TRANSPORT
ISSN 1648-4142 print / ISSN 1648-3480 online
2012 Volume 27(1): 60–66
doi:10.3846/16484142.2012.664827

POSSIBILITIES OF USING *CAMELINA SATIVA* OIL FOR PRODUCING BIODIESEL FUEL

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Submitted 5 April 2011; accepted 11 May 2011

Abstract. Biofuels for diesel engines are produced mainly from rapeseed oil in Lithuania and the Member States of the European Union. In order to minimise an adverse impact of biodiesel fuel on the food sector, it is necessary to look for alternative feedstocks for producing biodiesel fuel including the potential utilisation of the new kinds of oilseed crops and various fatty waste. Camelina (*Camelina sativa*) could be one of the kinds of such oilseed crops, and therefore the physical and chemical parameters of *Camelina sativa* oil and biodiesel fuel produced from this oil were determined and the conformity of quality parameters with the requirements of biofuel standard was evaluated. It was found that fatty acid methyl esters made from *Camelina sativa* oil had a high iodine value (164.6÷169.6 g I_2 / 100 g oil), and therefore could be used as fuel for diesel engines only in the mixtures with methyl esters produced from animal fat or used for frying oil. It has been established that similar mixtures can contain 50÷60% of *Camelina sativa* oil methyl esters. The possibilities of increasing oxidation stability as well as improving the cold flow properties of ester mixtures were investigated. The most effective antioxidant – Ionol (optimal dosage of 500 ppm) and the most efficient depressants Wintron XC-30 (optimal dosage – 1500 ppm) and Infineum R-442 (optimal dosage – 1200 ppm) were selected.

Keywords: *Camelina sativa* oil, biodiesel fuel, properties, antioxidant, depressants.

1. Introduction

Biodiesel fuel for diesel engines is produced mainly from rapeseed oil in Lithuania and the Member States of the European Union. With the intensive development of producing biodiesel fuel, demand for rapeseed feedstock production is constantly growing. Both in the EU Member States and Lithuania in particular, the expansion of land areas used for cultivating rape is limited due to crop rotation and the suitability of soil for rape cultivation. Another problem related to producing biodiesel fuel is that its production involves the use of rapeseed oil suitable for food purposes. The fact that an increasingly higher portion of rapeseed oil is directed for producing fuels has resulted in the deficiency of feedstock for the production of edible oil and growing market prices of rapeseed oil. A part of food-purpose rapeseed oil has to be replaced by the oil of other types, most frequently, with soybean oil imported from the USA (Haas *et al.* 2001; Clark *et al.* 1984). The production of biofuels from feedstocks suitable for food purposes causes dissatisfaction among the public and its prejudice against the production and use of biofuels.

In order to minimise an adverse impact of biodiesel fuel on the food sector, it is necessary to look for alternative feedstocks for producing biodiesel fuel including the potential utilisation of the new kinds of oilseed crops and various fatty waste. Camelina (*Camelina sativa*) (Zubr, Matthäus 2002) could be one of the kinds of such oilseed crops. Compared to rapeseed, Camelina is less demanding for the quality of soil, more resistant to unfavourable climatic conditions and can be used as an under-crop in crop rotation (Vollmann *et al.* 2007; Šiaudinis, Lazauskas 2009; Povilaitis 2008).

Because of the high content of unsaturated fatty oils, Camelina-seed oil belongs to the group of fast-drying oils; this is why it is used for producing environmentally friendly polymers, varnishes and paints. Besides, this type of oil is suitable for making some medicines. Like all other vegetable oils, Camelina-seed oil can be used in the production of biodiesel fuel; however, because of the composition of fatty acids (high content of polyunsaturated acids) (Peiretti, Meineri 2007), it is probable that biodiesel fuel will fail to comply with the requirement of Standard EN 14214:2003 in respect of the iodine value. This problem can be resolved by mixing Camelina-seed oil used in the production of biodiesel fuel with other fats or oils having a higher content of saturated fatty acids.

