

LOW LINOLENIC CANOLA OIL PILOT SCALE TESTING

J.Mikle^{1/}, R.A.Carr^{2/}, N.A.Eskin^{3/}

- 1/ POS Pilot Plant Corporation, Saskatoon, Canada,
2/ POS Pilot Plant Corporation, Saskatoon, Canada,
3/ University of Manitoba, Winnipeg, Canada.

ABSTRACT

Commercially available canola seeds, such as Tobin and Westar have a linolenic acid content of approximately 10%. The presence of this readily oxidized fatty acid is generally considered to be detrimental to the oxidative stability of canola oil and canola oil derived products. Canadian canola breeders have developed a canola strain containing low levels of linolenic acid (approximately 3%).

Comparative testing of the experimental crop versus commercially available Westar was performed in pilot scale. The experimental work included testing of the responsiveness to conventional processing, frying performance of a liquid shortening, monitoring the quality deterioration caused by frying and identifying the breakdown artifacts.

Processing the low linolenic canola oil (LL canola) did not reveal any significant differences in the analytical responses when compared with the control (Westar canola oil). Hydrogenation tests indicated that the LL canola oil required less time than the control oil to reach the desired endpoint Iodine Value (IV), due to its lower starting IV and favourable fatty acid composition.

In frying tests, both the LL canola oil and Westar control had a longer "fry life" than the second control (soybean oil). Oxidative stability of slightly hydrogenated oils (20% solids at 50°F) was checked by the Active Oxygen Method (AOM). AOM hours (Bomb) of 87, 74 and 24 were recorded for LL canola, Westar and soybean oil, respectively. Deodorized salad oil (unhydrogenated) samples recorded AOM hours (Bomb) of 32 and 24 for LL canola and Westar oils, respectively. After 240 hours of frying, the LL canola oil had a free fatty acid (FFA) content of 0.78% and red colour of 11.0 compared to Westar's FFA % of 1.2 and red colour of 20.0. These experimental data indicate that LL canola is a promising crop for both vegetable oil processors and end-users.

INTRODUCTION

B.R. Stefansson, P.B.E. McVetty, S.R. Rimmer and R. Scarth, of the Dept. of Plant Science, University of Manitoba, developed and produced a small quantity of Canola Strain S81-2716, which has a low linolenic acid content oil. The CCC requested POS Corporation to execute a pilot plant test crush and then evaluate the performance of the components produced.

The purpose of the test was to identify any particularities related to the processing of low linolenic type canola seed (LL canola), to evaluate the finished products derived from the oil component and to identify potential economic advantages that could benefit processors and users of end products alike.

The comparative testing of the experimental LL canola crop versus commercially available Westar, used as the control, included; 1) Responsiveness to processing/testing, 2) Frying performance of liquid shortenings, 3) Monitoring of the quality deterioration caused by extended frying and 4) Identification of the breakdown artifacts in the exhausted frying fats.

Processing unit operations performed on the experimental crop and the control included; 1) Prepressing, 2) Solvent extraction, 3) Degumming, 4) Caustic refining, 5) Bleaching, 6) Hydrogenation and 7) Deodorization.

During each processing and evaluation phase, the associated quality indicators were routinely monitored and recorded. The hydrogenated, deodorized stocks were subjected to a frying test in competition with a soybean oil stock of the same solids content at 50°F, as control. The exhausted frying oils were all analyzed for polar compound and polymer content in addition to the daily checks of free fatty acids content, colour and foam height.

EXPERIMENTAL WORK

1) Materials

The analytical data for the LL canola and Westar control is presented on Slide A. Oil contents of the two seed types are equal, at approximately 42%. The low linolenic variety shows 3.1% C18:3 vs. 11.5% present in the Westar. For the meal, protein content is higher and glucosinolate level is lower in the new variety.

The prepressed and solvent extracted oils were combined into a composited crude. The analytical data descriptive of these materials (LL canola and Westar crude is presented in Slide B. For all practical purposes, the two crudes are equivalent in quality except for their differing iodine values, which are fully supported by the Fatty Acid Compositions (FAC) presented on Slide C.

2) Unit Processes

All processing of the LL canola and Westar control seeds were performed in pilot plant scale equipment available at POS Corporation, 118 Veterinary Road, Saskatoon, SK, S7N 2R4, Canada.

Flaking/Cooking/Prepressing

The gap of the POS flaking mill (Turner) rated for 100-300 kg/hr, was set to 0.23 mm. Flakes with 0.25-0.29 mm thickness were generated. The cooker-prepress (Simon Rosedown; rated for 90-170 kg/hr) was fed at ca. 125 kg/hr. The top tray temperature was 67-73°C, the bottom tray was 87-93°C.

