q_3	1.23	0.22	5.49	0.00
q_3^2	0.12	0.31	0.40	0.70
$q_1 \cdot q_2$	0.66	0.32	2.09	0.07
$q_1 \cdot q_3$	-0.88	0.32	-2.79	0.03
$q_2 \cdot q_3$	-0.07	0.32	-0.21	0.84

q_1: Speed (mm min^{-1}); q_2: Temperature (^{o}C); q_3: Force (kN);

Table 6. Analysis of variance for the response surface of oil yield, OY (%) of sunflower seeds

Effect	Sum of Squares	Degree of freedom	Mean Square	F-value	p-value
Intercept	64.80	9	7.20	17.99	0.00
q_1	23.60	1	23.60	58.98	0.00
q_1^2	0.01	1	0.01	0.01	0.91
q_2	21.09	1	21.09	52.72	0.00
q_2^2	3.09	1	3.09	7.73	0.03
q_3	12.08	1	12.08	30.19	0.00
q_3^2	0.06	1	0.06	0.16	0.70
$q_1 \cdot q_2$	1.76	1	1.76	4.39	0.07
$q_1 \cdot q_3$	3.12	1	3.12	7.79	0.03
$q_2 \cdot q_3$	0.02	1	0.02	0.04	0.84
Error	2.80	7	0.40	-	-
Total	67.59	16	-	-	-

q_1: Speed (mm min^{-1}); q_2: Temperature (^{o}C); q_3: Force (kN);

$$OY_S = 19.14 - 1.72 \cdot q_1 + 1.62 \cdot q_2 - 0.86 \cdot q_2^2 + 1.23 \cdot q_3 - 0.88 \cdot q_1 \cdot q_3 \quad (18)$$

Table 7. Analysis of variance for the response surface of oil yield, OY (%) of rapeseeds

Model	Model coefficients	Standard Error	t-value	p-value
Intercept	14.22	0.83	17.04	0.00
Q_1	-2.48	0.66	-3.75	0.01
Q_1^2	2.87	0.91	3.16	0.02
Q_2	1.35	0.66	2.05	0.08
Q_2^2	-2.88	0.91	-3.17	0.02
Q_3	2.54	0.66	3.85	0.01
Q_3^2	2.24	0.91	2.47	0.04
$Q_1 \cdot Q_2$	-0.01	0.93	-0.01	0.99
$Q_1 \cdot Q_3$	0.64	0.93	0.68	0.52
$Q_2 \cdot Q_3$	0.97	0.93	1.04	0.33

Q_1: Speed (mm min^{-1}); Q_2: Temperature (^{o}C); Q_3: Force (kN)

Table 8. Analysis of variance for the response surface of oil yield, OY (%) of rapeseeds

Effect	Sum of Squares	Degree of freedom	Mean Square	F-value	p-value
Intercept	208.10	9	23.12	6.64	0.00
Q_1	49.01	1	49.01	14.07	0.01
Q_1^2	34.76	1	34.76	9.98	0.02
Q_2	14.63	1	14.63	4.20	0.08
Q_2^2	34.97	1	34.97	10.04	0.02
Q_3	51.51	1	51.51	14.79	0.01
Q_3^2	21.19	1	21.19	6.09	0.04
$Q_1 \cdot Q_2$	0.00	1	0.00	0.00	0.99
$Q_1 \cdot Q_3$	1.63	1	1.63	0.47	0.52
$Q_2 \cdot Q_3$	3.78	1	3.78	1.09	0.33
Error	24.37	7	3.48	-	-
Total	232.48	16	-	-	-

Q_1: Speed (mm min^{-1}); Q_2: Temperature (^{o}C); Q_3: Force (kN)

$$OY_R = 14.22 - 2.48 \cdot Q_1 + 2.87 \cdot Q_1^2 - 2.88 \cdot Q_2^2 + 2.54 \cdot Q_3 + 2.24 \cdot Q_3^2 \qquad (19)$$

For oil expression efficiency (%) (Table 9 and 10) for sunflower seeds, the linear terms of speed, temperature and force; quadratic terms of temperature and force as well as the interaction effects of speed and force were significant (F-values > P-values or P-values < 0.05). The rest of the terms were not significant (P-values > 0.05). The significant terms of the model (oil expression efficiency %) including intercept are given in equation (20).

For rapeseeds (Table 11 and 12), the linear and quadratic terms of speed, the quadratic term of temperature as well as linear and quadratic terms of force were significant (F-values > P-values or P-values < 0.05). The other terms including interaction effects were not significant. The significant terms of the model including the intercept for rapeseeds oil expression efficiency are given in equation (21).

Table 9. Analysis of variance for the response surface of oil expression efficiency, OEE (%) of sunflower seeds

Model	Model coefficients	Standard Error	t-value	p-value
Intercept	57.80	0.85	67.69	0.00
q_1	-5.19	0.68	-7.68	0.00
q_1^2	-0.10	0.93	-0.11	0.91
q_2	4.90	0.68	7.26	0.00
q_2^2	-2.59	0.93	-2.78	0.03
q_3	3.71	0.68	5.50	0.00
q_3^2	0.37	0.93	0.40	0.70
$q_1 \cdot q_2$	2.00	0.95	2.09	0.07
$q_1 \cdot q_3$	-2.67	0.95	-2.79	0.03
$q_2 \cdot q_3$	-0.20	0.95	-0.20	0.84

q_1: Speed (mm min^{-1}); q_2: Temperature (^{o}C); q_3: Force (kN)

