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RESEARCH ON POSSIBILITIES OF USING BY-PRODUCTS OF BIODIESEL PRODUCTION FOR BIOFUEL

Summary

More extensive development of biodiesel production is hampered by a relatively high production cost of this product. Together with external measures (organization of optimal production, minimization of transport costs, etc.) it is necessary to use the by-products of biodiesel as rationally as possible to reduce the production cost. The work deals with the possibilities of using by-products (rapeseed straw, oilcake and glycerol) for energy needs and is based on experiments. The experiments established fractional compositions, thermal properties and dependences of rapeseed oil, oilcake and crude glycerol. Equilibrium moisture content of rapeseed straw, keeping them in a closed room, is 15% and heating value is 17.5±1.9 MJ/kg; the heating value of cold-pressed oilcake with 8-10% of oil is 19.8±2.0 MJ/kg; the water content in green glycerol composition accounts to 12.5±0.6%, the one of methanol is 0.3±0.03% and their heating value is 22.1±1.7 MJ/kg. The research proved that the use of green glycerol while mixing it with sawdust and pressing is an efficient way to produce fuel. It was established that after adding 80% of crude glycerol to dust, the heating value of dry sawdust increases by 22-23% and the heating value of 8% moisture content sawdust increases by 31-32%. The amount of nitrogen oxides NO_x in fume does not exceed the permissible limit – 650 mg/m^3, CO amount in fume is minimal as is the amount of free oxygen and only traces of sulphur dioxide are found in the fume.

***Key words**: biodiesel; by-products; rapeseed straw; oilcake; glycerol; biofuel; heating value; emissions; experimentation*

1. Introduction

A decreasing demand of agricultural food products, problematic tasks of environmental protection, especially limitations of gas emissions causing greenhouse effect approved by Kyoto protocol make us search for alternatives in using agricultural produce. One of these is using rapeseed for biodiesel production. Directive 2003/30/EC of the European Parliament and of the Council regulates the production and development of using liquid fuels in the European Union (EU).

Taking these directives and the requirements of the Madrid Declaration into account in 2012 40 thou tons of biodiesel should be produced in Lithuania and for this it is required to grow 120 thou tons of rapeseed grain. Rapeseed growing will create reasonable number of workplaces in the country and while producing biodiesel in Lithuania there will be no need to import oil or its products for 50 mio LTL.

The raw materials for biodiesel production will have to be transported to factories from the farms of different country parts cultivating rapeseed. Environmental pollution and the price of biodiesel production depend on rational use of production materials and optimum organization of their transportation. Environmental pollution is considered proportional to the distance of product transportation.

Fast development of biodiesel production is mostly hampered by a relatively high production cost of this product. Together with external measures (preparation of optimal production organization scheme by minimizing transport costs) it is necessary to use the by-products of biodiesel as rationally as possible to reduce it. One of them is glycerol phase which is separated from the main product – rapeseed methyl ester and the degree of re-esterification.

With the increase of glycerol supply in the EU countries its price is decreasing and there are selling problems related to that. Therefore, it is necessary to search for new fields of using crude glycerol taking account of technical, economic and environmental requirements.

2. Literature Review

At a relatively high production cost of biodiesel it is necessary to use the by-products of rapeseed methyl ester – oilcake and glycerol phase in more optimal ways.

Rapeseed oilcake is mostly used for fodder. Economically, they are best suitable to produce protein mineral additives for cattle, pigs and poultry [1, 2]. There is a shortage of about 10-15% of digestible proteins in cattle rations. Due to poorer quality of green and succulent fodder animals are often short of energy. These problems can be solved by using rapeseed oilcake and meal. It is also worth it economically as the imported protein raw materials – soya, sunflower and cotton meal are rather expensive. In addition, due to the spread of bovine spongiform encephalopathy it is forbidden to use meat, meat meal, meat-bone and blood meal, therefore, it is necessary to search for the protein sources compensating them.

About 90-93% of dry matter, 10-20% of fat, 26-31% of proteins and 8-12% of fibre remain in rapeseed oilcake obtained by mechanical pressing. 1 kg of oilcake has 1.2-1.3 of a feed unit and 210-270 g of digestible proteins. There are a lot of irreplaceable substances (IS), amino acids (lysine, methionine, cysteine, tryptophan, etc.) in rapeseed proteins and irreplaceable fatty acids (olein, linol and linolein) in fat which are necessary for growing animals as they have a positive effect on their health and productivity [3]. Amino acids in rapeseed oilcake proteins are suitably balanced and they are equal to soy meal proteins in biological value. By chemical composition feeds from rapeseed match imported protein feeds (table 1).

