Biodiesel Fuel Quality of Canola Feedstock Grown on Saline Land

H. Steppuhn[1], T. McDonald[2], R. Dunn[3], M.A. Stumborg[1] and K.C. Falk[4]

[1]Semiarid Prairie Agricultural Research Centre, Agriculture and Agri-Food Canada, P.O. Box 1030, Swift Current, SK, S9H 3X2
[2]Biofuel Technology Centre, Olds College, 4500 – 50th Street, Olds, AB, T4H 1R6
[3]Alberta Agriculture and Rural Development, Agriculture Centre, 5401 – 1st Ave. South, Lethbridge, AB, T1J 4V6
[4] Saskatoon Research Centre, Agriculture and Agri-Food Canada, 107 Science Place, Saskatoon, SK, S7N 0X2

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Abstract

Vegetable oil from canola-grade feedstock ranks among the best in the production of fatty acid methyl esters (FAME or biodiesel). FAME produced from canola-quality oilseed grown on salt-affected lands offer new opportunities for increased production and counter fuel-versus-food concerns provided the biodiesel product meets quality standards. The American Society for Testing and Materials (ASTM) has set the North American fuel quality standards (D6751) for 100% biodiesel (B100) to be blended with petrodiesel fuel. Canola-quality feedstock yield oil low in free fatty acids, acids which are not bonded to parent oil molecules. These free acids may negatively affect diesel engine components, especially at biodiesel oil blends greater than 20%. Also, solid and dissolved impurities, alkali/alkaline earth metals, and oxidation stability are of concern to fuel injection equipment manufacturers. Ultimately, purity, composition, and biodiesel utility depend on the quality of the feedstock supplied. Processing can improve purity, but not composition. Contaminants in biodiesel fuel may include water, sediment, S, P, K, Na, Ca, Mg, carbon residue, and various other constituents in its sulphated ash. Canterra 1818 canola feedstock grown on negligibly, slightly, moderately, and severely salinized soil were crushed and tested for biodiesel fuel quality. All samples yielded biofuel within the ASTM International specifications except for free glycerol in the negligibly-saline sample.

Introduction

The advancing demand for biodiesel feedstock encourages Canadian producers to increase the area seeded to canola-grade oilseed crops. Biodiesel production facilities operating and announced alone will likely require over a million tonnes of canola feedstock per year. A federal requirement for 2% biodiesel in diesel fuel by 2012 could generate a demand for 500 million additional litres of the biofuel per year (Canola Council of Canada 2007). The Council has set an annual production target of 15 million tonnes of canola by 2015 to meet this demand. Industry experts identify two major constraints to the development of the Canadian biodiesel industry: (1) assured supply of oilseed feedstock, and (2) maintenance of the quality of the fuel produced (Kemp 2006).

Oilseed supply

Selected varieties of canola (*Brassica napus* L.) tolerate root-zone salinity equally to that of Harrington barley (*Hordreum vulgatre* L.) (Steppuhn & Raney 2005). This knowledge could serve to encourage producers to introduce or increase canola cropping in the seven million hectares of cultivated lands across the Canadian Prairies identified by Wiebe et al. (2007) and Steppuhn (1996) as slightly and moderately-affected or at risk of being affected by salinity (Figure 1); these lands typically consist of many small-to-medium size areas scattered among negligibly saline areas within individual fields. By including a salt-tolerant canola crop in rotation with barley or other forage crops in salt-affected fields, producers would gain new weed-control options, implement more efficient use of applied fertilizer, and contribute to biofuel feedstock supply without diminishing the production of food products. In addition, over three million ha of sodium-affected (solonetzic) lands might offer further canola cropping opportunities (Cairns and Bowser 1977). Together these 10 million ha of salt-affected lands represent a third of the total cultivated area across the Canadian Prairies and present

