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Effects of Temperature and Pressure on Hemp Oil Filtration Parameters and Peroxide Number

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Abstract: This article focuses on the quality of hemp oil processing, specifically the filtration that is an important part of the technological procedure of processing vegetable oils. The aim of the research was to determine the effects of pressure and temperature on the filtration parameters when using plate filters. The research was carried out on an experimental measuring device with adjustable static pressure. The qualitative properties of the oil were observed in terms of analytical composition, microbial content, and changes in peroxide value as the indicator of oxidation stability. The change in pressure affected the oil flow rate, especially at lower pressure values. The increase in temperature of the filtered oil had a negative impact on the oxidation stability.

Keywords: vegetable oils; oxidation stability of oils; oil storage; hemp oil; oil filtration; oil treatment; vegetable oil processing; plate filters; oil viscosity

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Citation: Souček, J.; Jevič, P.; Dědina, M.; Tománková, V.; Mašán, V.; Burg, P.; Szalay, K.; Jasinskas, A. Effects of Temperature and Pressure on Hemp Oil Filtration Parameters and Peroxide Number. *Processes* **2023**, *11*, 2281. https://doi.org/10.3390/pr11082281

Academic Editor: David Herak

Received: 22 June 2023
Revised: 20 July 2023
Accepted: 27 July 2023
Published: 28 July 2023

1. Introduction

This paper is an extended version of a paper published at the 8th International Conference Trends in Agricultural Engineering 2022, Prague, Czech Republic, 20–23 September 2022 [1]. Hemp is a plant with a wide variety of applications. According to many authors, it can be used as a technical or energy resource [2,3]. However, the most valuable use of hemp seed is currently considered to be for the food industry. It is most often used hulled, or pressed in the form of oil and press cake. It has a high dietetic value, which is retained even after the process of pressing, which means that the oils and press cakes contain a significant amount of unsaturated fatty acids. However, this dietetic advantage also has its drawbacks in the form of low oxidation stability of the final products [4]. For ensuring a high-quality oil, the most important parts of the process are storage, pressing, and filtration. The parameters of these have a significant effect on the composition and quality of the oil.

The subject of oil filtration has been addressed by a number of authors. The effect of filtration on the oil quality and storage is described by [5], who state that the filtration technology can improve the oxidation stability of the oil with positive effects on its nutritional quality and shelf life. The effect of filtration on reducing chlorophyll, carotenoids, tocopherols, and polyphenols has been reported. A collective of authors [6] carried out research on the process of filtration of sunflower oil using cellulose filters. They determined that the cellulose filters have a relatively high efficiency in filtering certain components of the pressed oil (99.20% of waxes, 74.88% of phospholipids, 100% of soap, 7.99% of carotenoids, 16.39% of Fe, and 18.33% of Cu). The authors [7] showed that filtration efficiency can be increased by introducing additives. In their research, they used licitin as the additive in the filtration process of linseed oil in order to study the effect of phospholipids on oil filtration at the temperature of 20 °C. The procedure was effective only at lower concentrations. The addition of more than 2% of lecithin reduced the filtration rate. At 20 °C, the lecithin precipitated on the particles and caused a sedimentation to such an extent that the filter cake became impenetrable to the oil. This was partially overcome by increasing the temperature of the filtered oil (up to 50 °C). The subject of filtration in terms of phospholipid content was also addressed by [8], who filtered soybean and sunflower oil using an SP015A membrane at a temperature of 40–60 °C, a pressure of 200–500 kPa, and a flow rate of 0.3–0.4 L/h. The phospholipid filtration efficiency was 70–77%. By adding water to the oil, the efficiency was increased to 90%.

Other undesirable components of vegetable oils that can be removed by plate filters are various types of mechanical impurities of organic and inorganic origin. Oil and water can be separated by filtration. Mainly membrane filters are used for this purpose [9].

The subject of comparing different methods of oil filtration has been addressed by [10]. They compared three oil filtration systems. The results showed that depth filtration was more effective in removing lipid oxidation products than paper membrane plate filters and filters with paper inserts combined with silica. The levels of free fatty acids were not significantly affected by the filtration methods used, but the disadvantage of the methods compared is the need to heat the filtered oil.

