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Research of Acidulation Parameters Affecting the Extraction of Acid Oil from Rapeseed Soapstock

Igors ASTRAUSKS[1], Svetlana ZHIZKUN[2], Indulis STIKĀNS[3], Lauma LAIPNIECE[4]*

[1,2,4]*Institute of Chemistry and Chemical Technology, Faculty of Natural Sciences and Technology, Riga Technical University, 3/7 Paula Valdena Street, LV-1048, Riga, Latvia*
[3]*Bio-Venta Ltd., 21F Ziemeļu Street, LV-3602, Ventspils, Latvia*

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***Abstract* – The problems of climate change, especially the greenhouse effect caused by transport exhaust gas emissions, make the issue of finding alternatives to fossil fuels increasingly urgent. One such alternative is biodiesel. The valorisation of industrial biomass based by-products such as soapstock, generated during vegetable oil refining, supports both waste reduction and sustainable biodiesel production. This study investigates the acidulation of rapeseed soapstock to obtain acid oil – a mixture of free fatty acids and glycerides suitable for advanced biodiesel synthesis. The effects of acid type (concentrated sulphuric acid, 50 % sulphuric acid, 25 % citric acid), pH (2–7), and temperature (20, 45, 70 °C) on purified acid oil yield and phospholipid removal were examined under controlled laboratory conditions. Rapeseed soapstock was acidified till defined pH using chosen acid at set temperature. Then extraction with cyclohexane followed by purification with cold acetone yielded acid oil. The optimal acidulation parameters were achieved with concentrated sulphuric acid at pH 5 and initial soapstock temperature around 20 °C, producing 34.7 % of purified acid oil. These results demonstrate that optimized acidulation of rapeseed soapstock provides a cost-effective route for converting industrial waste into a viable feedstock for biodiesel synthesis, contributing to circular bioeconomy principles and the reduction of fossil fuel dependence.**

***Keywords* – Acidification; biodiesel feedstock; industrial waste; sulphuric acid.**

1. INTRODUCTION

Transportation produces about 26 % of global carbon dioxide emissions, with majority coming from road transportation. Also, transport is the only sector with rising greenhouse gas emissions, highlighting the need for sustainable mobility [1]. Growing diesel demand and fossil fuel limits have driven interest into renewable and advanced biofuels. Biodiesel is a biodegradable, non-toxic fuel with properties similar to fossil diesel. Biodiesel also reduces exhaust gas emissions, reducing hydrocarbons by 67 %, particulate matter by 47 %, and greenhouse gases by 86 % [2], [3].

Biodiesel consists of fatty acid alkyl esters, that can be derived from vegetable oils, algae, or animal fats. Most often biodiesel is produced from annual food crops like rapeseed,

* Corresponding author.
E-mail address: lauma.laipniece@rtu.lv

Igors ASTRAUSKS https://orcid.org/0009-0009-3206-9014
Svetlana ZHIZKUN https://orcid.org/0000-0002-0568-1664
Lauma LAIPNIECE https://orcid.org/0000-0002-4190-0692

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soybean, and palm [4], [5]. However, reliance on food crops raises sustainability concerns. Rapeseed is widely used in Europe due to high oil yield and favourable fuel properties, especially in winter [6]–[8]. Using industrial waste as biodiesel feedstock offers dual advantages: it provides a sustainable raw material source while aiding in waste management, reducing environmental footprints, and promoting circular economy principles [4], [8]–[10].

Vegetable oil extraction (mechanical, solvent, or enzymatic) produces crude oil containing mostly triglycerides and impurities such as phospholipids and free fatty acids (FFA) [11], [12]. Vegetable oil refining begins with degumming to remove phospholipids and continues with neutralization converting FFA into sodium salts (soaps). After centrifugation, the process yields neutral oil and soapstock – a refinery side stream containing soaps, residual oil, excess alkali, and impurities [12], [13]. Both steps can be realized separately obtaining mostly phospholipids containing gums and soapstock or, alternatively, combined degumming and neutralization produce a mixed soapstock composed mainly of alkaline water (32–67 %), fatty acids as soaps (10–28 %), phospholipids (5–9 %), and acylglycerols (12–13 %) [14].

