



A novel method for determining peroxide value of edible oils using electrical conductivity

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ABSTRACT

In this study, a new method for determining peroxide value (PV) of edible oils was described. The basis of this method was the determination of changes in the electrical conductivity (EC) values of the aqueous phase during the reaction of potassium iodide (KI) with the hydroperoxides presented in oil samples. The effect of various analytical conditions was evaluated, such as temperature, holding time, oscillating time, stability of the saturated KI solution and oil types. The calibration was developed and validated using the EEC, AOCS, IUPAC, and AOAC methods. The results showed that the analytical conditions had no significant ($P > 0.05$) influence on the EC determination at room temperature; the calibration equation of PV was obtained as $y = 150.54x - 0.0327$ (where x is the change in EC values, S/m; y is PV, mmol/kg), with $R = 0.9980$ and $SD = 0.4045$. The linear correlativity between the proposed method and AOCS method was very high ($R = 0.9992$), and the standard deviation of the blind samples used in the experiments was slightly better than that of the EEC, AOCS, IUPAC, and AOAC procedures, which illustrates the feasibility of determining PV of edible oils using the proposed method.

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1. Introduction

Fats and oils are widely employed in the food industry, but they get easily oxidized during processing, circulation, and preservation. The oxidation of fats and oils in food can result in food toxicity (Gotoh et al., 2011; Gotoh & Wada, 2006; Gotoh et al., 2006). From the perspectives of food quality and safety, the determination of the peroxide value (PV) is one of the most important quality control measurements for edible oils because it is an indicator of the primary oxidation status of the oils (Akinoso, Aboaba, & Olayanju, 2010; Pizarro, Esteban-Díez, Rodríguez-Tecedor, & González-Sáiz, 2013). This indicator measures the concentration of hydroperoxides (the primary oxidation compounds) that are not stable and can easily decompose into secondary oxidation products (such as ketones and aldehydes), and are considered to be responsible for the off-flavor in oxidized oils (Choe & Min, 2006; Mohammadi, 2013).

As an indicator of oil quality, the quantification of PV is fairly important. PV indicates the quantity of the peroxides reported as milliequivalents of active oxygen in 1000 g of an oil sample. Over the past few years, a relatively large number of methods had been developed for determining PV of edible oils. The conventional American Oil Chemist's Society (AOCS) (2003) method used for

determining the PV involves iodometric titration that measures the iodine liberated from potassium iodide (KI) after reacting with the peroxides present in oil samples (Cirlini, Caligiani, Palla, De Ascentiis, & Tortini, 2012; Ruíz & Lendl, 2001), and other PV standard determination methods such as Association of Official Analytical Chemists (AOAC) (1990), Commission Regulation of European Economic Community (EEC) (1991), and International Union of Pure and Applied Chemistry (IUPAC) (1991) methods are similar to AOCS method; these methods are labor-intensive, time-consuming, requires a large amount of lipids, and more importantly, its accuracy depends strongly on the analytical conditions (Armenta, Garrigues, & de la Guardia, 2007; Zhao, Zhang, Dang, Wu, & Hu, 2013). To overcome the above disadvantages, a number of alternative methods and techniques for determining PV had been reported in the literatures, such as the chemiluminescent method (Bezzi, Loupassaki, Petrakis, Kefalas, & Calokerinos, 2008; Stepanyan, Arnous, Petrakis, Kefalas, & Calokerinos, 2005), flow-injection method (Dhaouadi, Monser, Sadok, & Adhoum, 2006; Saad, Wai, Lim, & Saleh, 2006), and high-performance liquid chromatography (Gotoh et al., 2011). These methods afford more simplicity as compared to iodometric titration at the expense of introducing more expensive instruments into the analysis. Further, the obvious disadvantage of the spectrophotometer method (Dermiş, Can, & Doğru, 2012; Muresan et al., 2010; Talpur, Sherazi, Mahesar, & Bhutto, 2010) was the obtained results were not accurate when the oil contained compounds that naturally absorb in the

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measurement wavelength range (Hornero-Méndez, Pérez-Gálvez, & Mínguez-Mosquera, 2001). Spectroscopy technologies were also applied to determine the PV, including near-infrared spectroscopy (NIR) (Wu, Xing, Xiao, Xu, & Zhang, 2012), and mid-infrared spectroscopy (MIR) (Pizarro et al., 2013; van de Voort, Ismail, Sedman, Dubois, & Nicodemo, 1994; Yu, Du, van de Voort, Yue, & Li, 2009; Yu, van de Voort, & Sedman, 2007). NIR and MIR spectroscopy had received considerable attention when it comes to the quality control of edible oils as they offer a simple, efficient, nondestructive, and low-cost solution for quality control in the industry (Ruíz & Lendl, 2001). Several studies on the determination of PV using these methods had been documented (van de Voort et al., 1994; Yu et al., 2009; Yu, van de Voort, & Sedman, 2007). Although the methods offer numerous advantages, they have found limited their applications because they require the use of expensive instruments (Yu, Yang, Du, & Gao, 2012).