Foreign scientists performed research in the area of utilising Camelina-seed and palm oil in the production of biodiesel fuel (Moser, Vaughn 2010; Fröhlich, Rice 2005); however, searching for other kinds of fatty feedstocks and waste, which may be used in the production of biodiesel fuel alongside with Camelina-seed oil, must be continued. Possible fatty materials for admixture with Camelina-seed oil in biodiesel fuel production could be animal fat used for frying oil.

The use of edible oil and fat, that oxidise when heated, in industrial and household cooking causes the formation of aldehydes. Such oil cannot be used for producing food (Falk *et al.* 2001); however, it can be used for technical purposes.

In the former EU countries, the annual consumption rate of oil ranges from 30 kg (in Austria) to 80 kg (in Holland) per capita. The consumption rate in Lithuania is 15.6 kg. With the application of the collection system, the actual amount of collected and used frying oil in a community would amount to 3 kg per capita. It can be forecasted that from 3 to 4.3 thousand tons of used for frying oil and approximately 1.5 thousand tons of animal fat could be processed in Lithuania into biodiesel fuel. The advantages of using these products include low cost and non-competition with products used for food purposes. However, the quality of used for frying oil varies significantly and is influenced by the presence of solid particles, water, salts, free fatty acids and polymers.

The use of the waste of animal origin, to the utilisation of which the EU pays much attention, is problematic. Several directives and regulations stipulating the general principles and methods of utilizing the waste of various types depending on their origin and risk to the human have been adopted. Regulation (EC) No 1774/2002 provides for the classification of animal waste and procedures for their accumulation, processing, trade and export. Stricter heat treatment requirements are set for dead animal waste that is allowed to be disposed of by direct burning or, after heat treatment according to the methods strictly defined in the Regulation, to be used for other purposes. It would be reasonable to use such waste for the production of biodiesel fuel in the mixtures with Camelina sativa oil. The use of the waste of animal origin as well as that used for frying oil has the potential to increase the profitability of biofuel production and to improve life cycle parameters of biodiesel fuel (Montrimaitė *et al.* 2010).

Before an industrial application of biodiesel fuel production from Camelina sativa oil in the mixtures with other suitable feedstock, it is necessary to determine the optimal composition of the above mentioned mixtures and to evaluate the physical and chemical properties of a new kind of biodiesel fuel as well as their compliance to the requirements of Lithuanian Standard LST EN 14214:2003. Also, it is necessary to look for new ways to improve the physicochemical parameters of the new kinds of biodiesel fuel by using industrially produced additives.

A complex evaluation of fuel quality also includes comparative engine tests and the measurement of the concentration of harmful components in engine emissions.

Some authors investigated the physical and chemical parameters of soybean oil methyl esters, tallow methyl esters or their mixtures (Lebedevas, Vaicekauskas 2006). Moreover, engine emissions when fuelling with mixtures containing camelina sativa oil and pork lard methyl esters in the mixture with fossil diesel fuel (Lebedevas *et al.* 2010) were evaluated.

These results are promising and demonstrate the possibilities of using Camelina sativa oil and methyl esters mixtures with beef tallow methyl esters and fossil diesel fuel in diesel engines. There is no data about the engine tests of the mixtures containing Camelina sativa oil used for frying oil methyl esters, the physical and chemical properties of fuel and the possibilities of improving them applying various industrial additives.

The aim of our research was to study the possibilities of using Camelina-seed oil in the production of biodiesel fuel.

2. Materials and Methods

Camelina sativa of winter variety 'Borowska' and spring variety 'Przybrodzka' was grown in Vėžaičiai Branch of the Lithuanian Institute of Agriculture in 2009–2010.

Oil was pressed out from seed by means of a mechanical screw press equipped with a heating element (Varmec RCV 252) while heating seed to a temperature of 150°C. The quality indicators of the obtained oil were studied in accordance with the methodology specified in Lithuanian Standard LST 1959:2005. Pork lard received from the trading network and used for frying oil received from the public catering network was used for taking tests.

