acids, iodine value and perhaps neutral oil loss, with little or no sample preparation. This technique holds considerable promise but must be thoroughly evaluated. As processors increase their production volumes, they will need to become more sophisticated in their process quality control in order to remain competitive. This means more samples analyzed and probably more instrumental analysis, using the classical wet methods as reference methods for calibrating instruments, rather than as routine methods.

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Qualih/Control in a Canola Crushing Plant

STEWART J. CAMPBELL, United Oilseed Products Ltd., P.O. Box 1620, Lloydminster, Alberta, Canada S9V 1K5

ABSTRACT

A strong emphasis on quality control provides the technical base on which *the reputation* for the oil and meal products from a crushing plant is established. Most crashing plants in Western Canada now process only canola, the new quality oilseed developed from the former rapeseed. Quality control procedures employed by these plants to contract, grade, purchase and segregate canola seed for processing are described. Quality products are manufactured to meet national standard specifications of quality with the aid of a regular schedule of sampling and laboratory analysis, combined with frequent communication between quality control personnel and plant operators. Operating procedures are established to minimize variability in the quality of oil and meal products resulting from the natural variation in the characteristics of the seed to be processed. Instrumental methods of analysis are being used increasingly **to** facilitate the analysis of process materials.

INTRODUCTION

Vegetable oils are extracted from a wide variety of oilbearing seeds and fruits in crushing plants ranging in size from a few tons per day to over 3,000 tons of seed per day. The unique physical and biochemical characteristics of individual species of seed or fruit have often necessitated the development of unique extraction processes for each. In addition, distinct national or cultural preferences in the quality of edible fat and oil products have developed in certain countries which have resulted in further specialization of processing parameters and equipment. The subject of quality control in crushing plants, therefore, covers a very broad field of interest. In this paper, the scope of the discussion will be confined to quality control in the processing of canola seed grown and processed in Canada.

Canola seed is a genetically engineered oilseed, developed in the 1960's and 1970's from the traditional rapeseed. Rapeseed, as it is known in world commerce, is a heterogeneous mixture of several distinct Brassicae species including *Brassica Napus, B. Campestris, B. Juncea, B. Sarson* and others. The composition of oil and meal components of the naturally occurring land races from each specie are known to differ in several important respects, i.e., fatty acid composition, protein content, hull color and the amount and composition of glucosinolates (1). These differences were further highlighted with the genetic isolation of seed within the *B. Napus* and *B. Carnpestris* species containing low levels of erucic acid and low levels of certain glucosinolates (2). With these latter developments, a new oilseed was created which, when processed, yielded oil and meal products uniquely different to products from the traditional rapeseed and mustards.

The name canola is the registered trademark of the Canola Council of Canada and may be used freely to reference the seed, oil and meal products obtained from B. *Napus* and *B. Campestris* containing less than 5% erucic acid and less than 3 mg/g glucosinolates. Glucosinolates included in this specification are only those which have been commonly analyzed to date, i.e., gluconapin, glucobrassicanapin, progoitrin and napoleiferin. Their contents are expressed in units of mg equivalents of 3-butenyl isothiocyanate released per gram sample (meal basis) or, more appropriately, as micromoles glucosinolates per gram sample (3 mg/g \simeq 26 micromole/g).

QUALITY CONTROL OF SEED FOR PROCESSING

Brassicae species currently grown in Western Canada are of the summer form of *B. Napus* and *B. Campestris.* In 1981, over 85% of the Brassicae varieties grown were of the canola type. Very recently, agronomic and plant breeding research was initiated to develop also winter canola varieties of *B. Napus* adapted to Southern Ontario. The general seed characteristics and yield relationships between the summer and winter types, when grown in Canada, are expected to be similar to those described recently for summer and winter biotypes grown in Europe (3).

The production and quality of canola grown in Canada is surveyed annually by the Canadian Grain Commission and is reported in an annual crop bulletin (4). These harvest surveys show that the erucic acid content of the oil has exhibited a consistent decline during the 1970s. Recent analysis for seed grown in 1981 indicates that canola oil obtained from commercial seed contains 0.5-2.0% erucic acid. Several new varieties are being released currently through the pedigree seed system which have erucic acid contents substantially below 0.2%.

