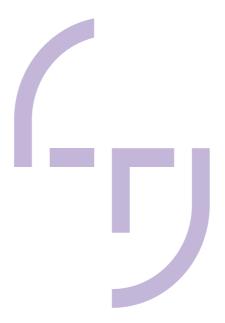
Tampere University of Applied Sciences



# Industrial Production of Rapeseed Oil and Its Application

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## ABSTRACT

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Rapeseed oil production has been increasing globally over the last couple of decades. The rapeseed oil and its by-product, the rapeseed meal, are actively used not only for human and livestock consumption, but also in major industrial applications. In an industrial scale, the rapeseed oil production involves several steps of preparation of seeds for preliminary pressing, followed by several steps of extraction of oil. There has also been an interest in the small scale oil production for food consumption and for fuel purposes. In this regard, the educational institution IUT Nancy-Brabois created a research project for small scale oil production using the screw press Farmet UNO FM. The thesis work was based on this research project, which was implemented during the practical work in spring 2019 in IUT Nancy-Brabois.

The aim of the work was to define the optimal combination of press nozzle of diameters 6, 8, 10 mm with frequency of 30, 50, 70 Hz of the screw press to obtain the high production of rapeseed oil and least production of oilcake. The obtained oil was subjected to centrifuge filtration. The oil from oilcakes with diameters 6 and 10 mm was extracted with propan-2-ol ( $C_3H_8O$ ) solvent. The soap was produced from mixture of obtained rapeseed oil with store-bought olive oil, followed by pH analysis. The biodiesel was produced from obtained rapeseed oil, followed by analytical chromatography analysis and viscosity testing. Both viscosities of biodiesel and rapeseed oil were compared.

The results showed the highest oil production was achieved with combination of 6 mm press nozzle and 30 Hz frequency. Pressing 38,65% of oil and 60,83% of oilcake per 500 g of seeds. The centrifuge filtration ran during 5 min and 10 min showed almost the same filtrated residue of 12,35 g and 12,57 g respectively. Therefore the set-up of 5 min was determined to be the most time efficient. The solvent extraction showed the 6 mm diameter oilcake contained the least amount of oil. The soap was produced with skin-safe pH of 8,5. The biodiesel was successfully produced with the viscosity close to standards. Analytical chromatography analysis indicated the formation of biodiesel. The obtained results were accepted and approved by IUT Nancy-Brabois and will be used as a base in future laboratory works and research projects.

Key words: rapeseed oil, Brassica napus, filtration centrifuge, solvent extraction, biodiesel production, vegan soap.

# CONTENTS

1	INTRODUCTION	6
2	LITERATURE REVIEW	7
	2.1 Origin and brief history of Oilseed rape	7
	2.2 Rapeseed cultivation and economic importance	7
	2.3 Composition of rapeseed oil	8
	2.3.1 Fatty acids	9
	2.4 Physico-chemical characteristics	11
	2.5 Production of rapeseed oil	11
	2.5.1 Preparation	12
	2.5.2 Solvent extraction	13
	2.6 Major industrial applications	14
	2.6.1 Biodiesel	14
	2.6.2 Biosolvents	17
	2.6.3 Biolubricants	17
	2.6.4 Soap	17
	2.7 Related research	18
3	METHODOLOGY AND APPROACH	20
	3.1 Production of rapeseed oil and oilcake	21
	3.2 Filtration of rapeseed oil	22
	3.3 Solvent extraction	23
	3.4 Production of soap	25
	3.5 Production of biodiesel	26
	3.5.1 Paper chromatography	28
	3.5.2 Viscosity measurements	30
4	RESULTS	31
	4.1 Production of rapeseed oil and oilcake	31
	4.2 Filtration of rapeseed oil	32
	4.3 Solvent extraction	33
	4.4 Production of soap	34
	4.5 Production of biodiesel	36
	4.5.1 Analytical chromatography	37
	4.5.2 Viscosity	38
5	DISCUSSION	
6	CONCLUSION	42
RE	EFERENCES	43
AF	PPENDICES	47

Appendix 1. Oil screw press Farmet UNO FM in operating manual. ...47

# ABBREVIATIONS AND TERMS

CH₃OH	Methyl alcohol, methanol
$C_2H_5OH$	Ethyl alcohol, ethanol
C <sub>3</sub> H <sub>8</sub> O	Isopropyl alcohol, propan-2-ol
EU	European Union
FDA	Food and Drug Administration
GRAS	Generally recognized as safe
IUT	Institut universitaire de technologie
КОН	Potassium hydroxide
K <sub>3</sub> PO <sub>4</sub>	Tripotassium phosphate
MMt	Million metric tonnes
NaOH	Sodium hydroxide, caustic soda
Na <sub>(s)</sub>	Solid sodium
Na <sub>2</sub> SO <sub>4</sub>	Sodium sulphate
Na <sub>3</sub> PO <sub>4</sub>	Trisodium phosphate
рН	Potential of hydrogen
rpm	Revolutions per minute

#### **1 INTRODUCTION**

This thesis work was based on experimental project, which was a part of preliminary study of an oilseed rape value chain for biofuel production. The experiment was conducted in spring 2019 in Institut universitaire de technologie (IUT) de Nancy-Brabois, France.

Rapeseed oil production has been increasing globally over the past decades. European Union (EU) is the world's biggest producer of oilseed rape, followed by Canada, China and India. Among all European countries, France is the leading producer. (Carré & Pouzet 2014) The prevalence of this cultivar is due to the high demand for two extracted products, rapeseed oil and remaining seed material, rapeseed meal. Rapeseed oil is used for human consumption and various industrial applications. Rapeseed meal is actively used for animal feed industry (USDA Foreign Agricultural Service 2018). Nowadays, the search for sustainable alternative oil to replace the petroleum based oil has been increasing. According to report by USDA Foreign Agricultural Service (2018), in Europe rapeseed oil is the leading oil for biofuels use, followed by palm oil, soybean and sunflower. Additionally, there has always been an interest in small scale oil pressing for on farm production of edible oil and for fuel purposes (Prior, Vadke & Sosulski 1991).

In this regard, IUT created an experimental project to define the optimal parameters and methods to be used in the oil extraction process using small capacity oil screw press Farmet UNO FM. The aims of this thesis work are to find the optimal combination of press nozzle and rotational speed parameters of oil press, to filtrate raw oil using the centrifuge filtration method, to extract the oil from oilcake using the solvent, to produce the soap from mixture of rapeseed oil with store bought olive oil, to produce biodiesel from obtained oil and to analyse it with analytical chromatography. Finally, the raw rapeseed oil and biodiesel are tested for viscosity.

#### 2 LITERATURE REVIEW

#### 2.1 Origin and brief history of Oilseed rape

The name "rape" from "rapeseed" is derived from the Latin word *rapum*, which means "turnip" (Gupta & Pratap 2007, 6). Rapeseed cultivars, also known as Brassica napus, are closely related to turnip, cabbage, mustard and others, which all belong to the diverse group of genus Brassica of Brassicaceae family also known as the Mustard family. (Fahey 2003, 606–615) In this thesis work the most common names of Brassica napus were used. These names are rape, rapeseed, oil rape and oilseed rape (Shahidi 1990, 4).

In Europe, early rapeseed cultivars became particularly popular between 18th and 19th centuries. Rapeseed oil was used for lightening homes and as a lubricant, but not as an edible oil due to high quantities of glucosinolates and erucic acid. The presence of these components was a health concern for both humans and livestock (Przybylski, Mag, Eskin & McDonald 2005, 62). In 1977 in Canada was introduced first low erucic acid and low glucosinolates cultivar. In 1978 the oil was registered under the "canola" name. Later on it was recognized by the FDA and granted the GRAS status (Shahidi 1990, 10). The term "canola" is mainly used in countries of South and North America and Australia. The term "rapeseed" is mainly used in European countries and the rest of the world (Przybylski et al. 2005, 62). Rapeseed cultivar, low in erucic acid and glucosinolates, differ in chemical and physical properties from cultivars high in erucic acid and glucosinolates. In this thesis, in the experiment were used seeds of low erucic acid and low glucosinolates cultivar of oilseed rape.

