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THE STUDY OF RAPESEED OIL PRODUCTION TECHNOLOGY OF REDUCED MACRONUTRIENTS CONTENT AS AN ENGINE FUEL

Summary

The evaluation of rapeseed oil production technology of reduced macronutrients content was executed on the specially made experimental production line. The scope of the study included the process of rape seed pressing and oil refining. Performance screw press FARMET DUO, level of oil yield and the impact of montmorillonite-based products to reduce phosphorus magnesium and calcium in raw rapeseed oil was evaluated. With the press capacity of about 10 kg of oil per hour, the oil yield was about 30%. The technology has allowed the reduction of phosphorus, magnesium and calcium to trace amounts <0.5 ppm. Based on the results, the compatibility of oil quality with the applicable quality standard of vegetable oil used as fuel for diesel engines DIN V 51 605 was proven.

Key words: *chemical technology, rape oil, oil refining, engine fuel, macronutrients content*

1. Introduction

In Polish agriculture, about 75% of transportation, farming and stock-farming works is performed using vehicles, machines and agricultural devices [1]. Such high level of mechanization of agricultural works means significant demand for fuel. Demand for liquid engine fuels reaches 50% of consumption of all energy carriers in farmsteads [2]. That is why research and development works are performed that lead to reduction of unitary consumption of Diesel oil and to substitute it with pure vegetable oil. The works are performed within the scope of oil plant cultivation, technologies of extracting oil and adaptation of vehicles to such fuel.

The most common oil plant in Europe is rape, that is why intensive works are being performed concerning the increase of yielding this plant. In 2008, average yield of tested plants amounted 48.5 dt/ha [3]. Rape oil, due to its physical and chemical properties, is a good bio-fuel or raw material to produce it [4,5]. Applying suitable technology of its yielding and refining, rapeseed oil can be a fuel used in agricultural machines [6]. This is proven also by research concerning application of pure vegetable oil or its processed form (bio-Diesel – fatty acid methyl esters) in military industry. The first attempts were made during interwar era and are continued now [7].

Examination performed by Hemmerlein and partners in 1991, has shown a major technical problem resulting from supplying the engines with pure rape oil. The problem consisted in negative impact of rape oil on Diesel engines with direct fuel injection [8]. The main reason for that phenomenon was high viscosity of rape oil as well as micronutrient content not present in petroleum derivative fuels. Hence, for a few years now, there are intensified research and development works performed, that concern adaptation of agricultural machines engines to rape oil. As a result, problems such accumulation carbon deposit in combustion chamber, thickening of lubricating oil as a result of its mixing with vegetable oil or difficult start-up at low temperatures were solved. In 2003, research project was initiated that was aimed at performing operational examination of agricultural vehicles adapted to pure rape oil [9]. In 2008, John Deere company started a research project which goal was to perform operational tests of John Deere agricultural tractors factory adapted to rape oil [10]. To supply new generation agricultural tractors, it is necessary to use fuel that meets quality standards imposed by manufacturer.

Due to lack of small systems for rape oil generation of quality that would meet the German standard DIN 51605 [11], works concerning preparation of technology and development of a test station to test it have been initiated, and this was the main goal of the discussed work [12]. The research problem in the undertaken work was development of low-cost and not technologically complex method of phosphorus, magnesium and calcium reduction as well as determination of operational capacity of the model system. Results of the research will allow implementing the developed system.

Oil pressing process at high temperature can be characterizes by high yield (ratio of produced oil weight to seeds weight) at the cost of significant increase of phosphorus content to over 150 ppm. That is why, refining of the produced oil is necessary. Moreover, the obtained rape oil-cake cannot be used to feed animals. Reduction of phosphorus content in vegetable oil is possible by pressing the seeds at low temperatures. In this case, phosphorus content does not exceed 15 ppm, however oil yield is relatively low. That is why it is not necessary to perform expensive refining processes [13]. The second method is less effective, but the manufactured products can be directly used to feed animals and the oil itself requires only filtration [14].

According to the standard DIN 51605, content of phosphorus in oil should not exceed 12 ppm, magnesium and calcium 20 ppm in total. There is no uncomplicated refining method that in connection with the aforementioned technologies would provide the required effect. Evaluation of phosphorus, magnesium and calcium reduction using the developed, new technology was an additional goal of the research.

2. Materials and methods

In order to perform the test, mode system for oil pressing was constructed (Figure 1). It consists of a hopper, magnetic filter, pug mill Farmet Duo, initial tank, mixer and dust filter. The test was performed in two stages. As a result of the first stage, rape oil was obtained, whereas during the second stage it was subjected to purification. Within the first stage, press capacity and oil yield was determined, during the second – level of phosphorus, magnesium and calcium reduction as well as the remaining parameters of rape oil, which are given in the standard DIN 51605.