The content of common glucosinolates in seed now also exhibits yearly declines following the release of the first low glucosinolate variety in 1974. Commercial seed in 1981 was found to contain about 3 mg/g of common glucosinolates. Further decreases in the glucosinolate content in the 1982 and 1983 crops occurred as the recently licensed canola varieties Tobin, Andor and Westar entered the commercial production system and completely replaced the remaining high glucosinolate varieties still in production.

For several years, most crushing plants located in Western Canada have only processed canola varieties. Several quality control procedures have been used to identify and segregate canola seed within the commercial system. First of all, crushing plants, through production contracts and certified seed sales programs have encouraged producers to plant only canola seed. Planting seed was frequently specified to contain less than 2% erucic acid and less than 2 mg/g of common glucosinolates. At the seed-receiving elevator, rapid chemical testing procedures have been utilized to identify the desirable characteristics of low erucic acid and low glucosinolate.

The monitoring and control of erucic acid content in canola is now practiced almost exclusively through the pedigree seed production system, starting with the release of the breeder's seed. Although contamination of canola seed with the older high erucic acid rapeseed varieties can still occur occasionally during seed multiplication and in commercial fields, the level and extent of contamination is now relatively insignificant. As a result, a rapid test procedure for erucic acid at the seed-receiving elevator (5) is no longer performed routinely. Fatty acid composition of canola oil, however, is monitored each year using common gas chromatography procedures (6).

To identify and segregate canola seed with the low contents of common glucosinolates, two rapid glucose test tape procedures developed by McGregor, of Agriculture Canada, have proven to be very satisfactory (7,8). In circumstances where more accurate determinations of the amount and composition of glucosinolates are required, the gas chromatographic procedure of Underhilt and Kirkland (9) has, until recently, been used to identify the common glucosinolates. In December 1981, a standard reference method for the analysis of glucosinolates was selected by representatives of the Canadian industry, government and universities. The method is based on the procedure of Heaney and Fenwick (10) which involves the pretreatment of glucosinolates with the enzyme sulfatase before the standard derivitization reaction with a sytation reagent. By the use of this procedure, greater precision and accuracy in the determination of common glucosinolates is expected. In this latter procedure, additional information is gained also concerning secondary glucosinolates such as glucoerucin, glucobrassicin, neoglucobrassicin and others which are present in both canola and rapeseed. The method may also be used to give subjective indications of possible admixtures of commercial mustard, wild mustard, stinkweed and other glucosinolate containing seeds within commercial canola seed or meal products. However, admixtures of meal from these seed contaminants with canola meal cannot be quantified reliably since the individual glucosinolates that might

TABLE I

Quality Characteristics of Canola Seed by Variety (4)

be used for identification and the common glucosinolates of canola are hydrolyzed to varying degrees during commercial processing.

Harvest surveys show that the quality of commercial canola seed is dependent on the variety/species of canola grown (Table I) and on the region in which the canola is grown. It is generally found that varieties of *B. Napus* are highest in oil and protein content and those of *B. Carnpestris* are lowest in chlorophyll and fiber content. The oil from current varieties of *B. Napus* generally contains 2-3% less linolenic acid than oil from the *B. Campestris* variety Candle. The species planted will depend upon the climate of the region and the timing of the farm operation. B. *Campestris* is a short season crop which is favored in northern regions in Canada and in circumstances requiring very early or late planting. The quality of the seed is also influenced by localized environmental conditions affecting the crop as it develops during the growing season, matures and is harvested. The result is that canola seed, as it is received directly from farms at crushing plants, can exhibit considerable variation in quality parameters such as oil, protein, chlorophyll and linolenic acid.