In practice, however, plate filters are the most commonly used for the filtration of vegetable oils. Their advantage lies in the simplicity of their design. The parameters of filtration can be easily changed by the number of plates, membrane type, and the size of the holes. The pressure of the filtered fluid can be adjusted within the operating mode of the filter. Plate filters can operate in a wide temperature range if the appropriate construction materials are used. They can be used to filter many types of liquids and are widely used in food processing, industry, and various waste management and environmental protection technologies.

The disadvantage of plate filters is that they clog and require frequent maintenance. Most of the membranes have a limited lifespan, which increases the cost of running the filters, as they need to be purchased and replaced regularly. From an operational point of view, it is usually necessary to stop the production for filter maintenance, which involves stopping and then restarting the production.

In the field of hemp applications, research has been carried out on oil and air filtration using hemp paper membranes. Through research and experimentation, the authors [11] were able to make a hemp bast paper suitable for practical applications. They reported that both the degree of grinding and the weight have a great influence on the membrane permeability, pore size, flow rate, and the filtration efficiency, which, for the hemp paper filter, was 99.75–99.975% for 0.33 μm particles when filtering oil, and 99.942–100% for 0.26 μm NaCl aerosol particles when filtering air. Compared to standard materials, the hemp paper has lower thickness, weight, medium pore diameter, porosity, better efficiency, and higher pressure drop. The advantage of hemp paper membranes is their environmental friendliness, as they are made from natural materials and are easily biodegradable. According to [12,13], hemp seed oil is valued primarily for its nutritional values, which are linked to beneficial health effects. Hemp seed contains up to 35% of fatty acids, a high amount of protein (25%), carbohydrates (30%), and fibre (10%). Hemp oil is rich mainly in linoleic and linolenic acids in a ratio suitable for nutrition, with the latter one contributing to the high nutritional values. Hemp oil is also widely used in medicine and cosmetics, because of the linolenic acid having a positive effect on the treatment of rheumatoid arthritis, atopic eczema, and allergies, and has anti-inflammatory, anti-hypertensive, anti-vasoconstrictive, and anti-cancer effects [14]. Due to its good absorption properties, hemp oil is used for the preparation of body creams. The tocopherols it contains are also used to lower the risk of cardiovascular diseases and changes associated with aging [15,16].

Under the right conditions, hemp seed is relatively easy to store; the most important storage atmosphere parameters are temperature and humidity. It is advised to regularly aerate the stored seed and to prevent any damage during handling.

Hemp oil is usually produced by cold pressing. To increase the yield, standard industrial procedures can be used; however, these negatively affect the quality of the oil [17]. Pressing is most often carried out on screw presses, with the outlet temperature of the oil not exceeding 50 °C. The Food Codex defines cold-pressed oils as oils obtained by only mechanical means, such as extrusion or pressing. They can be purified only by washing with water, settling, filtering, or centrifugation [12]. According to [18], cold pressing can extract up to 65% of the oil in the seeds, while the rest (35%) remains in the sludge. The cold-pressing process also extracts minor compounds naturally present in hemp, i.e., antioxidants, such as phenols and tocopherols [19]. The evaluation of the total phenolic and tocopherol content can be useful in assessing storage quality [20]. Another compound that can also be found in hemp oil is chlorophyll, which is a photosensitive pigment that changes the colour of the oil to green and promotes lipid oxidation [17,18], which can affect the quality of the oil during storage. The presence of chlorophyll and the high content of polyunsaturated fatty acids causes the hemp oil to be susceptible to oxidation [21].

In the literature [22], oxidation stability is most often defined by the peroxide value and acidity number. To reduce the oxidation process in hemp oil, one can use, for example, a rosemary extract, which was found to promote oxidation stability [23]. According to some authors [24], the stages associated with the processing of oil, such as filtration or bottling, highly affect the oxidation behaviour of hemp oil.