Rapeseed meal and oilcake are rich in mineral nutrients. They have more assimilated calcium, iron, manganese, phosphorus, magnesium and selenium whereas soya is richer in copper, zinc, and calcium. The use of rapeseed oilcake and meal in cattle and pig rations have been widely analysed by the researchers of the Western Europe, Canada and USA [4-6].

Table 1. Composition of rapeseed oilcake and other protein feeds

Feed	Dry matter, $g\cdot kg^{-1}$	Crude proteins, $g\cdot kg^{-1}$	Crude fat, $g\cdot kg^{-1}$	Crude fibre, $g\cdot kg^{-1}$	IS $g\cdot kg^{-1}$	Metabolic energy, $kJ\cdot kg^{-1}$
Soya meal	901	384	16	87	291	23.70
Sunflower meal	901	398	21	144	244	20.13
Rapeseed oilcake	900	274	80	90	229	26.60
Rapeseed meal	900	378	22	144	244	20.13

EU Regulation 1251/1999/EC provides for area payments for fallow allowing to grow there rapeseed for biodiesel production, i.e. agricultural produce for non-food purposes, therefore, rapeseed oilcake should not be used for animal feeding. The companies producing biodiesel equipment indicate that oilcake or meal can be used as biofertilizer or biofuel [7]. Proteins from meal are used in the production of bioplastics, covers, glues, paper, emulsifier and other biologically degradable products [8]. Fat should be removed from this raw material. In rapeseed meal fat must not exceed 1%. That changes oil production technology in principle. The oilcake with 8-12% of fat has to be extracted by chemical solvents. Usually hexane is used as an extrahent, which has to be eliminated from oil phase and meal after extraction. This process requires huge energy input [9].

Rapeseed meal has been used for years as fertilizer in Japan and China and as kitchen fertilizer in Europe. It can be used to fertilize winter rapeseed, incorporated during sowing (in August) as nitrogen does not leach from it in winter [10].

As biofuel rapeseed meal is similar to coal in terms of heating value and spontaneous combustion temperature [11]. However, these indicators depend on many factors; therefore, they have to be specified in view of technologies and equipment used in Lithuanian biodiesel production.

Different ways to separate the glycerol phase are used in different rapeseed methyl ester (FAME) production technologies depending on the requirements raised for the quality of by-products [12]. When producing biodiesel by "Feld-Hahn I" technology the glycerol phase is separated after the first and the second degree of re-esterification.

In an improved technology of biodiesel production "Feld-Hahn II" the glycerol phase from the first and the second degree reactors passes to phase of separators. H_3PO_4 is additionally supplied to the second degree separator which causes hydrochloric acid to enter the glycerol phase. The glycerol obtained by such method does not correspond to commercial requirements either (glycerol concentration 72-77%) [13].

According to "Lurgi" technology of biodiesel production [14] the glycerol phase is formed from the lower phase of the first degree re-esterification settling container and acidified washing waters of HCI rapeseed methyl ester. And the glycerol phase from the second degree re-esterification settling container is returned to the first degree re-esterification reactor.

A little bit better way of separating the glycerol phase is proposed by the technologists of "Voge&Noot" [15]. In this technology the glycerol phase is collected from the settling containers of the first and the second degree and the reactor of FAME acidic wash by water. The collected phase is firstly treated with phosphoric acid. The filtered down potassium phosphate is returned to this phase. After that the formed KH_2PO_4 (a valuable fertilizer) and free fatty acids are separated. Glycerol is neutralized by KOH, filtered, water and methanol evaporated. The vapour is directed to a methanol rectifying column from which pure methanol is returned for re-esterification and the water is used for FAME washing. The production of 1000 kg of FAME yields 101 kg of crude glycerol with 87-92% of pure glycerol. In addition, this technology of FAME production requires minimal energy consumption compared with other technologies. To produce 1 t of FAME 211 kWh heating energy and 16 kWh electric power are consumed.

When producing biodiesel by "Feld-Hahn I" technology the cleanness of separated crude glycerol is not higher than 62-69%. It is an impure glycerol phase with methanol and water after evaporating of which the average purity glycerol (crude glycerol) is obtained. Pure glycerol is obtained by distillation.

