



# A simplified UV spectrometric method for determination of peroxide value in thermally oxidized canola oil

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## ABSTRACT

The aim of present study was to develop a simple method on UV spectrometer for the determination of peroxide value (PV) of the frying oil. The basis of the PV determination was the stoichiometric reaction of triphenylphosphine (TPP) with the hydroperoxides present in frying oil to produce triphenylphosphine oxide (TPPO), which exhibits a readily measurable absorption band at 240 nm by ultraviolet region. The PV ranged between 0.15 and 11.66 meq. of active oxygen per kilogram of oil as the canola oil was heated from 0 to 12 h in the fryer at 180 °C. The proposed method was correlated with official AOCS titration method and best correlation coefficient ( $R^2 = 0.99525$ ) was achieved, proving that there is no significant difference in the results. Therefore, developed method could serve as an alternative to the titration method, for the determination of PV in frying oils.

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## 1. Introduction

Basically frying is a cooking method in which food is submerged in hot oil. During frying thermo-oxidative or lipid oxidation and hydrolytic reactions take place that results in deterioration of the frying oil [1–3]. The primary oxidation products that develop in triacylglycerols are hydroperoxides, which may later break down to produce lower molecular weight compounds, such as free fatty acids, alcohols, aldehydes, and ketones, eventually leading to rancid product [4]. Lipid oxidation is a main deteriorative process which has an important implication in stipulations of the quality and value of fats and oils, particularly in relation to the off-flavors that develop as an outcome of autoxidation [5]. The level of hydroperoxides is commonly determined by using well-established standard AOCS iodometric procedure [6], and the resulting peroxide value (PV) is expressed as milliequivalents (meq.) of peroxide oxygen per kilogram of oil. Besides the sensitivity and reproducibility, the titrimetric methods are laborious and need a lot of chemicals and solvents, hence are associated with environmental issues. In the literature some other methods have been reported for PV determination that were based on both direct and indirect measurement of hydroperoxides by spectrophotometry [7,8] and infrared spectroscopy [9,10,11,5]. The common spectrophotometric method is very complex and involves numerous steps with the use of several chemicals and reagents [12]. Subsequently, a very useful and

more accurate method was developed by Xiuzhu Yu et al. [5] on FT-IR spectroscopy using an established stoichiometric reaction of triphenylphosphine (TPP) with hydroperoxides developed in oil to form triphenylphosphine oxide (TPPO). Our present work was focused on to develop an effective UV spectrometric method based on a stoichiometric reaction involving hydroperoxides. According to well-recognized reaction (Fig. 1), hydroperoxides present in the oil react stoichiometrically with TPP to form TPPO and their respective alcohols.

## 2. Experimental

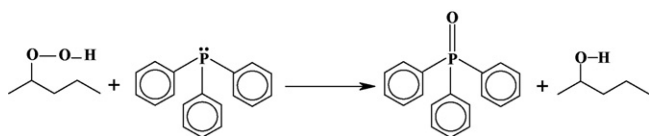
### 2.1. Reagents, standards and samples

All chemicals and reagents used were of analytical grade. Chloroform was purchased from Fischer scientific UK Ltd. Triphenylphosphine (TPP), Triphenylphosphine oxide (TPPO), potassium iodide, sodium thiosulphate and acetic acid were obtained from Sigma–Aldrich Chemie GmbH (Buchs, Germany). The concentrated stock solution for working standards was prepared by dissolving TPPO in the chloroform (40%). Fresh refined, bleached and deodorized canola oil was obtained from the local edible oil industry in Karachi, Pakistan.

### 2.2. Determination of peroxide value

Peroxide value, expressed in milliequivalents of active oxygen per kilogram of oil (meq./kg), was determined by standard procedure by a mixture of oil and chloroform/acetic acid which was

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**Fig. 1.** Reaction of hydroperoxides with triphenylphosphine (TPP) to form triphenylphosphine oxide (TPPO) and alcohol.

left to react with a solution of potassium iodide in darkness; and then free iodine was titrated with a sodium thiosulfate solution [13].

### 2.3. Standard and sample preparation

Stock solutions (0.7 g) of TPPO (40% in chloroform) were mixed into 100 g of zero-PV canola oil to produce base oils which contained 50 PV equivalents of TPPO [9]. Calibration standards ranging from 0.5 to 50 PV were then prepared by gravimetrically mixing various amounts of stock oils of 30 PV in the chloroform. Just for 30 s, all the standard solutions were heated in the microwave oven to dissolve the TPPO completely. Furthermore, the PV values of standards were also confirmed by standard AOCs titration method. Similarly the preparation of oil samples involved the mixing of thermally oxidized canola oil (0.7 g) with TPP (40% in chloroform) in stoppered Erlenmeyer flask and kept in microwave oven for 30 s to complete reaction of peroxide of oil with TPP to form TPPO.

