

CHARACTERIZATION OF CRUDE GLYCEROL FROM BIODIESEL PRODUCTION FROM MULTIPLE FEEDSTOCKS

J. C. Thompson, B. B. He

ABSTRACT. *Glycerol is the principal by-product of biodiesel production. For each gallon of biodiesel produced, approximately 0.3 kg of crude glycerol accompanies. Such crude glycerol possesses very low value because of the impurities contained. As the demand and production of biodiesel grow exponentially, the utilization of the glycerol becomes an urgent topic. The make-up of crude glycerol varies depending on the parent feedstock and the biodiesel production process. Before the crude glycerol could be considered for possible value-added utilizations, it is necessary to characterize it on its physical, chemical, and nutritional properties. This article reports the characterization of crude glycerol obtained from different seed oil feedstocks of mustard, rapeseed, canola, crambe, soybean, and waste cooking oils. Batch processes of biodiesel production were used as the means of crude glycerol preparation using unrefined vegetable oils, methanol, and sodium methylate as the catalyst. After separation from biodiesel, the crude glycerol from each of the oils was analyzed using ASTM and other standard test methods. Elemental impurities, nutritional value, and other chemical properties were tested.*

Keywords. *Biodiesel, Crude glycerol, Glycerin.*

Biodiesel production worldwide has been on an exponential growth curve over the past several years. The principal by-product of this production is glycerol, also known as glycerin. It occurs in vegetable oils at a level of approximately 10% by weight. For each gallon of biodiesel produced, approximately 0.3 kg (0.66 lb) of crude glycerol accompanies. Such crude glycerol possesses very low value because of the impurities. As the demand and production of biodiesel grows, the quantity of crude glycerol generated will be considerable, and the utilization of it will become an urgent topic. The make-up of crude glycerol varies depending on the parent feedstock and the biodiesel production process. Before the crude glycerol can be considered for possible value-added utilizations, it is necessary to characterize the crude glycerol on its physical, chemical, and nutritional properties.

Most industrial biodiesel processes utilize a 6-to-1 molar ratio of alcohol to oil, which is an excess of 100%, in order to drive the reaction to completion. Most of the excess alcohol (up to 80%) will end up in the glycerol layer after the reaction. As a minimum effort, producers will recover this alcohol for reuse. Further refining of the crude glycerol will depend on the economy of production scale and/or the availability of a glycerol purification facility. Larger scale biodiesel producers refine their crude glycerol and move it to markets in the food, pharmaceutical, and cosmetic industries.

It is generally treated and refined through filtration, chemical additions, and fractional vacuum distillation to yield various commercial grades, such as dynamite grade, yellow distilled, and chemically pure (*Bailey's Fats and Oils*, 1979). It can also be refined by a less energy intensive method of filtration through a series of ion-exchange resins (Miesiac, 2003). Small to moderate scale producers who cannot justify the high cost of purification find crude glycerol utilization or disposal to be a problem.

As more and more crude glycerol is continuously generated, researchers are studying economical ways to utilize it, thus further defraying the cost of biodiesel production. This article reports the characterization of crude glycerol obtained from different seed oil feedstocks of mustard, rapeseed, canola, crambe, soybean, and waste cooking oils. Doing so, the authors attempt to provide useful information for the further discovery and utilization of this very versatile by-product.

MATERIALS AND METHODS

Glycerol samples were obtained from batch transesterification of seven oils and methanol. Sodium methylate solution was used as the catalyst. The reaction was carried out in 250-mL baffled flasks placed in a 30×35-cm Lab Line orbital shaker-bath (Barnstead International, Dubuque, Iowa) set at 50°C and at 240 rpm for 60 min. Seven flasks were prepared with reactants for each set of experiments. After 1 h on the shaker bath the contents of each flask was transferred into a separatory funnel and allowed to settle overnight. The two layers were separated and the soaps, if any, at the interface were allowed to remain in the crude glycerol. Each layer was weighed and recorded. The ester layer was heated at 85°C for 1 h to remove the methanol. Samples were taken from the glycerol layer without any further treatment. The methanol content in the glycerol layer was calculated by the difference between the original amount