Based on the consideration of practicality, the present investigation focused on the proposal of a new method to determine the PV of edible oils. In this study, an electrical conductivity (EC) instrument was employed. The ability of a material or solution to conduct an electric current can be detected using this EC instrument. The EC of a solution is a very useful property because it depends mainly on the chemical composition and ionic concentration of its constituents (Yu et al., 2012). Ions are not present in edible oils, so the oils cannot conduct an electric current. However, inferior oils, deep fried oils, and adulterated oils contain several ions, such as total polar components, free fatty acids (FFA), metal ions, and so on. When these oils were dispersed in deionized water, the EC values of the aqueous phase were higher when compared to those of qualified oils. Based on this principle, Zhou, Chen, Mao, and Zhao (2011) developed a method for measuring quality of oils by determining the EC values of oils, and the results obtained using the EC method and those obtained using the other methods were highly consistent; moreover, the EC method showed good repeatability. However, the accuracy was not good, because the EC values were lower and the error in instrument reading was large. In addition, the results obtained using the EC method represented the total EC value, Yu et al. (2012) improved this method to determine the FFA content in edible oils based on the changes EC values of a potassium hydroxide solution layer during the reaction of potassium hydroxide with the FFA. These results indicated that the EC method could be applied to detect the quality of oils, and this method was simple and practicable.

In this work, the effects of analytical conditions on the EC values were investigated to establish a new method for determining the PV of edible oils. This method was based on the changes in the EC values of the aqueous phase during the reaction of KI with the hydroperoxides present in the oil samples. PV calibration was performed by using the relationship between the changes in the EC values and the PV; the calibration was then validated.

2. Materials and methods

2.1. Materials and reagents

Various edible oils were obtained from local retail outlets. Among these oils, refined rapeseed oil was used as the base oil for preparing the calibration standards, whereas the other oils included blind oil samples that were obtained from a local market. Isooctane was obtained from the Xi'an Chemical Company, Ltd, and hydrochloric acid and KI were purchased from the Tianjin Chemical Company, Ltd. All the used chemicals and reagents used were of the analytical grade.

2.2. Apparatus

The EC instrument DDSJ-308A obtained from the Shanghai Precision & Scientific Instrument Co., Ltd. was used.

2.3. Determination principle

The fundamental principle of PV determination was based on the changes in the EC values of the aqueous phase during the reaction of KI with the hydroperoxides present in the oil samples. When the temperature was constant, the EC value depended on the ionic concentration. After reacting with hydroperoxide using excessive KI, the EC value decreased with a reduction in the concentration of the KI solution. Then, the changes between the control groups (a control group was detected in the same way without the addition of oil samples) and experimental groups in the EC values were used to develop the calibration and to determine the PV of edible oils. This determination should be finished within a short time to protect from light and avoid the oxidation of KI.

2.4. Optimization studies

Optimization studies on the effects of temperature, holding time, oscillating time, stability of saturated KI solution and oil types were performed. The adopted experimental conditions were a compromise between the analytical conditions and changes in the EC values. A solution was obtained by 2 mL of isooctane, 0.06 mL of hydrochloric acid, and 4.000 g of oil samples, which was hereafter to as the "reference solution"

2.4.1. Effect of temperature

The effect of temperature on the EC value was determined after holding the reaction solution at the specific temperatures (24.5 °C, 25.0 °C, 25.5 °C, 26.0 °C, 26.5 °C, and 27.0 °C) for 160 s. One milliliter of saturated KI solution was added to the reference solution and stirred for about 60 s; then 50 mL of distilled water was immediately mixed with the reaction solutions.

2.4.2. Effect of holding time

At the ambient temperature, the EC values of the aqueous phase during the reaction were determined at different standing times. One milliliter of saturated KI solution was added to the reference solution; 50 mL of distilled water was immediately mixed with above reaction solution after the mixtures was stirred for approximately 60 s; this mixture was held for different times ranging from 0 to 480 s with 80 s intervals.

2.4.3. Effect of oscillating time

At the ambient temperature, the effect of oscillating time on the EC values of the aqueous phase during the reaction was determined. The reference solution was mixed with 1 mL of saturated KI solution. Then, 50 mL of distilled water was immediately mixed with this reaction solution and the mixtures was stirred for different times ranging from 30 to 180 s and held for 160 s in all the cases.

2.4.4. Stability of saturated KI solution

At the ambient temperature, the EC value of the aqueous phase during reaction was determined. Here, 1 mL of saturated KI solution standing for various times ranging from 5 to 180 min was added to the reference solution; 50 mL of distilled water was immediately mixed with this reaction solution; the mixtures was stirred for about 60 s and then held for 160 s.

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