Quality Control in Edible Oil Processing

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ABSTRACT

This paper describes a Q.C. program for the manufacture of edible products from crude degummed oil. Tests done at various stages in the production are discussed, including test methods used and what is considered as acceptable results. Processes covered include receiving oil, alkali refining, bleaching, hydrogenation, deodorization, bulk shipping and packaged finished product.

The purpose of quality control is sometimes viewed as ensuring that the outgoing products meet the specifications set out by the customer and agreed to by the producer. I feel that this definition is too limited and fails to take into consideration all of the responsibilities of quality control.

The objective of quality control is not only to ensure that product shipped is within specifications, but also to control process supplies and raw materials, and aid production in making the product at the lowest cost with minimum rejections. In this sense, quality control has failed in its function if a final product is rejected and must be reprocessed. This could be necessary because of low quality feed stocks, off-specification processing supplies or insufficient production checks.

To meet these objectives, quality control must work closely with production by checking current processing, incoming material and finished products. Communication must be two-way, with production personnel keeping quality control personnel informed on the progress of any orders through the plant and quality control personnel informing production of any problems or potential problems and supplying advice on causes and solutions. The quality control team must consist of all personnel, from the operators through to the managers, not just the laboratory staff. Only when quality control is everyone's responsibility can a plant produce a consistently high quality product at the lowest possible cost.

Different plants have different views on who has the responsibility to check product quality. In some cases, there is an attempt to make all testing the responsibility of the laboratory staff only. In other cases, all testing is performed by the production personnel. I feel that a compromise of these two extremes is the best. This would involve production handling the less technical checks such as color, free fatty acid, filter test and soap. Quality control then does a more complete testing of finished product quality, including a verification of the production tests. Advantages of this system include reduced laboratory staff and direct involvement of production staff with product quality. Therefore, if problems arise, the production department knows about them immediately. Unfortunately, there are also disadvantages to this system. Only less technical tests can be performed by the operator and the records will generally be kept outside the laboratory. The operator is put in a "conflict of interest" situation since he wants to produce as much as he can and is tempted to interpret borderline results with a bias, or simply to record typical results and not do the tests. Control of this situation is critical to the success of the program. If the laboratory checks show that excessive amounts of borderline product are passed, then steps must be taken to correct the situation quickly.

Since quality control in oil crushing has been discussed earlier, I will now deal with ensuring quality in the edible dry rendering process and refining.

Incoming fat should be inspected for mold, rotting fat, excessive moisture, red meat, bones and skin or other contaminants. The operator should recognize the fresh kill and cook it immediately. To ensure quality, the cooking time and temperature must be monitored. If the fat is overcooked, then the resulting tallow will be dark and of low quality. Each cooking should be tested for free fatty acids and color before the oil is pumped to storage. Oil with a free fatty acid content over 1% or darker than 4.0 red on a 5¼" cell should be kept separately until after bleaching and stability tests can be performed. After filtering or decanting to remove fines, the oil should again be checked for free fatty acids and color. At this stage, a filter should also be done to ensure that the filter is not passing any fines. The iodine value and solid fat index should also be checked before the product is sent to crude storage, as this information verifies the source of the fat. The filter test is done by filtering a large quantity of oil (usually 0.5 kg) through a small diameter (5.5 cm) standard filter. The color of the filter and quantity of solids retained are then judged, compared to a set of standards and given a number from 1 to 10. Ten is considered perfect, with no material retained by the filter, 1 is very bad (almost black) and a pass is a 7. Good judgment is required and the person conducting the test requires training to do it well.

Crude oil analysis is very important. Incoming oil quality determines the necessary processing and is a good indication of the treatment it will require. This is especially true now that physical refining is becoming more common. Also, crude oil quality often affects the price. Crude oil should be checked for color, free fatty acids, neutral oil, phosphorous, peroxide value, iodine value, moisture and impurities and acetone insolubles. The chlorophyll and sulfur in canola oil should also be checked on a regular basis. The chlorophyll, soap and anisidine value should be checked on an occasional basis with all types of incoming oils. Soap is often found in offshore imported oil traded on a free fatty acid basis.

For crude canola oil, the analysis results should be as follows: color—5.4 red max.—1" cell, PV (peroxide value)—2 max., Phosphorous for water degummed oil—less than 200 ppm, acetone insolubles—.5% max., moisture and impurities—0.09% max., iodine value—112-120, chlorophyll—25 ppm max., neutral oil—98.9% min., sulfur—15 ppm max.

The tests are all performed using the AOCS standards. The sulfur test is done by a modification of the Granatelli method.

At this point, recommendations can be made by the lab as to where the oil should be stored, what other incoming crudes can be mixed with it, and how it will be processed, e.g., what strength caustic or pretreatment is needed and whether the oil can be physically refined.

Before refining, the oil should be checked for free fatty acid content. This will accomplish two things. First, it verifies that the oil has received the correct pretreatment for refining and, secondly, it provides a check on the storage stability of the oil.

