



## Bruker White Paper Series (microESR for Oxidative Resistance in Edible Oil)

### Innovating to facilitate prediction of edible oil stability

#### Introduction

Edible oils consist of about 96% triacylglycerides, as well as free fatty acids, phospholipids, phytosterols, tocopherols, and a variety of other antioxidants. They are the primary source of unsaturated fats and vitamin E in the human diet and serve a range of essential functions, including vitamin metabolism and cell wall stability.

Across the food manufacturing industry, edible oils are widely used to enhance both flavor and functional properties. However, they must be used with care since all edible oils are prone to spoilage during storage. This presents the development of a rancid taste and smell as well as changes in color, viscosity, density, and solubility<sup>1</sup>.

Since lipids are readily oxidized at room temperature, this is the main factor determining how long edible oils can be stored without loss of quality<sup>2</sup>.

It is thus important to evaluate the oxidative stability of oil in order to determine its shelf life and evaluate whether this can be increased by the addition of antioxidants<sup>3</sup>. The storage test is the most reliable way to determine the shelf life of food, but this is not always economically viable as it can take many months to get the result.

Adequate evaluations can be obtained using a range of analytical techniques, but these often necessitate a series of analyses using different methodologies to provide information on all the important characteristics<sup>4,5</sup>. It now appears that the analysis of edible oils for the determination of their functional and storage properties could be revolutionized by the use of sophisticated spectroscopic techniques.

#### Oxidation of oils

Oxidation is a key mechanism of food spoilage and is consequently a reaction that food manufacturers strive to limit. In edible oils, the oxidation process results in the formation of short-chain aroma-active compounds that are responsible for the unpleasant taste and smell of deteriorating lipids.

The oxidation process in edible oils is essentially a free radical chain reaction and the extent to which an oil is susceptible to oxidative spoilage is determined by the composition of the constituent fatty acids<sup>7</sup>. The more unsaturated and less saturated a fat is, the faster the oxidation reaction proceeds<sup>8</sup>.

Determination of oxidative stability is one of the most important quality parameters of edible vegetable oils. Methodologies to

increase an oil's resistance to oxidation, such as the addition of antioxidants, are continually being explored. Mixtures of synthetic antioxidants, usually phenols, typically provide better protection against oxidation than a single antioxidant<sup>6</sup>.

Evaluation of the propensity of edible oil to undergo oxidation allows companies to understand how to control the oxidation process and ensure the best taste and appearance of their products and develop products that maintain their quality and freshness for longer after production. The ability to acquire this information readily enables more productive research and development, enabling companies to increase customer satisfaction whilst remaining competitive.

## Evaluation of oil oxidation

In the food industry, many methods have been used to determine the oxidative stability of oils. Although the storage test is the most reliable test of shelf-life, it is rarely used now since it takes too long. It has been replaced by thermostatic test and chemical determinations of stability, such as the Rancimat test, and the Schaal Oven Test.

The thermostat test uses temperatures of 30–63 °C and closely mimics the changes observed in natural storage conditions. However, it still takes up to several weeks for fat oxidation to occur at these temperatures.

The Rancimat test determines the oxidation stability of oils more quickly but has the disadvantage of requiring high temperatures to produce volatiles and intensive aeration, which can change the nature of the oxidation process<sup>9</sup>. Nonetheless, it has been shown to be a relevant analysis for evaluating the oxidative stability of oils in comparison with other methods, such as differential scanning calorimetry (DSC)<sup>4</sup>.

Estimations of oxidative stability can also be obtained through the measurement of free radicals, which begin oxidative degradation by reacting with the double bonds in the fatty acid chains. Free radicals can be detected by spectrophotometry, chemiluminescence, and fluorimetry method. Electron spin resonance (ESR) spectroscopy is the most direct and efficient means of detecting free radicals in edible oil and has been widely applied to the analysis of foodstuffs<sup>6,10,11</sup>.

## ESR in the evaluation of oil oxidative stability (Comparison to the Rancimat Test)

Electron spin resonance spectroscopy has been utilized to facilitate investigations of free radical production across a wide range of applications, from food science to cancer chemotherapy to nanotechnology<sup>4,12</sup>. The popularity of this technology is largely due to its high sensitivity and the ability to identify the generation of free radicals in situ.

ESR has now been developed as a rapid test to evaluate the oxidative stability of different edible oils and the effect of several antioxidants.<sup>13</sup> The concentration of free radicals present in peanut oil, palm oil, rapeseed oil, soybean oil, sunflower seed oil, and corn oil with and without the addition of a selection of antioxidants, was measured using a Bruker ESR spectrometer. Induction periods were determined using the Boltzmann equation and compared with those obtained using the Rancimat method.

High linear correlation was found between the two methods, indicating that a rapid ESR method can predict the oxidative stabilities provided by the Rancimat method<sup>13</sup>.

This latest research thus demonstrated that ESR is a sensitive method for the rapid evaluation of the oxidative stability of edible oils.

The authors concluded that ESR is an efficient and accurate method to study the shelf life of edible oils and other foodstuffs.

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info@bruker.com  
www.bruker.com