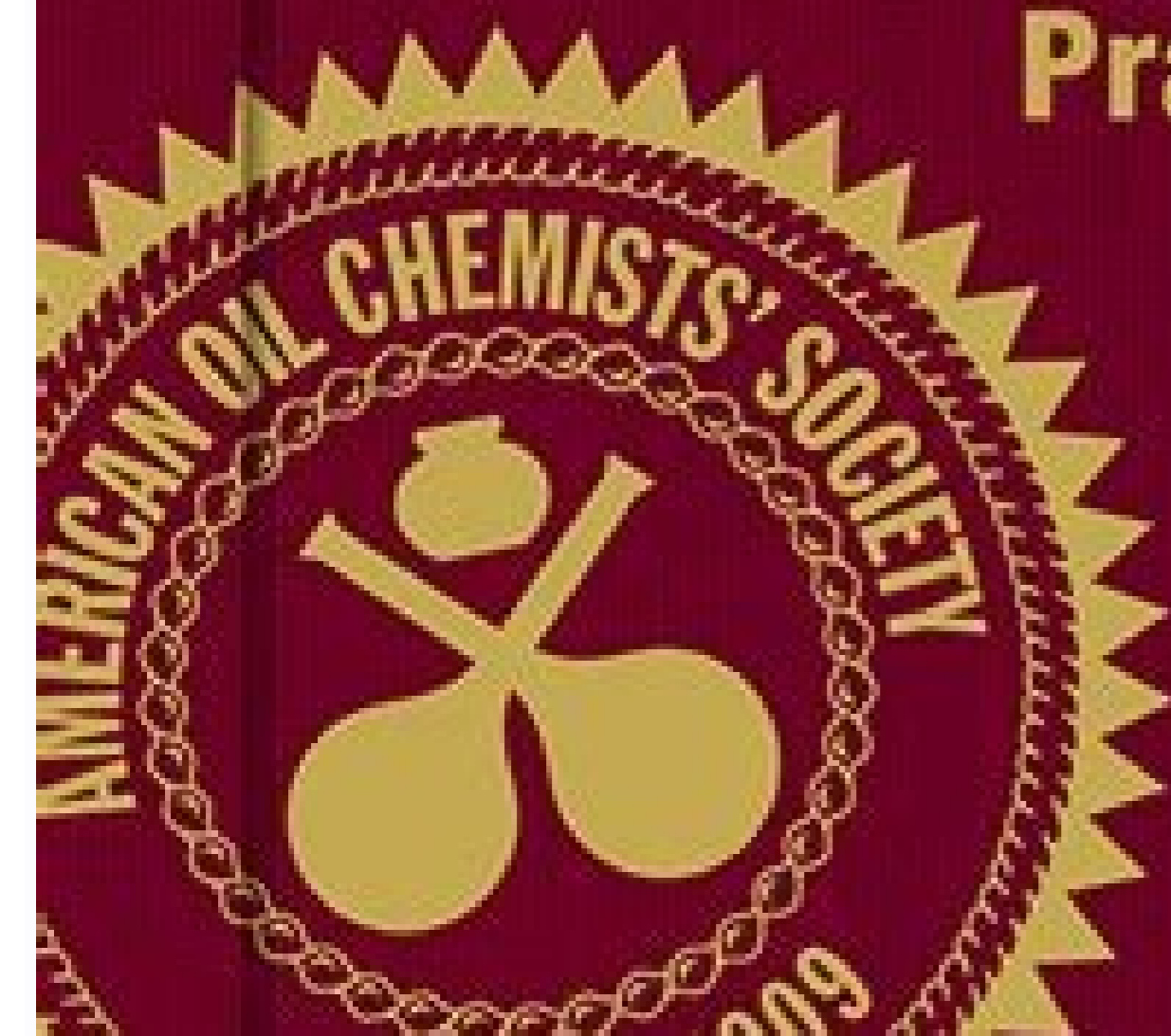


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Official Methods and Recommended Practices of the AOCS