Table 10. Analysis of variance for the response surface of oil expression efficiency, OEE (%) of sunflower seeds

Effect	Sum of Squares	Degree of freedom	Mean Square	F-value	p-value
Intercept	590.71	9	65.63	17.99	0.00
q_1	215.07	1	215.07	58.98	0.00
q_1^2	0.05	1	0.05	0.01	0.91
q_2	192.28	1	192.28	52.73	0.00
q_2^2	28.23	1	28.23	7.74	0.03
q_3	110.11	1	110.11	30.20	0.00
q_3^2	0.58	1	0.58	0.16	0.70
$q_1 \cdot q_2$	16.00	1	16.00	4.39	0.07
$q_1 \cdot q_3$	28.41	1	28.41	7.79	0.03
$q_2 \cdot q_3$	0.15	1	0.15	0.04	0.84
Error	25.53	7	3.65	-	-
Total	616.24	16	-	-	-

q_1: Speed (mm min^{-1}); q_2: Temperature (^{o}C); q_3: Force (kN);

$$OEE_S = 57.80 - 5.19 \cdot q_1 + 4.90 \cdot q_2 - 2.59 \cdot q_2^2 + 3.71 \cdot q_3 - 2.67 \cdot q_1 \cdot q_3 \quad (20)$$

Table 11. Analysis of variance for the response surface of oil expression efficiency, OEE (%) of rapeseeds

Model	Model coefficients	Standard Error	t-value	p-value
Intercept	44.60	2.62	17.03	0.00
Q_1	-7.76	2.07	-3.75	0.01
Q_1^2	9.01	2.85	3.16	0.02
Q_2	4.24	2.07	2.05	0.08
Q_2^2	-9.04	2.85	-3.17	0.02
Q_3	7.96	2.07	3.85	0.01
Q_3^2	7.04	2.85	2.47	0.04
$Q_1 \cdot Q_2$	-0.02	2.93	-0.01	0.99
$Q_1 \cdot Q_3$	2.00	2.93	0.68	0.52
$Q_2 \cdot Q_3$	3.06	2.93	1.04	0.33

Q_1: Speed (mm min^{-1}); Q_2: Temperature (^{o}C); Q_3: Force (kN)

Table 12. Analysis of variance for the response surface of oil expression efficiency, OEE (%) of rapeseeds

Effect	Sum of Squares	Degree of freedom	Mean Square	F-value	p-value
Intercept	2048.64	9	227.63	6.64	0.00
Q_1	482.21	1	482.21	14.06	0.01
Q_1^2	341.94	1	341.94	9.97	0.02
Q_2	143.99	1	143.99	4.20	0.08
Q_2^2	344.15	1	344.15	10.03	0.02
Q_3	507.37	1	507.37	14.79	0.01
Q_3^2	208.78	1	208.78	6.09	0.04
$Q_1 \cdot Q_2$	0.00	1	0.00	0.00	0.99
$Q_1 \cdot Q_3$	16.04	1	16.04	0.47	0.52
$Q_2 \cdot Q_3$	37.33	1	37.33	1.09	0.33
Error	240.08	7	-	-	-
Total	2288.72	16	34.29	-	-

Q_1: Speed (mm min^{-1}); Q_2: Temperature (^{o}C); Q_3: Force (kN)

$$OEE_R = 44.60 - 7.76 \cdot Q_1 + 9.01 \cdot Q_1^2 - 9.04 \cdot Q_2^2 + 7.96 \cdot Q_3 + 7.04 \cdot Q_3^2 \quad (21)$$

For the deformation energy of sunflower seeds (Table 13 and 14), the linear and quadratic terms of speed and force were significant (F-values > P-values) or (P-values ≤ 0.05) whereas the rest of the model terms were not significant. The significant terms of the model including intercept are given in equation (22).

For rapeseeds, (Table 15 and 16), the quadratic term of temperature, as well as the interaction terms of speed and temperature, speed and force and temperature and force, were not significant (P-values > 0.05) or smaller F-values. The significant terms of the model including intercept for rapeseed deformation energy are given in equation (23). The tests of the whole models were also assessed by the coefficients of determination (R^2) which were between 91 and 97% (Tables 17 and 18).

Table 13. Analysis of variance for the response surface of deformation energy, DE (J) of sunflower seeds

Model	Model coefficients	Standard Error	t-value	p-value
Intercept	427.66	4.50	94.97	0.00
q_1	-8.39	3.56	-2.36	0.05
q_1^2	0.09	4.91	0.02	0.99
q_2	3.52	3.56	0.99	0.36
q_2^2	0.25	4.91	0.05	0.96
q_3	49.91	3.56	14.02	0.00
q_3^2	0.72	4.91	0.15	0.89
$q_1 \cdot q_2$	-9.33	5.03	-1.85	0.11
$q_1 \cdot q_3$	-6.42	5.03	-1.28	0.24
$q_2 \cdot q_3$	6.35	5.03	1.26	0.25

q_1: Speed (mm min^{-1}); q_2: Temperature (^{o}C); q_3: Force (kN);

Table 14. Analysis of variance for the response surface of deformation energy, DE (J) of sunflower seeds