The refined and washed oil should be checked for soap, free fatty acids, color and moisture. The oil should have less than 200 ppm soap after washing, a color of 4.5 red on a 1" cell, a moisture of less than 1% and a free fatty acid content of less than 0.05% before production is started.

Soap can be determined usin the Wolf method. This procedure is a two phase titration of oil and a 1% solution of water in acetone which extracts the soap. The titration is done with a 0.1 N solution of hydrochloric acid with bromophenol blue indicator to a yellow end point. The acetone solution must always be standardized before use as it tends to become acidic with time. Methanol may be substituted for the acetone solution.

The moisture test can be done by the hot plate or oven method as outlined in the AOCS standards. A faster and easier check can be made by spinning the sample of oil for 1½ min on a high speed centrifuge, using a calibrated 10 cc tube. This will allow a quick estimate of moisture and provide a crude indication of soap. These tests should be done every hour to ensure that the product is consistent.

In addition to the above tests for product quality, quality control should also check the caustic and soapstock. The caustic should be checked for impurities and also to ensure that correct dilution has taken place. The use of high-strength caustic will increase losses whereas weak caustic could result in a low quality product. The soapstock should be checked to ensure that good separation is taking place and that excessive losses are not occurring. The oil content can be estimated by spinning the soapstock as outlined before. There are two other tests which will provide more quantitative results. The total fatty matter test will estimate all the fatty acids present in the soapstock. Neutral oil or cromo loss can then be performed on a dried sample of soapstock. The difference can then be used as an estimate of the soap in the stock. These results will allow a verification of the measured loss by mass balance if the soapstock output is known.

It is also wise to check the free fatty acid content occasionally before the first separator but particularly after the caustic addition and mixing. This will indicate if the correct caustic treatment has been given to the oil. Excessive treatment with caustic can lead to high saponification losses, whereas too little caustic will lead to high free fatty acid content or low product quality.

Every batch, or at least every 150,000 lbs. of oil, should be checked for color, free fatty acid, moisture and soap, plus phosphorous. The color, free fatty acid content and soap should be the same as the production analysis results. The moisture will be lower due to vacuum drying of the product. This moisture should be less than 0.1%. The phosphorous must be reduced to less than 30 ppm if standard bleaching practices are to be followed. If any of the above tests indicate a problem in the product, then steps can be taken to correct it in bleaching, thus eliminating possible problems before they occur.

Since the advent of physical refining, it is more important than ever that the exact quality of the feed oil is known. For tallow and animal feedstocks, the oil should meet the specifications stated earlier. For refined vegetable oil, the previously mentioned specifications should be met. Before being physically refined, oil must have a phosphorous of less than 80 ppm and a sulfur of less than 15 ppm. If the absorption isotherm for the clay is known and the initial and desired final oil quality is known, it is possible to calculate the minimum amount of clay needed in bleaching. This is an example of how accurate knowledge of feedstock quality, processing supplies and final product specifications can save money in processing.

The tests to be performed in bleaching should include an occasional check of the feedstock to the filter press for clay content. This can be done by filtering out the clay, washing with hexane and drying the cake to a constant weight. It is also possible to ash the sample and weigh the ash as an indication of the clay content. This will ensure that clay metering is accurate, since excessive clay increases losses and insufficient clay reduces product quality.

The oil should be recirculated through the press until the desired bleached oil color is achieved, the soap is removed and the filter test is acceptable. Immediately after bleaching has started, the oil should be heat tested. This test involves heating a small quantity of bleached oil under a vacuum or nitrogen blanket to 260 C. After the oil has cooled, the color should be checked. If a reading of less than 2 red is observed, then the bleaching is usually sufficient. Dark colors after heating can indicate incomplete removal of phosphorous, chlorophyll or protein fines. The color and filter test should be performed every hour to ensure that the press has not broken a cake and that oil quality is consistent.

Every batch, or at least every 150,000 lbs. of oil for continuous bleachers, should be checked for color, soap, filter test, phosphorous, free fatty acids, chlorophyll, peroxide and given a heat test. The free fatty acid content should be 0.1-0.2% higher than the feed oil, especially when acid-activated clay is used. The peroxide value of freshly bleached oil should be very low; generally below 0.1 me/mg.

Phosphorous readings for vegetable oils like soy or canola must be lower than 5 ppm if the oil is to be successfully hydrogenated or deodorized into a stable product. The chlorophyll content should be less than 6 ppm. For canola, the sulfur level should be checked for every batch as it must be reduced to less than 5 ppm if hydrogenation is to proceed unhindered.

If a variety of oils are produced from the same bleacher, then the products should be examined for contamination. Contamination is indicated by changes in the iodine value. For hard products, the SFI should also be checked. If the end product is to be a salad oil, then the cold test will quickly point out if the product was contaminated with any hard fat.