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The authors are **Joseph Charles Thompson**, Research Support Scientist, and **Bingjun Brian He**, **ASABE Member Engineer**, Assistant Professor, Biological and Agricultural Engineering, University of Idaho, Moscow, Idaho. **Corresponding author:** B. Brian He, 81A James Martin Laboratory, Moscow, ID 83844-2060; phone: 208-885-7435; fax: 208-885-8923; e-mail: bhe@uidaho.edu.

added and the amount measured in the ester layer. The experiments and subsequent tests, with the exception for nutrient analysis, were performed in triplicate.

The formula of reaction mixture make-up was calculated on a molar basis using a 6:1 MeOH to oil ratio and a 0.085:1 NaOCH₃ to oil ratio. The catalyst sodium methylate (also called sodium methoxide, NaOCH₃) was prepared from a 25% weight stock solution (Occidental Petroleum, Los Angeles, Calif.). Approximately 4 L of the catalyst solution was prepared by adding 289.2 g of 25% sodium methylate to 2975 g of methanol. The amount of methanol/catalyst solution for each batch was set at 20 g (19.54 g MeOH + 0.46 g NaOCH₃) to make it easy to dispense. The amount of oil was added according to its molecular weight based on the needed molar ratio (table 1).

Viscosities were conducted on a Koehler Model K-233 oil bath (Koehler Instrument Company, Bohemia, N.Y.) using Cannon-Fenske Routine viscometers (#100 for the glycerol and #150 for the oil samples) according to ASTM standard method (ASTM D445). Viscosity measurements were made at 40°C and reported in centistokes (cs). Heats of combustion were measured on a Parr 1341 Oxygen Bomb Calorimeter (Parr Instrument Company, Moline, Ill.) according to ASTM standard method (ASTM D240).

The Holm Food Research Laboratory at the University of Idaho conducted analyses of crude oil and glycerol samples on macro elements and other components. Macro elements (Ca, K, Mg, Na, P, S) were determined by ICP-AES (Perkin Elmer Optima 3200, Perkin-Elmer Inc., Wellesley, Mass.). Carbon and nitrogen contents were determined by combustion using a LECO CNS Analyzer (LECO Instrumente Plzen, Czech Republic).

RESULTS AND DISCUSSIONS

Analytical tests were conducted on the parent oils and on the crude glycerol as it was recovered after separation from the fuel without further processing. Measurements include fatty acid profile, viscosity, heat of combustion, macro elements, carbon and nitrogen content, and food nutrient

analysis. The fatty acid profiles of the oils were analyzed using GC method (Hammond, 1991) and are summarized in table 2. The measurement was in duplicate and the molecular weight of each oil sample was determined based on the average of its fatty acid profile.

It can be seen from the table that three or four of the fatty acids dominates each of the oils. Canola, soybean, and waste vegetable oils (WVO) are concentrated more heavily on the shorter chain molecules while the mustard, rapeseed, and crambe oils shift toward the longer, heavier ones. This is also reflected by the higher molecular weights.

Table 3 represents the data used to calculate the mass balance of the inputs of table 1 and the two layers obtained after the reactions. The accounting, of the mass balance was indicated as a percent loss or gain of the original inputs. On average, over all of the trials, there was less than 1% loss or gain due to experimental error. Assuming half of the 19.54 g of methanol was used up in the reaction, of the remaining 9.77 g, approximately 46% of it was in the ester layer and 54% went with the glycerol phase. The methanol partition coefficient is an indicator of this split. The higher the coefficient, the more methanol in the glycerol layer. The crude glycerol yield for the six first-use oils ranged from 8.8 to 12.3 g per 100 g of input oil, while the WVO crude glycerol yield, which included more soaps and impurities, averaged around 22 g/100g of oil. The fuel yield, given in grams of fuel per 100 g of input oil, was near 100% for the first-use oils and about 92% for the WVO.