The equipment for crude glycerol distillation and absorption cleaning is offered by several European and American companies. One of them is "Chemco" (Italy) [16]. The greatest problems for pure glycerol distillation method are caused by mono-glycerides, which are difficult to separate from glycerol.

Pure glycerol obtained by the mentioned methods can be used in many areas: food, drinks, medicine, cosmetics, tobacco and textile industries [17]. However, its production requires huge energy consumption. Such production is cost-effective, if at least 10 thou t/year of crude glycerol is produced and such amount can be obtained only when you produce more than 100 thou t/year of FAME.

The most attractive method seems to be mixing of an unseparated glycerol phase with liquid fuel, e.g. fuel oil, and burning in boiler-houses. However, glycerol and other phase components are not mixed with oil products and become layered; therefore, their use is problematic [18].

At small volumes of FAME production the most economical glycerol use method is the production of fuel briquettes. The glycerol phase with methanol removed can be used for that as methanol can create dangerous conditions regarding fires and explosions on the briquette production line [19]. The experiments in this field was started by the Austrian Institute of Agricultural Engineering [20], however, no data was found in the literature regarding the technological parameters of such briquette production and heating value of the briquettes. There is no data on the maximum glycerol amount possible to work in them either.

On the basis of the literature analysis made it can be stated that briquette production from sawdust or rapeseed straw with glycerol additives has not been thoroughly investigated.

The work objective is to investigate and substantiate the possibilities of using biodiesel production by-products for energy needs.

3. Experimental Section

The density of crude glycerol was established by an aerometer according to ISO 2099:1972 requirements [21]. When measured glycerol is heated to 55±1°C to have it liquid. The water content in glycerol was established by Karl Fisher method according to ISO 2097:1972 requirements

[22]. Methanol amount in the sample of a glycerol fraction was established in a capillary column of gas chromatography by using a flame ionization detector. The amount of fatty acid methyl ester (FAME) in crude glycerol was established by gas chromatography method according to EN14103 [23]. The experimentation was carried out in the chemical laboratory of the Lithuanian University of Agriculture using the existent experimental facilities and the prepared methodologies.

The heating value of solid fuel was established by using calorimeter V-08MA. The experiments were carried out in a special room with invariable temperature at ±1°C accuracy. Fuel sample mass was 0.8-1.5 g. Invariable temperature of 24°C was maintained in the calorimeter. Temperature fluctuations must not exceed ±0,2°C.

A comparative heating value of the sample is established using the formula:

$$Q = \frac{C_i \Delta t}{m} \tag{1}$$

here: Q – sample heating value, kJ·kg^{-1};

C_i – energy equivalent of calorimeter, kJ·K^{-1};

Δt – change of calorimeter system temperature during experiment, K;

m – sample mass, kg.

Energy equivalent of the calorimeter is 15±0.15 kJ·K^{-1}. Error is ±0.1% at 0.95 reliability.

In order to analyse fuel combustion, combustion temperature, the amount of CO and NO_x in fume an experimental heating device was used with a fireplace type furnace, the diagram of which is shown in fig. 1.

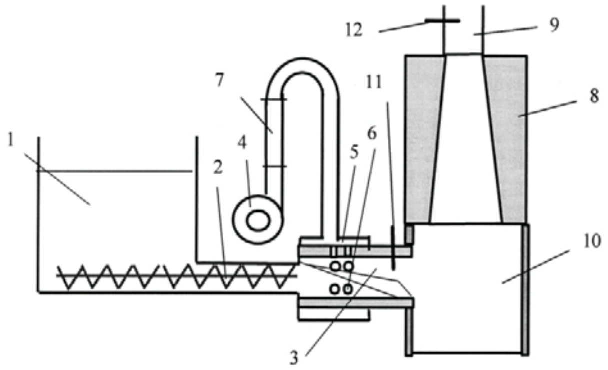

Fig. 1. Scheme of biomass heating device: 1 – fuel bunker; 2 – screw fuel feeder; 3 – furnace; 4 – fan; 5 – air chamber; 6 – air feeding inputs; 7 – rotameter; 8 – heat exchanger; 9 – fume outlet; 10 – ash chamber; 11– thermal sensor; 12 – probe of combustion analyzer

Fuel is supplied to the furnace by cycles of fixed rhythm. A cycle consists of a working period when fuel feeder is switched on and a pause when the feeder is switched off. While changing the duration of the working period and pause it is possible to change the amount of fuel supplied to the furnace.