In the case of vegetable oils, in addition to the filtration technology used, the variety from which the oil was extracted also plays an important role in the stability of the oil. For example, the authors of [25] have shown that the stability of olive oil differs between oils stored at room temperature (25 °C) and oils stored at elevated temperatures (40 °C). Single-variety olive oils from the Arbequina, Colombaia, Cornicabra, Picual, and Taggiasca varieties were studied. All of them were stored in the variants a—untreated, b—filtered, and c—dehydrated. The results showed that filtration and dehydration reduced the rate of hydrolysis of the triacylglycerol matrix, especially at higher temperatures, and delayed the appearance of rancidity defects in oils with higher initial free acidity. The formation of simple phenols due to the rate of the hydrolysis of their secoiridoid derivatives was also higher in the unfiltered oils. As a result of the research, it was concluded that the stability of oils of different varieties differs under the same storage conditions. Filtration and especially dehydration could help to extend the shelf life of high-quality and less stable oils.

Virgin hemp oil has a high content of unsaturated fatty acids and, if all technological processes are carried out accordingly, retains all nutritional values while not containing the psychoactive substance THC.

The aim of this research was to verify and test the influence of the filtration conditions on the operational parameters and oxidation stability of the oil, and, hence, contribute to improving the overall quality of vegetable oil processing.

2. Materials and Methods

Unfiltered hemp oil of the Bialobrzeskie variety grown in the Částrov municipality (Vysočina region, the Czech Republic) was used in the experiment. The harvested seed was treated post-harvest and stored for 30 days. Subsequently, it was pressed on the Farmet Duo (manufacturer Farmet, a.s.) low-tonnage screw press with the oil temperature on the outlet being 43 ± 3.5 °C. The pressed oil was stored for 4 days at a temperature of 8 ± 1.2 °C, during which gravity separation of the solid part (sludge) and liquid part (oil) took place. As part of the elemental composition analysis, these two parts were separated, analysed, and then subjected to experimental filtration.

For the determination of the filter curves of the tested specimens, the experimental equipment of VÚZT (Research Institute of Agricultural Engineering, p. r. i., Czech Republic) was used, the basis of which is the FARMET plate filter (FARMET a.s., Czech Republic). The experimental setup allowed the setting of operation parameters and online monitoring of physical quantities at nodal points. To ensure uniform flow, the device was equipped with a system of adjustable static oil pressure in the range of 0–300 kPa (limited by the filter design).

The experiments were carried out with Riftelen Oil N15 filter cartridges, manufactured by Pardam nanotechnology. The pilot filtration tests were carried out with N15-8, N15-25, and N15-50 membranes. They showed that the N15-25 membrane with a hole size of 25 μm is best suited for hemp oil filtration. When the N15-8 membrane was used, the filter clogged quickly, and the oil flow rate was very low. When the N15-50 membrane was used, it took a long time for the filter cake to form, and the filtered oil contained a greater amount of contaminants.

During the experiment, the oil was forced through a membrane to the plate filter by static pressure. The pressure was set to the desired value before the beginning of the experiment. For the filter curve measurement, a sample of 15 kg of oil was prepared. Control temperature measurements and logging were carried out during the preparation of the sample. Immediately after the preparation, the sample was gravity fed into the inlet tank and, after settling, transferred to the pressurised storage tank. After the desired pressure value was reached, the air in the tank was removed and the recording of values started, which could be monitored in real time on a PC connected to the measuring station. The sample was considered filtered when the measured oil flow was zero for 30 s.

The following quantities were measured during the experiment:

- Flow velocity—measured by mass using a KERN FTC 60K2 strain gauge (KERN&SOHN, Balingen, Germany) with a measuring range of 60 $\pm$ 0.002 kg and a digital online output. The oil flow rate was determined by the increase in weight, measured using the aforementioned KERN FTS 60K2 scale;
- Oil pressure before the filter—measured using a Greisinger DMP 331 110 pressure transmitter (GHM Messtechnik GmbH, Remscheid, Germany) with a measuring range is 0–4 MPa, accuracy 14 kPa;
- Oil temperature—measured with sheathed thermocouples of type K $\times$ 0.25 mm with a time constant of *1 s. The oil temperature was measured in the inlet tank, at the filter inlet and filter outlet, and in the outlet tank;
- Relative humidity and ambient air temperature—measured by a Testo 6651 temperature and humidity transmitter (Testo SE & Co. KGaA, Titisee-Neustadt, Germany) with an accuracy of 0.5 °C, $\pm$2.5% relative humidity. The relative humidity was only an indicative figure to characterise the measurement conditions. It ranged from 60 to 85% and had no effect on the results.