7th Edition



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Short Report

A Green Potentiometric Method for Determination of the Acid Number of Oils and Fats

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Um método potenciométrico verde é proposto para a determinação do número de acidez de óleos vegetais e de gorduras animais. A amostra é dissolvida em mistura etanol-água (1:1 v/v) e titulada, usando um eletrodo combinado de vidro, com uma solução aquosa padrão 0,02 mol L⁻¹ de NaOH. Analisaram-se óleos de canola, girassol, linhaça, mamona, milho e soja além de gordura de porco, em um total de doze amostras. Os resultados obtidos com o método proposto foram estatisticamente comparados com aqueles resultantes dos métodos AOCS Cd 3d-63, ABNT NBR 14448 e de titulação verde com detecção visual, através de procedimento de regressão linear em nível de confiança de 95%, não sendo encontrada diferença sistemática. O desvio padrão relativo médio observado para o método proposto foi de 2,7%, enquanto que para os procedimentos ABNT e AOCS foi de 5,4% e para o método verde de titulação visual foi 4,8%.

A green titrimetric method using potentiometry is proposed for determination of the acid number of vegetable oils and animal fats. The sample is dissolved in a water-ethanol mixture (1:1 v/v) and potentiometrically titrated with a 0.02 mol L⁻¹ aqueous NaOH standardized solution using a glass pH combined electrode. Canola, sunflower, linseed, castor, corn and soy oils as well as swine lard, a total of twelve real samples, were analyzed. The results were compared with those from the application of the procedures AOCS Cd 3d-63 and ABNT-NBR 14448 and a green visual titrimetric method, through a statistical linear regression procedure at the 95% confidence level. No evidence for systematic differences was observed. Mean relative standard deviation for the proposed procedure was 2.7%, whereas that for the AOCS and ABNT methods 5.4% and for the visual green method 4.8%.

Keywords: acid number, vegetable oil, animal fat, potentiometric method, green chemistry

Introduction

Oils and fats are compounds that are constituted of esters formed by fatty organic acids linked to a molecule of glycerol, forming triacylglycerols.^{1,2} Due to their structural composition, they are susceptible to diverse degradation reactions.³

In the hydrolytic rancidity reactions, the carboxylic group suffers the action of enzymes of a microbial order commonly found in oleaginous seeds. It can also react with water. These reactions cause the breaking of the triacylglycerol molecule leading to the formation of free fatty acids which are responsible for the unpleasant flavor characteristic of the oxidized fats as for example, rancid butter.^{3,4} The oxidative rancidity reactions are characterized

by reactions of the unsaturations in the fatty acid chain and by the formation of degradation compounds such as alcohols, aldehydes, ketones, etc.^{5,6} The oxidation reactions are influenced by several external factors as, for example, oxygen from the air, temperature, the presence of metallic cations, light and humidity.^{7,8}

The presence of free fatty acids is undesirable in oils and fats as it reflects the nutritional quality of the product. The quantity of these acids indicates how the feedstock was treated during industrial processing and during the storage. High concentrations mean loss of money as the rancid of the product decreases. Therefore, the determination of their concentrations throughout the refining process and during the storage is important for monitoring the occurrence of degradation reactions.^{9,10,11,12}

During the cooking of the food submitted to frying, the triacylglycerols are degraded as a consequence of the

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Iodine Value in Partially Hydrogenated Castor Oil (Ricinus Oil) as determined by AOCS Official Method Cd 1-25 (Wijs' Method)

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Research Article

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Abstract

The Iodine Value (Iodine Number) is an important analytical characteristic of fats and oils. The iodine (I₂) required saturating the fatty acids present in 100 grams of the oil or fat. Iodine is essential element of human nutrition. A third of the global population has insufficient iodine intake and is at risk of developing Iodine Deficiency Disorder. Oils rich in saturated fatty acids have low iodine value, while oils rich in unsaturated fatty acids (α-linolenic acid) have high iodine value. Several variations of iodine value have been developed, although Iodine Monobromide Method or Hanus Method, Iodine Monochloride Method or Wijs' Method, and Pyridine Bromide Method or Iodine-Mercuric Chloride in alcohol (Hubl). The based on American Oil Chemists' Society (AOCS) Cd 1-25 describes the determination of the iodine value (a measure of unsaturation) in Partially Hydrogenated Castor Oil (COH); the specification is 28-32 g I₂/100 g sample.