For esterification tests, oil with acidity exceeding 2% was poured into a thermostatically controlled three-neck flask with an addition of methanol (18%) and catalyst (1.5%) – concentrated sulphuric acid (H_2SO_4). A reflux condenser, a thermometer and a pressurised mixer were fitted on the flask. The mixture was homogenised while maintaining constant temperature and rotation speed. After the reaction, having separated the fatty phase (triglycerides and fatty acid methyl esters) and having removed methanol residues by distillation, the sample was treated with barium carbonate ($BaCO_3$) and left for settling for 10 minutes, which was followed by decanting the fatty phase. The acidity of the fatty phase was tested applying the titrimetric method according to Lithuanian Standard LST EN ISO 660:2000. The relation between the content of free fatty acids in the resulting product and the content of free fatty acids in the raw material demonstrate the efficiency of the esterification process.

The usual biodiesel fuel production method, i.e. transesterification with methanol using sodium hydroxide, was applied for the transesterification of Camelina-seed oil used for frying oil and fat. The quality indicators of fatty acid methyl esters were determined in accordance with the methodology set forth in Lithuanian Standard LST EN 14214:2003.

Depressants Wintron XC-30 (Biofuel Systems Group Limited, UK) and Infineum R-442 (Infineum GmbH, UK) were used for reducing the cold filter plugging point of biodiesel fuel (fatty acid methyl esters). Oxidation stability was enhanced by applying the antioxidant Ionol (JSC Pemco Chemicals).

3. Results and Discussion

Taking into account the fact that the efficiency of producing biodiesel fuel and the quality of the resulting product are significantly influenced by the quality of fat and oil used for its production, the physical and chemical characteristics of the oil obtained from Camelina-seed were evaluated. For comparison purposes, the quality of pork lard, oil used for frying as well as winter and spring rapeseed oil were analysed (Table 1).

The results of the studies show that the content of polyunsaturated fatty acids in Camelina-seed oil is considerably higher than that of rapeseed oil, that of animal fat and that of used for frying oil. Camelina-seed oil has an especially high content of polyunsaturated – linolenic acid which reaches as much as 38.2% in winter variety Camelina-seed oil and 34.3% in spring variety Camelina-seed oil.

Vollmann *et al.* (2007) reported that Camelina-seed oil was 90% composed by unsaturated fatty acid, including 25÷42% of alpha-linolenic acid (18:3), 13÷21% of linoleic acid (18:2), 14÷20% of oleic acid (18:1), 12÷18% of eicosenoic acid (20:1) and 2÷4% of erucic acid (22:1).

According to the requirements of Lithuanian Standard LST EN 14214:2003, the content of linolenic acid in biodiesel fuel should not exceed 12%. Therefore, it can be stated that biodiesel fuel produced from Camelina-seed oil will fail to meet the requirements of the standard in regard to the aforementioned parameter. A high content of linolenic acid also predetermines a high iodine value of Camelina-seed oil and its lower oxidation stability compared to rapeseed oil.

There is no doubt that biodiesel fuel produced from spring or winter variety Camelina-seed oil will fail to comply with the requirements of Lithuanian Standard LST EN 14214:2003 in respect of the iodine value that should not exceed 120 g I_2/100 g of fat or oil, since the iodine value of Camelina-seed oil, which is the subject of our studies, showed the value of 164.6÷169.6 g I_2/100 g of oil.

This characteristic as well as the content of linolenic acid may be adjusted by preparing the mixture of Camelina-seed oil with fat or oil with a higher content of saturated fatty acids. It is known that animal fat possess-