The efficient extraction of phospholipids and acid oil from soapstock – a low-value residue from vegetable oil refining – is a promising approach for advancing circular economy principles [15]. Hence soapstock valorisation supports integrated biorefinery and circular economy concepts. For example, two acid oil components are suitable for the synthesis of biodiesel: FFA and glycerides [16]–[18]. Phospholipids inhibit chemical catalysts in biodiesel synthesis, and cause emulsions that complicate biodiesel and glycerol separation [19], but separated phospholipids may serve as a raw material for obtaining other valuable products [20]–[22]. Components of soapstock need to be separated by economically justified techniques. In earlier attempts, lyophilization was used to remove moisture, but difficulties in biodiesel synthesis occurred because other impurities remained [23]. Also, total hydrolysis of glycerides and phosphatides was elaborated to obtain FFA, which were subjected to esterification [24]. But most commonly acidulation or soap-splitting is used to produce FFA and glycerides containing acid oil, which is converted to biodiesel [18], [24]–[29]. Acidulation is usually performed till pH 2–3 at 80–95 °C temperature [18], [24], [27]–[30] without optimization of acidulation conditions, but phospholipids are usually not met or are hydrolysed during acidulation in investigated soapstocks and acid oils. Typical method for separation of phospholipids is precipitation with cold acetone [21], [27].

Previous studies of acidulation conditions have focused mainly on soapstocks from rice bran [31], buriti [32], sunflower [33], and soybean oils [25], leaving limited data on rapeseed soapstock and the relationship between acidulation conditions and acid oil yield. Amount of sulphuric acid used for acidulation was expressed as mole ratio to soaps [31] or soapstock [32] and optimized or maintained in excess as controlled by methyl orange [33]. Optimized parameters differed in each report, but overall acidulation parameters have been found as temperature in the range of 90–95 °C for 40–90 min, but these conditions are harmful for phospholipids [11], that are valuable by-products. Rice bran soapstock was acidified to obtain γ-orizanol in acid oil but yield of acid oil was not reported [31]. Burity soapstock conversion into fatty acids reached 94 % [32], although used soapstock contained unusually low amount of moisture. Sunflower oil soapstock yielded 79 % of acid oil [33]. Acidulation of soybean soapstock was optimized very differently, optimal amount of water for dilution of soapstock was determined to be 60 % of soapstock mass [25]. Soybean soapstock was acidified at 25 °C temperature and addition of sulphuric acid was monitored till easy separation of two layers, as formation of emulsion was a problem caused by phospholipids. Reported yield of acid oil at optimized dilution and acidulation speed reached 45 % [25]. Reported yields in these studies were practically incomparable, because soapstock composition could be very different

depending mostly on oilseed species and refining technology [13], but also many other conditions could be different from day to day in the same refinery.

Soapstock use as raw material for biodiesel synthesis was extensively explored in the first decade of 21st century, but there is scarce information in the last decade, now our group renewed research on rapeseed soapstock use in sustainable advanced biodiesel synthesis. First step to proceed from soapstock to advanced biodiesel is acidulation which is not extensively studied for rapeseed soapstock, but occasionally just mentioned as one procedure from many without investigation. In our present research we are exploring acidulation parameters to evaluate acid oil yield and purity with emphasis on phospholipid content, which should be minimal. Valorising soapstock aligns with the EU Bioeconomy Strategy and Renewable Energy Directive (RED III) [34] goals to improve biomass resource efficiency and reduce industrial waste. This research will enable use of industrial waste as a low-cost feedstock leading to a more cost-effective production process and making advanced biodiesel a more competitive alternative to fossil diesel.

2. MATERIALS AND METHODS

2.1. Materials

Soapstock is obtained from combined degumming and neutralization steps in refining process of rapeseed oil production in BioVenta, Ltd. BioVenta is a full cycle biodiesel producer in Latvia, producing biodiesel started from rapeseeds, vegetable oil, or waste oils. Commercial reagents were used without purification: sulphuric acid (98 wt%, Chempur), citric acid monohydrate (99.5 %, Chempur), cyclohexane (99.5 %, SigmaAldrich), acetone (99 %, Chempur), petroleum ether (40–60 °C, pure, Chempur). Acid solutions were prepared in weight percent. Solvents were recovered and reused in process evaluation to support sustainable valorisation.