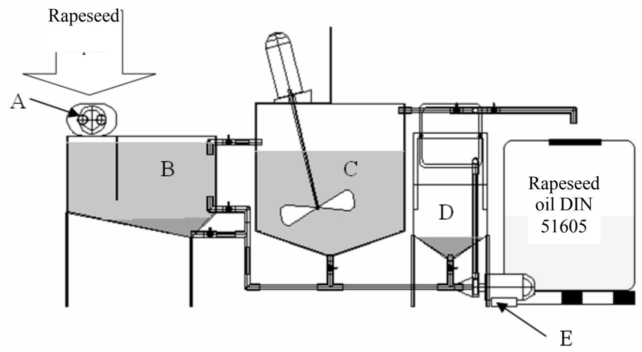

Fig. 1. Schematic diagram of oil press system, where: A- press, B- initial tank, C- mixer, D- platen press , E- hydraulic pump

Low erucic acid, double zero rapeseeds were used for the tests. Before the test, the following parameters of the seeds were determined: oil-content, humidity and contamination for each batch used for tests. Based on the results, seeds suitability for the tests was determined.

Batch of rape was introduced to the buffer tank, then the press was started. The procedure consisted in: heating the extractor press heads to about 100°C and starting the press. Using the G-fore and change of the seed buffer tank bolt location, quantity of seeds introduced to the press was uniformly increased. During this process, heads electrical heaters were turned off. If they were turned on, this would lead to sintering of cake in the head. The pressing process is an exothermic process, thus the generated heat is sufficient to extract oil from the seeds. The start-up procedure has ended at the moment of full opening of the buffer tank discharge bolt. Time of effective pressing was measured after 10 minutes from the moment of producing first rape pellets. Oil from the press was directly fed with the G-force to the tank under the press placed on extensometer scales. Beginning and end of the scales scale was recorded. Based on gathered results, pressing time and amount of produced oil, press capacity and oil quantity was calculated.

After pressing 1500 kg of rapeseeds and producing 450 kg of oil, the second stage begun. 10 dm^3 of oil was sampled. Half of it was subjected to filtering in order to separate solid contamination from the pressing. Oil in quantity 0.4 kg from each test was introduced to 6 glass containers, three samples of filtered oil and three samples of contaminated one. Montmorillonite-based product of weight 8 g was introduced to each sampling container. Three different products were used for the tests, which physical and chemical characteristic is given in table 1. Such samples were mixed until disappearing of all visible solid particles in the suspension. Process time was 15 minutes for each sample. The ready suspension was put aside for two hours, and them using cellulose filter, solid contamination was filtered. In the obtained samples, the following elements concentration was determined: phosphorus, magnesium and calcium.

Table 1. Physical and chemical properties of products for oil filtering

Parameter	M 300	M 100C15	M 100W10
form	dusty, odourless powder		
colour	pale beige	pale beige	dark grey
loose bulk density [$g*dm^{-3}$]	500-650	450-550	450-550
thick bulk density [$g*dm^{-3}$]	550-700	500-600	500-600
pH	2,9-3,5	3-6	3-6
free acid content (HCl) [%]	0,9	0,9	0,9
active carbon / cellulose content [%]	-/-	-/15	10/-

The next stage was the preparation of filtering charge, consisting of three examined products and introduction to the main mixer. Then, the aforementioned procedure was applied. Having filtered contamination from the oil, oxidising stabilizer based on butylated hydroxytoluene (BHT) and butylated hydroxytoanisole (BHA) was added to the storage tank, in quantity according to manufacturer's recommendations 1:1000. The above compounds cannot be used to refine food oils. After the completed production cycle, samples of oils were examined in an accredited laboratory, that performed the analysis of required parameters in order to determine oil quality conformity with the standard DIN 51605.

3. Tests results

Table 2. Rapeseed characteristics

Batch No	Rapeseed quantity [kg]	Water content [%]	Contamination [%]	Oil content [%]
1	500	7,4	1,5	44,0
2	500	7,0	1	44,5
3	500	6,5	1	44,0

Source: own tests

Humidity of seeds directly affects the water content in oil, that according to DIN 51605 should not exceed 750 ppm. Content of solid contamination in the initial material influences indirectly the chemical composition of oil and directly the pressing process. Too much content of contamination causes that the press clogs leading to its emergency stopping. Content of oil in rapeseeds is 39-46% and depends on many factor, mostly on its species. Seeds used during the tests met the basic quality requirements, i.e. humidity below 7.5% and contamination below 2% and were characterized by high content of oil.

According to the adopted methodology, pressing parameters were measured separately for each batch of tested material. The results are given in table 3.