Table 1. The physical and chemical characteristics of oils and fats

Parameter	Spring variety Camelina oil	Winter variety Camelina oil	Rapeseed oil	Used for frying oil	Pork lard
Density (15°C), kg/m^3	925	926	917	904	–
Viscosity (40°C), Pa·s	0.03	0.029	0.014	0.04	–
Acid value % (m/m)	0.26	0.76	0.52	6.4	0.6
Iodine value g I_2/100 g	164.6	169.6	98	60.3	63
Oxidation stability, h	8.23	7.7	9.5	3.4	2.3
FAME composition %					
Saturated:	15.3	15.1	4.7	38.2	53
C14:0	0.5	0.4	n. d.	1.1	2
C15:0	n. d.	0.2	n. d.	0.1	0.5
C16:0	11.7	12.7	3.5	24.3	28.1
C17:0	n. d.	n. d.	n. d.	0.4	1.4
C18:0	3.1	2.2	1.2	12.3	21
Monounsaturated:	30.2	31.5	61.2	45.3	39.9
C14:1	n. d.	n. d.	n. d.	2.1	0.9
C6:1	n. d.	n. d.	n. d.	4.2	5.5
C18:1	14.5	15.2	61.2	36.4	33.5
C20:1	13.3	13.5	n. d.	1.9	n. d.
C22:1	2.4	2.8	n. d.	0.7	n. d.
Polyunsaturated:	54.5	53.4	34.1	16.5	7.1
C18:2	20.2	15.2	24.6	15.4	4.8
C18:3	34.3	38.2	9.5	1.1	2.3

Notes: FAME − fatty acid methyl esters; n. d. – not detected es such characteristics (Schwab *et al.* 1987; Tashtoush *et al.* 2004).

The results of our studies have shown that the iodine value of pork lard amounted to 63 g I_2/100 g, while that of used for frying oil was 60.3 g I_2 /100 g. However, the one used for frying oil has high acidity. When producing esters from oil and fat with higher acidity (more than 2%), the catalyst is spent for the generation of soap while the generated soap forms emulsion and impedes further transesterification. Therefore, when using oil or fat with high acidity in the production of biodiesel fuel, the usual transesterification process should be preceded by the esterification process of free fatty acids with the use of various types of acid catalysts (Ma, Hanna 1999; Srivastava, Prasad 2000). The most frequently used process of esterification, which also has the most promising prospects, is the esterification process where sulphuric acid acts as a catalyst (Sendžikienė *et al.* 2004).

To sum up the results of the studies on the quality of raw materials, one can state it is impossible to produce biodiesel fuel meeting standard requirements from pure Camelina-seed oil; therefore, the possibilities of producing and composing the mixtures of Camelina-seed oil with pork lard and used for frying oil to produce biofuel were studied. For that purpose, the mixtures of various concentrations were prepared and the iodine value of the mixtures containing Camelina-seed oil and pork lard or used for frying oil were analysed. Fig. 1 shows the dependence of the iodine value on the composition of the mixtures of Camelina-seed oil and pork lard.

The iodine value of pure winter variety Camelina-seed oil is 169.6 g I_2/100 g of fatty matter, whereas that

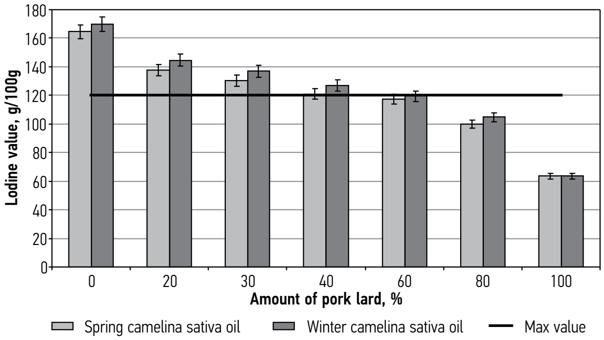

Fig. 1. The dependence of the iodine value on the composition of the mixtures of *Camelina sativa* oil and pork lard

of spring variety Camelina-seed oil is lower and reaches 164.6 g I_2/100 g of fatty matter. The iodine value of pure pork lard amounts only to 63 g I_2/100 g of fatty matter. With a growing content of pork lard, the iodine value of the mixtures decreases almost on a pro rata basis. The requirements of Lithuanian Standard LST EN 14214:2003 are met only by the mixtures the composition of which contains no more than 50% of winter variety Camelina-seed oil, while the content of spring variety Camelina-seed oil may reach as much as 58%. For studies on biodiesel fuel production, the mixtures of Camelina-seed oil and pork lard at a ratio 1:1 were selected. They underwent transesterification with methanol, and thus the physical and chemical characteristics of the produced methyl esters and their compliance with the requirements of the standard were evaluated (Table 2).