#### 2.2 Rapeseed cultivation and economic importance

In most of Europe, rapeseed is cultivated mainly during the winter season, but in the northern Europe the spring forms are also suitable (Snowdon, Lühs & Friedt 2007, 55–56). Comparing both forms, the spring cultivar is observed to have a higher seed oil percentage compared to the winter cultivated rape (Rad & Zandi 2012). Regarding to weather sensitivity the plant shows the decline in acreage

during the drought, extremely high or low temperature seasons (USDA Foreign Agricultural Service 2019). Therefore, in countries with a continental climate, rapeseed farming is quite risky.

In EU, France and Germany are the largest rapeseed producers, followed by Poland, United Kingdom and Romania. In 2018 and 2019, the agriculture has struggled with poor weather conditions, excessive spring rains and summer drought. This caused a reduction in crop production throughout the whole EU. France had been impacted the most as well due to the EU's ban on effective neonicotinoid pesticides, which increased the population of Cabbage Stem Flee, the most problematic pest for rapeseed. As a result, the plant gets so damaged that it ends up dying. Currently, the forecast of rapeseed harvest for 2019 and 2020 is at 17.8 MMt. (USDA Foreign Agricultural Service 2019)

## 2.3 Composition of rapeseed oil

The typical composition of rapeseed oil is presented in Table 1.

Component	Rapeseed		
Triacyglycerols, %	91.8 - 99.0		
Phospholipids, %			
Crude Oil	up to 3.5		
Water - degummed	up to 0.8		
Acid - degummed	-		
Free Fatty Acids, %	0.5 - 1.8		
Unsaponifiables,%	0.5 - 1.2		

TABLE 1. Constituents of rapeseed oil (Przybylski et al. 2005, 63).

The seed of rape contains approximately 44% of oil (Przybylski et al. 2005, 80). Rapeseed oil typically consists of 99.0% triacylglycerols, also called triglycerides (EFSA European Food Safety Authority 2005). Figure 1 demonstrates the structure of triglyceride molecule, where  $R_1$ ,  $R_2$  and  $R_3$  are long chains of carbons and hydrogen atoms of fatty acids.

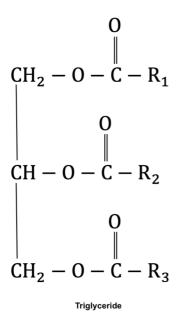


FIGURE 1. Structure of triglyceride molecule.

Triacylglycerol is an ester composed of molecule of glycerol (C<sub>3</sub>H<sub>8</sub>O<sub>3</sub>) bonded to three molecules of fatty acids (Fahy, Cotter, Sud & Subramaniam 2011). The remaining 1% is composed of phospholipids, free fatty acids, unsaponifiables and other minor components. Phospholipids, also known as polar lipids, are important components of cell membranes. Molecule of phospholipids is composed of molecule of glycerol, two molecules of fatty acids and one phosphate group (Przybylski et al. 2005, 67–68). Free fatty acids are found in oils in a form of detached fatty acids from oil molecules. The high presence of free fatty acids of more than 5% in unrefined oils usually indicates the deterioration of oil quality due to damage of plant during processing or poor oil processing. (Wiesman 2009, 18) Unsaponifiable substances represent a fraction of oil that cannot form soaps in a reaction with sodium hydroxide (NaOH). The majority of polar lipids, free fatty acids and unsaponifiables are removed during the process of oil refining. (Dijkstra & Segers 1995, 143, 144, 152)

## 2.3.1 Fatty acids

Fatty acids are carboxylic acids with long aliphatic chains, containing even number of carbon atoms. The length of the chain, the number and the location of carbon-carbon double bond in the chain make fatty acids differentiate from each other. Saturated fatty acids have no double bond, unsaturated have one or more double bonds, monosaturated have one double bond and polyunsaturated have two or more double bonds. (Ouellette & Rawn 2018, 1020) The melting point of fatty acids depends on number of carbon atoms and carbon-carbon double bonds in a molecule. The melting point increases with increasing number of carbons and decreases with increasing unsaturation (Przybylski et al. 2005, 75). Therefore, the liquid state of rapeseed oil indicates a dominance of unsaturated fatty acids in it (Przybylski et al. 2005, 64). There are over a hundred fatty acids discovered in living organisms. However, there are number of essential fatty acids that cannot be metabolized naturally by human bodies and therefore have to be ingested with food. Rapeseed oil is a great source of essential, omega-6 and omega-3, fatty acids, which are linoleic and linolenic respectively. (Kapalka 2010, 99) Table 2 demonstrates the typical composition of fatty acids of rapeseed oil.

Fatty acid	Symbol	Composition,% wt
Myristic acid	C14:0	0.05
Palmitic acid	C16:0	4.84
Palmitoleic acid	C16:1	0.06
Heptadecanoic acid	C17:0	0.14
Stearic acid	C18:0	0.14
Oleic acid	C18:1	62.73
Linoleic acid	C18:2	22.40
Linolenic acid	C18:3	7.50
Arachidic acid	C20:0	0.50
Eicosenoic acid	C20:1	1.25
Behenic acid	C20:0	0.30

TABLE 2. Composition in fatty acids of rapeseed oil (Cristea et al. 2018, 3).

As it was mentioned before, the early rape cultivars had high levels of erucic acid, which were obstructing the oil to be consumed by humans. This is due to discovery of adverse effects on heart caused by erucic acid (Sissener et al. 2018). Erucic acid is a monosaturated fatty acid with twenty two atoms of carbon and one double bond. In virtue of discovery of oxidation process, the erucic acid chain had been shortened to oleic acid form. (Przybylski et al. 2005, 64) Oleic acid consists of eighteen atoms of carbon and one double bond. In comparison with erucic acid, oleic acid was discovered to have a positive effects on heart's health (Teres et al. 2008). Currently, different types of rapeseed oil with slightly varying fatty acids composition can be found on the market. Such feature was developed through genetic modifications depending on usage purpose of oil.

## 2.4 Physico-chemical characteristics

Chemical characteristic of oil predetermines the physical behaviour of it, for example, whether the oil will remain in a liquid or solid state at room temperatures. Table 3 shows the physical chemical properties of rapeseed oil according to Przybylski et al. (2005, 74).

Parameter	Rapeseed oil
Relative density at 20 °C, g/cm <sup>3</sup>	0.914 - 0.917
Kinematic viscosity at 20 °C, mm <sup>2</sup> /sec	78.2
Flash point, open crucible,°C	275 - 290
Specific heat at 20 °C, J/g	1.910 - 1.916
Saponification number	188 - 192
Iodine value	110 - 126

TABLE 3. Physicochemical properties of rapeseed oil (Przybylski et al. 2005, 74).

Relative density and viscosity parameters were the most substantial for the experiments presented in this thesis work. Relative density between 0.914-0.917 g/cm<sup>3</sup> make the rapeseed oil practically insoluble in water. This happens because water is polar and fatty molecules are non-polar. Thereby an important property of oils is solubility in non-polar organic solvents, such as gasoline. Kinematic viscosity indicates the resistance of oil to flow due to gravity. The viscosity increases with the increasing weight of the molecule. Rapeseed oil is very viscous comparing to kinematic viscosity of water, which is approximately 1 mm<sup>2</sup>/s at 20°C. (Przybylski et al. 2005, 74) Vegetable oil subjected to heat treatment changes its physicochemical properties. Rapeseed oil produced by hot pressing method experiences reduction in antioxidant activity (Siger, Józefiak & Górnaś 2017). On the other hand, the heat treatment does not affect main vitamins A, D and E, which are resistant to heat (Oliveira, Desai, Favaro & Ferreira 1994). According to Idu et al. (2018, 056–066), hot pressed vegetable oil showed better nutritional composition and cold pressed oil showed better antioxidant activity.