Effect	Sum of Squares	Degree of freedom	Mean Square	F-value	p-value
Intercept	21263.91	9	2362.66	23.30	0.00
q_1	562.97	1	562.97	5.55	0.05
q_1^2	0.03	1	0.03	0.00	0.99
q_2	98.98	1	98.98	0.98	0.36
q_2^2	0.27	1	0.27	0.00	0.96
q_3	19925.07	1	19925.07	196.53	0.00
q_3^2	2.16	1	2.16	0.02	0.89
$q_1 \cdot q_2$	348.01	1	348.01	3.43	0.11
$q_1 \cdot q_3$	164.87	1	164.87	1.63	0.24
$q_2 \cdot q_3$	161.42	1	161.42	1.59	0.25
Error	709.68	7	101.38	-	-
Total	21973.59	16	-	-	-

q_1: Speed (mm min^{-1}); q_2: Temperature (^{o}C); q_3: Force (kN);

$$DE_S = 427.66 - 8.39 \cdot q_1 + 49.91 \cdot q_3 \qquad (22)$$

Table 15. Analysis of variance for the response surface of deformation energy, DE (J) of rapeseeds

Model	Model coefficients	Standard Error	t-value	p-value
Intercept	512.56	8.42	60.89	0.00
Q_1	-28.20	6.65	-4.24	0.00
Q_1^2	-24.84	9.17	-2.71	0.03
Q_2	22.53	6.65	3.39	0.01
Q_2^2	13.20	9.17	1.44	0.19
Q_3	43.55	6.65	6.54	0.00
Q_3^2	-32.97	9.17	-3.59	0.01
$Q_1 \cdot Q_2$	-5.35	9.41	-0.57	0.59
$Q_1 \cdot Q_3$	-4.67	9.41	-0.50	0.64
$Q_2 \cdot Q_3$	-0.83	9.41	-0.09	0.93

Q_1: Speed (mm min^{-1}); Q_2: Temperature (^{o}C); Q_3: Force (kN)

Table 16. Analysis of variance for the response surface of deformation energy, DE (J) of rapeseeds

Effect	Sum of Squares	Degree of freedom	Mean Square	F-value	p-value
Intercept	33779.14	9	3753.24	10.59	0.00
Q_1	6363.05	1	6363.05	17.96	0.00
Q_1^2	2597.17	1	2597.17	7.33	0.03
Q_2	4061.71	1	4061.71	11.46	0.01
Q_2^2	733.53	1	733.53	2.07	0.19
Q_3	15169.34	1	15169.34	42.81	0.00
Q_3^2	4577.21	1	4577.21	12.92	0.01
$Q_1 \cdot Q_2$	114.49	1	114.49	0.32	0.59
$Q_1 \cdot Q_3$	87.05	1	87.05	0.25	0.64
$Q_2 \cdot Q_3$	2.79	1	2.79	0.01	0.93
Error	2480.17	7	354.31	-	-
Total	36259.31	16		-	-

Q_1: Speed (mm min^{-1}); Q_2: Temperature (^{o}C); Q_3: Force (kN)

$$DE_R = 512.56 - 28.20 \cdot Q_1 - 24.84 \cdot Q_1^2 + 22.53 \cdot Q_2 + 43.55 \cdot Q_3 - 32.97 \cdot Q_3^2 \quad (23)$$

Table 17. Test of whole model for the response surface of the responses of sunflower seeds

Responses	R^2	F-value	P-value
Oil yield, OY (%)	0.96	17.99	< 0.05
Oil expression efficiency, OEE (%)	0.96	3.65	< 0.05
Deformation Energy, DE (J)	0.97	101.38	< 0.05

R^2: Coefficient of determination

Table 18. Test of whole model for the response surface of the responses of rapeseeds

Responses	R^2	F-value	P-value
Oil yield, OY (%)	0.91	6.64	< 0.05
Oil expression efficiency, OEE (%)	0.91	6.63	< 0.05
Deformation Energy, DE (J)	0.93	10.59	< 0.05

R^2: Coefficient of determination

4.4 Optimal compression factors of sunflower seeds and rapeseeds

The optimal factors for predicting the responses (oil yield %, oil expression efficiency % and deformation energy J were determined for sunflower and rape bulk oilseeds. For sunflower seeds, the optimal factors were speed of 5 mm/min, the temperature of 75 ^{o}C and force of 100 kN (Table 19) whereas for rapeseeds the optimal factors were speeds of 5 and 7.5 mm/min, the temperature of 75 ^{o}C and forces of 90 and 100 kN (Table 20). Using the regression models (equations (18) to (23)), the responses were predicted, and based on the optimal factors; the responses were then validated (Tables 19 and 20). Percentage difference (%) and percentage error (%) were used to evaluate the predicted and validated values of the responses (Tables 21 to 23). The models for the responses were adequately suitable with a percentage error between 5.12% and 27.98% (Tables 21 and 22). Profiles for predicted values and desirability level for different factors for optimum responses of sunflower seeds and rapeseeds are presented in Figures 9 to 14. The desirability of the responses is between 0 and 1, and higher values show the adequacy of the models.