2.2. Acid Oil Extraction from Soapstock

Full process scheme of acid oil isolation from rapeseed soapstock is shown in Fig. 1. The soapstock sample (25.00 g) was heated to a certain temperature (if it was necessary) in a water bath. When the required temperature was reached, the sample was acidified to a certain pH value with concentrated or 50 % sulphuric acid or 25 % citric acid. The mixture was vigorously shaken for two minutes. Cyclohexane (25 mL) was added to the resulting mixture, then it was extracted in an ultrasonic bath (Bandelin Sonorex (160/640 W power, 35 kHz frequency) for 30 minutes. The mixture was centrifuged for 10 min (8000 rpm; Ohaus Frontier 5916) to separate the obtained phases. The top layer was decanted and evaporated to yield crude acid oil.

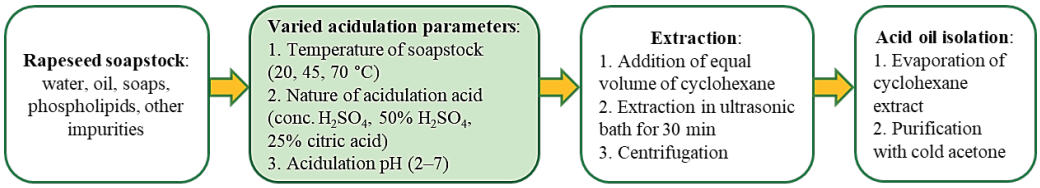

Fig. 1. Step by step process scheme of acid oil isolation from rapeseed soapstock.

Cooled acetone (5 °C) was added to the crude acid oil in an amount exceeding the crude acid oil five times by weight to separate phospholipids from the sample. The sample was stirred and placed in the refrigerator for one hour. Then the purified acid oil was filtered, evaporated and collected.

2.3. Analysis Methods

Soxhlet extraction of soapstock was performed [35] using petroleum ether as solvent. Prior to Soxhlet extraction SS was acidulated with sulphuric acid to pH 2 and water was removed by centrifugation, the residue was mixed with clean sand and dried at 103 °C.

Titration curve was obtained using PASCO wireless drop counter PS-3214 with PASCO wireless pH sensor PS-3204 and SPARKvue v.4.10.0.6 Software. Soapstock sample (4.000 g) was dissolved in 20 mL of isopropanol and titrated with 0.10 M H_2SO_4 in water.

Acid oil components (FFA, monoglycerides, diglycerides and triglycerides) were quantified using gas chromatography system (Agilent technologies 7890A) equipped with 7683B automatic liquid sampler and temperature programmable splitless injector. DB5-HT column (15 m × 0.32 mm × 0.10 μm) was used with carrier gas He flow rate 2 mL/min; detector temperature 390 °C. Column oven and injector temperature program: 50 °C (5 min) → 180 °C (15 °C/min) → 230 °C (7 °C/min) → 370 (10 °C/min, 5 min). In the gas chromatography analysis, derivatisation reagent *N*-trimethylsilyl-*N*-methyltrifluoroacetamide and two internal standards, 1,2,4-butanetriol and tricaprine, have been used.

The moisture content of soapstock was determined using a moisture analyser Precisa EM 120-HR. PerkinElmer Spectrum 100 Fourier Transform Infrared spectrometer (FTIR) connected with Universal Attenuated Total Reflectance Sampling Accessory was used for phospholipid analysis in mid-infrared spectral range 650–4000 cm^{-1}.

MATLAB R2024a (24.1.0.2628055) software was used to perform calculations.

The yields (%) were calculated as the product to rapeseed soapstock mass ratio expressed in percents.

Accuracy and precision of measurements and instruments, variations in operating conditions, calibration of the instruments, and actions of the scientists cause an uncertainty of experiment results. Estimation of uncertainty of measurements is important to validate the experimental results [36], [37]. Experiments were performed several times and averaged. Each analytical measurement was carried out twice and the average value was calculated and used. Gas chromatography results of standard solution measurements were used in linear regression model to determine acid oil composition. Table 1 shows the uncertainties of the measured and calculated parameters.

TABLE 1. UNCERTAINTIES OF MEASURED AND CALCULATED PARAMETERS

Parameter	**Measuring range**	**Uncertainty**
pH	0–14	0.1
Amount of acid	–	0.001 mmol H^+/g
Triglycerides	10–100 %	2.0 %
Diglycerides	0–20 %	0.5 %
Monoglycerides	0–20 %	0.5 %
FFA	0–100 %	1.9 %

3. RESULTS AND DISCUSSION

Soapstock cleavage using mineral acid, called acidulation, is the simplest, cheap and widespread method of fatty acid extraction. In order to find optimal economic conditions for the process of producing acid oil from rapeseed soapstock, an assessment was made of the influence of the following three parameters on the yield of acid oil and phospholipids: the nature of the acid, the required pH value of acidulation, and the initial temperature of the soapstock.