Table 4 shows the average of three measurements of viscosity and heat of combustion for each of the feedstock and its corresponding glycerol. The crude glycerol in this study contained 23.4% to 37.5% methanol when these tests were made, hence, the relatively low viscosity. The viscosity or the heat of combustion did not vary significantly among the oil and glycerol samples except for the WVO. Because the free fatty acid content of the WVO was 4.4%, the single-stage base reaction was compromised by the formation of soaps, which tend to emulsify a portion of the unreacted oils and esters and allow them to be carried into the glycerol. Consequently, the reaction was incomplete and the

Table 1. Formulation and calculation of the experiments.

Feedstock	IdaGold	PacGold	Rapeseed	Canola	Soy	Crambe	WVO ^[a]
Oil (g)	97.1	94.8	99.3	90.5	89.5	100.4	89.0
MeOH (g)	19.54	19.54	19.54	19.54	19.54	19.54	19.54
NaOCH ₃ (g)	0.46	0.46	0.46	0.46	0.46	0.46	0.46
Total	117.1	114.8	119.3	110.5	109.5	120.4	109.0

^[a] Waste vegetable oil.

Table 2. Fatty acid profiles of vegetable oils used in this study.

Fatty Acids	Composition (%wt)													
	IdaGold		PacGold		Rapeseed		Canola		Soybean		Crambe		WVO	
Palmitic (16:0)	2.8	2.7	3.1	3.1	2.8	2.9	4.4	4.5	10.7	10.7	2.0	1.9	18.6	18.8
Stearic (18:0)	1.0	1.0	1.6	1.6	1.0	1.0	1.8	1.8	4.3	4.3	0.9	0.9	6.3	6.3
Oleic (18:1)	24.9	24.6	23.9	23.9	13.6	13.7	60.9	60.5	24.9	24.9	17.8	18.0	40.4	40.5
Linoleic (18:2)	10.4	10.2	21.6	21.6	11.8	11.7	19.1	19.1	51.6	51.6	8.1	8.1	28.0	28.0
Linolenic (18:3)	9.4	9.3	9.9	9.9	7.5	7.5	9.5	9.5	7.3	7.3	4.5	4.5	1.5	1.5
Eicosic (20:1)	10.7	10.7	12.1	12.1	8.6	8.7	1.8	1.8	0.2	0.2	3.7	3.7	-	-
Erucic (22:1)	34.3	35.1	22.1	21.8	47.9	49.1	0.8	1.0	-	-	54.2	53.9	-	-
Avg. MW (kg/kmol)	946.3		924.6		968.5		882.1		872.8		978.5		867.2	

Table 3. Results and calculation of the experiments.

Feedstock	IdaGold	PacGold	Rapeseed	Canola	Soy	Crambe	WVO
Biodiesel layer							
Fuel (g)	98.18 ± 0.23	95.09 ± 0.40	99.39 ± 0.46	90.25 ± 0.09	89.58 ± 0.06	100.76 ± 0.57	81.81 ± 0.63
MeOH (g)	4.72 ± 0.31	4.85 ± 0.24	4.54 ± 0.25	4.63 ± 0.17	4.57 ± 0.07	3.17 ± 0.72	3.86 ± 1.12
Sub-total	102.9 ± 0.31	99.9 ± 0.17	103.9 ± 0.32	94.9 ± 0.08	94.2 ± 0.12	103.9 ± 0.19	85.7 ± 1.19
Glycerol layer							
Glycerol+MeOH+Cat (g)	13.61 ± 0.19	13.27 ± 0.40	15.23 ± 0.20	15.94 ± 0.27	16.16 ± 0.47	17.58 ± 1.07	25.26 ± 0.62
Glycerol (g)	8.56 ± 0.35	8.35 ± 0.16	10.01 ± 0.06	10.80 ± 0.26	10.96 ± 0.48	10.98 ± 0.40	19.35 ± 0.82
Glycerol concn. (% wt)	62.9 ± 2.30	62.9 ± 0.65	65.7 ± 1.19	67.8 ± 1.02	67.8 ± 1.12	62.5 ± 2.16	76.6 ± 4.11
Calculations							
Mass balance (%)	+0.71 ± 0.18	+0.83 ± 0.48	+0.29 ± 0.36	-0.01 ± 0.25	-0.29 ± 0.44	-0.07 ± 0.74	-0.86 ± 0.76
Glycerol yield ^[a] (g/100g)	8.82 ± 0.22	8.81 ± 0.05	10.08 ± 0.07	11.93 ± 0.22	12.25 ± 0.25	10.94 ± 0.23	21.74 ± 1.25
Glycerol yield ^[b] (g/mol)	81.5 ± 3.29	83.4 ± 1.62	97.6 ± 0.54	105.3 ± 2.54	106.9 ± 4.71	107.0 ± 3.87	188.5 ± 7.98
MeOH partition coeff. ^[c]	11.2 ± 2.23	10.5 ± 0.99	10.4 ± 1.33	8.45 ± 0.70	8.89 ± 0.53	17.8 ± 4.10	8.79 ± 5.40
Fuel yield (g/100g) ^[d]	101.1 ± 0.00	100.3 ± 0.00	100.1 ± 0.00	99.7 ± 0.00	100.1 ± 0.00	100.4 ± 0.01	91.9 ± 0.00