Keywords: Iodine Value; Partially Hydrogenated Castor Oil; Wijs' Method

Introduction

Castor oil occurs in the seed of the castor plant, *ricinus communis* L. (Euphorbiaceae Family), growing in most tropical and subtropical areas. Castor seeds are toxic, containing a highly poisonous protein, ricin, and highly allergenic material. Castor oil is non-toxic, a renewable resource, and biodegradable [1]. Castor oil is viscous, a colorless to pale yellow and non-drying vegetable oil with a bland taste and it is sometimes used as purgative [2]. Its boiling point is 313°C (595°F) and its density is 961 kg/m³ [3]. It is a triglyceride in which approximately ninety percent of fatty acid chains are ricinoleic acid. Oleic acid linoleic acids are the other significant components [4]. Castor oil is essentially triricinolein, which is a triglyceride of ricinoleic acid, CH₂(CH₂)₇CH(OH)CH₂CH=CH(CH₂)₇COOH. The expected iodine value for castor oil is 83-88 g/100g I₂ and for hydrogenated castor oil it is 28-32 g/100g I₂, based on AOCS Cd 1-25 Wijs' Method. The American Oil Chemists Society

(AOCS) methods are widely used for contrast purposes in the trade of oils and fats. The traditional method for determining iodine value (AOCS official method Cd 1-25), make use of solvent carbon tetrachloride. In a number of countries this solvent is now banned for use in laboratories because of its carcinogenic properties. Consequently this method of analysis has been modified using cyclohexane as a solvent (AOCS) recommended practice Cd 1b-87 [5].

Hydrogenated Castor Oil (HCO) occurs as a fine, almost white or pale yellow powder or flakes. Hydrogenated castor oil as the oil obtained by hydrogenation of virgin castor oil. Empirical formula C₅₇H₁₀₄ and molecular weight is 939.50 g/mol. Some of Synonyms are castorwax; castorwax MP 70, Castorwax 80; Croduret; ricini oleum hydrogenatum. HCO is refined, bleached, hydrogenated, and deodorized Castor Oil [6]. Chemical name of hydrogenated castor oil is Glyceryl-tri-(12-hydroxystearate). Structural formula has been below (Figure 1):

Iodine Value in Partially Hydrogenated Castor Oil (Ricinus Oil) as determined by AOCS Official Method Cd 1-25 (Wijs' Method)

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Determination of the Oxidative Stability of Fats and Oils: Comparison between the Active Oxygen Method (AOCS Cd 12-57) and the Rancimat Method

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Oxidative stability is an important parameter in the characterization of fats and oils. The determination of this parameter with the Active Oxygen Method (AOM; AOCS Method Cd 12-57) is both very costly and labor intensive, owing to the repeated peroxide value determinations involved. The alternative rancimat method is based on the conductometric determination of volatile degradation products and features automatic plotting of the conductivity against time. The evaluation is performed graphically after completion of the experiment. The labor required for this method is considerably less as it is not necessary to perform titrations at regular intervals. In the determination of the peroxide values of six samples at three temperatures, ca 15 l mixed solvent and chemicals valued at SFr. 400 (ca 180 US) were consumed.

The induction times (t_i) determined with both methods using six different fats and oils show a good correlation (slope 1.005, correlation coefficient 0.987). The rancimat method thus yields results equivalent to the AOCS Method Cd 12-57, but offers a real alternative for the determination of oxidative stabilities owing to the appreciable saving in labor.

Oxidative stability is an important parameter for the quality assessment of animal and vegetable fats and oils. Autoxidation is effected by atmospheric oxygen; the oxidation process is initiated by radical reactions involving unsaturated fatty acids (1-3). The primary products formed are hydroperoxides, which then break down in a series of complex reactions, the exact nature of which is still under investigation; the secondary products include alcohols and carbonyl compounds (1-3). These can be oxidized further to carboxylic acids (4).