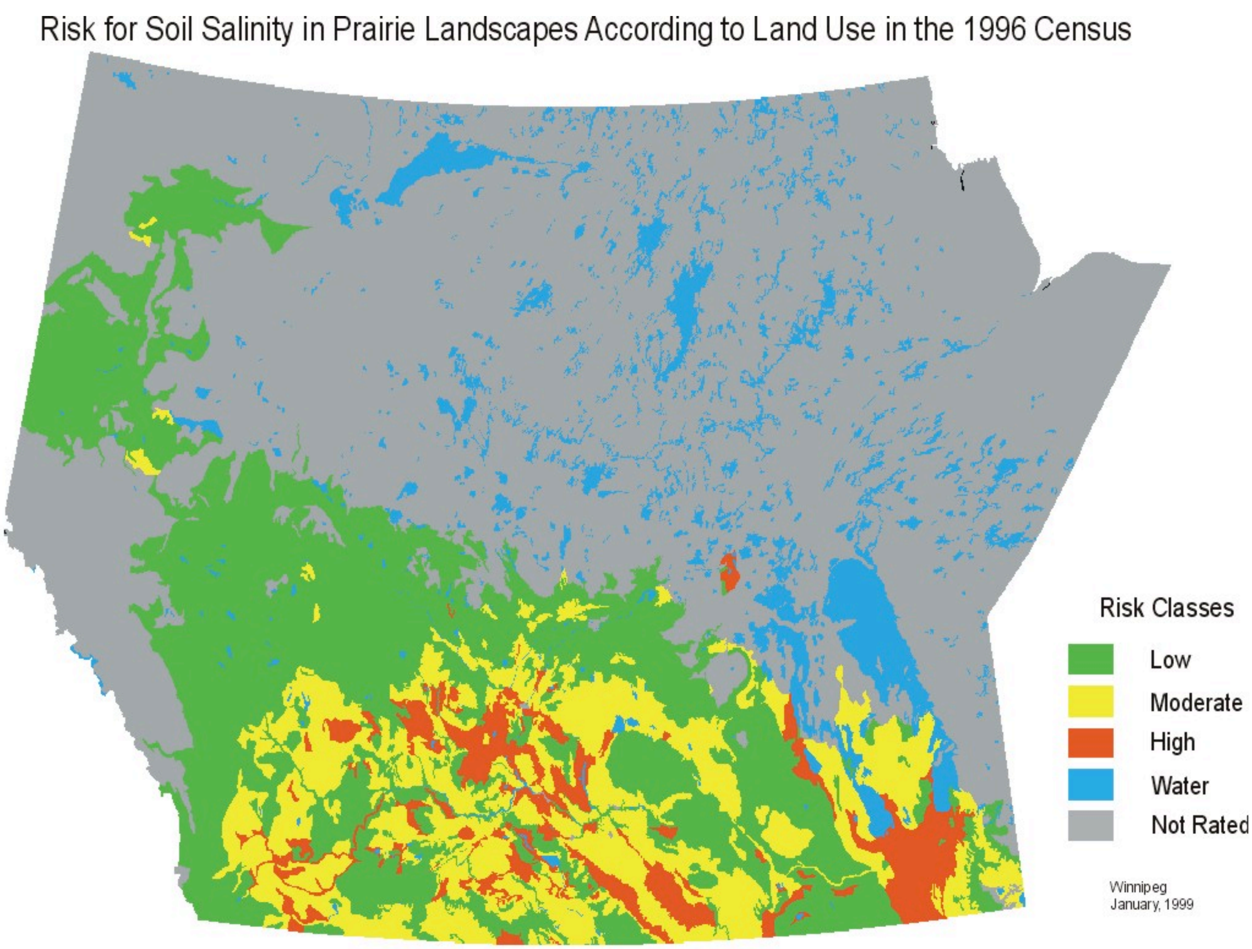

Figure 1. View of the Canadian Prairies and the 1996 soil salinity risk evaluation based on factors which include the existence of root-zone salts (Taken from Wiebe et al. 2007).

opportunities to the biodiesel industry for growing fuel crops in environments detrimental to wheat and many other crops.

Biodiesel fuel quality

Canola oil purity, composition, and biodiesel fuel quality depend on the quality of the oilseed feedstock crushed. Processing can improve purity, but not composition. Also, blending oils from different feedstock grown under varying conditions may work to circumvent some deficits in composition. However, the best assurance for maintaining a uniform supply of biodiesel feedstock of consistently high canola-grade quality rests with knowledge of the biodiesel fuel quality expected from feedstock grown in salt-affected soils. This knowledge serves to identify production limits and contributes to cost-effectiveness and profitability.