All the listed instrumentation was connected to the COMET MS6D (Comet AG, Wünnewil-Flamatt, Switzerland) measuring and recording control unit, which allows for online data logging with a predetermined interval to a connected PC, or in a recording mode with a subsequent data export to a PC. The data are stored in standard MS Excel file format.

The agrochemical laboratory of the VÚZT determined the following properties of the measured samples, which are important for further use:

- Analytical composition according to [26,27];
- Microbiological analyses of the total microbial count, mould and yeast content [28–31];
- Oxidation stability of the oil expressed as a value of a peroxide number [32]. The value was determined for the stored oil before the experiment and after the filtration process. The increase in peroxide value was determined as the difference between the values at the inlet and outlet of the filtration equipment. Samples with a higher inlet peroxide number value (>10 meq O_2/kg) were excluded from the evaluation as the effect of filtration on the change in the value is not very conclusive;

- The measurement uncertainties were determined according to the CIA European Cooperation for Accreditation EA4/02 document. The measuring equipment meets the requirements of [33].

The measured data were processed in MS Excel and statistically evaluated in STATISTICA 13 software.

3. Results

All the data and results obtained according to the described procedures were processed and statistically evaluated.

3.1. Composition of the Oil and Sludge

During storage, prior to the measurement, the oil and the sludge were separated by gravity. Both parts were subjected to analytical analyses. The results of the analyses are given in Table 1. Quantities are given in weight percent, based on the total weight.

Table 1. Composition of hemp oil and sludge according to analyses.

Parameters	Oil	Sludge
amount of carbon, %	68.07 ± 1.12	77.86 ± 1.14
amount of hydrogen, %	10.15 ± 0.45	11.31 ± 0.42
amount of sulphur, %	0.26 ± 0.001	0.08 ± 0.001
amount of nitrogen, %	1.82 ± 0.1	0.05 ± 0.001
amount of chlorine, %	0.069 ± 0.004	26 ± 0.004
amount of silicon %	0.80 ± 0.003	0.24 ± 0.004
amount of calcium, %	0.253 ± 0.025	<0.07
amount of magnesium, %	<0.05	<0.04
amount of potassium, %	0.814 ± 0.016	0.07 ± 0.020
amount of phosphorus, %	0.650 ± 0.004	0.005 ± 0.002
amount of zinc, mg/kg	0.002 ± 0.75	0.002 ± 0.75
amount of cadmium, mg/kg	<0.2	<0.2
amount of lead, mg/kg	<0.002	<0.002
amount of chromium, mg/kg	<0.002	<0.002
amount of copper, mg/kg	<0.001	<0.001

Table 1 clearly shows that the composition of oil and sludge differed in several parameters. The oil had a significantly higher content of nitrogen, sulphur, silicon, and nutrients (calcium, magnesium, potassium, and phosphorus). On the contrary, the sludge had a higher content of chlorine. The levels of heavy metals in both the oil and the sludge were below the detection limits of the instrumentation.

3.2. Microbiological Analyses

Microbiological analyses were carried out on the filtered oil to determine the total number of microorganisms, yeasts, and moulds, the content of *Escherichia coli*, and the content of intestinal enterococci. The results are shown in Table 2.

Table 2. Results of microbiological analyses of oil.

Parameters	Oil	Sludge
total number of microorganisms, CFU/mL	4.9×10^2	8.3×10^6
yeast and mould count, CFU/mL	<10	2.5×10^4
Escherichia coli, CFU/mL	0	0
intestinal enterococci, CFU/mL	0	0

From the results in Table 2, it can be concluded that the microbiological contamination of the oil and the sludge was different. For the sludge, the total number of microorganisms and the number of yeasts and moulds were one order of magnitude higher than for the oil. Neither *Escherichia coli* nor intestinal enterococci were detected in either sample.

3.3. Pressure and Flow of Oil during Filtration

The oil filtration experiments were carried out at oil temperatures between 15 and 20 °C. The sample temperature increased slightly during the filtration due to the increase in pressure.