Table 2. The characteristics of various types of fatty acid methyl esters (FAME) and their compliance with the requirements of LST EN 14214:2003

Parameter	FAME (LST EN 14214: 2003)	SCFME	WCFME	SCOME	WCOME	RME
Ester content, % (m/m)	min 96.5	96.5	96.8	96.5	96.6	99.2
Density at 15 °C kg/m^3	860÷900	883	883	884	884	882
Viscosity at 40°C mm^2/s	3.50÷5.00	4.4	4.38	4.38	4.36	4.88
Water content, mg/kg	max 500	320	330	310	315	280
Oxidative stability at 110°C h	min 6.0	4.6	3.98	5.03	4.08	8.2
Acid value, mg KOH/g	max 0.50	0.4	0.37	0.48	0.47	0.3
Iodine value, g I_2/100 g	max 120	109.8	113.3	118.4	119.5	95
Linolenic acid methyl ester content, %	max 12	10.6	10.9	11.3	11.5	10.1
Monoglyceride content, % (m/m)	max 0.80	0.36	0.52	0.35	0.38	0.42
Diglyceride content, % (m/m)	max 0.20	0.008	0.02	0.1	0.06	0.022
Triglyceride content, % (m/m)	max 0.20	0.002	0.003	0.006	0.0004	0
Total glycerol, % (m/m)	max 0.25	0.24	0.23	0.18	0.11	0.14
Cold filter plugging point (CFPP), °C	min −5	+6	+4.6	+0.8	+1.4	−9

Notes: SCFME – methyl esters of spring variety Camelina sativa seed oil (50%) and pork lard (50%); WCFME – methyl esters of winter variety Camelina sativa-seed oil (50%) and pork lard (50%); SCOME – methyl esters of spring variety Camelina sativa seed oil (60%) and used for frying oil (40%); WCOME – methyl esters of winter variety Camelina sativa seed oil (60%) and used for frying oil fat (40%); RME – rapeseed oil methyl esters

Taking into account the fact that the quantity of used for frying oil generated in Lithuania is considerable, the possibilities of using it in a mixture of Camelina-seed oil were studied. Fig. 2 shows the dependence of the iodine value on the composition of the mixture of Camelina-seed oil and used for frying oil.

The results of the studies show that in this case, the mixtures having a higher content of Camelina-seed oil show the iodine value meeting the requirements of the standards as compared to the mixtures containing pork lard. The iodine value of the mixtures containing 40% and more of used for frying oil was 120 g I_2/100 g of fatty matter and lower.

In order to use as big amount of Camelina-seed oil as possible for biodiesel fuel production, it is reasonable to use a mixture containing 60% of Camelina-seed oil and 40% of used for frying oil. Taking into account the fact that the acidity of pure used for frying oil was high, i.e. 6.4%, the acidity of the mixture of the established optimal composition was studied prior to producing biodiesel fuel. It reached 2.75% in case of the mixture with spring variety Camelina-seed oil and 3.46% in case of the mixture with winter variety Camelina-seed oil, i.e. exceeded the maximum permissible acidity of feedstock if applying conventional biodiesel fuel production processes by transesterification with methanol. This is why the sample for further studies was prepared by applying an additional pre-esterification procedure under optimal conditions established above (Sendžikienė *et al.* 2004).

After producing methyl esters of spring variety Camelina-seed oil (50%) and pork lard (50%) (SCFME), winter variety Camelina-seed oil (50%) and pork lard (50%) (WCFME), spring variety Camelina-seed oil (60%) and used for frying oil (40%) (SCOME) and winter variety Camelina-seed oil (60%) and used for frying oil (40%) (WCOME), their physical and chemical characteristics and compliance with the requirements of the standard were determined. They were compared to the corresponding characteristics of rapeseed oil methyl esters (RME). The obtained data are presented in Table 2.