^[a] Grams of crude glycerol yielded per 100 g of oil input (g/100g).

^[b] Grams of crude glycerol yielded per mol of oil input (g/mol).

^[c] The partition coefficient of methanol between crude glycerol and fuel layer is calculated as MEOH concentration in fuel (g/g fuel):MEOH concentration in glycerol (g/g glycerol) excluding catalyst NaOCH₃.

^[d] Grams of fuel yielded per 100 g of oil input (g/100g).

ester yield was poor compared to the other oils. The high viscosity and energy content of the glycerol layer from the WVO suggest the presence of soaps and other matters dissolved in it, including glycerides, and esters. The interface between the two layers was treated as part of the crude glycerol, which was consistent with the sampling procedure as for the other oils. There was no significant difference between the heat of combustion of crude glycerol from different feedstock as well, except for the WVO, which was significantly higher for the same reason mentioned above. The heat of combustion of the first use oils averaged 19,560 kJ/kg, approximately 10% higher than that of pure glycerol, which was 17,961 kJ/kg (Lide, 1999). Based on the heat of combustion, a direct thermal use of crude glycerol is an obvious choice. Studies have reported that co-firing of the raw glycerol with other biomass would yield favorable results (Oberberger and Jauschnegg, 1998).

Table 5 shows the analytical results from the macro element screening tests. As expected, the carbon and nitrogen levels in both the oil and the glycerol samples were very homogeneous with the carbon at about 77% in the oil and about 25% in the glycerol, and nitrogen at 0.22% and 0.05%, respectively. The only exception to this observation was that the carbon content in the WVO glycerol sample was 50% higher than that of the average. This was probably due to the presence of soaps and dissolved unreacted glycerides and esters. Sodium, which was quantified as an ion, represents the catalyst (sodium methylate, NaOCH₃) used in the reaction. It is interesting to note the high levels of elements such as calcium and phosphorus in the crambe glycerol compared to the others. This most likely was due to the soil conditions in

North Dakota where the seeds were grown (Montola Growers, Inc., 2004). Additionally, the elements in all of the samples, with the exception of sulfur in the WVO, were concentrated in the glycerol phase. This is a desirable effect, as it would tend to remove the minerals from of the fuel and allow them to be drained off with the glycerol.

From conversations with an animal scientist, it was believed by the researchers that crude glycerol might be used as an animal feed additive or supplement. To investigate this further and to add to the general knowledge about the makeup of crude glycerol, food nutrient analysis was made on the parent oils and their resulting glycerols. One hundred gram samples were sent to Midwest Laboratories in Omaha, Nebraska to have the basic proximates quantified. Due to the large sample size and the high cost, these tests were only run once. Table 6 shows the moisture, protein, fat, ash, carbohydrates in percent and calories for 100 g. The efficacy of using crude glycerol as an animal feed supplement is unclear. A typical materials safety data sheet for crude glycerol found on the Biodiesel Industries web site (2004) deemed it non-toxic, however, there are issues of ingestion that should be worked before a feeding trial is attempted.

