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Application Notes
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Peroxide value measurement in edible oils

Introduction

Peroxide is a useful indicator in determining the levels of oxidation in fats and oils. Its level of existence is called peroxide value (PV) and is a measure of total amount of peroxide in a substance. It is one of the main reasons behind the rancidity, production of toxins in edible oils. The autooxidation reaction between atmospheric oxygen and lipids generally leads to oxidative degradation of lipids. Enzymatic, thermal and photo oxidation process are also responsible for oil degradation.

Edible oils with high level of PV values have the ability to potentially cause the initiation of degenerative disease, cancers etc. This application note describes a process to analyze PV in edible oils using UV spectrophotometer.

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Principle

$Fe^{(III)}$XO (XO = Xylenol) complex formation is used to detect the concentration of hydroperoxides. $Fe^{(II)}$ can be oxidized by H2O2 to $Fe^{(III)}$ which will sequentially coordinate with XO to yield purplish red $Fe^{(III)}$XO complex as shown below

$$Fe^{(III)} + H_2O_2 + H^+ \rightarrow Fe^{(III)} + H_2O + {}^{\cdot}OH$$
$${}^{\cdot}OH + \text{xylenol} + H^+ \rightarrow \text{xylenol}^{\cdot}$$
$$\text{xylenol}^{\cdot} + Fe^{(II)} \rightarrow \text{xylenol} + Fe^{(III)}$$
$${}^{\cdot}OH + Fe^{(II)} + H^+ \rightarrow Fe^{(III)} + H_2O$$
$$Fe^{(III)} + \text{xylenol} \rightarrow Fe^{(III)} - \text{xylenol}$$
(Purplish red)

These reactions are fast, sensitive and reproducible. The concentration of H2O2 can usually be determined by spectrophotometrically measuring purplish red color of Fe(III)XO complex in solution.

Reagents

- Xylenol orange sodium salt
- Hydrogen peroxide
- Barium chloride dehydrate
- Ferric chloride solution
- Ferrous sulphate heptahydrate
- Chloroform
- Methanol
- Hydrochloric acid

Preparation of $Fe^{(II)}$ chloride solution

- 0.5 g $FeSO_4$, 7 H_2O dissolved in 50 mL of distilled water to prepare an iron sulphate solution.
- Barium chloride (0.4 g) is dissolved in 50 mL of distilled water.
- The resultant solution is added to the $Fe^{(II)}$ sulphate solution slowly with constant stirring.
- Then, 2 mL of 10 N HCl was added to the solution. Barium sulphate recipitate is formed.
- The barium sulphate precipitate is then filtered off to obtain a clear $Fe^{(II)}$ solution.
- It is kept in the dark in a brown bottle. The solution is stable for up to 1 month.

Procedure

- Edible oil samples equivalent to ~ 0.2 g are added with 9.8 mL of a mixture of chloroform and methanol (mixed in 7:3 ratio), in capped vials on a vortex mixer for 5 s.
- Then, 100 mL of 10mM xylenol orange is mixed and vortexed for 5 s.
- 50 mL of 36 mM $Fe^{(II)}$ solution is added and mixed on a vortex mixer for 5 s.
- After 5 min of incubation at room temperature, the absorbance of the sample is determined at 560 nm by a UV-Vis spectrophotometer.

Calibration curves for 2 - 40 µg/mL $Fe^{(III)}$ are prepared by the use of 100 µL xylenol orange.