Soapstock was received as mustard coloured viscous emulsion, which was formed in combined degumming and neutralization steps of rapeseed oil refining, pH value was 8.5. The dry matter content of soapstock was 43.6 % and total fat content as determined by Soxhlet extraction was 37.9 %. pH titration curve (Fig. 2) showed one step attributed to soap conversion to FFA. No distinct changes were found that could be attributed to phospholipid protonation. A 0.122 mmol of acid per gram of soapstock was needed to neutralize the soapstock and further 0.06 mmol of acid was needed to convert soaps to acids.

Three acids were chosen for acidulation of soapstock: concentrated sulphuric acid, 50 % sulphuric acid and citric acid. In research of soapstock acidulation, sulphuric acid is widely used both in concentrated form [24] and as 50 % solution [16], [26]. Vegetable oil refining plants usually exploit citric acid solutions in chemical refining [12], therefore, citric acid was also chosen for acidulation of soapstock to compare performance of this moderately strong organic acid opposed to strong inorganic sulphuric acid.

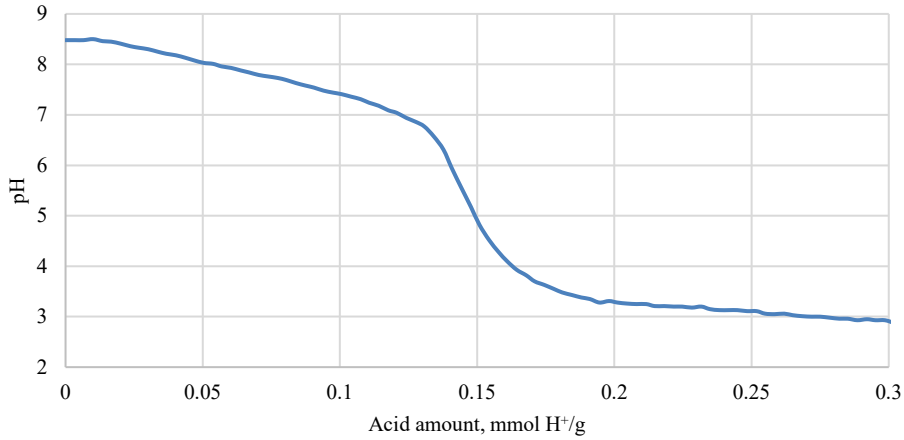

Fig. 2. Titration curve of rapeseed soapstock with 0.10 M H_2SO_4.

The titration curve allowed to calculate the amount of acid required to reach a certain pH, which was in good agreement with the actual pH achieved. A set of experiments was performed to determine the optimal acid and pH value (Table 2). These experiments were performed at room temperature around 20 °C. Crude acid oil was obtained by acidulating soapstock and extracting the lipid phase with cyclohexane under ultrasound. Ultrasound ensures fast and energy efficient extraction even at room temperature [38]. Extraction with solvents was essential to avoid acid oil losses due to inseparable emulsion formation as reported by Wang *et al*. [25], they overcame this by dilution of soapstock. In addition, Choi *et al*. performed acidulation at room temperature and used isopropanol and *n*-hexane as

extraction solvents [26]. Use of both cyclohexane and ultrasound were found to be suitable in previous studies from our scientific group [30]. The crude acid oil was purified by acetone precipitation to yield purified acid oil with reduced phospholipid content and acetone-insoluble fraction, which is mainly composed of phospholipids. It was possible to obtain all pH values in range from 2 to 7 using sulphuric acid. Citric acid did not allow to reach pH 2 and yielded inseparable emulsion in extraction step at pH 7, therefore, acid oil could not be obtained.