In order to determine oxidative stability, a fat is exposed to a stream of dry air at a temperature of 100-140 °C. The progress of the oxidation curves can be followed by periodic determination of the peroxide value (PV) (Active Oxygen Method, AOM; AOCS Method Cd 12-57) or other parameters. The curves comprise an induction phase, in which practically no secondary products are formed, and an oxidation phase, during which a large increase in peroxide value and volatile products is detected.

The method developed by Hadorn and Zürcher (5) and which is used in the 617 Rancimat (METROHM AG, CH-9100 Herisau, Switzerland) (6) (Fig. 1) utilizes the fact that the greater part of the volatile products consists of formic acid (7). These volatile components are trapped in distilled water, measured conductometrically

and the conductivity plotted automatically. The progress of the oxidation curves determined in this manner virtually parallels the development of the peroxide value (5). The t_i (point of greatest inflection) is determined graphically after completion of the experiment (tangential intersection point, see Fig. 2). The apparatus requires no supervision during the course of an experiment (e.g. overnight).

On the other hand, in the procedure involving the AOM, the peroxide value must be determined at regular intervals throughout the whole experiment. The end of the t_i is considered as the attainment of a PV of 100 µeq/kg and is evaluated by interpolation of two experimental points between PV = 75 µeq/kg and PV = 175 µeq/kg.

In what follows, it will be shown that the results obtained with the rancimat method correlate extremely well with those of the AOM test.

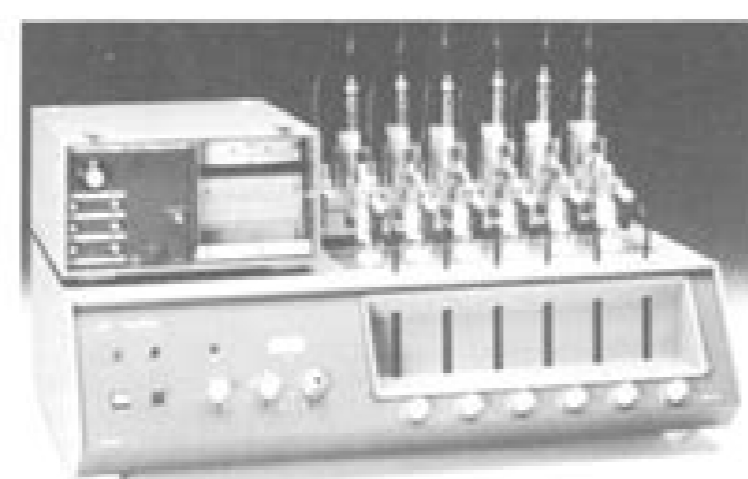


FIG. 1. 617 Rancimat.

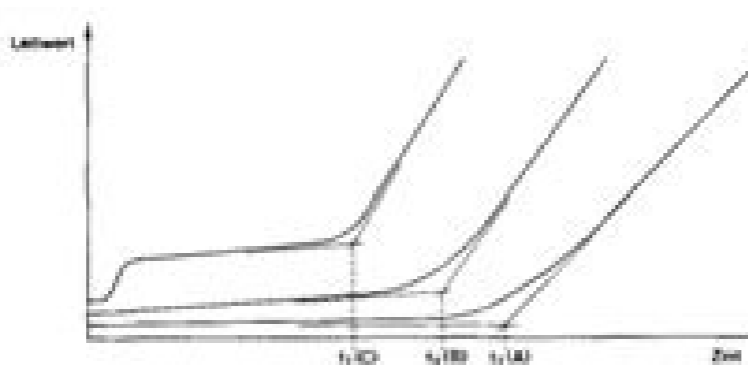


FIG. 2. Graphic determination of the induction time (t_i) by the tangent method. A and B, typical conductivity curves; C, curve with initial step.

*To whom correspondence should be addressed.

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