Table 19. Predicted and validated values for optimum factors of oil yield, oil expression efficiency and deformation energy of sunflower seeds

Factors			Predicted values			Validated values		
q_1 (mm min^{-1})	q_2 (^{o}C)	q_3 (kN)	OY (%)	OEE (%)	DE (J)	OY (%)	OEE (%)	DE (J)
5	75	100	23.73	71.68	477.57	22.51 ±1.34	56.01 ±3.32	503.33 ±2.72

q_1: Speed; q_2: Temperature; q_3: Force; OY: Oil Yield; OEE: Oil Expression Efficiency; DE: Deformation Energy

Table 20. Predicted and validated values for optimum factors of oil yield, oil expression efficiency and deformation energy of rapeseeds

Factors			Predicted			Validated		
Q_1 (mm min^{-1})	Q_2 (^{o}C)	Q_3 (kN)	OY (%)	OEE (%)	DE (J)	OY (%)	OEE (%)	DE (J)
5	75	100	21.47	67.33	549.03	25.44 ±1.99	79.81 ±6.23	596.74 ±4.13
7.5	75	90	15.13	47.43	556.51	23.39 ±0.83	71.54 ±0.11	547.67 ±12.85

Q_1: Speed; Q_2: Temperature; Q_3: Force; OY: Oil Yield; OEE: Oil Expression Efficiency; DE: Deformation Energy

Table 21. Evaluation of predicted and validated values of sunflower seeds for factors (q_1: = 5 mm min^{-1}; q_2= 75 (^{o}C) and q_3= 100 (kN)

Predicted/ validated values	Percentage difference (%)	Percentage error (%)
OY (%)	5.28	5.42
OEE (%)	24.54	27.98
DE (J)	5.25	5.12

OY: Oil Yield; OEE: Oil Expression Efficiency; DE: Deformation Energy

Table 22. Evaluation of predicted and validated values of rapeseeds for factors (Q_1: = 5 mm min^{-1}; Q_2= 75 (^{o}C) and Q_3= 100 (kN)

Predicted/ validated values	Percentage difference (%)	Percentage error (%)
OY (%)	16.93	15.61
OEE (%)	16.96	15.64
DE (J)	8.33	8.00

OY: Oil Yield; OEE: Oil Expression Efficiency; DE: Deformation Energy

Table 23. Evaluation of predicted and validated values of rapeseeds for factors (Q_1: = 7.5 mm min^{-1}; Q_2= 75 (^{o}C) and Q_3= 100 (kN)

Predicted validated values	Percentage difference (%)	Percentage error (%)
OY (%)	42.89	35.31
OEE (%)	40.53	33.70
DE (J)	1.60	1.61

OY: Oil Yield; OEE: Oil Expression Efficiency; DE: Deformation Energy

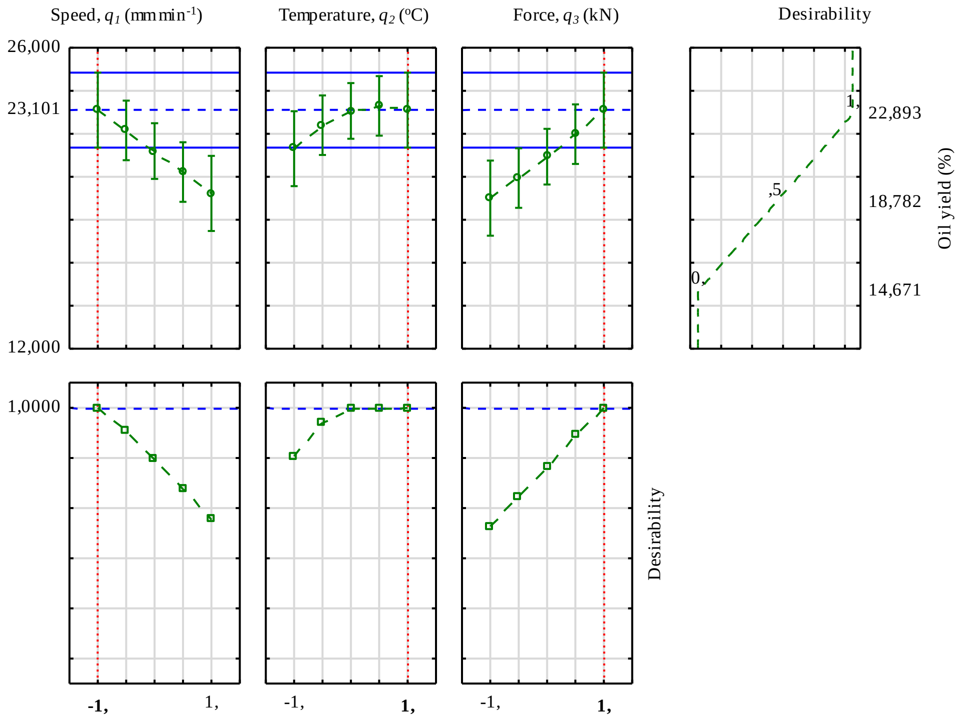

Figure 9. Profiles for predicted values and desirability level for different factors for optimum oil yield of sunflower seeds.