It can be noticed that some quality parameters (oxidative stability, CFPP, etc.) of the mixtures containing methyl esters produced from winter and summer variety Camelina-seed slightly differ. It could be explained by a different ester content of mixtures as well as by a slightly different fatty acid composition of Camelina-seed oil of winter and summer varieties.

The results also show that the produced esters do not comply with all requirements of the standard. The cold filter plugging point (CFPP) of the methyl esters of fatty acids obtained from the mixture of Camelina-seed oil and pork lard or used for frying oil is too high while their oxidation stability is too low.

Oxidation stability is expressed by means of the induction period (IP) that should last not less than 6 h at a temperature of 110°C according to the requirements of the standard for biodiesel fuel. Oxidation stability depends on the structure of fatty acids, the content of natural antioxidants and storage conditions (temperature, light, and moisture). Oxygen is highly significant for the initiation and progress of the autoxidation process where hydrocarbon chains are connected with the formation of polymers through oligomers.

Polymers formed among methyl esters impede the operation of the engine, especially the fuel injection system of the engine. The oxidation stability of fatty acid methyl esters (FAME) can be enhanced by introducing special additives. The possibility of enhancing the oxidation stability of ester mixtures that contained Camelina-seed oil by applying the industrial antioxidant Ionol was studied. For that reason, various amounts of the oxidant were added to esters and its influence on the induction period of ester mixes was evaluated (Fig. 3).

The obtained results showed that the minimum dosage of antioxidant Ionol ensuring the oxidation stability of fatty acid methyl esters (SCFME, WCFME, SCOME, and WCOME) was 500 ppm.

The cold filter plugging point (CFPP), i.e. the temperature when fuel plugs the filter partially or fully due to the formation of crystal agglomerates, is another important parameter. The method of determining the cold filter plugging point referring to moderate temperatures is widely used in Europe and other countries. The EU accepts two standards applicable to diesel fuels: EN 590:2004 is intended for fossil diesel fuel which, however, allows introducing only up to 5% of fatty acid methyl esters (FAME), and EN 14214:2003 intended for neat biodiesel fuel.

The CFPP of diesel fuel used during the summer period should be not higher than −5°C, and that of die-

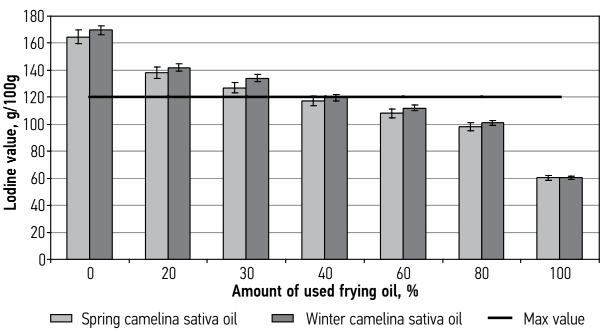

Fig. 2. The dependence of the iodine value on the composition of the mixture of *Camelina sativa* oil and used for frying oil

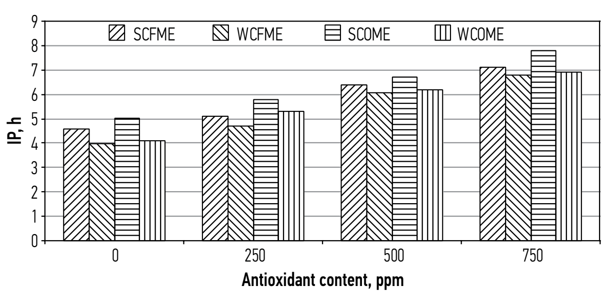

Fig. 3. The dependence of the induction period on the concentration of antioxidant (Ionol)

sel used during the winter period should be not higher than −32°C (EN 590:2004). The CFPP of the produced fatty acid methyl esters ranged from +1.4 to +6°C except for RME (Table 2). Industrial depressants Wintron XC-30 and Infineum R-442 were applied in order to reduce the CFPP.