## 2.5 Production of rapeseed oil

A high-quality raw rapeseed oil production includes several extraction and processing steps. The whole process is divided into two major stages, preparation of seeds and extraction of oil from oilcake. All processes involved in the production of rapeseed oil are demonstrated in Figure 2.

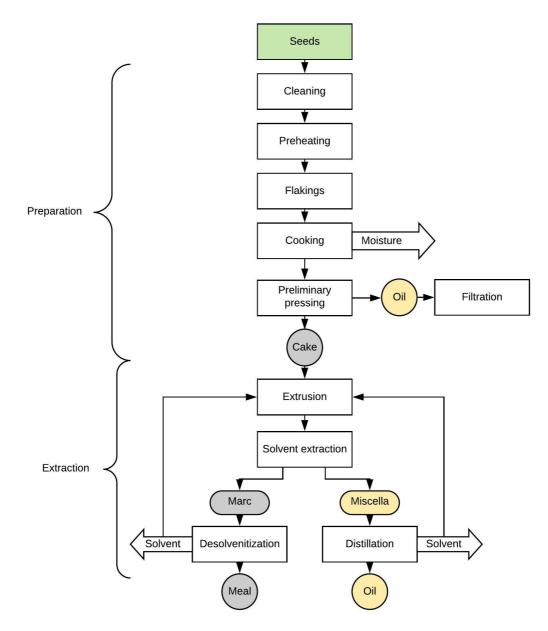


FIGURE 2. Processed of rapeseed oil production.

#### 2.5.1 Preparation

The preparation stage includes a number of steps, which are described as follows. The first step is seeds cleaning. Seeds are delivered from farms to production factory. During the preparatory processes, seeds are cleaned from impurities and foreign objects. Such objects range from small particles like dust and hull to big particles like stones. The following step is preheating. Seeds are loaded into

the heater and heated up to 30-40°C. Moisture content is reduced to approximately 9%. As a result, seeds lose the hardness and become more elastic. This state contributes to the effective seeds flaking, which is the next step. Seeds are fed to the rolling mills and rolled into flakes with approximate thickness of 0.3 mm. The purpose of flaking is to break the cell walls of seed for better oil extraction. Next is the cooking step. Flaked seeds are fed into the heating bunkers with inside temperature of 70-100°C. The goal is to achieve the moisture content of 5-7% of flaked seeds. The application of high heat on flaked seeds also contributes to efficient oil extraction. However, the temperature levels must be carefully controlled to prevent the quality degradation of oil. The next step is the preliminary oil pressing. Flaked seeds are conveyed from the cooker to the oil screw press. During the process, up to 70% of oil is pressed out of the seeds. As a result of the oil extraction two valuable products were obtained, rapeseed raw oil and oilcake. The unfiltered raw oil released during the preliminary pressing is left aside for further processing. The oilcake still has an oil content of 25-30% to be pressed out. In this moment the preparation stage is finalized. (Przybylski et al. 2005, 77-80)

## 2.5.2 Solvent extraction

Just like the preparation stage, the extraction stage includes several steps described as follows.

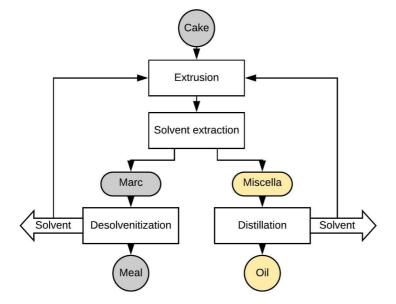


FIGURE 3. Processing of oilcake.

The first step is the mechanical extraction of the remaining oil from the oilcake obtained after the preliminary pressing. The oilcake is fed into extruders, where it is crushed and heated to 100°C. After that, crushed cake is conveyed onto the continuous belt extractor. In this process the oilcake is mixed with hexane, the most used oil extracting solvent. During the process, the solvent penetrates the oilcake and diffuses within the molecular cells and dissolves the oil. As a result two products are formed, the oil-solvent mixture, called miscella, and the remaining matter, called marc. (Bart, Palmeri & Cavallaro 2010, 94–96) Miscella usually constitutes of 30% of oil and 70% of solvent, whereas marc contains 10 g of oil per kg (Mosenthin et al. 2016, 4). To finalize the extraction stage, the remaining of the solvent are stripped from miscella and marc by distillation and desolventizing processes. Marc is conveyed to desolventization tank heated to around 105°C. Marc is constantly mixed under the low pressure. In this way, hexane starts evaporating leaving the dry residues of the marc, called meal (Mosenthin et al. 2016, 4, 5). During the distillation process, miscella is fed into the evaporator where it is heated to around 100°C. As a result, hexane starts evaporating and condensing into solvent separator. (Patidar, Sethiya & Ghosh 2015, 1–7). The recovered solvent can be used all over again back in the solvent extraction step. As a final result, solvent free crude rapeseed oil is obtained and stored inside oil tanks. The crude rapeseed oil has to further undergo processes to become an edible product.

#### 2.6 Major industrial applications

The chemical composition, natural origin, biodegradability, non-toxic property and other specific properties of vegetable oils have allowed them to be used not only as edible products, but also as a sustainable alternative to petroleum based products (Acaroglu, Oguz & Ögüt 2001). Nowadays, the main focus has been on technologies and methods that embody plant oils as biodiesels, biolubricants and biosolvents (Salimon, Salih & Yousif 2012).

## 2.6.1 Biodiesel

Fatty acid methyl ester, also known as biodiesel fuel, is a product of transesterification reaction between vegetable oil and alcohol in the presence of a catalyst (Jeong et al. 2004, 748–749). Pure vegetable oil can also be used as a fuel, but only in engines with specific parameters (Sidibé, Blin, Vaitilingom & Azoumah 2010). Whereas the biodiesel fuel can be used in almost all existing engines in a form of a mixture with petroleum based diesel fuel or in a pure form (Van Gerpen et al. 2004, 1; Xue, Grift & Hansen 2011). The simplified transesterification reaction for obtaining rapeseed methyl ester is shown in Figure 4, where  $R_1$ ,  $R_2$  and  $R_3$  are long chains of carbons and hydrogen atoms of fatty acids. The ratio of oil to methanol (CH<sub>3</sub>OH) is approximately 9 to 1. (Van Gerpen et al. 2004, 1)

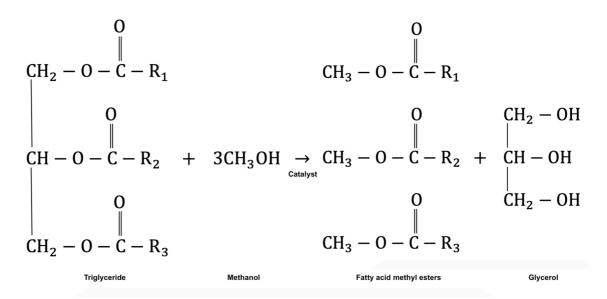


FIGURE 4. Transesterification reaction with methanol as catalyst.

As it is shown in Figure 4, during the reaction of transesterification, triglyceride (vegetable oil) reacted with methanol in presence of alkaline catalyst, for example, commonly used NaOH. The catalyst is added to increase the reaction rate. Methanol is commonly used due to having a short chain and low cost (Jeong et al. 2004, 748).

The reaction takes place at 60°C. As a result, one molecule of triglyceride reacts with three molecules of methanol and produces three molecules of fatty acid methyl ester (biodiesel) and one molecule of glycerol. Usually the final product of the reaction is recommended to contain approximately 0.24% total glycerol. The yield of biodiesel can be up to 90% (Van Gerpen et al. 2004, 5). Kinematic viscosity of biodiesel is approximately 6.78 mm<sup>2</sup>/s at 20°C and kinematic density is approximately 0.8798 g/cm<sup>2</sup> (Esteban et al. 2012, 167–168).

In industrial scale, the biodiesel fuel production involves multiple steps, as shown in Figure 5.