Oil yield expressed as a ratio of raw oil weight to rapeseed weight, was in average 29% ± 0.82%. Cake produced as a result of pressing was characterized by high content of oil (21% in average). This speaks for low efficiency during cold pressing. However, cake obtained as a result of cold pressing is a very good, high-protein and high-energy component of feeds for breeding stock. Rapeseeds were pressed at temperature 21-25°C, oil from the press was at temperature ca. 32°C, and the rated temperature of the press (temperature of head) did not exceed 70°C. Based on the tested pressing temperature, it can be said that pressing took place using the cold method. Efficiency of the press was tested due to lack of manufacturer's data, who guaranteed capacity at the level 25 $kg*h^{-1}$. It was found that this value depended on a series of factors, among other things, seeds temperature, outlet nozzle diameter and distance of press piston to the head. As a result of the test, it was found that actual capacity of the press was from 36 to 38 $kg*h^{-1}$ of seeds, with the diameter of hole in the cake forming head fi 6 mm, distance of the press from the piston 1.5 mm and seeds temperature >20°C. This information is valid for correct construction of the installation and selection of proper support devices.

Table 3. Rapeseed pressing process parameters

Batch No	Pressing time	Oil weight	Oil cake weight	Oil content in cake	Capacity	Oil yield
	[h]	[kg]	[kg]	[%]	[$kg*h^{-1}$]	[%]
1	14	146	354	21	10,5	29
2	14	150	350	20	10,7	30
3	13	140	360	22	10,8	28
Average	13	145	355	21	10,7	29
(±SD)	(±0,5)	(±4)	(±4)	(±0,01)	(±0,13)	(±0,82)

Source: own tests

Effective, average capacity of the press was 10.7 $kg*h^{-1}$ of raw rape oil as a result of which 25-27 $kg*h^{-1}$ of cake was produced. This information is important due to selection of proper volumes of individual tanks and planning the production scale. Vegetable oil according to this technology is fed to tank B (Figure 1), where it is subjected to sedimentation and cooling process. Having filled tank B, oil is poured to mixer C where previously batched, montmorillonite-based product is added. Mixing process is not a continuous process, it is performed after topping the mixer and adding the montmorillonite-based product. According to the test methodology, oil was mixed for two hours and then it was subjected to filtering on platen filter. Volume ratio of these two tanks is very important, where properly selected sizes allow continuous pressing of oil and enable limiting the time within which oil is in raw condition, which fact contributes to formation of free fatty acids. It was assumed that the developed solution would allow reaching the required quality of oil with maximum simplification of the whole installation for its production. The discussed solution was reported for patent protection in 2010.

Oil generated as a result of pressing was used in the second stage of the tests. Initial and basic tests were performed. The results are given in table 4.

Each of the tested products, used for elements reduction (P, Mg, Ca) in oil, regardless of whether the oil was filtered or not, reduced the content of phosphorus below the range of measurement apparatus, i.e. 0.5 ppm. In relation to the remaining important elements, coarse-grained montmorillonite M 300 reduced calcium content most effectively, however its reaction to magnesium reduction in the tested oils was low. Application of M 100C15 affected the reduction of magnesium in average by 50% in both cases. Based on gathered results, it was found that the mixture 1:1 M 300 and M100C15 will most effectively reduce the three analysed elements. Based on the results, batch of filtering products was prepared in ratio 1:1, maintaining the proportion of total weight to oil weight. This mixture was poured through the top cover to the mixer C.

Having completed the whole production cycle, oil sample was tested in an accredited laboratory. The results are given in table 5.

Table 4. Results of analyses of examined elements content in oil sample

*Test code product No / oil type**	*Ca mg/L*		*Mg mg/L*		*P mg/L*	
	value	*reduction*	*value*	*reduction*	*value*	*reduction*
Raw oil	2,26	-	0,29	-	1,0	-
M 300/NF	0,20	91%	0,20	31%	<0,4	>60%
M 100C15/NF	0,37	84%	0,17	41%	<0,4	>60%
M 100W10/NF	0,66	71%	0,14	52%	<0,4	>60%
M 300 /F	0,19	92%	0,27	7%	<0,4	>60%
M 100C15/F	0,35	85%	0,13	55%	<0,4	>60%
M 100W10/F	0,66	71%	0,27	7%	<0,4	>60%

* NF – not filtered oil; F – filtered oil

Source: own work and analyses performed in the Environmental Laboratory of Unique Chemical Apparatuses at the Adam Mickiewicz University in Poznań

Table 5. Parameters of oil produced based on the tested technology in the model installation