As expected, the oils or triglycerides are mostly all fat plus a small amount of protein and carbohydrates with a little dissolved water. The small discrepancies that exist were related to the relative amounts of gums and other impurities in the different crude oils. Nutritional data from the glycerol of the first-use oil samples show that it is mostly carbohydrate and could reasonably be mixed with high protein meal and used as a feed supplement. The WVO glycerol, on the other hand, had a much higher fat content making it more

Table 4. Average viscosity and heat of combustion for feedstock and crude glycerol.

Feedstock	IdaGold ^[a]	PacGold	Rapeseed	Canola	Soybean	Crambe	WVO
Viscosity at 40°C (cs)							
Oil	43.86 ± 0.02	39.03 ± 0.03	46.69 ± 0.0	35.42 ± 0.02	31.45 ± 0.02	50.16 ± 0.02	38.85 ± 0.05
Crude glycerol	8.80 ± 0.02	8.67 ± 0.01	8.50 ± 0.02	8.46 ± 0.01	8.65 ± 0.02	8.50 ± 0.01	26.50 ± 0.19
Heat of combustion (kJ/kg)							
Oil	40,104 ± 93	39,854 ± 100	39,748 ± 67	39,508 ± 93	39,223 ± 67	40,184 ± 123	38,877 ± 126
Crude glycerol	18,600 ± 374	19,428 ± 228	19,721 ± 270	20,510 ± 263	19,627 ± 393	19,472 ± 340	25,176 ± 193

^[a] Data shown are in the format of "average ± standard deviation."

Table 5. Analysis results of macro elements, carbon and nitrogen.

Feedstock	IdaGold	PacGold	Rapeseed	Canola	Soybean	Crambe	WVO
Measurements on oils							
Calcium (ppm)	2.7 ± 0.2	4.9 ± 0.5	15.7 ± 0.6	7.6 ± 0.9	3.1 ± 0.2	47.7 ± 0.6	BDL ^[a]
Potassium (ppm)	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Magnesium (ppm)	0.8 ± 0.0	1.3 ± 0.1	2.1 ± 0.1	1.6 ± 0.0	1.2 ± 0.1	28.3 ± 2.1	BDL
Phosphorus (ppm)	BDL	8.0 ± 0.9	13.0 ± 0.9	10.0 ± 0.8	10.0 ± 0.9	256.7 ± 5.8	BDL
Sulfur (ppm)	26.0 ± 2.2	23.0 ± 1.5	24.0 ± 1.1	22.0 ± 1.8	22.0 ± 0.6	44.0 ± 1.2	28.0 ± 2.7
Sodium (% wt)	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Carbon (% wt)	77.3 ± 0.58	77.0 ± 0.00	77.3 ± 0.58	77.0 ± 0.00	77.0 ± 0.00	77.7 ± 0.58	76.3 ± 0.58
Nitrogen (% wt)	0.22 ± 0.03	0.21 ± 0.02	0.17 ± 0.12	0.22 ± 0.01	0.22 ± 0.06	0.22 ± 0.01	0.25 ± 0.03
Measurements on crude glycerol							
Calcium (ppm)	11.7 ± 2.9	23.0 ± 1.0	24.0 ± 1.7	19.7 ± 1.5	11.0 ± 0.0	163.3 ± 11.6	BDL
Potassium (ppm)	BDL	BDL	BDL	BDL	BDL	216.7 ± 15.3	BDL
Magnesium (ppm)	3.9 ± 1.0	6.6 ± 0.4	4.0 ± 0.3	5.4 ± 0.4	6.8 ± 0.2	126.7 ± 5.8	0.4 ± 0.0
Phosphorus (ppm)	25.3 ± 1.2	48.0 ± 2.0	65.0 ± 2.0	58.7 ± 6.8	53.0 ± 4.6	136.7 ± 57.7	12.0 ± 1.5
Sulfur (ppm)	21.0 ± 2.9	16.0 ± 1.4	21.0 ± 1.0	14.0 ± 1.5	BDL	128.0 ± 7.6	19.0 ± 1.8
Sodium (% wt)	1.17 ± 0.15	1.23 ± 0.12	1.06 ± 0.07	1.07 ± 0.12	1.20 ± 0.10	1.10 ± 0.10	1.40 ± 0.16
Carbon (% wt)	24.0 ± 0.00	24.3 ± 0.58	25.3 ± 0.58	26.3 ± 0.58	26.0 ± 1.00	24.0 ± 0.00	37.7 ± 0.58
Nitrogen (% wt)	0.04 ± 0.02	0.04 ± 0.01	0.05 ± 0.01	0.05 ± 0.01	0.04 ± 0.03	0.06 ± 0.02	0.12 ± 0.01