The cake discharge should occasionally be checked for oil content. This is to ensure that correct blowing and steaming practices are being followed. Excessive blowing will reduce oil content to less than 30%. If a cake is overblown, it can be a fire hazard and the final steamings can be low enough in quality to ruin the rest of the batch. Insufficient blowing is indicated by a high oil content in the cake and, of course, is a very wasteful practice.

Hydrogenation must be controlled carefully as off-specification products from hydrogenation can present blending problems and, in times of full capacity utilization, result in an inability to meet shipping deadlines. If bleached oils do not meet the specifications, then long reaction times and excessive use of catalyst can result. If it is known that the bleached oil contains catalyst poisons, it is best to manufacture products which require high hydrogen pressures during production. The high pressure encourages the reaction, reducing the need for excessive catalyst.

During hydrogenation, the tests performed should include an iodine value by refractive index, followed by a verification using the standard test when the end point is approached. If the product is made often, it may be possible to send it to storage before the solid fat index is checked. However, some companies have the policy of not releasing any product to storage until the SFI curve is known accurately. The value of this added information must be balanced against the cost of the lost production time. Compromises involving tempering solids to release the

product are also possible. In any case, the SFI of each hydrogenated batch and mixtures of the same product from different batches should be verified.

Before the filtered product is released to storage, the oil should be checked for color, free fatty acid content and nickel content. The test for nickel is qualitative only; ammonium sulfide is added to a sample of the filtered product. If a black precipitate forms, then nickel is present. This test is sensitive to ca. 2 ppm nickel. If more than just an indication of nickel is desired, then atomic absorption testing is required.

The color of hydrogenated oil should be less than 1.5 red on a 5¼" tube. A dark color could indicate an incomplete filtration process. The free fatty acid content of hydrogenated oils should be only slightly increased from the bleached oil stocks unless a long reaction time was necessary.

The filter cake should occasionally be tested, as in the bleaching process, to ensure that proper blowing practices were followed. Since the price of nickel was increased in past years, testing the cake for nickel content is occasionally necessary. The nickel content of the cake can affect its disposal in some cases as well as the saleability of the cake to nickel reclaimers.

The customer requirements, as well as in-house specifications, must be considered during the blending stage, i.e., the customer specifications determine which tests are essential. Often, a customer will specify not only the SFI but also the I.V., melting point and often a fatty acid profile. These specifications, to a large extent, dictate the acceptable base stocks to use in a blend. All the components of the blend must be checked for these parameters and then the blend itself must be checked. The free fatty acid content of the blend should be analyzed to ensure that no changes in deodorizing procedures are needed.

The first oil from the deodorizer should be checked for free fatty acids, color, P.V. and flavor before it is sent to storage. Each deodorized batch of oil should be checked for all of the customer's specifications. For time-consuming analyses like active oxygen stability, cold tests or solid fat index tests, the shipping schedule may not permit completion of the tests before the product leaves the plant. For this reason, all testing of deodorized batches should be given priority and testing should begin as soon as possible after the batch is complete. Minimum test requirements before loading are: color, free fatty acid content, peroxide value, flavor, odor, iodine value and smoke point, if necessary. If the customer's specifications call for melting point and/or SFI, it is best if these are also verified before shipping. With any shipment, a complete analysis report should be included, giving the results of analyses completed. The time-consuming test results should follow as soon as possible. An oil sample should be taken from the truck or railcar before it leaves the plant. This sample should receive the complete analysis even after the truck is released to ensure that it was not contaminated during loading.

If deodorized oil is stored on site for more than a day, it should be tested on a daily basis for color, peroxide value, flavor and free fatty acid content. This daily testing will provide a forewarning of reversion problems.

Packaged goods should receive all the testing that a bulk shipment of oil receives, and should also be tested for the presence of additives, such as monoglycerides, salt, flavoring, coloring and antioxidants. Procedures for these tests can be found in either the AOCS standard methods or AOAC standard methods. The performance of shortening should also be tested under "use" conditions. Baking tests to determine water absorption, batter density and icing density should be conducted, as well as some frying tests.

All bulk shipments and each batch of packaged goods should have samples retained. The bulk oil sample should be taken from the truck or car before it is released. Packaged goods should be sampled at the filling station. These samples should be retained for at least 3 times longer than the customer would have the product in storage.

Warehouses and storage areas should be inspected periodically for old packaged goods and expired code dates.

The tests and procedures described in this paper may be reduced or increased as the need arises. An increase may be required when problems arise with incoming raw material or if there is a specification change. A decrease may be practical if the incoming crude oils are the same type and quality, or if the same finished product is produced in long runs.

This paper is only a summary of quality control practices from a laboratory view. There is much more involved in the quality control process than I have discussed here. For instance, another complete paper could be written on record keeping during processing alone. These records will provide both quality control and production staff with explanations of problems and give easy verification of the processing parameters.

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