Fuel mass is established by weighs RP-50S11 at ±0.01 kg error. The amount of air supplied for combustion is measured by the rotameter. The rotameter was tested by measuring air movement velocity in the furnace at ±0.1 m·s^{-1} error by flow sensor FVA915-S120 and device ALMAMO-2290-8. Combustion temperature in the furnace is measured by thermal element NiCroSil-NiSil (N) and registered device ALMAMO-2290-8 at 1°C accuracy. The experiment duration was established by the time readings of the mentioned device. The combustion quality was established by combustion analyser BACHARACH PCA-65 while measuring the amounts of oxygen, carbon monoxide, nitrogen oxide in fume and fume temperature. Measurement accuracy: oxygen – 0.3%; CO – 10 ppm; NO – 5 ppm; fume temperature 1°C.

During the experiments the furnace feeder operated at maximum capacity. To maintain combustion 30.6 m^3·h^{-1} – 10.4 m^3·h^{-1} of air was blown into the furnace by reducing the amount in intervals. The duration of each mode was 300 s.

Combustion temperature is recorded every 10 s and fume composition – every 20-30 s. Combustion temperature is calculated as the average value of 18 measurements per each working mode. The fume composition and air surplus rate are calculated as the average value of the measurements carried out per 180 s period of each working mode corresponding to the period of temperature measurements.

4. Results and Discussion

The composition of glycerol fractions obtained by re-esterification of rapeseed oil is presented in table 2.

Table 2. Composition of glycerol fraction

Indicators	Glycerol fraction	Crude glycerol
Water, %	26.8±1.6	12.5±0.6
Glycerol, %	69.3±2.3	86.8±3.6
Methanol, %	3.2±1.7	0.3±0.03
FAME residues, %	0.7±0.08	0.4±0.08
Density at 20°C, g·cm^3	1.12±0.09	1.18±0.11

Table 2 also presents the composition of crude glycerol after evaporating water and methanol from the glycerol fraction. As we can see from the data submitted there are 26.8% of water and 3.2% of methanol in the glycerol fraction. The latter is especially dangerous when using glycerol in the composition of fuel briquettes as it poses threat to human health. In addition, a higher concentration of methanol vapours can cause fire or explosion in a room. The experiment established that there are 1.05% of phosphorus, 2.2% of potassium and 0.11 % of sodium in the composition of crude glycerol. Elemental composition of rapeseed straw and oilcake is presented in table 3.

As we can see there is a very small amount of environmentally dangerous elements chlorine and sulphur in the composition of rapeseed oilcake and straw. Therefore, harmful compounds in their combustion products will not exceed permissible rates [24].

Table 3. Elemental composition of rapeseed oilcake and straw

Indicators	Elemental composition of dry matter, %						The moisture content, %	The fat content, %
	C	H	N	S	O	C1		
Oilcake	41.74	5.31	5.47	0.54	31.20	0.04	9.21±0.61	2.2±0.12
Straw	43.38	5.20	0.37	0.11	39.04	0.15	9.37±0.73	–

The established heating value results of the glycerol fraction, rapeseed oilcake (8% of oil) and rapeseed straw (moisture content of 12%) are presented in fig. 2.
We can see from the results presented that the highest amount of energy is obtained when glycerol is burnt: 22.1±1.7 MJ·kg^{-1}; and the least amount – when rapeseed straw is burnt: 16.8±1.2 MJ·kg^{-1}.

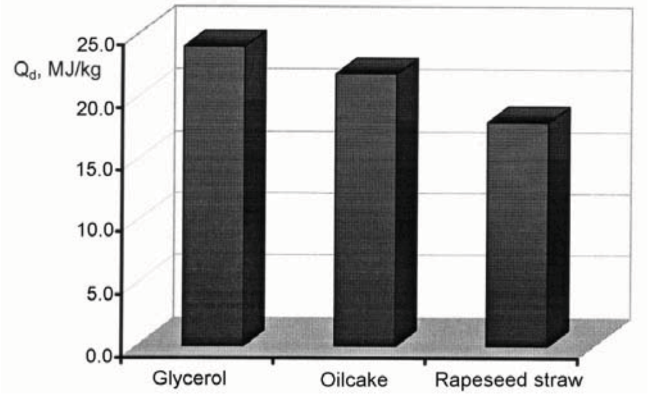

Fig. 2. Heating values of biodiesel production by-products

It was established by experiments that the dependence of oilcake heating value Q_{oc} on the amount of oil M_a in it at correlation ratio 0.92 can be expressed by a logarithmic equation:

$$Q_{oc} = 2.05 \ln M_a + 14.98 \qquad (2)$$

Heating value of rapeseed straw is directly dependent on its moisture content (fig. 3).