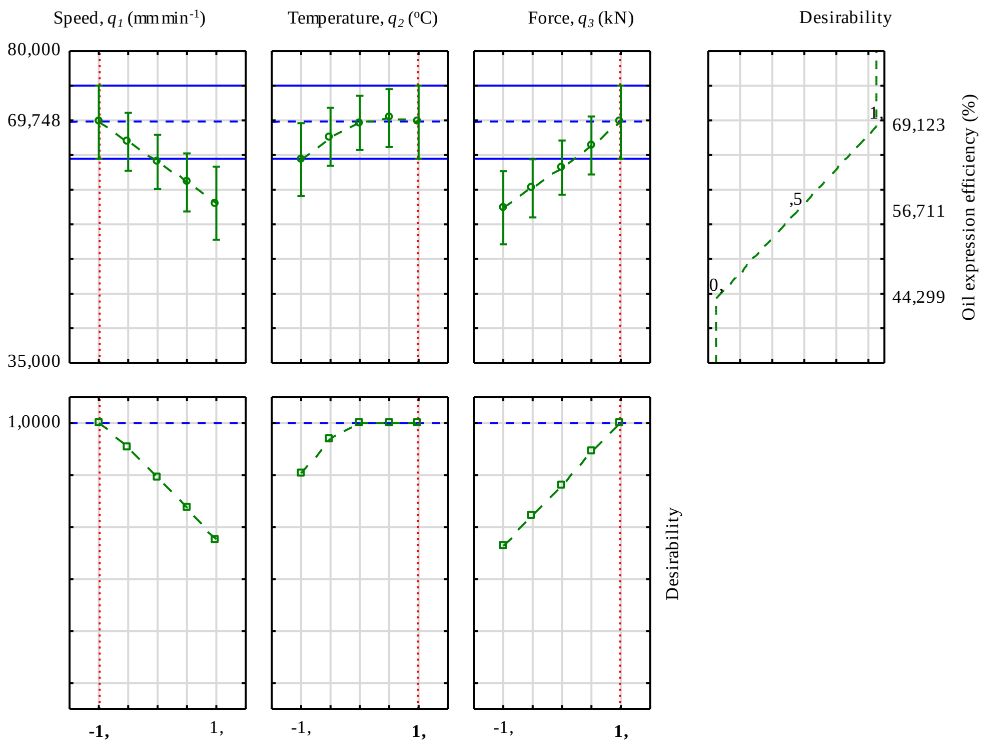

Figure 10. Profiles for predicted values and desirability level for different factors for optimum oil expression efficiency of sunflower seeds.

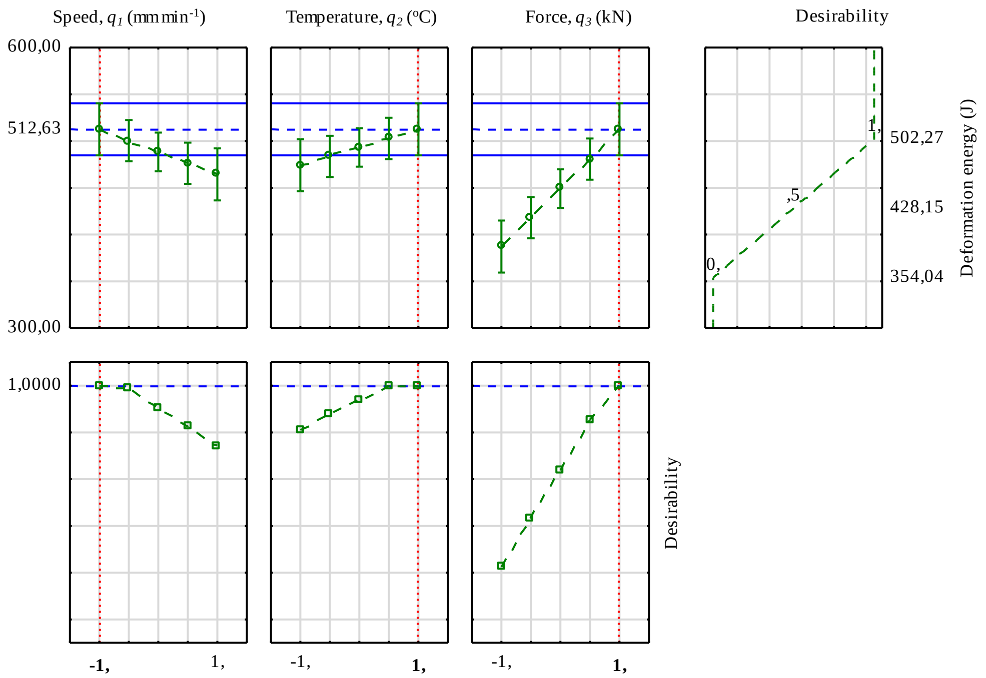

Figure 11. Profiles for predicted values and desirability level for different factors for optimum deformation energy of sunflower seeds.

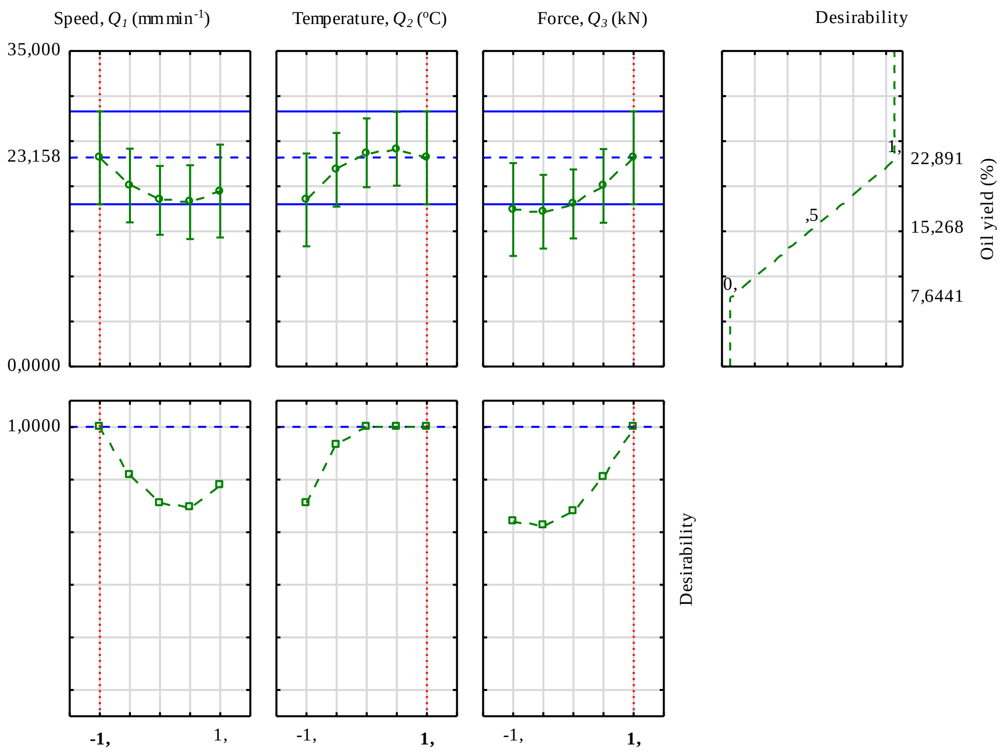

Figure 12. Profiles for predicted values and desirability level for different factors for optimum oil yield of rapeseeds.