The grading system for canola in Canada is based primarily on a visual inspection for immaturity, damaged or. heated seed and admixtures as well as for moisture content (Table II). Oil and protein content per se are not grading factors in the Canadian system even though the ratio of oil and meal products is important economically. Immaturity, as indicated by the percentage of green seeds in a test lot, is the major degrading factor of the Canadian system (11). Despite the subjectiveness of the grading procedure for immaturity, the visual inspection of seed for percentage green seeds is used effectively to segregate or exclude seed from processing which has a high chlorophyll content. Canada No. 1 seed in each of the species of canola generally provides the highest oil content and the lowest content of chlorophyll/pheophytin, free fatty acids and non-hydratable phospholipids in the crude, degummed oil. Canada No. 2 seed yields a meal with the highest protein content.

Most crushing plants receive seed directly from producers by truck in 7-20 ton lots. Wide variations in the gross composition of seed from tot to lot, particularly with respect to oil and moisture content, can lead to unstable cooking, expelling and solvent extraction processes. To stabilize plant capacities, quality and performance, plant operators often will receive, clean and store canola seed of the *B. Napus* specie separate from seed of the *B. Campestris* specie. They may also segregate seed further according to grade. Subsequently, the seed of each species may be processed separately. However, in our experience, it is

a8.5% moisture basis.

bOil free meal, 8.5% moisture basis.

preferable to blend seed of the two species after cleaning and to process the mixture at a known and relatively constant proportion. By following this procedure, quality variations in the oil and meal products are reduced substantially compared to the quality differences indicated between varieties and species of canola in Table I.

The moisture content of seed received at the crushing plant has an important impact on the selection of processing and quality parameters. Compared with rapeseed, canola seed has demonstrated an increased tendency to shatter on flaking, particularly at low seed moisture contents (12,13). The moisture content of commercial seed from 1965 to *1979* varied within a relatively narrow range of 8.2-9.8% moisture. However, in 1980 and 1981, harvesting of crops in Western Canada proceeded under very dry conditions with the result that seed moisture contents were reduced to 8.1 and 7.8% moisture, respectively. These low moisture contents have made it more difficult for plant operators to condition seed adequately for oil extraction. On the other hand, the 1980 and 1981 crops were not exposed to physiological weathering in the field or swath and yielded crude, degummed oils with low free fatty acid and residual lecithin contents and canola meals with high protein content. The processing of a constant mixture of the

TABLE II

Canadian Statutory Grades for Rapeseed a

two species of canola has helped to reduce the variation in the moisture content of seed for processing, helping to stabilize plant capacity and operating performance.

QUALITY SPECIFICATIONS FOR PRIMARY CANOLA OILS AND MEAL

Crude and crude, degummed canola oil are produced from canola seed according to the specifications of the National Standard of Canada CAN 2-32.300M-76 for a low erucic acid rapeseed oil (Table III). In 1981, a new "special" degummed canola oil containing extremely low amounts of phosphorus (<30 ppm) and low sulfur was announced by several companies in Canada.

Our corporate specifications for special degummed oil indicated in Table III demonstrate that this new oil is distinct from crude, degummed canola oil by having had substantially all of the residual phosphorus content of the crude oil removed. However, compared with a refined canola oil which has had substantially all of the phosphorus and acidity removed by alkali refining, special degummed canola oil typically contains 0.4-0.7% of free fatty acids. The three types of primary canola oils are differentiated from each other on the basis of the neutral oil content and

aAdditional specifications for canola: $<$ 5% erucic acid and $<$ 3 mg/g glucosinolates.

TABLE III

Specifications for Primary Canola Oils

neutral oil loss and by refining yield and performance criteria.

Refining tests of special degummed oil in the tab and, more recently, in commercial refineries indicate that the oil is a suitable feedstock for physical refining. Special degummed canola oil may be prepared for physical or steam refining by bleaching using the normal amounts of an acidactivated bleaching earth. Hydrogenation tests of bleached, special degummed oil have also shown that bleaching can remove effectively residual amounts of phosphorus and sulfur which can contribute to the poisoning of hydrogenation catalyst. A national standard specification for special degummed canola oil is expected to be developed once further experience is gained in the production and refining of this oil, particularly by physical or steam refining processes.