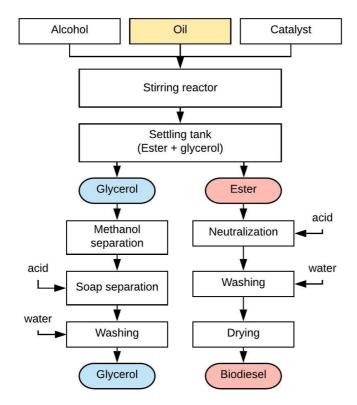


FIGURE 5. Processes of biodiesel production.

Firstly, oil, alcohol and catalyst are mixed in a stirring reactor at temperature between 60-80°C for 1-2 hours. Catalyst, NaOH or potassium hydroxide (KOH), is usually added in excess to make sure that triglycerides dissolve. Obtained fatty esters with by-product glycerol are collected into the settling tank for further separation by decantation. The esters are neutralized with the addition of acid, trisodium phosphate (Na<sub>3</sub>PO<sub>4</sub>) or tripotassium phosphate (K<sub>3</sub>PO<sub>4</sub>) and subjected to washing with water and dried. At the same time, the obtained glycerol is in a form of a mixture of glycerol, methanol and catalyst. In the methanol separation tank, the glycerol is separated from the methanol. All recovered methanol can be used again for biodiesel production. In the next step, glycerol is separated from free fatty acids by addition of acid. Finally, two end products are obtained, biodiesel and pure glycerol. Glycerol is not a waste end product, but usually it is sold further to refiners. (Dimian & Bildea 2008, 410–416)

#### 2.6.2 Biosolvents

Rapeseed oil esters are considered to be a good base for production of biosolvents. Main advantages are due to low viscosity, high penetration, high boiling and flash points, non-toxic property and low volatility of organic compounds (Przybylski et al. 2005, 112). Most importantly due to natural origin and recyclability, biosolvents produce less environmental impact, which makes them a "green" substitution to petroleum-based solvents (Yara-Varón et al. 2017). The application area of biosolvents is the same as for petroleum-based solvents, for example, in ink production (Pan et al. 2018), cleaning products, production of pesticides, paints, coatings and other similar products (Przybylski et al. 2005, 112).

#### 2.6.3 Biolubricants

Lubricant is a liquid substance used to reduce the friction between mating surfaces. Various vegetable oils are used as a base to produce biolubricants. Biolubricants just like biosolvents are an environmentally friendly substitution to petroleum-based lubricants. Due to natural origin, vegetable oils are biodegradable, have higher viscosity and do not produce toxic effect on living organisms once release to the environment. However, not all vegetable oils can be used as a base for lubricants due to poor oxidative stability caused by high levels of unsaturated acids (Salimon, Salih & Yousif 2010). The rapeseed oil can be used as it is low in polyunsaturated acids, which makes it resistant to oxidation and corrosion (Przybylski et al. 2005, 112). Biolubricants are used on surfaces exposed to friction, such as lubrication of human prostheses, mechanical engines and others (Salimon et al. 2010).

## 2.6.4 Soap

Natural soaps are derived as a result of chemical reaction between triglycerides with NaOH, known also as caustic soda. This reaction is called saponification, also known as the cold soap production. (Félix, Araújo, Pires & Sousa 2017) As it was mentioned before, triglycerides are composed of one molecule of glycerol bonded to three molecules of fatty acids. In the chemical reaction, triglyceride

reacts with NaOH producing glycerol and fatty acid salts, which are a soap. Figure 6 shows the saponification reaction, where  $R_1$ ,  $R_2$  and  $R_3$  are long chains of carbons and hydrogen atoms of fatty acids.

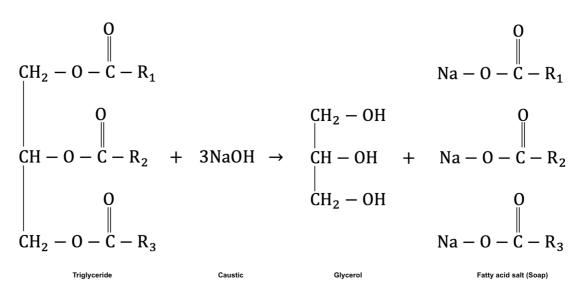


FIGURE 6. Saponification reaction.

Rapeseed oil is rich in unsaturated fatty acids, such as oleic and linoleic acids. Soap produced from such oil possesses a high water solubility properties at 20°C. (Przybylski et al. 2005, 86–87). In terms of potential of hydrogen (pH), soaps are recommended to have pH within the range of adult human skin pH, which is 5.4-5.9. However, a pH range between 4-11 is also acceptable (Tarun et al. 2014).

#### 2.7 Related research

The main focus of this thesis work is the production of rapeseed oil using the small capacity single-stage cold stamping screw press. Obtained oil is further used to produce biodiesel and soap. In this regard, several research works had been studied. In this section, methods and results of three scientific research works are presented and discussed as follows.

The research published by Wcisło (2006), was implemented as a single-stage cold stamping rapeseed oil production on fourteen cultivars of oilseed rape. The difference in cultivars was based on weather and soil conditions. The oil press machine used was Farmet UNO FM. In the results, the average mass of oil per 1 kg of seeds after pressing and filtering was 335 g/kg, 33.5%. The biggest mass

of oil obtained from single cultivar was 389 g/kg. The lowest mass was 275 g/kg. The average density measured was 0.89 kg/dm<sup>3</sup> at 15°C.

The research published by Rashid and Anwar (2008) implemented small scale rapeseed oil production followed by production of biodiesel. Results demonstrated the optimal parameters for biodiesel production. Catalyst used was KOH, ratio of CH<sub>3</sub>OH to oil was 6 to 1, temperature of the reaction was 65°C and reaction time was 2 hours. The yield of 95% of biodiesel was obtained.

The aim of the research published by Pasyniuk and Golimowski (2013) was a small-scale cold pressing production of rapeseed as a biodiesel. The obtained oil yield as a ratio of oil mass to seeds mass was approximately 29%. The oil content in the oilcake with diameter 6 mm was 21%. The temperature inside the press was above 70°C. Measured kinematic viscosity was 34.44 mm<sup>2</sup>/s at 40°C. The results showed that chosen parameters produced oil that can be used for biodiesel production.

The knowledge obtained from these research works is further used in this thesis work to compare and discuss the final results.

## 3 METHODOLOGY AND APPROACH

The whole experiment is divided into five stages: production of rapeseed oil and oilcake (stage 1), filtration of rapeseed oil (stage 2), solvent extraction (stage 3), production of soap (stage 4), production of biodiesel (stage 5). Figure 7 demonstrates the sequence of these five stages. Each stage is located inside the dotted-line rectangle and numbered in the top left corner.

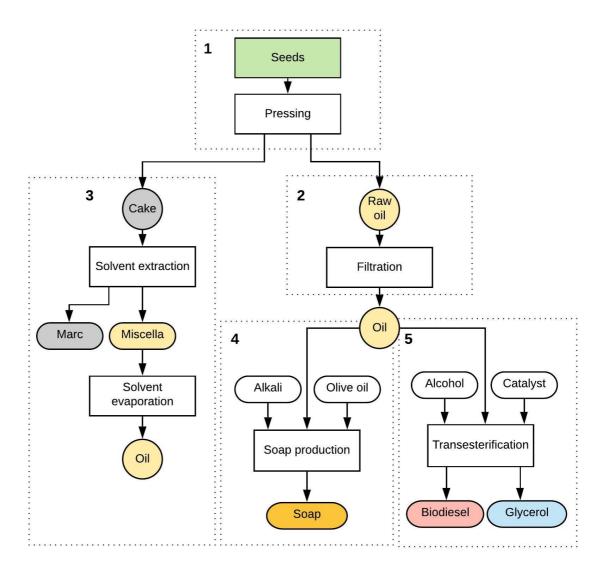


FIGURE 7. Five stages of experimental research.