According to Kemp (2006), oil from canola-grade feedstock ranks among the best in the production of fatty acid methyl esters (FAME or biodiesel). The American Society for Testing and Materials (ASTM International) has set the North American fuel quality standards (D6751) for 100% biodiesel content (B100). Canola-grade feedstock yield oil low in "free fatty acids," acids which are not bonded to parent oil molecules. These free acids may negatively affect some diesel engine components, especially in blends greater than 20%. Also, solid and dissolved impurities, alkali/alkaline earth metals, and oxidation stability are of concern to the fuel injection equipment manufacturers.

The aim of this study is to evaluate canola biodiesel quality from feedstock grown in soil affected by sulphate salinity. Jia and Xu (2006) suggested that growing biodiesel feedstock on saline-alkaline lands would assist in maintaining non-saline lands for food production. But, they provided no examples of where this was currently being practiced, nor did they identify any evaluations of the biodiesel quality of the fuel produced from salt-affected soils. To our knowledge, neither the practice of growing biodiesel fuel in saline environments nor checks on the biodiesel quality of the resulting fuel have been reported. The preliminary study described herein was conducted to obtain insight into the approximate limits in root-zone salinity associated with biodiesel fuel produced from canola grown in sulphate salt-affected soil. The specific objectives of the study were to determine percent oil recovery and the standard quality of the pure B100 biodiesel fuels derived from canola grown in soils rated as negligible, slight, moderate, and severe with respect to root-zone salinity. These objectives identify feedstock production limits related to biodiesel fuel quality.

Materials and Methods

Canola feedstock test crop

In 2007, Mr. Ron Svanes grew a commercial canola crop (variety: Canterra 1818) in a salinity-affected field near Carmangay, Alberta. Using a surface electromagnetic induction propagator/sensor and a differential field geo-positioning system, Alberta Agriculture and Rural Development technical staff estimated and surveyed the root-zone salinity across the field following methods adopted from Wollenhaupt et al. (1986). According to the survey, the canola field contained saline soils classified by the United States Salinity Laboratory Staff (1954) as negligible ($EC_e<2$ dS m^{-1}), slight ($2\leq EC_e<4$ dS m^{-1}), moderate ($4\leq EC_e<8$ dS m^{-1}), and severe

($EC_e \geq 8$ dS m^{-1}). Three replicate areas per salinity class were identified, marked, and harvested separately under guidance of the Alberta Agriculture and Rural Development technical specialists. Threshing was completed in the field, but precise grain yields could not be obtained. Twelve oilseed samples (representing the four salinity classes each replicated from three field locations) were air-dried and kept in protected, non-heated storage first at Lethbridge, Alberta and later at Oyen, Alberta. In January of 2008, the samples were transported to Olds, Alberta, and again stored in a cool environment until they were processed.

Feedstock crushing and processing

The new biodiesel pilot plant at Olds College in Alberta forms part of the School of Innovation Biofuel Technology Centre. The canola oilseed feedstock samples from the test field at Carmangay were crushed, carefully processed, and analyzed in the Centre's laboratory facilities utilizing bench-size equipment and procedures. The feedstock samples representing each of the four saline conditions were obtained from three replicate locations for each condition in the test field, weighed in equal amounts, and bulked into one sample per salinity class and crushed.

The four composite Carmangay canola oilseed samples, arrayed by field salinity levels, were measured for initial water content using a Sartorius MA100 Analyzer. The technique involved recording the change in mass with time while each sample was subjected to a temperature of 105^{o}C. To increase crushing efficiency and make the crushed oil less viscous, each oilseed sample was tempered by adding sufficient water to reach approximately 8% in water content before crushing. Each oilseed sample was crushed using a Komet Seed Oil Extractor. The temperature of the oilseed was measured in the hopper as the samples moved into the press and again in the expressed oil leaving the extractor. Temperatures of both the oilseed and the oil were maintained below 55^{o}C to prevent oil degradation. The crushed oil was cleansed using a Beckman Coulter J6-M1 centrifuge to remove the heavier solid particles remaining in the oil after the crushing process. The remaining oil was allowed to stand and further settle in order to remove any unwanted constituents prior to transesterification.