The pressure range during the filtration was set between 30 and 250 kPa. The system pressure was maintained at the set value using a reduction valve with an accuracy of ±10 kPa.

Figures 1 and 2 show the filtration curves at the pressure values 200 and 250 kPa, respectively.

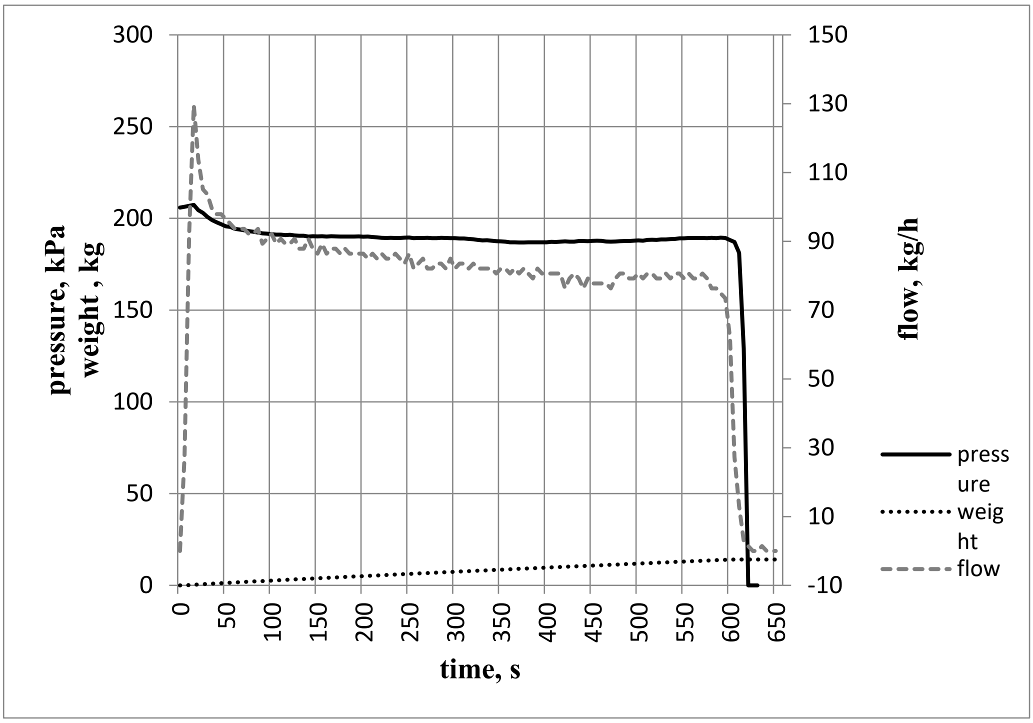

Figure 1. Graph of oil pressure, flow through the plate filter, and the weight of the filtered oil for the experiment-200 kPa-filter N15-25.

From the graphs in Figures 1 and 2, it is clear that the highest flow rate through the filter occurred at the beginning of the measurement, and then it slowly decreased due to the fouling of the filter plates by sludge.

In the same way as in the above examples, the filtration parameters were determined for other pressure values in the range of 0–250 kPa. The dependence of oil flow through the filter on the pressure can be seen in Figure 3.

From Figure 3, it can clearly be seen that the change in pressure at a lower level (up to about 100 kPa) has a greater effect on the value of the flow than at higher levels. For values above approximately 150 kPa, the change in pressure is not so significant. The slight variations are caused by the differences in the temperature of the filtered oil and the degree of contamination of the filter (pressure drop).

Another measured parameter was the oil temperature and in the monitored range of 15–25 °C, it had no significant impact on the filtration rate.