### 2.4. Instrumentation

#### 2.4.1. UV–visible spectrophotometer

UV–vis spectra were recorded the respective absorbance value at specific wavelength on a PerkinElmer Lambda 35 UV/visible spectrophotometer, using standard 1.00 cm quartz cells.

#### 2.4.2. Domestic microwave oven

A domestic microwave oven, Pell-PM 023 (Japan) with power settings range from 100 to 900 W was used in the present study. All standards of TPPO in canola oil and chloroform as well as samples with TPP in chloroform containing glass vial were subjected to full power (900 W) of microwave irradiation. After 30 s microwave heating the vial was taken out and shaken vigorously

to cool. Then the UV spectra of each standard and sample were taken.

#### 2.4.3. Heating procedure

The west point deep fryer (E-2016) was used for carrying out the thermal oxidation of canola oil at frying temperature. The capacity of fryer was 3 L with thermostatic temperature control up to 180 °C. 2 L pure canola oil was initially heated to about 180 °C, then kept at constant frying temperature i.e. 180 °C for 12 h. After each hour heating interval, about 10 mL of the heated canola oil was filtered into a screw cap vial and stored at 4 °C until analysis. The volume of oil was not refilled during the heating process.

#### 2.4.4. Calculations and statistical analyses

Each standard and sample was analyzed twice. The data obtained were put into OriginPro 7.5 program and reported as mean ( $n = 1 \times 2 \pm SD$ ).

## 3. Result and discussion

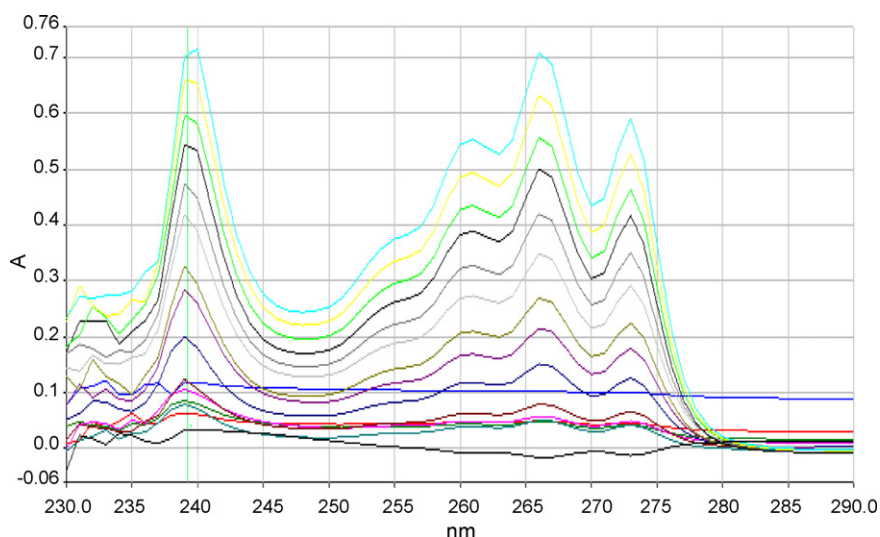
### 3.1. General spectroscopy

The UV spectral characteristics of TPPO in canola oil at various concentrations were investigated by adding the TPPO to zero-PV canola oil. The recorded UV spectra of the standards for the calibration with 0, 0.5, 1, 2, 4, 6, 8, 10, 15, 20, 25, 30, 35, 40, 45 and 50 PV are shown in Fig. 2. Sophisticated chemometric technique i.e. PLS was used to achieve the regression equation for the quantification of PV in the thermally oxidized canola oil.

### 3.2. PLS calibration

A PLS calibration model was developed to predict PV based on the quantitation of TPPO using the absorbance at 240 nm. Excellent correlation coefficient of 0.99885 was achieved which proves the good accuracy of proposed method as shown in Fig. 3. Following regression equation was achieved from the plot of various PV of the standards versus UV absorbance at 240 nm (Fig. 3) by using Origin-Pro 7.5 program to predict the unknown PV in thermally oxidized canola oil.

$$Y = 0.03193 + 0.01385X$$



**Fig. 2.** Spectra showed the calibration range of TPPO concentration in canola oil vs absorbance for PV from 0, 1, 2, 4, 10, 15, 20, 25, 30, 35, 40, 45 and 50 PV.

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