Minimum quality criteria for canola meal are established by the specifications of the National Standard of Canada CAN 2-32.301-76 for protein, fiber and moisture content (Table IV). In commercial practice, corporate specifications have been established which guarantee protein content at 37% minimum (as is basis) and 3 mg/g maximum common glucosinolates.

QUALITY CONTROL METHODS AND PROCEDURES

Canola seed is processed using conventional pre-press/ solvent (14,15) or direct solvent extraction processes (16) similar to those used commonly for rapeseed and other high oil content seeds. The increased tendency of the *B. Napus* varieties of canola to shatter on flaking and the lower fiber content of the yellow seeded *B. Campestris* canola variety Candle, have necessitated changes in cooking parameters and in the design of the worm assembly for prepress expellers (12,13). Furthermore, demands by refiners for improvements in the quality and consistency of the primary canola oils have further stimulated crushers to seek alternatives and improvements in the extraction and oil recovery processes.

All oilseeds contain several functional proteins or enzymes which will interact with seed constituents once the oil has been freed from the cell structure by flaking. Cooking has been used traditionally to inactivate or denature these functional proteins so that the quality of oil is preserved as the flakes are conditioned for expelling or direct solvent extraction. Brassicae seeds are unique among oilseeds in containing the complex group of compounds called glucosinolates. These sulfur-containing compounds are hydrolyzed readily by several native enzymes, including myrosinase, to yield undesirable products in both the oil and meal. Cooking of rapeseed, frequently at temperatures as high as 110 C, has been used to inactivate the myrosinase, thereby eliminating the breakdown of glucosinolates by enzymatic processes. The decomposition of glucosinolates, however, is not completely eliminated as several are thermally and chemically labile at elevated cooking temperatures. In a survey several years ago, rapeseed oils were found to contain 15-57 ppm sulfur (17), with the result that the oil was difficult to hydrogenate due to sulfur poisoning of the nickel catalyst.

Today, canola processors employing prepress/solvent processes flake preheated seed at temperatures of 20-50 C, then cook the canola flakes at temperatures of only 80- 95 C for 15-30 min. Generally, water or steam is not added to flakes during the cooking process as this can accelerate the hydrolysis of glucosinolates. Under these milder cooking conditions, which are sufficient to inactivate myrosinase, the thermal decomposition of the glucosinolates is minimized and sulfur contents of 2-3 ppm in degummed canola

TABLE IV

Specifications for Rapeseed and Canola Meal/Pellets

Screen analysis (meal) - 90% by mass shall pass through a 1.70 mm (No. 12) sieve and 100% through a 2.00 mm (No. 10) sieve.

aSpecification of the Canola Council of Canada.

oils can be obtained consistently. This significant reduction in sulfur content has contributed to substantial improvements in the hydrogenation performance and reaction rates of refined and bleached canola oil (18).

To this point in the discussion, characteristics and features of canola seed and canola processing have been highlighted which tend to differentiate canola within itself and from other oilseeds. As noted, the compositional characteristics of canola can have a significant impact on the selection of procedures and processes that may be used to condition seed for solvent extraction, i.e., seed pre-heating, flaking, cooking and expelling. Solvent extraction and distillation equipment used in canola crushing is similar to that described in the literature for the solvent extraction of soybean. Operating procedures and quality control checkpoints for the operation of these processes are, in most respects, identical for both types of seed material extracted.

The yield of oil recovered from seed delivered into the process is a significant efficiency factor ultimately affecting the economics of crushing. Flaking is perhaps one of the more important processes affecting oil recovery as unflaked or poorly flaked seed can pass through all subsequent processes relatively unchanged and contribute to high residual oil in the finished meal. The quality of flake preferred for efficient processing will vary from plant to plant given different constraints for seed quality and processing equipment. Experimentation at each plant will establish the appropriate flake thickness and particle size after flaking. The extent of cell disruption after flaking, cooking and expelling can be estimated in the laboratory using a double extraction procedure. In this method, free oil is obtained in a first extraction with solvent whereas oil remaining bound within the intact cell structure is recovered only after severe grinding of the previously extracted residue. Expeller cake of canola that has been appropriately flaked and cooked will preferably contain less than 1% cell-bound oil (percentage of dry solid) in the second extraction. The performance of the solvent extraction process can be evaluated by comparing the residual oil of extracted cake sampled before desolventizing with this estimate of cell-bound oil.