Solvent Extraction

The cake from the prepress operation was subjected to solvent extraction in the Crown unit rated to 25-150 kg/hr with a retention time of 60 minutes. The meal was sparged in the top tray of the Desolventizer-Toaster with 5% (feed rate based) steam.

Degumming, Caustic Refining, Water-washing

Water/acid degumming and caustic refining were combined into one operation. First 0.15% phosphoric acid (concentrated) was added and held under agitation for 30 minutes, followed by addition of 2.5% water. Thirty minutes of contact time was allowed with water, then the caustic treat was added. The caustic was allowed 15 minutes contact time with the oil, then separation was started.

The Westfalia (SA-14) deslugger centrifuge (rated capacity 370-1000 kg/hr) was used, with a throughput set at ca. 200 kg/hr.

Adsorptive Bleaching

This step was carried out in a 500 L reactor. Citric acid of 50% concentration and activated clay were added at 0.04% and 1.0% levels respectively. The mixture was heated to 120°C and maintained between 118-122°C for 30 minutes under vacuum (ca. 50 mm Hg residual pressure). After 30 minutes elapsed contact time, the cooling sequence was initiated. When the oil temperature reached 65°C, the vacuum was broken with inert gas and the filtration sequence initiated to remove the spent adsorbent.

Partial Hydrogenation

An 80 lb size pilot convertor, equipped with turbine type impeller and turning at 190 rpm was used. The gas was admitted when temperature reached 160°C in the slurry. The catalyst used was type HK4 from Harshaw, added at 0.05 (% Ni) level. Gas pressure was maintained at 25 psig.

Deodorization

A 100 lb, batch type, stainless steel deodorizer was used to deodorize the salad oil and partially hydrogenated stocks, under the following conditions:

Sparge steam rate: 4-6 lbs/hr
Vacuum: 2-4 mm Hg absolute
Low cycle time: 2 hrs
Temperature: 176°C
High cycle time: 2 hrs
Temperature: 232°C

3) Frying Test

Deep Fat Frying Test (French Fried Potatoes):

Definition: A method to determine the acceptability of a fat for commercial frying purposes by comparative means.

Scope: Applicable to any shortening or frying fat.

Normal Length of Test: Until Continuous foam at 360°F or 182°C.

Evaluation Criteria

Observe and record frying appearance after initial moisture has been driven off (approximately 1 to 1 1/2 minutes). Examples: off-color, odor, smoking, foaming, etc.

The fry test is terminated when persistent foam exists which is confirmed by a foam test height of at least 1 inch.

RESULTS & DISCUSSIONS

Unit Processes

The flaking, cooking, prepressing operations were staged and conducted as per standard procedures. No unusual or unexpected problems were observed while processing the experimental crop (LL canola). The same observation is valid for the solvent extraction step. The composited crude oils from the control and the experimental crop are of equivalent quality, (Slide B) with the exception of the I.V. and FAC (Slide C).

In the meal derived from the experimental crop the glucosinolates level was low (Slide D), and this feature should increase the acceptance of the product. The analytical tracking of the degumming, refining and bleaching operations is presented in Slide E. Both materials responded well to processing to yield refined, bleached, (R,B) oils of practically equivalent quality. Prior to bleaching both oils had the same colour, similar chlorophyll and trace metal levels. Both were bleached with the

same amount of clay (approximately 1.0%) under identical conditions.

During the pilot scale bleaching, the following Lovibond colour values were recorded:

	Low Linolenic	Westar
Refined colour (1" cell)	70.OY 3.7R	70.OY 3.7R
Bleached colour (5 1/4" cell)	35.OY 2.9R	35.OY 2.7R

The refined oil was too dark to read in 5 1/4" cell, the bleached oil was too light to read in 1" cell. However, it was observed that both materials responded well to standard bleaching. The experimental crop did not exhibit any properties that would have warranted bleaching optimization studies, eg. attempts to use less clay to get colour identical to that of standard bleached control/Westar.

Partial hydrogenation was the first unit operation to yield qualitatively different products from the seemingly identical materials. In order to view the data obtained in this process in the proper context, it should be remembered that the (RB) Westar contains 3.5 ppm sulfur, while the experimental material only had 0.5 ppm (Slide E).

The analytical indicators of the partially hydrogenated materials, along with a soybean oil based control, are listed in Slide F. The LL canola, Westar and soy controls were all hydrogenated to 2.5-3.5% solids at 50°F, yet the AOM stabilities observed are quite different. The LL canola oil produced an outstanding 87 hours AOM, compared to 74 hours for Westar. Both canola stocks are far superior when compared to the similar solids content soybean oil based stock, with its 24 hours AOM stability. The LL canola stock is clearly a superior product, with only 16.3% transisomer content and 12.0% linoleic acid content in its composition. The lower linolenic content is most probably the explanation for the additional 13 hours of AOM stability, as compared to the Westar stock. Slide G contains the processing parameters for the pilot scale tests (9 through 11) and for each bench scale test with the low linolenic and Westar oils.