Parameter	*Method DIN*	*Result*	*DIN V 51605*	*Unit*
Density (15°C)	DIN EN ISO 12185	920,2	900-930	$kg{\cdot}m^{-1}$
Ignition temp.	DIN EN ISO 2719	262,5	> 220	°C
Kinematic viscosity (40°C)	DIN EN ISO 3104	34,44	< 36	$mm^2{\cdot}s^{-1}$
Calorific value	DIN 51 900-2	37320	> 36000	$kJ{\cdot}kg^{-1}$
Cetane index	IP 498	46,2	> 39	-
carbon residue	DIN EN ISO 10370	0,34	< 0,40	% ($m{\cdot}m^{-1}$)
Iodine number	DIN EN 14111	112	95-125	$10gJod{\cdot}kg^{-1}$
Sulfur content	DIN EN ISO 20884	5,9	< 10	$mg{\cdot}kg^{-1}$
Contamination content	DIN EN 12662	51	< 24	$mg{\cdot}kg^{-1}$
Acid number	DIN EN 1404	0,890	< 2,0	$mgKOH{\cdot}g^{-1}$
Oxidizing stability (110°C)	DIN EN 14112	6,7	> 6,0	h
Phosphorus content (P)	DIN EN 14107	< 0,5	< 12	$mg{\cdot}kg^{-1}$
Content of calcium and magnesium (Ca+Mg)	DIN EN 14538	< 0,5	< 20	$mg{\cdot}kg^{-1}$
Ash content	DIN EN ISO 6245	0,004	< 0,01	% ($m{\cdot}m^{-1}$)
Water content	DIN EN ISO 12937	600	< 750	$mg{\cdot}kg^{-1}$

Source: own work and results of laboratory tests performed at ASG Analytik-Service Gesellschaft mbH, Neusass, Germany

4. Summary

Rape oil used to supply agricultural vehicles must meet specified quality standards, which fact results from guarantee law. In 2006, in Germany draft (pre-standard) was prepared DIN V 51605, currently adopted as DIN 51605 that specify parameters, that rape oil should meet, in order to represent independent bio-fuel. Standardization of fuels is a very important item while introducing new vehicles on the market. Table 5 presents characteristic of oil obtained based on the production technology developed at Institute of Technology and Life Sciences. The major problem to solve is the method o phosphorus, magnesium and calcium reduction in oil. As a result of reaction of oil refining products based on montmorillonite, trace amounts of these elements were found to be below the measurement apparatus range. Other parameter, that would not be possible to achieve without subjecting the oil to complete refining, i.e. free fatty acids reduction, was the oxidizing resistance interval. The problem was solved by adding stabilizing agents, thus the oxidizing stability exceeded 6h.

The only parameter that was not still achieved at a satisfactory level, is the oil contamination level. The filtering system applied in the discussed model required improvement. Operation characteristic of the platen filter depends mostly on its level of filling with suspension, the higher the level, the purer oil is, however filter capacity drops. Oil contamination level can be improved by connecting the filter with mixer. First few litres of oil should be returned to the tank, then after its partial filling with suspension, it can be directed to storage tank.

5. Conclusions

- Vegetable oil can be a bio-fuel on condition that it is not a food product. Vegetable oil manufactured according to technology developed at the Institute of Technology and Life Science cannot be used for food purposes, because it does not meet the quality requirements, concerning e.g. BHT and BHA content. Oil parameters meet the requirements of DIN 51605, i.e. it can be used as fuel for agricultural vehicles.
- Test of the rape oil production technology, performed of a model especially prepared for this purpose, showed that parameters of the obtained oil meet the requirements of DIN V 51065 except the contamination content, which is only a technical problem that can be easily solved. However, it is necessary to improve this part of the installation by introducing additional oil circuit in the existing installation.
- Initial tests showed high efficiency of montmorillonite-based products during reduction of phosphorus, magnesium and calcium in raw rape oil. Initial results confirm that the research must be still continued.
- High stability of raw rape oil results from the addition of stabilisers based on butylated hydroxytoluene (BHT) and butylated hydroxytoanisole (BHA), which fact automatically disqualifies the oil as a food product.
- Cake obtained as a result of cold pressing are valuable, high-protein and high-energy additive of feed for breeding stock and can be directly applied. The tested production technology is practically non-waste process, because all manufactured products are used for production purposes within a farmstead.
- Benefit of this solution is satisfactory efficiency and option to run continuous production, which fact translates directly to low process costs. Relatively low efficiency of the line allows avoiding problems with storing the cakes and their becoming rancid. It also allows the production of oil on current basis, according to the needs, which fact is very important in the face of short life of oil as fuel, that does not exceed a few months. Size of presses and minimization of additional devices quantity is beneficial, because this reduced the costs of purchase and servicing.

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