Transesterification, methyl ester B100 production

Transesterification and washing processes convert crushed canola oil to 100% biodiesel fuel (B100). The expressed canola oil is transformed into fatty acid methyl esters (FAME), the raw biodiesel fuel, by the addition of methanol in the presence of sodium methoxide, a catalyst (Kemp 2006). The result is a separable mixture of FAME and glycerol:

$$10 \text{ feedstock oil} + 1 \text{ methanol} \ \underline{\rightarrow CH_3ONa.2CH_3OH \rightarrow} \ 10 \text{ FAME} + 1 \text{ glycerol} \qquad [1]$$

Separation of the glycerol from the FAME is accomplished by allowing the heavier glycerol to settle to the bottom of the reaction vessel and the fatty acid methyl esters to be decanted from the top. At this point, the raw biodiesel FAME contains various contaminants including methanol, catalyst reactants, glycerol, soap, gums, etc. Adding water to the raw biodiesel "washes" these contaminants from the raw FAME rendering a more refined B100 product.

Samples of FAME were produced from each Carmangay oilseed test sample over a period of three days. Each sample received the same percentage of catalyst and methanol (6% and 24% by volume of oil, respectively). All processes were completed at the Olds College Biofuel Technology Centre using bench-top hot plates and glassware. The test oils were measured and heated to 60°C in uncapped 1000L flasks with stir bars to remove any excess water prior to adding the catalyst and methanol. At 60°C, catalyst and methanol were added to the oil flask and covered with a foil cap. Temperatures were maintained above 60°C for 45 minutes and the mixture stirred vigorously during that time. Once the reaction was completed, the raw product was removed from the hot plate and the stir bar withdrawn. The mixture was then poured into a separatory flask to allow the glycerol to settle by gravity. The glycerol was removed and its volume recorded. The methyl ester FAME was gravity "washed" with de-ionised water while the flask was gently shaken and then left standing to allow the contents to settle. Four separate washings to remove excess methanol and impurities were completed. The final washed product was dried by re-heating to 60°C until all water dissipated. Lastly, vacuum filtration was applied to remove any excess soap, gums, or other impurities from the final B100 product.

Biodiesel fuel quality testing

A series of biodiesel fuel quality evaluations, following the American Society for Testing and Materials approved-protocols (ASTM International 2007), was employed to evaluate the merits of salinity-influenced canola oilseed feedstock:

ASTM D2500 Cloud point This is the temperature at which wax crystals begin forming as the FAME is cooled. Fuels which are operated below their cloud point are likely to cause filter plugging and subsequent fuel starvation of the engine.

ASTM D4530 Carbon residue (100% sample) A fuel sample is combusted and the remains constitute the carbon residue; excessive levels of glycerol are the likely cause of a test failure.

ASTM D5185 (EN 14538, European standard) Metals (sodium, potassium, calcium, & magnesium) Saline soil solutions commonly contain metal cations, such as Na^{+}, K^{+}, Ca^{++}, and Mg^{++}; these tests indicate the degree of contamination from these ions in the biodiesel fuel produced from salt-affected soils.

ASTM D4951 Phosphorus This is a measure of contaminants resulting from the refining process for feedstock oils as well as from the use of phosphoric acid during the production process. Vegetable oil feedstock should have very low levels of phosphorous contamination.

ASTM D664A Acid number The acid number describes the free fatty acids in the FAME, which are known to lead to corrosion. Water in the fuel may be symptomatic of a high reading; the value is calculated by titrating a one-gram sample of FAME with a quantity of potassium hydroxide, and is measured in milligrams of the KOH base.

ASTM D130 Copper-strip corrosion Fuels which have high levels of free fatty acids will cause specially polished strips of copper to corrode when subjected to elevated temperatures; the degree of corrosion is compared to a series of reference strips to determine a pass/fail condition.