^[a] BDL indicates values that are below the detection limit for corresponding analytical method. The detection limits in ppm were as follows: calcium – 2, potassium – 40, Magnesium – 0.20, sodium – 80, phosphorus – 5, sulfur – 15, carbon – 200 and nitrogen – 100. Data shown are in the format of “average ± standard deviation.”

Table 6. Food nutrient analysis for oil and crude glycerol samples.

Feedstock	IdaGold	PacGold	Rapeseed	Canola	Soybean	Crambe	WVO
Oils							
Fat (%)	98.2	99.8	98.9	97.3	97.0	98.7	99.9
Carbohydrates (%)	1.36	BDL	1.05	2.36	2.91	1.18	BDL
Protein (%)	0.08	0.07	0.06	0.09	0.09	0.10	0.10
Calories (kJ/kg)	37.2	37.6	37.4	37.1	37.0	37.4	37.7
Ash (%)	BDL	BDL	BDL	BDL	BDL	BDL	BDL
Crude glycerol							
Fat (%)	2.03	1.11	9.74	13.1	7.98	8.08	60.1
Carbohydrates (%)	82.8	83.8	75.5	75.2	76.2	78.6	26.9
Protein (%)	0.14	0.18	0.07	0.06	0.05	0.44	0.23
Calories (kJ/kg)	14.6	14.5	16.3	17.5	15.8	16.3	27.2
Ash (%)	2.80	1.90	0.70	0.65	2.73	0.25	5.50

useful as a fat supplement. This data also confirms the presence of unreacted glycerides in the WVO. The ash contained in crude glycerol is mainly sodium from the catalyst.

CONCLUSIONS

Crude glycerol as a by-product of biodiesel production was recovered from seven different vegetable oils. In the final analysis there was very little variation in the chemical and physical properties. The only exception was the WVO, which was not exactly representative of the others because of the poor conversion rate under the same operating conditions. The viscosity of the crude glycerols prior to any treatment ranged from 8.46 to 8.80 cs and 26.5 cs for the WVO. The heat of combustion of the crude glycerols ranged from 1.86×10^3 to 20.5×10^3 kJ/kg and 25.2×10^3 kJ/kg for the WVO.

A macro element screen was completed on all of the samples. The carbon content averaged about 25% and the metals Ca, K, Mg, Na, P, and S were present in small quantities from 4 to 163 ppm with the exception of sodium, which averaged just over 1%. This was due to the residual sodium methylate catalyst.

The basic proximates for nutrition were measured for each of the samples as well. There was some variation in the data among the neat oils, and the WVO was again the outlier on fat and carbohydrates. Protein levels ranged from 0.06% to 0.44% with crambe being the highest. Fat content ranged from 1% to 13% and carbohydrates ranged from 75% to 83%. The same values for WVO were 60% and 27%, respectively.

When refined to a chemically pure substance, glycerol would be a very valuable by-product of the biodiesel production process with hundreds of uses. Purifying it to that stage, however, is very costly and generally out of the range of economic feasibility for the small to medium sized plants. Alternative uses for the crude glycerol should be explored to make biodiesel more competitive in the growing global market.

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