Comparing the conditions for tests 9 and 10 leads one to observe that the Westar required 11.5 minutes longer to reach the same end point I.V. (88-89) as the LL canola. In other words, a plant using LL canola exclusively could increase its hydro capacity by ca. 39% (valid for I.V. 88 stock example, where drop tanks and additional filtration capacity are available), without installing additional hardware.

In addition to production capacity increase, the LL canola oil offers yet another advantage, reduced hydrogen consumption. The LL canola oil needs 27% less hydrogen gas than Westar. The bench scale tests (1 through 8) were performed in order to collect information on the behavior of the two oils when preparing harder stocks (I.V. ca. 64).

The following observations were made during these experiments (Slide G).

- a) The I.V. drop per minute is comparable for the LL canola and the Westar control oils, when the processing conditions are similar.
- b) The LL canola will need less time on gas to reach the end point. This reinforces the observation made during the preparation of the soft stocks (I.V. ca. 88).
- c) When adiabatic conditions are applied (heat rise present) along with standard gassing temperatures (tests 2c, 2d) the end products are nearly identical (FAC, MMP°C).
- d) With standard catalyst loading and isothermic conditions, along with high (195°C) gassing temperature, the LL canola oil will yield a harder product, at similar I.V. endpoint (Slide H) (tests 2g and 2h). The harder stock is the result of the stearic content, which is ca. 6% higher in the LL canola oil based stock.

The deodorization of the salad oil samples did not reveal observable differences between the two stocks. Pertinent analytical data is presented in Slide I. The oxidative stability of the low linolenic oil was found to be 32 hours, to be compared with 24 hours found for the control. This fact can be equated with a measurable economic advantage in favour of the users. Thirty three percent improvement in the oxidative stability is significant, by any standard.

Finished Products - Salad & Frying Oils

The previous paragraph concerned itself with the performance of the materials through the different processing steps. Deodorization was last in the sequence to be applied to the end products (I.V. 88 for the liquid frying oil and 111.3 and 120.4 respectively, for the LL canola and Westar salad oils). Data regarding the salad oil finished products (low linolenic and Westar) are tabulated and presented in Slide I.

Schaal oven test results, TBA, TVCC, odor intensity and acceptability and Cold Test data are presented in Slide J.

Frying evaluation - finished products

The oils subjected to this evaluation are fully characterized in Slide F. The scope of the test was expanded beyond comparing the LL canola and Westar oils. The two hydrogenated canola oils were evaluated in a frying performance testing procedure alongside a 110 I.V. soybean oil. The method measures relative breakdown differences by comparing rates of colour, free fatty acid and foam development under high temperature usage conditions.

Fresh sliced potatoes were fried three times in each 24 hour period in fat held at 360°F ± 5°F. The oil was sampled daily and the remaining fat was not replenished to maintain the 14 pound level. Daily analysis consisted of Lovibond Red colour (3" mark), free fatty acid percentage,

and foam measurement.

The comparison of data (Slide K), suggests that both canola oils have improved resistance to foam development when compared to the 110 I.V. SBO control. The LL canola variety exhibited highest resistance to Lovibond Red colour rise and free fatty acid formation, when compared to the Westar canola oil. The attached graphs (Slides L, M) illustrate the relative resistance profiles.

Peroxide value of the used frying oil was not run as this analytical point is more dependent upon the time of sampling than upon the stability of the source oil.

Analytical evaluation of the exhausted frying oils

Polar compounds, total triglycerides and polymerized compounds were analyzed by HPLC.

Slide N summarizes the peak area in m AU's and area percent of the polar compounds, total triglycerides and polymerized compounds present in each sample.

The results in Slide N are calculated on the assumption that the coefficient of extinction for triglycerides, polar compounds and polymers is the same.

The LL canola and Westar oils both had ca. the same ratio of triglyceride to polymer in both the RBD oils and the hydrogenated and deodorized oils (Slide N). After frying to the one inch foam end point, the soybean oil had developed the least amount of polar compound, while the LL canola had developed the most. Westar developed the least amount of polymer, while the soybean and the LL canola both had developed about 20% more than was present in the Westar.

Polar compounds in all three products were well within the acceptable definitions used in France. They were below the 20% limit, above which oil is considered as altered, as well as the 25% level, above which the oil is considered as unfit for consumption.

CONCLUSIONS

- 1) Experimental LL canola processed quite similarly to the Westar control.
- 2) Major improvements in hydrogenation capacity and reductions, hydrogen usage can be obtained when LL canola is hydrogenated to identical solids content endpoints for Westar canola, and more so for soybean oil.
- 3) LL canola provides significant end-user benefits from improved salad/cooking oils AOM stability, liquid frying oil AOM stability and improved "fry life" for frying shortenings.