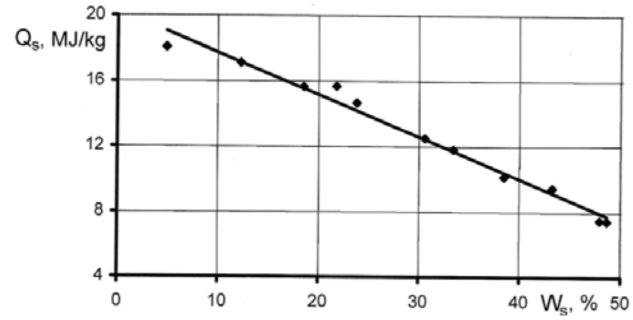

Fig. 3. Dependence of rapeseed straw heating value Q_s on its moisture content W_s

As we can see the heating value of rapeseed straw decreases as its moisture content increases. Equilibrium moisture content of rapeseed straw makes about 15% and the heating value of such straw reaches 17.5±1.4 MJ·kg^{-1}, if it is stored in a closed room.

Having compared the heating value of extracted oilcake and rapeseed it can be observed that it differs little. That can be easily explained while analysing the elemental composition (table 3) of the dry mass of these products. Oilcake has less carbon – 41.74% compared with 43.38% in straw, the hydrogen amount is the same – 5.31% and 5.2% and the oxygen is lower – 31.2% and 39.04%. A higher heating value of rapeseed oilcake can be explained by its lower moisture content (9.2% compare with 15% of straw).

By bomb calorimetry experiments the general heating value of sawdust with crude glycerol additive was established. In combustion experiments the sawdust of coniferous trees was used, its moisture content made 8.0±1.5%. The heating value of dry and moist fuel mass was established from the experimental values achieved (see fig. 4).

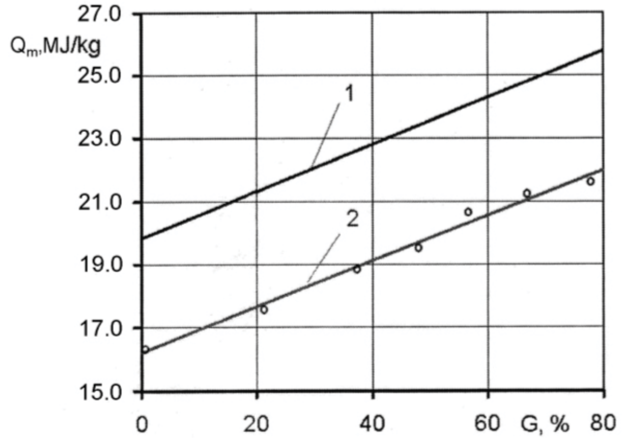

Fig. 4. Dependence of heating value of sawdust and crude glycerol mixture Q_m on glycerol amount in it G: 1 – the calculated heating value of totally dry mass mixture; 2 – 8% moisture content sawdust mixture heating value (experimental values)

We can see from the data submitted that the heating value of the totally dry mass mixture is 19.7±1.35 MJ·kg^{-1} and it is 16.28 MJ·kg^{-1} at 8% moisture content. Having added 80% of crude glycerol into the sawdust, the dry fuel heating value increases up to 25.5±1.83 MJ·kg^{-1} and the one of moist fuel – up to 23.7 MJ·kg^{-1}.

Having carried out the analysis of submitted experiments it can be stated that the most rational alternative to use for fuel is a by-product of biodiesel production – crude glycerol mixed with sawdust.

The experiments established that combustion temperature increases, when fuel mixture is burnt with a higher amount of glycerol (see fig. 5).

Temperature rising is explained by a higher glycerol heating value (see fig. 2).