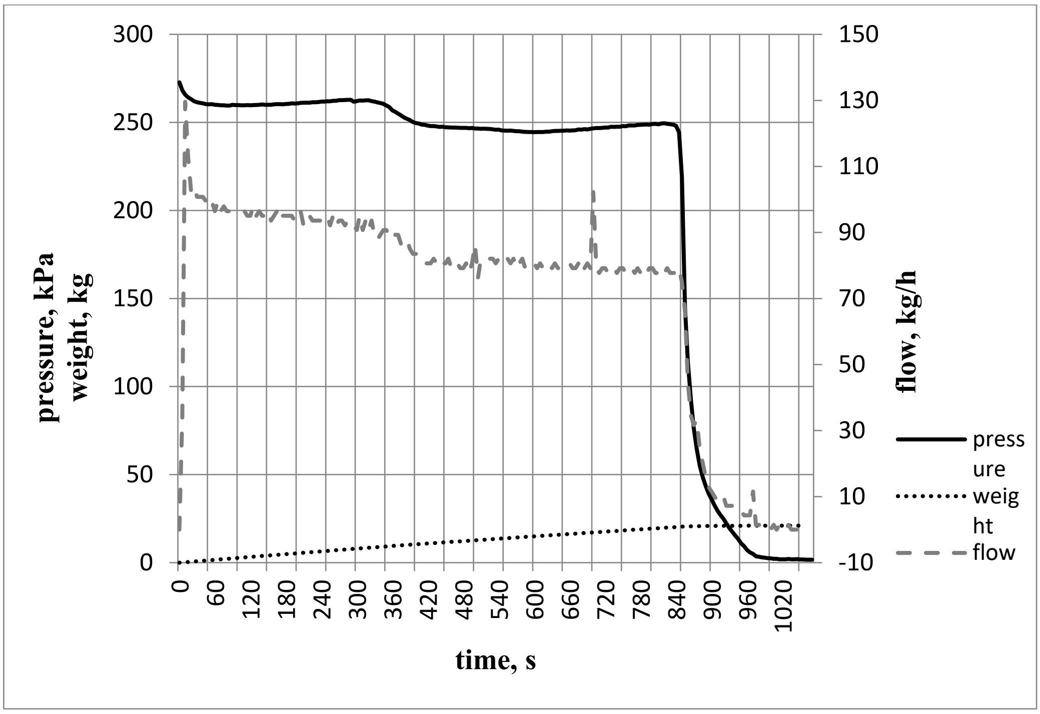

Figure 2. Graph of oil pressure, flow through the plate filter, and the weight of the filtered oil for the experiment-250 kPa-filter N15-25.

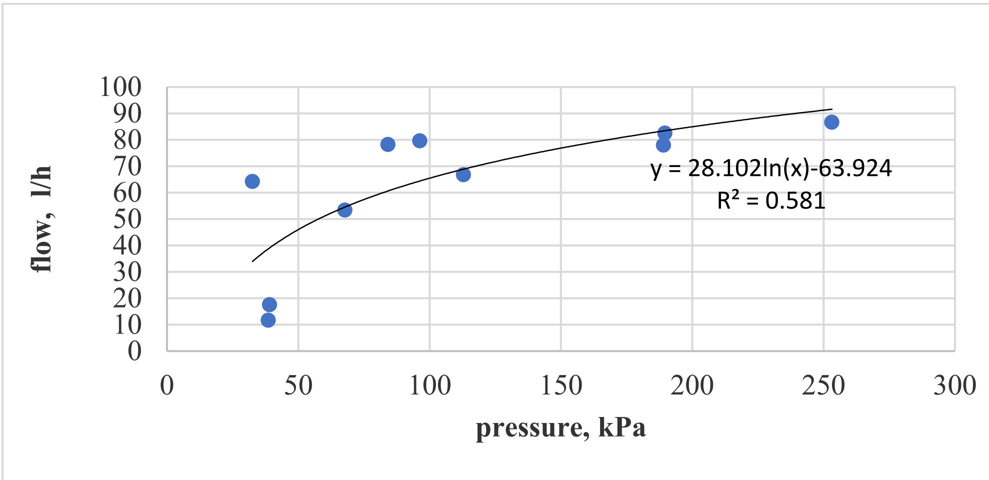

Figure 3. The dependence of oil flow through the filter on the pressure—filter N15-25.

3.4. Effect of Filtration on Peroxide Value

Figure 4 shows the values of the peroxide number for various levels of oil pressure during the filtration, while Figure 5 shows the values of the peroxide number in dependence on the temperature during the filtration.

From the statistical evaluation and the graphical representation of the results in Figure 4, it is clear that the oil pressure during the filtration does not have, in the observed range, a significant impact on the peroxide number.