ASTM D93A Flash point FAME fuels are classified as non-flammable as their flash or ignition points (at atmospheric pressures) are above 130^{o}C. This test is primarily a measure of residual alcohol which likely results from incomplete methanol recovery and/or washing of the raw FAME. Methanol is highly toxic, flammable, and can be easily inhaled because of its low vapour pressure.

ASTM D6584 Free glycerol Free glycerol refers to suspended glycerol compounds that remain in the raw FAME as a result of improper washing. Excessive glycerol causes carbon deposits on fuel injection components, engine valves, valve seats, pistons, and rings, which leads to degraded engine performance and eventual engine failure. Free glycerol forms sludge in fuel storage tanks, resulting in plugged filters and engine starvation.

ASTM D6584 Total glycerol This is the sum of the free (suspended) glycerol and the bonded glycerol present in the mono-, di-, and tri-glycerides in the FAME; elevated levels of total glycerol result from incomplete reaction of the feedstock oils during transesterification and will compound the problems noted under "free glycerol."

ASTM D445 Kinematic viscosity This is a measure of a fluid's resistance to flow under gravitational forces. Highly viscous fluids will become less resistant to flow when heated.

ASTM D874 Sulphated ash This test indicates the quality of metallic residue left over from the catalyst used in the transesterification process. A sample of FAME is combusted and the residue is treated and massed to determine the residual non-combustible mineral ash.

ASTM D5453 Total sulphur The total sulphur test determines the amount of sulphur contained in the FAME. Reducing the sulphur content of all fuels reduces the quantity of sulphur compounds released to the lower atmosphere.

ASTM D2709 Water content (including that dissolved) Free or bonded water in FAME fuels will lead to the formation of free fatty acids and corrosion of engine and fuel storage tanks and will also promote microbial growth.

ASTM D2709 Sediment Sediment can plug fuel filters and, if the sediment is small enough to pass through filters, can abrade fuel injection and other high-tolerance engine components.

ASTM D1160 Vacuum distillation end point This is the temperature under conditions of reduced pressure at which 90% of the fuel sample will be distilled, allowing a determination of the makeup of the FAME.

Oxidation stability EN 14112 (European standard) FAME fuels are oxidized by atmospheric oxygen and therefore have a relatively limited storage life. Also, the oxidation products could damage vehicle engines. This is an accelerated oxidation test where a sample is held in a sealed reaction tube at a constant temperature of 110^{o}C while a continuous flow of air is passed through the sample. With time, the air transports the oxidation products to a measuring vessel containing distilled water as an absorption solution. An increase in electrical conductivity of this water indicates the presence of secondary oxidation products (mainly formic and acetic acids) signalling a loss of stability. The time it takes for the conductivity to increase quantifies the oxidation stability of the fuel. The above tests were conducted by Maxxam Analytics, Inc. in Edmonton, Alberta.

The **cetane number (ASTM D6890)** test was performed by the Saskatchewan Research Council Biofuels Testing Centre in Regina, Saskatchewan. The cetane number is a direct indication of the ignitability of the fuel. A measured quantity of the test fuel powers a specially developed compression ignition engine or instrumented simulation apparatus. The fuel's performance is compared to calibrated standards measured according to reference values or numbers.

Because of fuel quality testing costs, the procedure following oil extraction and B100 biodiesel production in this study was to array the final biodiesel products into two groups in preparation for quality tests. The B100 biodiesel produced from the negligible and severe salinity feedstock were submitted separately for analyses to which the complete series of 18 ASTM D6751-07 evaluations were applied. The B100 products from the slight and moderate salinity feedstock were submitted for only seven of the 18 evaluations; these seven were selected as those most appropriate and important for salinity-related oilseed and biofuel production: sulphated ash, sulphur content, phosphorus content, cloud point, micro-carbon residue, and metal cations: (Na + K) and (Ca + Mg).

Results

Canola oil recovery

The initial water content of the four test oilseed samples from feedstock grown in soil classified as negligibly, slightly, moderately, and severely saline, ranged from 3.3 to 4.3% (Table 1). De-ionized water was added to each sample bringing the water percentages to 8±1%. The oilseed samples were then crushed to obtain canola oil and meal. The temperature of the oilseed feedstock when entering the crusher-extractor measured within 5°C (41 – 46°C) for all the samples (Table 1). The average temperature of the expressed canola oil ranged from 40 through 47°C. At these temperatures, degradation of the oil would assuredly have been lessened.