All instructions and equipment were provided by the educational institution where the project took place. Manipulations performed in each stage are described in subsections.

## 3.1 Production of rapeseed oil and oilcake

Educational institution, where the project took place, provided 4500 g of standard rape seeds. The seeds were already in a washed and cleaned state. The flaking and cooking procedures were not performed due to a lack of machinery equipment. For oil pressing procedure was used a small capacity screw press Farmet UNO FM. As it can be seen in Picture 1, the oil screw press machine had frequency converter, oil outlet, oilcake outlet and the entry for the seeds. The picture from official manual can also be found in appendix.



PICTURE 1. Farmet UNO FM seed oil press design.

The oilcake outlet is located at the press head with the press nozzle installed at the end of the outlet hole. This nozzle came in three diameters: 6 mm, 8 mm and 10 mm. These three diameters were used in the experiment. On the top of the engine is located the frequency converter with adjustable frequencies. For the experiment were set three rotational speed frequencies: 30 Hz, 50 Hz and 70 Hz. The aim of this stage was to determine an optimal combination of parameters, press nozzle dimeter and frequency, to obtain as much oil as possible with minimum oilcake. Therefore, a total of nine experiments have been conducted, using three variations of press nozzle diameters and three variations of frequency. For each combination of these variations a 500 g of seeds were fed into the screw press machine. The type of pressing was cold, maintaining the temperature at 60°C. In the process of oil pressing the raw oil was coming out of the oil outlet and

streaming into the glass container. The oilcake was coming from press nozzle and collected into the plastic bucket.

# 3.2 Filtration of rapeseed oil

A filtration centrifuge Hettich EBA 200 (Picture 2) was used to filtrate the part of the obtained raw oil from impurities. The capacity of centrifuge was 8 centrifuge tubes of 15 ml volume each. The rotational speed of sentrifuge was set to 5000 rpm. The first aim of this experiment was to see how much impurities the unfiltered raw oil contains.



PICTURE 2. Hettich EBA 200

A total of 500 g of produced oil from stage 1 was taken to perform this experiment. This specific quantity was chosen, because only this quantity of oil needed to be filtrated to be used in the rest of following experiments. The remaining raw oil was set aside to be used in another project. The experiment was performed in two combinations: 250 g of oil was filtrated for 5 min and 250 g was filtrated for 10 min. In one spin was filtrated around 60 g of oil. The second aim of this experiment was to see which time set up would give the best ratio of filtrated oil to impurities. The filtration was performed until all 500 g of oil was filtrated. After the filtration was complete the oil was poured into separate glass container. The filtrated impurities were not used in further experiments.

## 3.3 Solvent extraction

From the seed pressing stage were produced raw oil and oilcake. For solvent extraction experiment were taken oilcakes of two diameters of 6 mm and 10 mm (30 Hz), 50 g each (Picture 3). The aim of this experiment was to extract oil from two oilcakes of smallest and biggest diameters, to analyse how much oil they contained and to make a comparison.



PICTURE 3. Oilcakes of 6 mm and 10 mm diameters.

The solvent was provided by educational institution, where the project took place. The solvent used was isopropyl alcohol, also known as propan-2-ol with empirical formula  $C_3H_8O$ . Masses of each product are shown in Table 4.

Diameter, mm	Oilcake, g	Solvent, g	
6	50	100	
10	50	100	

The further described procedure was performed in the same way on oilcakes of 6 mm and 10 mm diameters. A 50 g of oilcake was mashed until fine crumb consistency with mortar and pestle. To extract the oil from the oilcake, the obtained mashed oilcake was transferred into glass beaker and 100 g of solvent was added. The solvent-oilcake substance was mixed with a magnetic stirrer for 10 minutes (Picture 4).



PICTURE 4. Production of marc and miscella.

At the end of mixing process, the miscella (solvent-oil mix) was formed at the top part of the beaker and the marc (seed residue) was precipitated at the bottom. The miscella and marc were carefully separated. The miscella was poured into a round-bottom flask for further solvent evaporation. To perform this, a Heidolph rotary evaporator was used (Picture 5).



PICTURE 5. Heidogh rotary evaporator.

The set-up parameters were as follows: 150 rpm rotary speed, 400-450 mbar maintained pressure, 80°C degrees temperature. The process was completed when the solvent completely evaporated and condensed into a large round-bot-tom flask.

## 3.4 Production of soap

The aim of this experiment was to produce a soap with a pH value safe for human skin. For soap making experiment was used a mix of filtrated rapeseed oil and a store bought extra virgin olive oil. The olive oil was chosen to be added because of its positive effects on human skin (Badiu, Luque & Rajendram 2010). The mix of oils was 1:1. To perform a saponification reaction was used NaOH. A mass of NaOH to be added to oils depends on mass of oil mixture. In order to calculate the masses, the number of moles of each component had to be calculated. The number of moles was calculated using the formula (1).

$$n = \frac{m}{M},\tag{1}$$

where *n* is number of moles (mol), *m* is mass of substance (g), *M* is molecular weight (g/mol). When all values were calculated, the experimental manipulations

had started. Firstly, a NaOH solution was prepared. The solution was carefully stirred with the stirring magnet until complete dissolution of NaOH. Finally, NaOH solution was added to the oil mixture and was stirred with magnetic stirrer for 15 minutes. As a final step, a liquid soap was poured into the five glass container to air dry for four weeks (Picture 6).



PICTURE 6. Produced soap at week 1.

## 3.5 Production of biodiesel

To perform a reaction of transesterification were used rapeseed oil, ethanol  $(C_2H_5OH)$ , solid sodium  $(Na_{(s)})$  and sodium sulphate  $(Na_2SO_4)$ . The transesterification reaction to obtain the rapeseed ethyl ester (biodiesel) is shown in Figure 8.

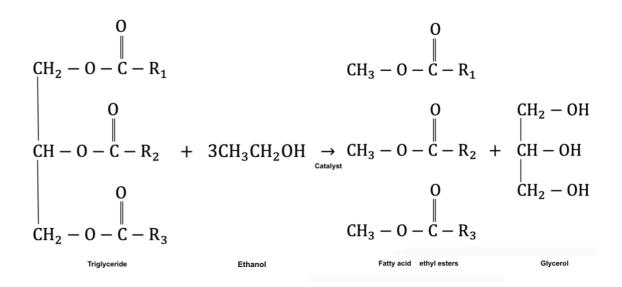


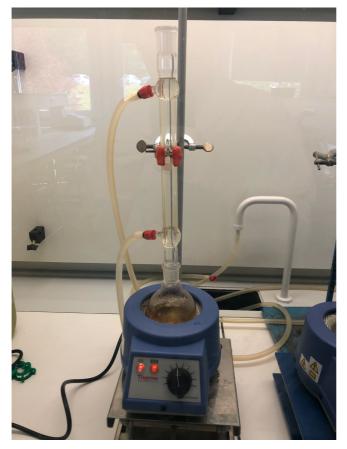
FIGURE 8. Transesterification reaction.

First, a blend of  $Na_{(s)}$  and  $C_2H_5OH$  was prepared to obtain a solution of ethoxide ions. This manipulation has to be done to create an active catalyst to produce ethyl esters. A  $Na_{(s)}$  was chosen over the aqueous NaOH in order to avoid the production of water molecule. Water is unwanted substance for the production of biodiesel, because it causes a saponification reaction. The reaction of ethanol with sodium occurs as shown in Figure 9.

$$2CH_3 - CH_2 - OH + 2Na \rightarrow 2CH_3 - CH_2 - O^{-}(aq) + 2Na^{+}(aq) + H_2(g)$$

FIGURE 9. Reaction of alcohol with sodium.