On the contrary, Figure 5 shows that the temperature of the filtered oil has a noticeable effect on the peroxide number, even in the relatively narrow monitored range of 17–22 °C. For this reason, the filtration methods described, for example, by [5,10], which require heating of the oil, appear to be inappropriate.

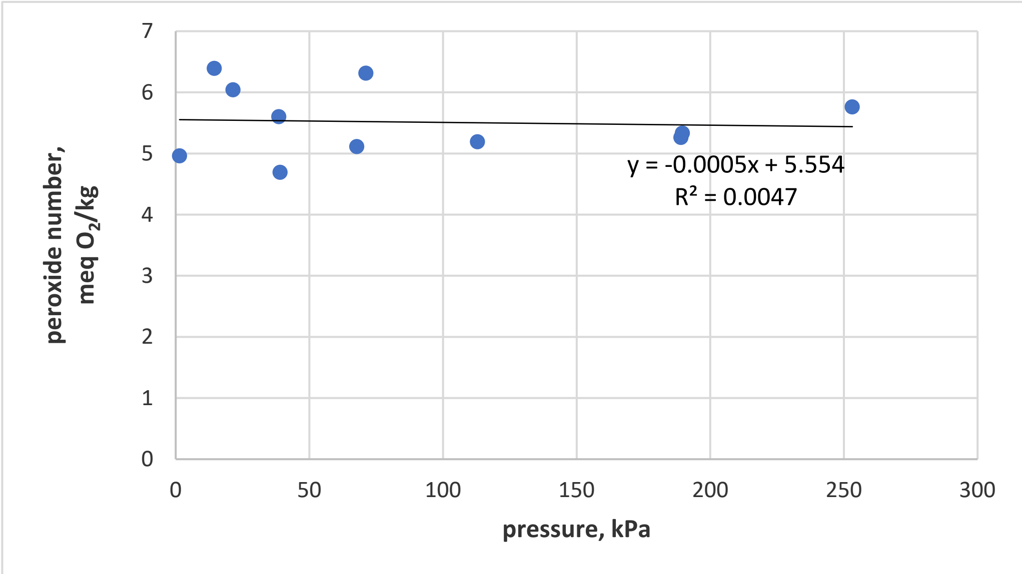

Figure 4. Dependence of the peroxide number increase on the oil pressure during filtration—filter N15-25.

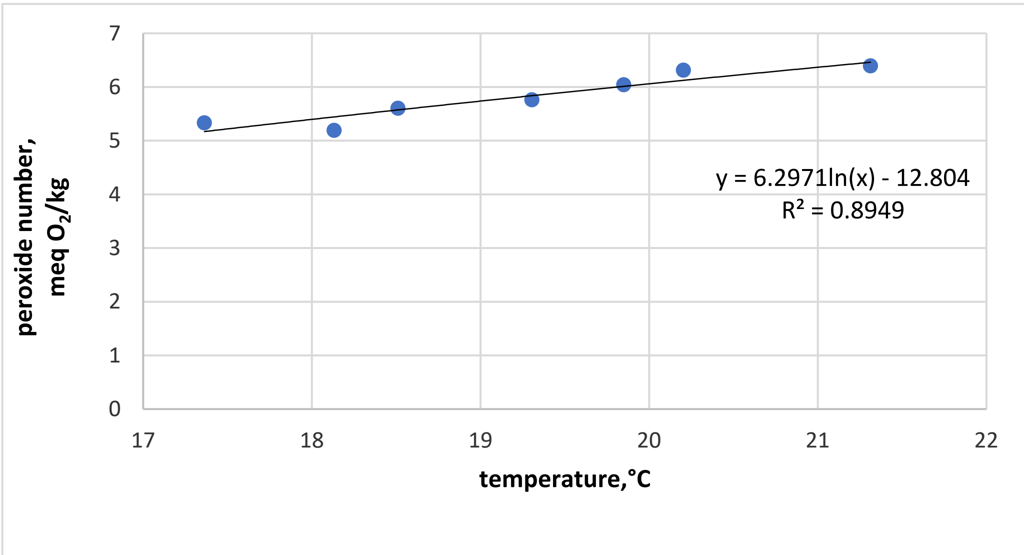

Figure 5. Dependence of the peroxide number increase on the temperature during filtration—filter N15-25.