Experience has shown that a regular schedule of sampling and laboratory analysis, combined with frequent communication and liaison with plant operators, provides a good technical base on which to establish an efficient crushing process producing high quality products. At regular intervals in a 24-hr period, the residual oil, protein and moisture contents of expeller cake and finished meal are determined using near infrared reflectance (NIR) spectroscopy. The instrument is calibrated with samples whose composition have been determined according to AOCS methods. As sampling and grinding techniques can have a significant influence on the precision and accuracy of the NIR determination of solid materials, care is taken to follow standardized procedures for sample preparation.

The quality of canola meal is established primarily on the basis of protein, moisture and gtucosinolate content. The residual oil content of canola meal is typically 2-3% (11% moisture basis). This level is higher than the amount of cell-bound oil referred to earlier as wet gums from the degumming processes are returned back to the meal in canola crushing and contribute ca. ½ of the residual oil content of the meal.

Variation in the digestability and quality of commercial rapeseed meals has been demonstrated in animal feeding studies (19). It is thought that excessive heating of the meal in the desolventizer:toaster contributes to this variability. Various laboratory procedures have been proposed to assess the affects of processing on protein quality of oil meals. The solubility of protein in water and alkaline solutions has been considered as criteria to estimate heat damage in canola protein. However, animal feeding studies of canola meal have shown that protein solubility in 2% KOH is unreliable as an index of protein quality in canola meal (20). Estimations of tysine availability or dyebinding capacity of the protein appear to be more suitable for estimating protein quality in canola meals than protein solubility. Significant correlations between the dyebinding capacity of the protein, total lysine and available or reactive lysine suggest that dyebinding capacity may be used as an index of protein quality $(21,22)$. Commercial feeding trials and feed use ultimately determine the appropriateness of various desolventizing parameters. Today, Canadian crushers employ milder desolventizing conditions with canola than with the older rapeseed so as to minimize heat damage to the meal protein (23) ,

Hexane used in the extraction process is monitored very closely at various stages in the distillation and solvent recovery systems to minimize safety and health hazards and solvent losses. Gas chromatography (27), involving the volatization of hexane into the headspace above a weighed sample, is used to monitor residual hexane in oil, meal and process water.

The primary canola oils are sampled in the crushing process at regular intervals. Official AOCS methods are used to determine free fatty acids, insoluble impurities and neutral oil content. Moisture is determined using a Karl Fischer titrater. Phospholipids in the oil are determined according to an acetone insoluble method adapted from the Canadian Government specification 2-32-300M-76 for rapeseed oil. An oxygen bomb procedure is preferred to a perchloric acid digestion procedure for the preparation of the samples for phosphorus determination (24), Phosphorus to acetone insoluble (lecithin) content conversion factors in primary canola oils are found to range from X18 to X35 depending on the source and characteristics of the canola oil. Oils with greater amounts of hydratable phospholipids require higher factors to convert phosphorus to acetone insoluble content. Chlorophyll content of oil extracted from unprocessed seed and pheophytin content of processed oils are estimated by absorption spectroscopy according to the AOCS official method Cd 13d-55 as modified by Kelly and Yuen (25). Sulfur content is measured by the Raney Nickel absorption method (I7). Other parameters that establish the quality of primary canola oils are similar to those described for soybean oil (26).

Near infrared reflectance spectroscopy has facilitated the routine measurement of oil, protein and moisture in canola process meal products. The application of reflectance spectroscopy to the analysis of phospholipids and free fatty acids in canola oil would be a welcome advancement. Instrumentation involving the combination of thin layer chromatography on glassrods and flame ionization detection (TLC-FID) appears to be of limited use for the quantitative determination of these constituents.

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