A 100 ml of C<sub>2</sub>H<sub>5</sub>OH was poured into a glass beaker and 0.46 g of Na<sub>(s)</sub> was added and mixed carefully. The reaction was bubbling producing the hydrogen gas. The indication of complete reaction was the end of gas production. Into another beaker was poured 120 ml of filtered rapeseed oil. A pinch of Na<sub>2</sub>SO<sub>4</sub> was added to absorb any possible excess water in the oil. As shown in Picture 7 solution with ethoxide ions and rapeseed oil were transferred into a round bottom flask and placed into a reflux apparatus for heating a chemical reaction for 45 minutes.



PICTURE 7. Boiling solution in reflux apparatus.

Afterwards, the flask was cooled down with ice and poured into a separating funnel. A 120 ml of salt saturated cold water was added into the separating funnel, shaked well for few minutes and left in the stand for further decantation for the next 24 hours. The total volume of final solution in separating flask was around 340 ml.

## 3.5.1 Paper chromatography

Paper chromatography was performed to identify the formed biodiesel and rapeseed oil solutes. This method was chosen for its cost efficiency and simplicity. To better understand the manipulations performed in analytical chromatography testing Figure 10 was created.

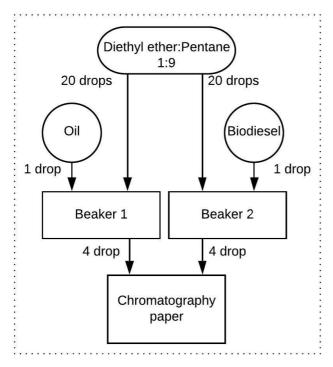


FIGURE 10. Sequence of processes in analytical chromatography.

In a glass beaker was prepared a 10 ml of liquid solvent. The solvent was a mixture of diethyl ether ( $C_4H_{10}O$ ) with pentane ( $C_5H_{12}$ ) in a ratio 1 to 9 respectively. Therefore, 1 ml of C<sub>4</sub>H<sub>10</sub>O was mixed with 9 ml of C<sub>5</sub>H<sub>12</sub>. As a next step, two empty glass beakers were prepared and named beaker 1 and beaker 2. The solvent was added to each beaker in number of 20 drops per each. To beaker 1 was added 1 drop of filtrated rapeseed oil and to beaker 2 was added 1 drop of ethyl ester. Both solutions were mixed well. From each solution were taken 4 drops and placed onto a strip of a chromatography paper, making two spots. The paper was placed into a glass beaker with a shallow layer of solvent and sealed until the solvent met the product samples and travelled up the paper until it reached 8 mm from the top. Then, the paper was taken out, and placed into developing chamber with iodine crystals. Iodine oxidized the products on the paper turning it into brown colour, making results visible to an eye. A clearly distinguished spots appeared on a paper, which characterized biodiesel and oil components present in the mixture. To better interpret the paper chromatography results, a retention factor  $(R_f)$  had to be calculated.  $R_f$  is defined as a ratio of distance travelled by each component to distance travelled by solvent.  $R_f$  is calculated as shown in formula (2).

$$R_f = \frac{h}{H},\tag{2}$$

30

in which h is distance travelled by component (cm), H is distance travelled by solvent (cm).

## 3.5.2 Viscosity measurements

The viscosity of filtrated rapeseed oil and biodiesel were measured using the digital viscometer with set up of 60 rpm and the small sized rotating spindle. The following procedure was performed in the same way for both liquids, oil and biodiesel. The liquid was placed into a glass beakers and warmed up to 20°C using the laboratory round heating plate. The liquid was placed under the viscometer and rotating spindle was submerged into the liquid. The viscosity value was shown on the display of the viscometer.

## 4 RESULTS

## 4.1 Production of rapeseed oil and oilcake

A total of 2834.32 g of raw oil and total 1655.89 g of oilcake were produced from 4500 g of seeds. The total loss of 10.79 g of product occurred during the pressing process. Figure 11 demonstrates the occurred process.

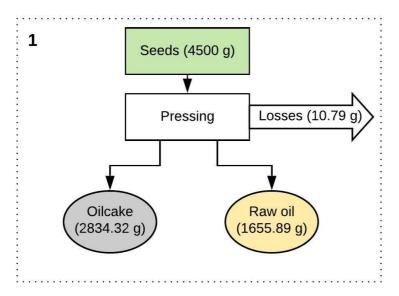


FIGURE 11. Sequence of processes and masses of raw material and products in stage 1 of oil pressing.

As it can be seen from Table 5 and Table 6 and the combination of press nozzle with diameter 6 mm and 30 Hz frequency gave the best oil to cake ratio. From 500 g of seeds was obtained 193.27 g of oil and 304.17 g of oilcake. The sum mass of two products gives 497.44 g, which is equivalent to 99.48% produced products out of 500 g seeds.

	Oil, g	Oilcake, g	Oil, g	Oilcake, g	Oil, g	Oilcake, g
Diameter	30	Hz	50	Hz	70	Hz
10 mm	185.09	300.32	185.28	312.16	175.74	324.06
8 mm	190.30	306.45	183.88	318.35	175.10	324.69
6 mm	193.27	304.17	187.75	318.01	178.48	326.11

	Oil	Oilcake	Oil	Oilcake	Oil	Oilcake
Diameter	30	Hz	50	Hz	70	Hz
10 mm	37.02%	60.06%	37.06%	62.43%	35.15%	64.81%
8 mm	38.06%	61.29%	36.78%	63.67%	35.02%	64.94%
6 mm	38.65%	60.83%	37.55%	63.60%	35.70%	65.22%

TABLE 6. Percentage of obtained raw oil and oilcake using different press nozzle.

## 4.2 Filtration of rapeseed oil

From 500 g of raw rapeseed oil obtained was filtrated off 24.92 g of impurities. The oil became clearer and slightly lighter on colour. Figure 12 demonstrates the sequence of the process.

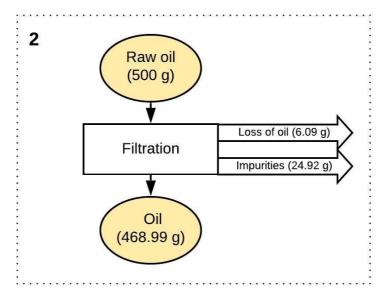


FIGURE 12. Sequence of processes and masses of raw materials and products in stage 2 of filtration centrifuge.

As it can be seen from Table 7 the time spin of 5 min and 10 min did not produce significantly different results. Therefor its was concluded that the set-up of 5 min spin will be used for future projects of raw oil filtration.

Spin time, min	Raw oil, g	Filtrated oil, g	Impurities, g	Loss of oil, g
5	250	234.19	12.35	3.46
10	250	234.80	12.57	2.63
Total	500	468.99	24.92	6.09

TABLE 7. Masses of filtrated oil and residue after two spin times.

## 4.3 Solvent extraction

The sequence of the process, masses of raw materials and products are demonstrated in Figure 13. The results of solvent extraction of oil can be observed in Table 8. The 6 mm diameter oilcake contains 4.37 g of oil per 50 g of oilcake. It can be seen that not all 100 g of solvent was recovered, this is because the part of the solvent was eliminated with the marc. The 10 mm diameter oilcake contains 6.57 g of oil per 50 g of oilcake. Therefore, 10 mm diameter oilcake contained more oil than 6 mm diameter. This indicates that 6 mm diameter press nozzle is more efficient to use, because more oil is produced directly from the oil press and the oilcake contains less oil.

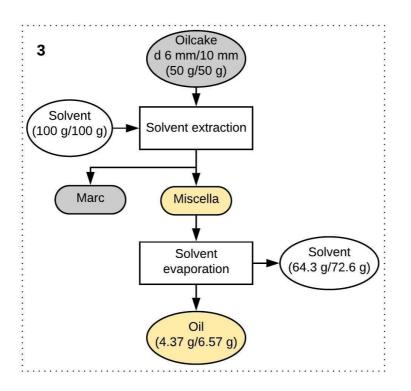


FIGURE 13. Sequence of processes and masses of raw materials and products in stage 3 of solvent extraction.

TABLE 8. Masses of extracted oil and recovered solvent from oilcakes.