Within the framework of the experiment, the values characterising the oil filtration process in terms of process parameters and properties of the filtered oil were measured and evaluated. The results of the analyses of the oil and sludge confirmed the initial assumptions and the findings of other authors [12,13] that hemp oil contains many beneficial constituents. Nutritionally valuable substances (nitrogen, calcium, magnesium, potassium, phosphorus) were more present in the oil, while a higher content of chlorine could be found in the sludge, which also contained a higher number of microorganisms. The analyses showed no heavy metal content for either the oil or sludge, which, indirectly, confirmed the conclusions of authors [15,16] claiming that hemp oil can be used as a suitable ingredient in creams and ointments.

Also confirmed was the assumption that with increasing pressure, the oil flow through the filter also increases. However, the dependence is not linear, with the highest increase in oil flow being measured at lower pressure levels, and for higher levels, above approximately

150 kPa, the pressure had little effect on the flow. The effect of the pressure level on the values of the peroxide number was not statistically significant.

However, temperature was found to have a significant effect on the peroxide number, even in the relatively narrow range observed. This finding confirmed the conclusions of [4,21] that hemp oil, and vegetable oils in general, are highly oxidatively unstable and suffer from the deterioration of their properties during processing [24]. In agreement with [22], it was confirmed that determining the peroxide number is a relatively easy method for the basic indication of the oxidation stability of vegetable oils.

4. Conclusions

This research has produced important findings, some of which have implications for operational practice, and has identified a number of issues that should be addressed in further research. One important finding is that, above a certain threshold, increasing the pressure during filtration does not have a significant effect on the filtration rate, and, therefore, on the efficiency of the process. It can even lead to an unwanted increase in temperature or damage to the plate filter.

Another parameter measured was the oil temperature, which had no effect on the filtration rate within the narrow range observed. However, it is logical to assume that at higher temperatures, the viscosity of the oil will change significantly, and the effect on the filtration rate will be more pronounced. This will be the subject of future research.

Temperature has a significant effect on the oxidation stability of the oil, which is one of the quality indicators. From the results obtained, it is clear that even in the relatively narrow range of measured temperatures, the oil temperature at the outlet of the filter had a positive effect on the increase in peroxide number value. In practice, this means that it is not advisable to underestimate the temperature increase, even if it is in the order of magnitude of degrees °C. In order to preserve the valuable properties of the oil, its oxidation stability, and shelf life, a temperature increase during the filtration is not recommended. Some filtration methods which appear to be effective in terms of performance and efficiency in removing undesirable components may therefore not be suitable. This means that in practice, it will be necessary to find the compromise between the desired effectiveness of the processing technology and maintaining the required quality.

Vegetable oils are an important natural resource, which can be used in a number of ways, especially as essential components or supplements in nutrition.

Within the framework of the research, the operating parameters of oil filtration and their effects on the stability of hemp oil, represented by the values of the peroxide number, were studied. When filtering with a plate filter, it is advisable not to increase the oil temperature excessively in order to maintain the oil pressure before the filter at approximately 150 kPa.

If the products obtained, including the oil, are well processed, oilseeds, including hemp, are very useful crops in terms of their usability in nutrition, cosmetics, medicine, and other areas of human life. Their great advantage is their environmental friendliness, both in terms of saving non-renewable material resources and in terms of agriculture, as part of a more diversified cultivation practice with a positive impact on agricultural land.

Author Contributions: Conceptualisation, J.S., P.J. and M.D.; methodology, J.S.; software, J.S.; validation, V.T.; data curation, P.B.; writing—original draft preparation, J.S.; writing—review and editing, J.S.; visualisation, V.M.; supervision, K.S. and A.J.; project administration, J.S. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the National Agency for Agricultural Research of the Ministry of Agriculture of the Czech Republic, grant number QK21010151, and the project of long-time development of the Research Institute of Agricultural Engineering, p.r.i. no. RO0623.

Data Availability Statement: Not applicable.

Acknowledgments: The authors thank Farmet, a.s., Czech Republic for support in the design of the experimental equipment.

Conflicts of Interest: The authors declare no conflict of interest.

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