The initial mass of each test oilseed sample measured 5.65±0.05 g just before entering the crusher/extractor (Table 2). Upon exiting the crusher, the raw canola oil was massed and its recovery calculated as a percent of the initial seed mass (Table 2). The oil recovered from severe-salinity feedstock measured somewhat less than the other three. Based on the centrifuged oil, recovery equalled 30.5, 33.4, 34.9, and 34.8% for the severe, moderate, slight, and negligible salinity test stock, respectively. The salinity-grown oil samples required substantially more settling and centrifugation than other canola oil samples previously processed at Olds College.

Table 1. Water content and temperature of oilseed and raw canola oil during crushing associated with feedstock grown in soil of the indicated salinity.

Salinity	Water content Initial	Water content Tempered*	Temperature Seed	Temperature Oil
	(%)	(%)	(°C)	(°C)
Severe	4.26	8.44	41-46	42-47
Moderate	4.20	7.42	42-46	40-47
Slight	4.02	7.02	42-46	39-47
Negligible	3.32	8.26	43-46	39-47

* "Tempered" refers to the addition of water to the oil to increase crushing efficiency.

Table 2. Mass of the crushed oil and meal and the percent oil recovered from canola feedstock grown in soil of the indicated salinity.

Salinity	Mass Initial seed	Mass Oil	Mass Meal	Oil recovered	Centrifuged oil
	(g)	(g)	(g)	(%)	(g)
Severe	5.70	1.79	3.91	31.3	1.74
Moderate	5.65	1.94	3.71	34.4	1.89
Slight	5.65	2.02	3.63	35.8	1.97
Negligible	5.60	1.99	3.61	36.1	1.95

Fuel quality tests

Transesterification was repeated with three separate sets of the oilseed feedstock grown on the salt-affected soils (Table 3). The sets involved either (1) different quantities of input constituents (but with the Eq. 1 proportions maintained) in the esterification and washing processes, or (2) selected repetition. The biodiesel B100 produced was bright orange in color rather than golden, and the glycerol was green, rather than dark brown.

Table 3. Inputs and results for the reactions and selected component volumes for three sets of salinity-influenced canola feedstock.

Set	Salinity	Raw oil	Catalyst	Methanol	Glycerol	Gums visible	Glycerol	Wash water
		(ml)	(ml)	(ml)	(ml)		(%)	(ml)
1	Severe	300	18	72	42	No	14	500
1	Moderate	300	18	72	70	Yes	23	500
1	Slight	300	18	72	37	No	12	500
1	Negligible	300	18	72	51	Yes	17	500
2	Severe	500	30	120	69	Yes	14	800
2	Moderate	500	30	120	75	Yes	15	800
2	Slight	500	30	120	73	Yes	15	800
2	Negligible	500	30	120	80	No	16	800
3	Severe A	500	30	120	71	No	14	800
3	Severe B	500	30	120	78	Yes	16	800
3	Negligible A	500	30	120	82	Yes	16	800
3	Negligible B	500	30	120	69	Yes	14	800

The ASTM D6751-07 series involved some 18 tests or evaluations (Table 4). The severely and negligibly salinity-influenced B100 fuels were each evaluated in all 18 tests. No significant differences in any of the evaluations were detected between these two test fuels, except for the free glycerol content (0.058% for the negligible and 0.017% for the severe). Except for the free glycerol measured in the negligibly saline sample (0.038% above the ASTM specification), both fuels met all of the D6751-07 specifications classifying both as acceptable in B100 quality. The elevated free glycerol percentage was likely caused by incomplete washing. The moderately and slightly salinity-influenced B100 fuel was subjected to only seven of the 18 evaluations. In these, the results neither deviated significantly nor trended differently among the fuels for the three samples from feedstock grown in salinity-affected environments; the test values all fell within the acceptable D6751-07 specifications for North American B100 biodiesel fuel (Table 4).