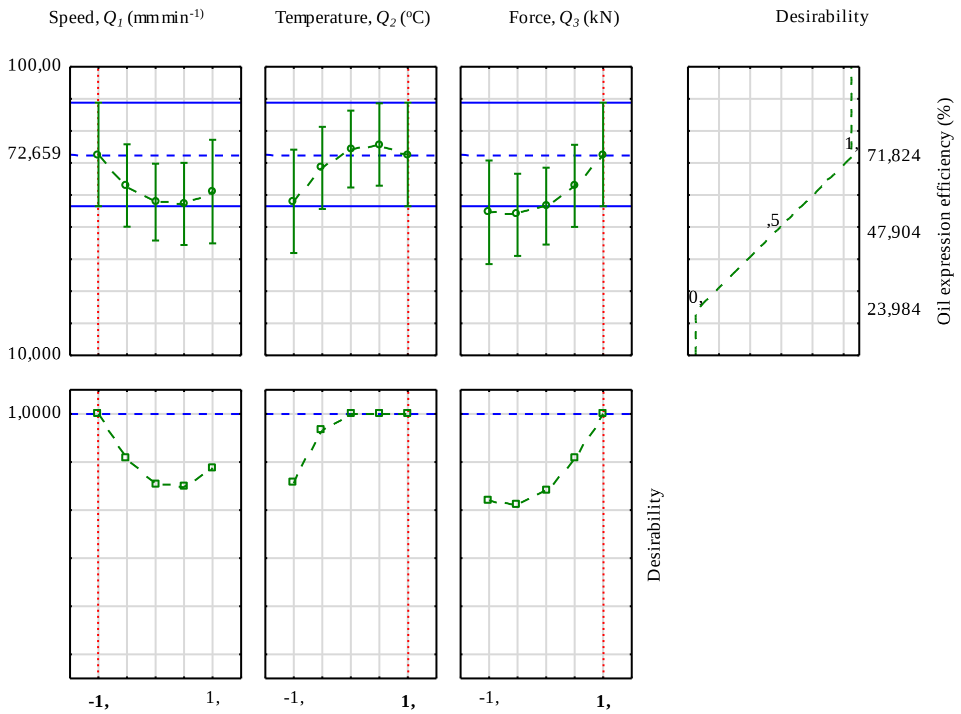

Figure 13. Profiles for predicted values and desirability level for different factors for optimum oil expression efficiency of rapeseeds.

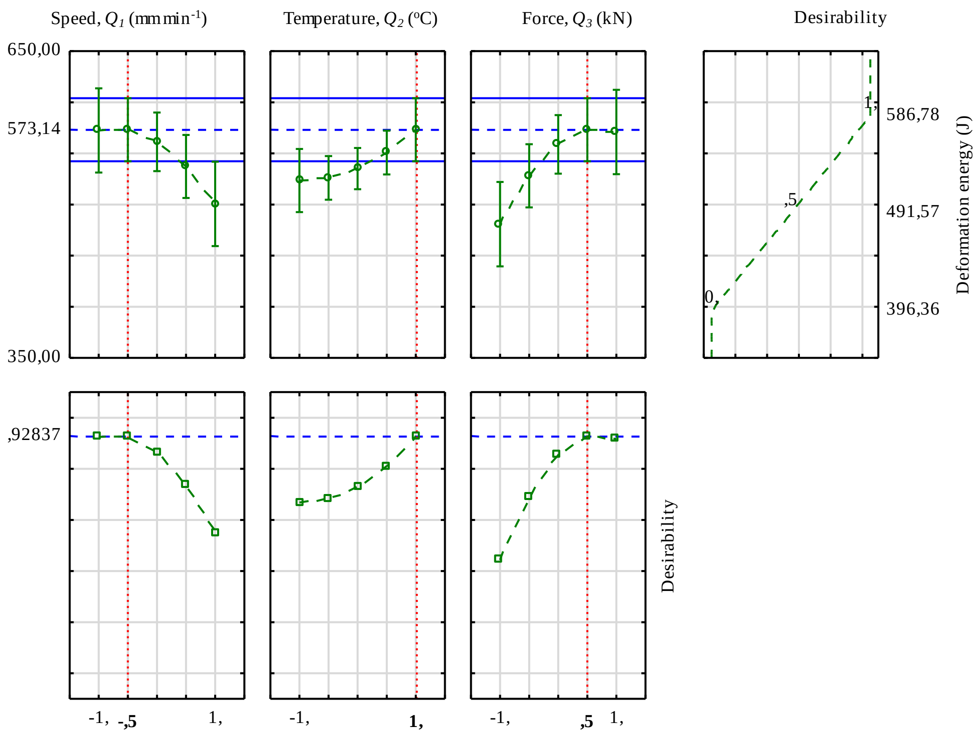

Figure 14. Profiles for predicted values and desirability level for different factors for optimum deformation energy of rapeseeds.