It was noticed (Fig. 4) that it is difficult to reduce the CFPP of those esters containing pork lard because most depressants that reduce the CFPP are designed for fatty acid methyl esters of rapeseed oil. Even very high concentrations (up to 25000 ppm) of depressants reduce the CFPP of SCFME and WCFME only to 2.1÷3°C correspondingly. The CFPP of fatty acid methyl esters of rapeseed oil and used for frying oil is reduced to the level meeting the requirements of the standard by adding 1500 ppm of Wintron or 1200 ppm of Irganox (Fig. 5).

It can be stated that it is impossible to obtain a product meeting the requirements of the standard in respect to the low-temperature parameters of fuels even adding depressants, in case of producing fatty acid methyl esters from the mixtures of Camelina-seed oil and pork lard or when it is necessary to use extremely high amounts of additives for the purpose that is economically unreasonable. It can be ascertained it is more reasonable to the blend used for frying oil into mixtures containing Camelina-seed oil.

To sum up the obtained results, it could be concluded that Camelina-seed oil is prospective raw material for producing biodiesel fuel. The mixtures containing methyl esters of Camelina-seed oil and pork lard or used for frying oil of optimal composition could be used as an additive to fossil diesel fuel or to ordinary biodiesel fuel. Further research is required to evaluate the physical and chemical exploitation as well as environmental characteristics of such fuel.

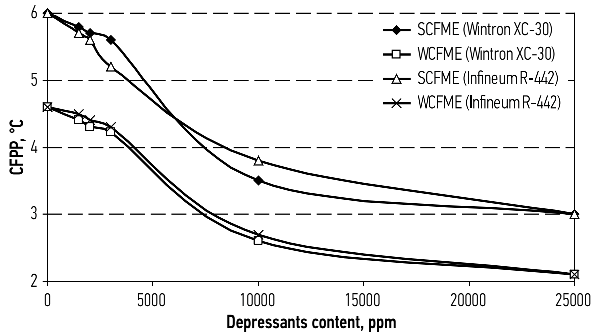

Fig. 4. The dependence of depressant concentration on the CFPP of fuel mixtures containing fatty acid methyl esters of Camelina sativa oil and pork lard (SCFME and WCFME)

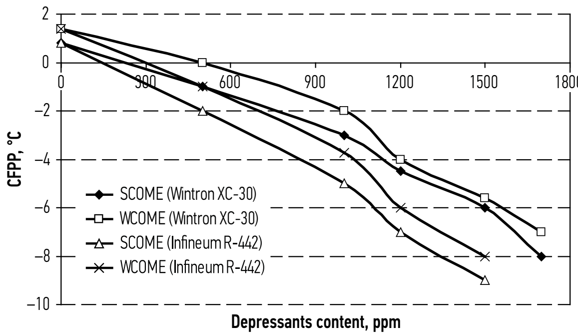

Fig. 5. The dependence of depressant concentration on the CFPP of fuel mixtures containing fatty acid methyl esters of Camelina sativa oil and used for frying oil (SCOME and WCOME)

4. Conclusions

1. Camelina-seed oil has a high iodine value (164.6÷169.6 g I_2/100 g oil); therefore, it cannot be used for producing biodiesel fuel directly.
2. Requirements for the iodine value are met by a composition containing 50% of spring or winter variety Camelina-seed oil and 50% of pork lard and a composition containing 60% of spring or winter variety Camelina-seed oil and 40% of used for frying oil, which can be utilised as feedstock for producing biodiesel fuel
3. The oxidation stability of fatty acid methyl esters produced from the mixtures of the established optimal composition can be enhanced by applying antioxidant Ionol with an optimal dosage of 500 ppm.
4. The cold filter plugging point of fatty acid methyl esters produced from Camelina-seed oil and used for frying oil can be reduced to −5°C by adding 1.500 ppm of depressant Wintron XC-30 or 1200 ppm of Infineum R-442. In Lithuania, such product can be used as fuel during the summer period.

Acknowledgment

For the research which was carried out under the project No VP1-3.1-ŠMM-06-V-01-003 (E!4018 CAMELINA-BIOFUEL) the authors sincerely appreciate the assistance of the Agency for Science, Innovation and Technology of Lithuania and the European Social Fund Agency.

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