Table 4. Comparisons of ASTM D7651 test results to ASTM specifications for the salinity-related B100 biodiesel samples.

Test variable	ASTM method	Salinity Severe	Moderate	Slight	Neg.*	Units	ASTM D6751 specification
Flash point	D93A	138.0	NT**	NT	138.0	°C	130 min
Acid number	D664A	0.03	NT	NT	0.03	mgKOH/g	0.5 max
Cloud point	D2500	-3	-3	-4	-4	°C	
Water & sediment	D2709	0.0080	NT	NT	0.0050	% vol	0.050 max
Free glycerol	D6584	0.017	NT	NT	0.058	% Mass	0.020
Total glycerol	D6584	0.097	NT	NT	0.13	% Mass	0.240
Sulphated ash	D874	<0.001	<0.001	<0.001	0.001	% Mass	0.020
Total sulphur	D5453	2.5	1.9	1.5	2.2	ppm	15
Copper strip corrosion	D130	1A	NT	NT	1A	No.	3A max
Cetane number	D6890	56.7	NT	NT	56.3	No.	47 min
Carbon residue	D4530	<0.010	<0.010	<0.010	<0.010	% Mass	0.050 max
Kinematic viscosity	D445	4.492	NT	NT	4.548	Mm^2/sec	1.9-6.0
Oxidation stability	EN14112	5.5	NT	NT	4.8	hours	3 hours min
Phosphorus	D4951	<0.5	<0.5	<0.5	<0.5	mg/L	10
Sodium + Potassium	D5185	<1	<1	<1	<1	mg/L	5 ppm
Calcium + Magnesium	D5185	<1	<1	<1	<1	mg/L	5 ppm
Absolute density @15°C	D4052	881.8	NT	NT	881.9	kg/m^3	
Distillation End Point Temp. (90% recovery)	D1160	349	NT	NT	359	°C	360 Max

* Neg. = Negligible salinity
** NT = Not tested

Discussion

Assurance of biodiesel quality was achieved by testing selected properties and characteristics of the B100 product fuel. In Canada, the American Society for Testing and Materials (ASTM International) D6751 is the quality standard applied to biodiesel (B100) used in a blend with petrodiesel. All the B100 biodiesel FAME produced from saline soil samples conformed to the ASTM specifications in every test performed, indicating that the chemical reaction was complete and that the level of salinity in the environment where the canola feedstock was produced did not adversely impact the quality of biodiesel product derived from the raw canola oil. The importance of this finding rests with the indication that biodiesel canola fuel produced from saline soil can meet the North American standard for acceptable quality. This implies that canola grown on saline land will yield market-grade biodiesel fuel.

The results presented herein are only preliminary. Only one canola crop from one field was sampled. Furthermore, the limited funds available for this study prohibited a full-scale evaluation of all the D6751 evaluations in all the treatment samples. Insufficient funding also limited the number of replicate samples that could be tested. This restricted the application of statistical analyses.

The results also indicate that oil recovery for Canterra 1818 canola suffered a 13% decline when the feedstock was grown in severely saline soil. Oil quantities recovered from this feedstock grown in moderately and slightly saline soil appeared not to have declined in comparison to crops grown on negligibly-saline soils. This agrees with the earlier results obtained by Steppuhn and Raney (2005) with InVigor 2573 and Hyola 401 canola.

Conclusions

Four oilseed feedstock samples from a 2007 canola field near Carmangay, Alberta representing soil root zones rated as negligibly, slightly, moderately and severely salinized were pressed to recover oil for biodiesel production. Oil recovery was favourable (within 31-36%) for all samples. Oilseed grown under conditions of the greatest salinity had the lowest oil recovery (31%). The raw oils were successfully converted to a B100 biofuel fuel of bright orange color and the glycerol produced showed a greenish tint. The quality of the biodiesel produced from all four oilseed samples was consistently within the ASTM International D6751 specifications (except for excessive free glycerol (0.058% compared to 0.020%) in the fuel produced from the negligibly-saline soil), indicating that there was no reduction in quality associated with canola feedstock grown in saline environments. This finding suggests the acceptance of the expanded use of saline lands for biofuel production.

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