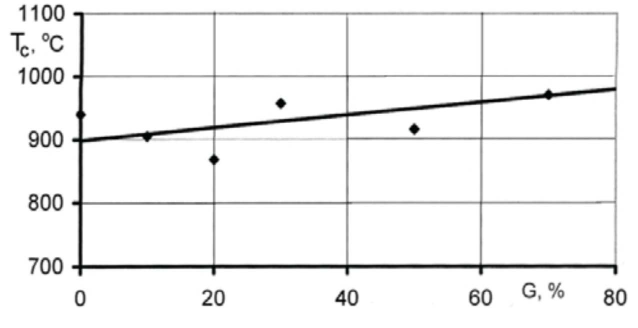

Fig. 5. Dependence of combustion temperature T_c on crude glycerol amount G in sawdust at comparative amount of air supplied V_o = 5 m^3·kg^{-1}

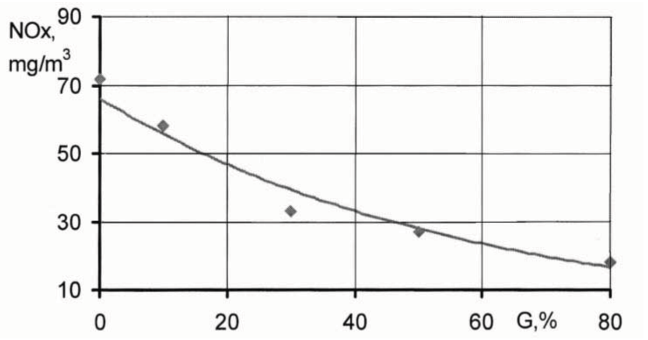

Fig. 6. Amount of nitrogen oxides NO_x in fume depending on crude glycerol amount in fuel mixture G. Comparative amount of air supplied V_o = 4 m^3·kg^{-1}

Fuel combustion temperature determines the amount of nitrogen oxides NO_x in fume. The variation in the amount of nitrogen oxides in fume depending on crude glycerol amount in the mixture is shown in fig. 6.

This dependence at correlation ratio 0.96 can be expressed by an exponential equation:

$$NO_x = 66.1\exp(-0.017G) \qquad (3)$$

With an increase of crude glycerol amount in fuel mixture combustion temperature increases and the amount of nitrogen oxides NO_x in fume decreases. Environmentally it is a favourable factor as nitrogen oxides are considered dangerous compounds for human health.

In the process of burning sawdust briquettes with an additive of 10% of crude glycerol it was achieved that in the beginning of the experiment the amount of CO in fume was minimal as was the amount of free oxygen. After 30 minutes the combustion process set in as well as the amount of free oxygen. This indicates the excess of the air blown into the boiler. The amount of nitrogen oxides NO_x in fume was minimal during the entire combustion experiment and it did not exceed the permissible limit – $650\ mg \cdot m^{-3}$. Only traces of sulphur dioxide SO_2 was recorded in fume. During the trial the temperature in the furnace was 1050 ± 30^oC, the temperature of oxides in the smoke vent was 67 ± 7^oC and the boiler's heating capacity was 35.2±2.1 kW. The ash-content of the examined fuel briquettes was 1.2±0.15%.

As a result of the experiments we can state that the use of crude glycerol mixing it with sawdust and pressing it is an efficient way of solid fuel production. The optimal amount of crude glycerol in the mixture is determined by pressing technologies used, as the amount of glycerol affects the features of fuel physical hardiness signally.

5. Conclusions

Most rational way of using rapeseed oilcake is to feed it to animals and the surplus can be used as fuel. The heating value of rapeseed cake depends on the amount of oil in it. The heating value of cold-pressed oilcake with 8-10% of oil is $19.8\pm2.0\ MJ{\cdot}kg^{-1}$ and if the oil amount decreases to 2.6% – the heating value is $17.8\pm1.5\ MJ{\cdot}kg^{-1}$. The heating value of rapeseed straw depends on its moisture content. Equilibrium moisture content of rapeseed straw, keeping them in a closed room, is about 15% and heating value is $17.5\pm1.4\ MJ{\cdot}kg^{-1}$. The glycerol fraction obtained after the second degree of re-esterification has 26.8±1.6% of water and 3.2±1.7% of methanol. Such glycerol cannot be used for fuel, without evaporating the mentioned components. Crude glycerol has 12.5±0.6% of water and 0.03% of methanol in its composition. Combustion temperature of sawdust and crude glycerol mixture varies depending on the mixture composition and the air amount supplied to the furnace. The amount of nitrogen oxides NO_x in fume depends on the amount of glycerol in the mixture. With an increase of glycerol amount combustion temperature increases and the amount of NO_x in fume decreases. When pressing sawdust the amount of glycerol has a signal effect on mechanical hardiness and heating features of the prepared fuel.

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