

A Modified Method for Determining Free Fatty Acids from Small Soybean Oil Sample Sizes

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ABSTRACT: A modification of the AOCS Official Method Ca 5a-40 for determination of free fatty acids (FFA) in 0.3 to 6.0-g samples of refined and crude soybean oil is described. The modified method uses only about 10% of the weight of oil sample, alcohol volume, and alkali strength recommended in the Official Method. Standard solutions of refined and crude soybean oil with FFA concentrations between 0.01 and 7.5% were prepared by adding known weights of oleic acid. The FFA concentrations, determined from small sample sizes with the modified method, were compared with FFA percentages determined from larger sample sizes with the Official Method. Relationships among determinations obtained by the modified and official methods, for both refined and crude oils, were described by linear functions. The relationship for refined soybean oil had an R^2 value of 0.997 and a slope of 0.99 ± 0.031 . The values for crude soybean oil are defined by a line with $R^2 = 0.9996$ and a slope of 1.01 ± 0.013 . JAOCS 75, 563–568 (1998).

KEY WORDS: Crude soybean oil, free fatty acid, Official Method, refined soybean oil, titration.

In the soybean industry, the presence and development of any amount of free fatty acid (FFA) in soybean oil is undesirable. The FFA content of oil in soybeans indicates how well the beans have been treated during the period between harvest and processing. The eventual consequence of high FFA content is monetary loss because processing soybeans with high FFA content results in more refining loss. Specifically, refining loss is the sum of oil weight lost during removal of FFA and other impurities plus the weight of entrained neutral oil in soapstock during the normal alkali-refining process. The percentage of oil lost during refining can be estimated as a multiple of the FFA percentage. Norris (1) estimates the potential loss at about three times the FFA content. Because of the economic impact of high FFA, it is important to determine the initial level of FFA in soybean oil and also to monitor its concentration during processing.

The standard method for FFA determination in extracted crude and refined soybean oil samples is based on an acid-

base titration technique in a nonaqueous system. The method commonly used is AOCS Official Method Ca 5a-40 (2). The procedure prescribes an oil sample of maximum 56.4 g and minimum 3.53 g for the titration, depending on the level of FFA in the oil sample. When oil quantities are limited, as in some storage studies or when experimental use of oilseed crops is evaluated, sample sizes of this magnitude are not possible. A simple and inexpensive method is needed that accurately quantifies FFA content from a smaller sample size.

The use of smaller amounts of chemicals will have an impact, especially in developing countries, where chemicals are scarce and expensive. Also, it is prudent to limit the use of resources, particularly those hazardous to the environment. For example, hexane, a solvent used in the titration, is a hazardous, highly flammable compound, and its disposal can be harmful to the environment. A smaller sample size also would result in savings of storage space in laboratories that handle large numbers of samples, or if the protocol warrants that oil samples be kept after analysis.

Lanser *et al.* (3) developed a user-interactive computer-assisted Fourier transform infrared (FTIR) method to estimate FFA content in soybean oil samples. The method requires only one drop of soybean oil. The limitation of the method, and other similar spectrometric methods, such as that described by Canham and Pacey (4), is the cost of the instruments; it is prohibitive for most laboratories. Lanser *et al.* (3) also evaluated a modified version of the Official Method in which between 6 and 7 g of oil is used, but no further description of the method was given. The coefficient of determination (R^2) of the modified method was 0.999. The high correlation indicates that the officially recommended sample size can be scaled down without losing accuracy of FFA determination.

The objective of this research was to develop and evaluate a method for determining FFA concentration within the range of 0.01 to 7.5% in soybean oil by using an oil sample of about 10% (between 0.3 and 6.0 g) of the weight recommended in the AOCS Official Method.

MATERIALS AND METHODS

Different amounts of oleic acid were added to refined-bleached-deodorized (RBD) and crude soybean oil samples

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