In the literature, Belhachat et al., (2018), used response surface methodology (RSM) to find out the best values of extraction time, ultrasonic power and plant material to water ratio for the extraction of essential oil from the ripe berries of *Pistacia lentiscus* using ultrasonic pretreatment. The optimum extraction conditions for extraction yield were found to be extraction time of 60 min, ultrasonic power of 60 W and the solid-liquid ratio of 1g:12 ml dependent variables. Shahedi et al., (2019) used RSM to study the effects of reaction temperature, methanol/oil ratio, reaction time, t-butanol concentration and CALB:RML ratio on the fatty acid methyl esters (FAME) yield to improve the biocatalytic biodiesel production from palm oil. The optimum combinations for the reaction were CALB:RML ratio (2.5:1), t-butanol to oil (39.9 wt%), temperature (35.6 °C) methanol: oil ratio (5.9), reaction time 33.5 h. Kandasamy et al., (2019) utilized RSM to enhance the bio-oil yield and quality of bio-oil by optimizing the hydrothermal liquefaction (HTL) of *Spirulina platensis* catalyzed by Fe_3O_4 nanostructures. The effects of three independent parameters such as temperature, holding time and catalyst dosage on oil yield were studied. A maximum bio-oil yield of 32.33% was observed for the high temperature at 320 °C, 0.75 g of catalyst dosage and 37 min of resident time. Rahimi and Mikani., (2019) also studied the optimization of lycopene ultrasound-assisted extraction from wastes of tomato with sunflower oil as a green solvent. The effect of ultrasonic intensity (30–70 W/m), solid/oil ratio (3.18–36.82% w/v), and the extraction time (1.59–18.41 min) were optimized by using RSM. The optimum ultrasound-assisted extraction (UAE) situation was accomplished with 70W/m2 ultrasonic intensity of and 10 min extraction time. The solid/oil ratio was not a substantial parameter on the yield of extraction. Agu et al., (2020) studied modeling and optimization of *Terminalia catappa* L. kernel oil extraction using response surface methodology and artificial neural network. The highest oil yield of 62.92% was obtained at optimum processing conditions of temperature (55°C), extraction time (150 min) and particle size (0.5 mm). The oil yield based on the optimum conditions was validated as 60.34%. Peng et al., (2020) applied supercritical carbon dioxide (SC-CO2) technique for extraction of oil from Roselle seed at temperatures from 40 °C to 80 °C and pressures from 20 MPa to 30 MPa. The best extraction conditions for Roselle seed oil corresponded to pressure and temperature values of 30 MPa and 40 °C. Mohammadpour., (2019) used RSM to optimize ultrasound-assisted extraction of *Moringa peregrina* oil and compared the result with Soxhlet extraction method. In the study, liquid-to-solid ratio (5–20 mL/g), extraction time (5–30 min), optimum ultrasound power (348 W) and extraction temperature (30 °C) were evaluated. The optimum process conditions which were determined by central composition design (CCD) to reach the maximum oil extraction yield (53.101%) were found to be 26.3min for extraction time and 17.8mL/g for liquid-to-solid ratio.

5 CONCLUSIONS

A Response Surface Methodology (RSM) using Box Behnken Design (BBD) was applied to study the effects of the independent variables: speeds (5, 10, 15) mm min^{-1}, heating temperatures (45, 60, 75) ^oC and forces (60, 80, 100) kN on the dependent variables: oil yield (%), oil expression efficiency (%) and deformation energy (J) of sunflower seeds and rapeseeds under compression loading. The results showed that the optimum conditions for compression of sunflower seeds and rapeseeds were found to be the speed of 5 mm min^{-1}, the heating temperature of 75 ^oC and a force of 100 kN. Based on the optimized factors, rapeseeds obtained the highest percentage of oil and energy than sunflower seeds. The MSc. Thesis objectives were entirely fulfilled where the force-deformation curves of sunflower and rape bulk oilseeds concerning speeds, heat treatment temperatures and forces were described. The response surface regression models for describing the deformation energy, percentage oil yield and oil expression efficiency of sunflower and rape bulk oilseeds with speeds, heat-treatment temperatures and forces were determined. The optimal speed, force and heat-treatment temperature of deformation energy, percentage oil yield and oil expression efficiency of sunflower and rape bulk oilseeds were determined.

6 RECOMMENDATIONS

(i) The percentage error approximately between 16 and 28% among sunflower and rapeseeds showed the reliability of the data of percentage margin between 84 and 72%. However, repeatability of study in future would be necessary to reduce the error margin and/or to confirm the findings.

(ii) Varieties of sunflower seeds and rapeseeds should be considered for evaluation of the oil expression efficiency about the processing factors.

(iii) Other compression factors such as moisture content, heating time and vessel diameters should be considered using a similar approach to evaluate the oil yield (%) oil expression efficiency (%) and deformation energy (J) of sunflower seeds and rapeseeds under linear compression.

(iv) Chemical properties such as peroxide value, free fatty acid and compositions among others should be determined to assess the oil quality with heat-treatment temperatures of oilseeds under compression loading tests.

(v) Central Composite Design (CCD) should also be considered for optimization of responses depending on the processing factors of oilseeds under compression loading tests.

(vi) Similar design of experiment should be applied to the non-linear compression process involving a mechanical screw press to optimize the oil processing factors and their responses.