Diameter, mm	Extracted oil, g	Extracted oil, %	Extracted solvent, g	Extracted solvent, %
6	4.37	8.74	64.3	64.3
10	6.57	13.14	72.6	72.6

Table 9 shows the total raw oil and oilcake obtained from oil pressing (stage 1). The total oil extracted from oilcake of each diameter was calculated based on mass of oil extracted from 50 g of oilcake. Therefore, the sum of total extracted oil with the total extracted oil from oilcake was calculated. The total oil produced from 500 g of seeds was calculated.

	Stage 1. Oil pressing		Stage 2. Solvent extraction		
Diameter,		Oilealia a	Total oil extracted from	Total oil	Oil produced/500 g
mm	Raw oil, g	Oilcake, g	oilcake, g	produced, g	seeds, %
6	193.27	304.17	26.58	219.85	43.97
10	185.07	300.32	39.46	224.53	44.91

TABLE 9. Masses of total extracted oil from oilcakes per 500 g of seeds.

These results show how much of total oil was produced from oil pressing (stage 1) and solvent extraction (stage 2) together for two different nozzle diameters. It is important to mention that the calculations presented in Table 9 give only an approximate results. The 10 mm diameter (30 Hz) showed that more total oil was produced from 500 g of seeds, than 6 mm diameter. However, the difference is not that significant. The 10 mm diameter oilcake still had more oil inside than 6 mm diameter, which makes 6 mm diameter still more efficient.

# 4.4 Production of soap

The sequence of the process of the soap production, masses of raw materials and products are shown in Figure 14.

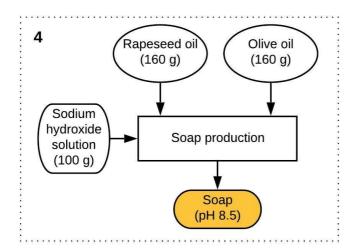


FIGURE 14. Sequence of processes and masses of raw materials and products in stage 4 of soap production.

The calculate the mass of NaOH further calculations were done. The mass of rapeseed oil is 150 g. The molar mass of rapeseed oil is 885 g/mol, this data was provided by supervising professor. The mass of olive oil is 150 g. The molar mass of olive oil is 884 g/mol (Clever 1980, 241). In the saponification reaction one molecule of triglyceride reacts with three molecules of NaOH. Therefore, the number of moles was calculated for rapeseed and olive oils respectively using formula (1):

$$n = \frac{150 \text{ g}}{878 \frac{\text{g}}{\text{mol}}} = 0.1708 \text{ mol}$$

$$n = \frac{150 \text{ g}}{884 \frac{\text{g}}{\text{mol}}} = 0.1697 \text{ mol}$$

Since two oils were mixed together into one substance, a total number of moles were calculated:

$$n_{\text{rapeseed oil}} + n_{\text{olive oil}} = n_{\text{total}} = 0.1708 \text{ mol} + 0.1697 \text{ mol} = 0.3405 \text{ mol}$$

The next step was to find the number of moles of NaOH. As it was mentioned before one molecule of triglyceride reacts with 3 molecules of NaOH. The calculation was following:

$$n_{\text{NaOH}} = n_{\text{total}} \cdot 3 = 0.3405 \text{ mol} \cdot 3 = 1.0216 \text{ mol}$$

The molar mass of NaOH is 39.9 g/mol (National Center for Biotechnology Information 2020). Now, knowing the number of moles of NaOH and the molar mass, a mass of substance needed for saponification reaction can be calculated as follows:

$$m = M \cdot n = 39.9 \frac{g}{\text{mol}} \cdot 1.0216 \text{ mol} = 41 \text{ g}$$

All obtained data is collected into Table 10.

Component	Mass, g	M, g/mol	n, mol
Rapeseed oil	150	878	0.1708
Olive oil	150	884	0.1697
NaOH	41	39.9	1.0216

TABLE 10. Mass, molar mass, number of moles of components.

Now, when all masses are known, the experimental manipulations can be started. First, 1 mol of NaOH solution had to be prepared. A glass beaker was filled with 1 L of water and 41 g of NaOH pellets were slowly added to the water and stirred until complete dissolution. To make sure that NaOH is completely used during the saponification reaction, it was decided to increase the mass of oil mixture by 7%. This made the mass of total oil mixture equal 320 g. Finally, 100 g of NaOH solution was added to the oil mixture and was stirred with magnetic stirrer for 15 minutes. After four weeks of maturing, the pH of soap was measured with pH paper. The pH of soap was approximately 8.5. This pH is considered to be safe for use for human skin (Tarun et al. 2014).

## 4.5 Production of biodiesel

Figure 15 demonstrates the sequence of the process, masses of raw materials and products of biodiesel production.

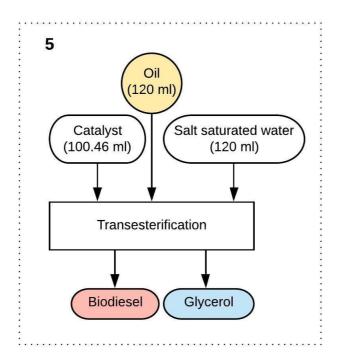


FIGURE 15. Sequence of processes and masses of raw materials and products in stage 5 of biodiesel production.

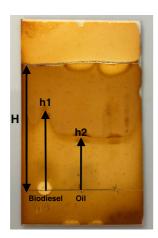
After 24 hours a formed biodiesel was gathered as the top layer and at the bottom was formed glycerol (Picture 8). The glycerol and biodiesel were carefully extracted from the separating funnel into two separate beakers. It was very difficult to manually separate to substances. The filtration processes of both substances could not be performed due to the lack of equipment. It was approximated, that the ratio ethyl ester to glycerol was 5:1. The ethyl ester was further tested with analytical chromatography and for viscosity.



PICTURE 8. Decantation process in separating funnel.

# 4.5.1 Analytical chromatography

Results of paper chromatography with distances travelled by solvent and two spots are demonstrated in Picture 9.



PICTURE 9. Paper chromatography results.

The distance travelled by solvent (*H*) was 3 cm, the distance travelled by biodiesel spot ( $h_1$ ) was 2 cm and the distance travelled by rapeseed oil spot ( $h_2$ ) was 1.4 cm.

Calculations of  $R_f$  were performed using formula (2):

Biodiesel: 
$$R_f = \frac{2}{3} = 0.66$$
  
Rapeseed oil:  $R_f = \frac{1.4}{3} = 0.46$ 

The value of the retention factor of biodiesel is greater than of rapeseed oil, so the values found by the technique were considered satisfactory and indicative of biodiesel formation.

#### 4.5.2 Viscosity

The measured viscosity value of rapeseed oil was 66.3 mm<sup>2</sup>/s. The measured viscosity value of biodiesel was 9.4 mm<sup>2</sup>/s. The obtained viscosity value of rapeseed oil was compared to the value from Table 3, where the viscosity of rapeseed oil is 78.2 mm<sup>2</sup>/s. The obtained viscosity of biodiesel was compared to the value provided by Esteban et al. (2012, 167–168), where the viscosity is 6.78 mm<sup>2</sup>/s. Both viscosities are different from required standard viscosities. The differences are discussed in the discussion section.