TABLE 2. YIELDS OF PURIFIED ACID OIL AND ACETONE-INSOLUBLE FRACTION FROM RAPESEED SOAPSTOCK AFTER ACIDULATION WITH 25 % CITRIC ACID AND CONCENTRATED H_2SO_4 AT 20 °C TEMPERATURE

Entry	pH	Acidulation with conc. H_2SO_4		Acidulation with 25% citric acid	
		Purified acid oil yield, %	Acetone-insoluble fraction yield, %	Purified acid oil yield, %	Acetone-insoluble fraction yield, %
1	7	33.0	4.4		
2	6	34.0	1.7	29.5	4.3
3	5	34.7	1.1	29.2	2.6
4	4	34.9	1.4	28.9	2.9
5	3	35.0	1.4	29.1	2.9
6	2	35.3	1.7		

The yields of acid oil were 5–6 % lower when using citric acid compared to use of sulphuric acid, but the yields of acetone-insoluble fraction were approximately twice higher. The effect on the composition of acetone-insoluble fraction had not been studied. Using concentrated sulphuric acid, acidulation to pH 7 resulted in a lower yield of purified acid oil and a higher yield of acetone-insoluble fraction, which could be explained by the incomplete conversion of soaps into fatty acids, resulting in separation of soaps together with phospholipids. The same reason could be proposed for high acetone-insoluble fraction yield at pH 6 using 25 % citric acid. From the results shown in the Table 1, it could be concluded that the use of sulphuric acid was more optimal than the use of 25 % citric acid, since a higher yield of purified acid oil was obtained. Acidulation to pH 4–5 was completely sufficient in aspect of high yield, sulphuric acid economy, and formation of less acidic wastewater.

The obtained data demonstrated that the purified acid oil yield did not depend on the pH value using citric acid, in the range from 3 to 6 and the average value was 29.2 % with a standard deviation of 0.3 %. The results of experiments using sulphuric acid showed the influence of pH on purified acid oil yield (Fig. 3), which could be expressed as regression dependence of purified acid oil yield on activity of hydrogen ions (a_{H+}) as power Eq. (1):

$$y\left(\text{purified acid oil yield},\%\right) = -0.0266 \cdot a_{H+}^{(-0.2756)} + 35.25\,. \tag{1}$$

The regression analysis showed an excellent fit (R^2 = 0.9950, Root Mean Squared Error = 0.076), confirming that the proposed model reliably describes the dependence of purified acid oil yield on activity of hydrogen ions (acidity). The high correlation and low error indicate that the acidulation process is highly predictable within the investigated pH range 2–7.

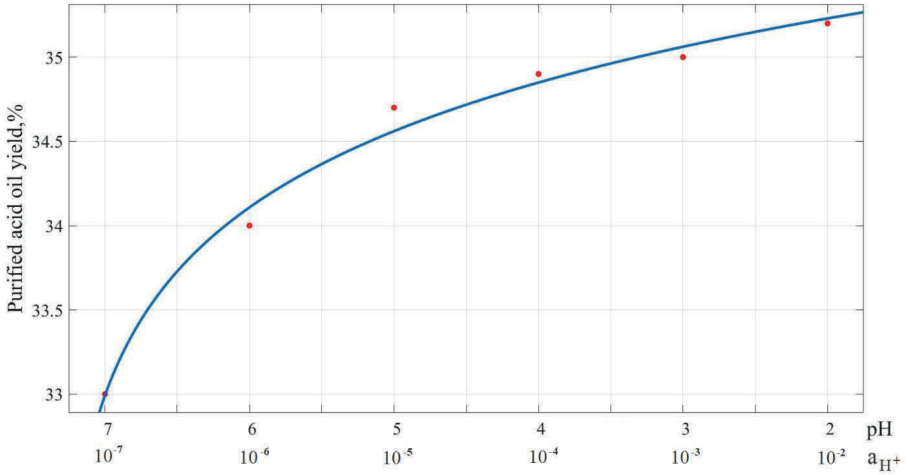

Fig. 3. Dependence of the yield of purified acid oil on the a_{H+} of the soapstock using concentrated H_2SO_4 at 20 °C temperature.

This dependence indicated that with an increase in acidity, the purified acid oil yield also increased. The above data indicated that the yield of purified acid oil at a pH value of 7 was the lowest. And the increase of purified acid oil yield from pH 5 to 2 was quite insignificant, although stable. Inflection point of titration curve was around pH 5. Thus, a pH value of 5 could be considered the stoichiometric point of the reaction and was optimal in terms of acid quantity, representing the minimum required.