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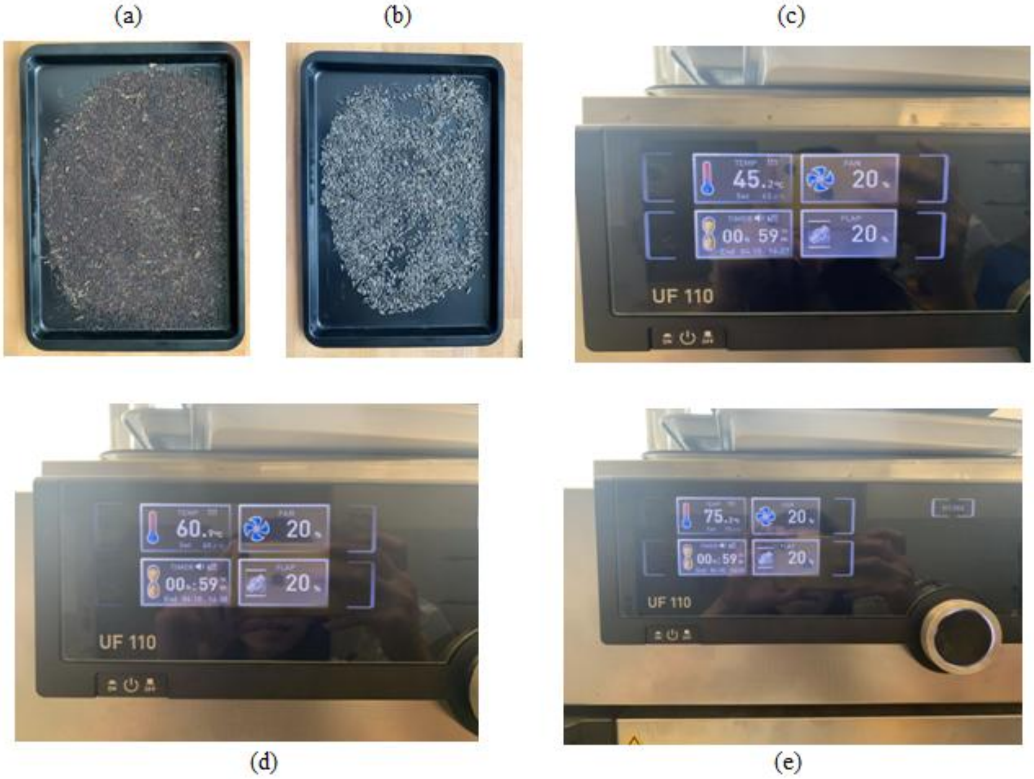
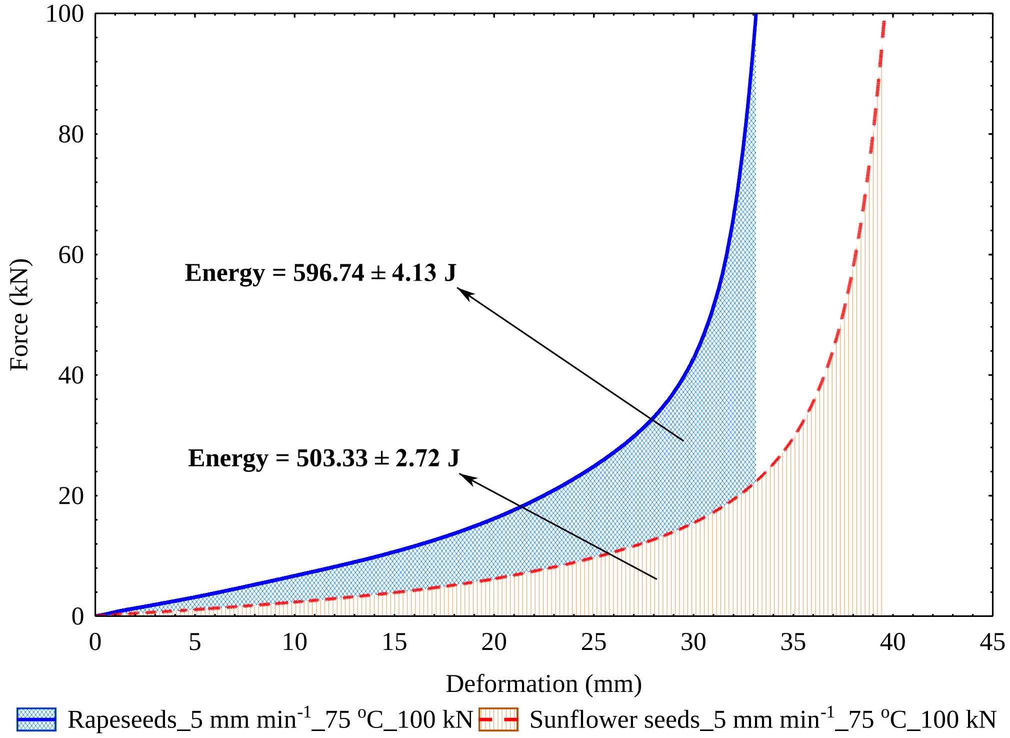
8 APPENDIXES



Appendix 3. (a): Rapeseeds (b): Sunflower seeds (c), (d) and (e): Pre-treatment of seeds at 45, 60 and 75 ^{o}C for heating time of 60 min using Memmert oven, type UF110, Germany.



Appendix 4. Relationship between force and deformation curve of sunflower seeds and rapeseeds at optimum pressing factors showing the energy requirements.