#### **5 DISCUSSION**

In stage 1, it was found that the best combination of nozzle head and frequency was 6 mm and 30 Hz respectively. This combination produced the biggest quantity of oil of 193.27 g and the least quantity of oilcake of 304.17 g. This can be explained, that the small diameter of the nozzle head creates a pressure inside the press head on producing oilcake. Oilcake gets pressurized and realises the its oil. This process gets enhanced when we add slow speed of the screw. With slow speed the pressure on oilcake increases and therefore more oil is extracted. However, following this logic, it cannot be concluded that the smaller the press nozzle the more efficient the oil production is. The minimum press nozzle size is usually provided by the manufacturer of the screw press machine. The diameter of nozzle depends on the composition of pressed raw material. Correctly selected nozzle helps to avoid the oilcake getting stuck inside the screw press and causing the breakdown of the machine. In stage 1 was also detected total loss of products of 10.79 g. Loss was discovered, because the sum of masses of produced oil and oilcake did not match the total mass of fed seeds. It can be explained, that oil pressing was done nine times, each time feeding the small portion of 500 g of seeds. There was no continuous pressing. Therefore, during the pressing process not all of the product was extracted from the screw press and therefore some of it remained inside. The loss can be avoided if the screw press machine operates continuously with the constant supply of seeds. In this way the loss can be minimized. It is also important to mention, that the quantity of seeds provided was limited, therefore the experiment could not be performed several times. In a related research article by Wcisło (2006) the biggest mass of oil obtained was 389 g per 1000 g of seeds. Comparing both results it can be concluded that the mass of produced oil in this thesis work is almost identical to the results of related research. This can be considered as a positive outcome, but it is important to keep in mind the differences in both methods. In another related research article by Pasyniuk and Golimowski (2013) the yield of obtained oil mass to seeds mass was 29%. As it was mentioned in section 2.3, according to Przybylski et al. (2005, 80) the seed of rape on average contains 44% of oil. In this thesis work the maximum yield was obtained as 38.65%. This also shows that the set-up of parameters was very efficient in this thesis work.

In stage 2, the two set-ups of 5 min and 10 min of spinning time did not produce significantly different results. Therefore it was decided that the set-up of 5 min spin will be used for future projects of raw oil filtration. It is important to mention, that used method fits only the small quantities of oil or any other liquid. The quantity of oil filtrated was 250 g, which can be considered as a big amount. Due to the lack of laboratory equipment, the centrifuge filtration was used in this process. The viscosity of filtrated oil and the standard viscosity of rapeseed oil were 66.3 mm<sup>2</sup>/s and 78.2 mm<sup>2</sup>/s (Table 3) respectively. The comparison shows that the oil obtained in this thesis work is less viscous. This can be due to possible temperature errors, poor filtration of oil or other similar errors.

In stage 3, oilcakes of only 6 and 10 mm diameters were taken for the solvent extraction. This was done in order to compare the oil content in oilcakes of smallest and biggest diameters and to compare them. This comparison also showed how much more efficient it was to use the press nozzle of 6 mm in combination with frequency of 30 Hz. Results showed that oilcakes of 6 mm diameter contained less oil inside, than oilcakes of 10 mm diameter. This indicated that the majority of oil was pressed from the seeds directly from the oil screw press and the oilcake contained less oil. In this way, this is more cost efficient for production. For example, individuals who produce oil in a farm scale, usually would feed the cattle with produced oilcake. It is not cost efficient if oilcake contains too much of excess oil. The oil can be used in many application areas and it is not good to waste it in a form of feed for livestock. Therefore the correct combination of press nozzle and frequency will help to avoid the loss of valuable oil. It is also important to mention, that in the process of oil extraction from oilcakes, not all solvent was recovered. This happened, because the part of the solvent was eliminated when marc and miscella were separated. The part of solvent was eliminated with the marc. The average total mass of oil produced from oilcakes with 6 and 10 mm diameters, was 222.19 g per 500 g of seeds.

As a result of stage 4 of soap production were produced five small soaps. The achieved pH of 8.5 of soap was considered to be very good. This result indicated that all calculations were performed correctly and a correct ration of oils to caustic soda was found. It was also a correct to add oils in a small excess to make sure

that NaOH completely dissolved. This decision positively affected on obtained pH value. The excess of NaOH could make soap much more alkaline, which is not safe for human skin. The produced soap was decided to be used in the laboratory for hygienic purpose.

Stage 5 of biodiesel production was the most challenging and complicated process. The experiment was performed two times, first time was unsuccessful. The formation of biodiesel did not happen at all. It was tested with analytical chromatography and the R<sub>f</sub> of biodiesel was equal to R<sub>f</sub> of rapeseed oil, indicating no formation of biodiesel. The results of first trial were not presented in this thesis, because of critical computational errors in calculations. The results of second trial were considered successful. The biodiesel was formed, analytical chromatography demonstrated the formed biodiesel against the rapeseed oil. However it was very hard to define the yield of formed biodiesel, thus the biodiesel to glycerin ratio was approximated to be 5:1. It is important to mention that in industrial scale production, the produced biodiesel is subjected to a number of purification stages. Whereas in the IUT laboratory was a lack of such equipment. The obtained biodiesel cannot be considered to be pure. A comparison was done between the viscosity of obtained biodiesel and the required standard viscosity of biodiesel. It was concluded that the obtained viscosity of 9.4 mm<sup>2</sup>/s is too viscous at temperature of 20°C and cannot be used for fuel purposes. The biodiesel is required to be in a very viscous state, maximum 6.78 mm<sup>2</sup>/s at 20°C.

To summarize, the process of performed oil production in the laboratory and the process of industrial pressing had many differences. In this thesis work, raw cleaned seeds were directly fed into the screw press. Preheating, flaking and cooking steps in preparation stage (Figure 2) were omitted, due to a lack of equipment in the laboratory. These steps could have increased the oil production. Therefore, results were affected of how much oil was pressed out and the content of oil in oilcakes. In the process of laboratory solvent extraction the solvent used was isopropyl alcohol, not hexane. IUT provided only isopropyl alcohol for its low-cost and safety. As a future suggestion, the experiment could be performed two times using both solvents. The results with both solvents could be observed and compared with each other. Overall the addition of machinery equipment and chemicals would have brought more experimental data for diverse discussion.

#### 6 CONCLUSION

It can be concluded that this thesis work has achieved set objectives. The optimal combination of parameters had been identified as press nozzle of 6 mm diameter combined with 30 Hz of rotational speed of screw press. The time spin of 5 min was identified as the best parameter for filtration centrifuge. The solvent extraction demonstrated that 6 mm oilcake contained the least amount of the oil, therefor proving again that press nozzle of 6 mm diameter combined with 30 Hz of rotational speed of screw press produces the best yield of oil. The goal to produce soap with pH safe for human use was achieved. The produced soap had pH of 8.5. The transesterification reaction had been done and biodiesel was produced. However the biodiesel viscosity of 9.4 mm<sup>2</sup>/s was not ideal, it was still considered as a good result taking into consideration the lack of some equipment and possible errors.

As for future suggestions, for filtration centrifuge experiment it is strongly recommended to use other methods for the same or higher quantities of raw oil. For biodiesel production experiment it can be recommended to find ways to filtrate and purify the biodiesel as it is done in industrial scale. In this way, the viscosity of biodiesel might improve. Additionally, chemical reactions might be further modified, by changing the type of alcohol, catalyst, temperature and duration of boiling. Moreover, seeds of different rapeseed cultivars can be tested for oil pressing and further soap and biodiesel production.

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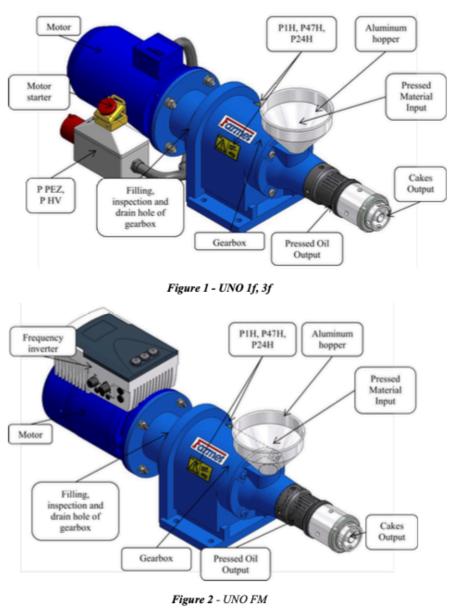
## **APPENDICES**

Appendix 1. Oil screw press Farmet UNO FM in operating manual.

OPERATING MANUAL



OILSEED SCREW PRESS FARMET UNO



# 3. DESCRIPTION OF THE EQUIPMENT

Page 17 of 53