Few experiments using 50 % sulphuric acid instead of concentrated sulphuric acid were performed and it was concluded that the concentration of sulphuric acid (concentrated or 50 %) was irrelevant from the point of view of the extract yield. But the preparation of 50 % sulphuric acid by dilution was an additional operation that involved safety risks due to the heat of dissolution of water and sulphuric acid [39]. The solution must be cooled or the acid must be added slowly, allowing the solution to cool when preparing solutions with the concentration higher than 35 %. The total volume was another factor: the volume of 50 % sulphuric acid required for acidification was approximately 60 % larger compared to the volume of concentrated sulphuric acid, which did not result in a significant increase in the total volume of acidulated soapstock. These findings suggested that commercially available sulphuric acid solutions of varying concentrations may be used with comparable effectiveness to concentrated sulphuric acid, provided that their use offers practical advantages such as improved safety, cost-efficiency, or convenient handling. Strong oxidizing properties speak against use of concentrated sulphuric acid [40], although influence was not observed in laboratory scale experiments, but caution should be maintained upon scale-up in industry. Concentrated 98 % sulphuric acid was used in further studies.

The degumming and neutralization processes are usually carried out at elevated temperature: crude oil is heated to 60–65 °C, but water-degummed oil to 85–90 °C [41]. The soapstock is hot just after production, thus it is important to evaluate the need of cooling prior to acidulation or need of heating in case soapstock cools upon storage. To study the effect of the soapstock temperature on the process of obtaining purified acid oil, experiments at 9 sets of process parameters were conducted with three different temperatures (20, 45, 70 °C) and pH values (2, 3, 5) using concentrated sulphuric acid for acidulation. The experimental data are shown in Fig. 4.

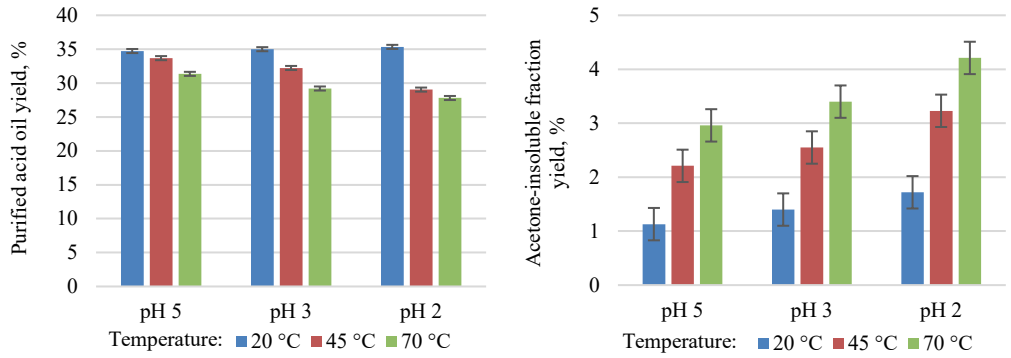

Fig. 4. The purified acid oil (left) and acetone-insoluble fraction (right) yield depending on pH and temperature as parameters using concentrated sulphuric acid for acidulation of rapeseed soapstock.

The yield of purified acid oil decreased with increasing initial soapstock temperature and decreasing acidulation pH value. Conversely, the yield of acetone-insoluble fraction increased under the same conditions. The yield of purified acid oil generally mirrored the trend of crude acid oil yield, although the variations were less pronounced. At elevated initial soapstock temperatures, acetone-insoluble fraction was contaminated with oily residues, even though precipitation with acetone occurred under identical conditions in all experiments. Decrease in purified acid oil and crude acid oil yields as well as increase in phospholipid oiliness with increasing temperature are unfavourable. Hence, it could be concluded, that most optimal temperature for extraction of acid oil is around room temperature of about 20 °C. This set of experiments further proved pH 5 as optimal for acidulation because purified acid oil yield was high at all temperatures. These optimized parameters allow efficient recovery of lipid-rich fractions from biomass residues, minimizing chemical consumption and enabling integration into biodiesel production processes.

Acid oil composition did not vary significantly regardless of sulphuric acid concentration in pH range 2–5 for acidulation at 20 °C temperature. The average composition of purified acid oil was 10.7 wt% FFA, 3.8 wt% monoglycerides, 1.8 wt% diglycerides and 83.4 wt% triglycerides. At low temperatures, the acetone-insoluble fraction of lipids from vegetable oil consists mainly of phospholipids [32]. Our separated acetone-insoluble fraction was found to contain mostly phospholipids as shown by FTIR spectrum (Fig. 5). Strong absorption bands of phosphate groups were found at 1200–970 cm^{-1} of separated phospholipids corresponding to known characteristic of pure phospholipids [42], [43], but bands were overlapped in our sample because it contained a mixture of natural phospholipids.

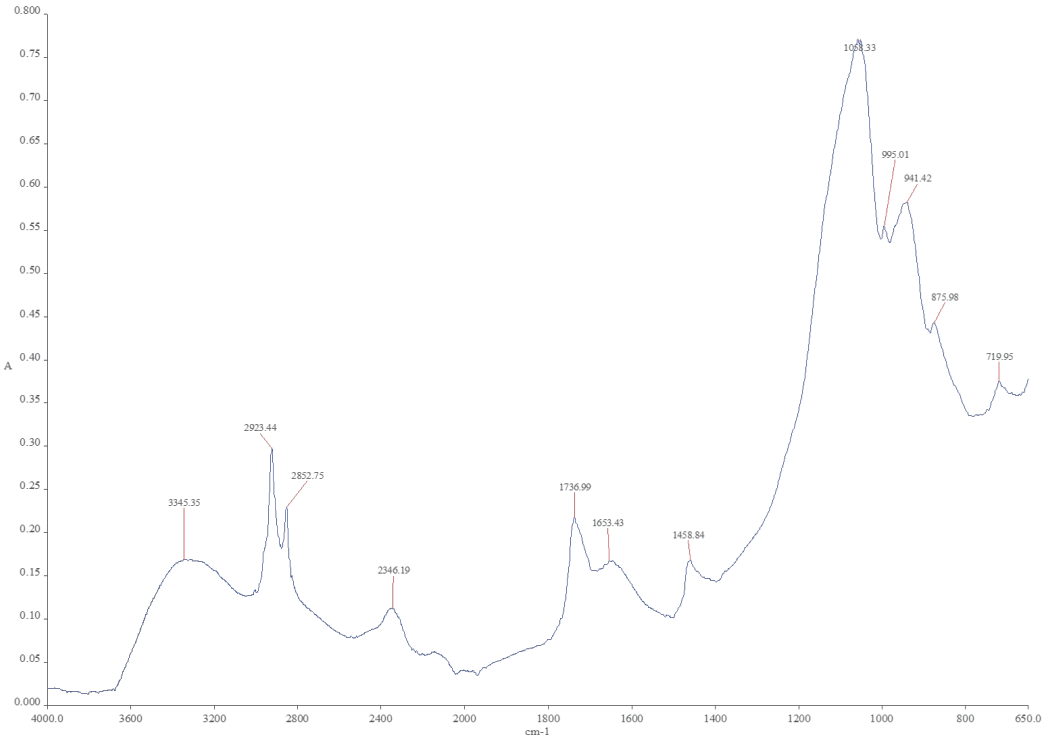

Fig. 5. FTIR absorption spectrum of acetone insoluble fraction from acid oil derived from rapeseed soapstock.

4. CONCLUSIONS

Acidulation process of rapeseed soapstock, that contains 56.4 % moisture and volatile compounds and 37.9 % total fat and is obtained from degumming and neutralization steps of rapeseed oil refining, was thoroughly investigated. A conclusion was made that the most optimal parameters for obtaining the highest yield of purified acid oil is the use of sulphuric acid in an amount sufficient to achieve a pH of 5 at temperature around 20 °C. Crude acid oil was extracted with cyclohexane in ultrasound environment and treated with cold acetone to obtain a high yield of 34.7 % of purified acid oil and separated acetone-insoluble fraction, that is mostly composed of phospholipids. The extraction and purification solvents should be regenerated and used repeatedly employing circular economy principles. Use of citric acid and higher temperatures resulted in lower yields. The acidulation pH values lower than pH 5 were not beneficial, because slight increase of purified acid oil yield may not balance use of higher amount of sulphuric acid and required treatment of acidic wastewater. The developed method contributes to the sustainable valorisation of rapeseed refining residues as a renewable biomass resource, supporting the principles of circular bioeconomy and leading to greener manufacturing. Acid oil, that is obtained using optimized method parameters, could be further used for biodiesel synthesis in a two-step procedure using esterification and transesterification reactions.

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