Review Article

Evaluation of Stability against Oxidation in Edible Fats and Oils

Selin Şahin[*]

Istanbul University-Cerrahpaşa, Engineering Faculty, Department of Chemical Engineering, Istanbul, Turkey

[*]**Corresponding Author**

Selin Şahin, Istanbul University-Cerrahpaşa, Engineering Faculty, Department of Chemical Engineering, 34320 Avcilar, Istanbul, Turkey, Tel: +90-212-4737070; Fax: +90-212-4737180; E-mail: selins@istanbul.edu.tr

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Abstract

There is a lipid oxidation problem in edible oils and fats due to the fact that they are continuously exposed to various storage conditions or overheating. Therefore, investigation of the stabilization of edible oil products is an area worth researching due to consumer-related health concerns. Consequently, thermal oxidative deterioration might be prevented if the stability of the product is comprehended fairly. Thus, food formulations can be regulated depending on the findings of the stability measurements. In this review article, the mechanism and structure of lipid oxidation process have been explained. Furthermore, the significance of prediction of shelf-life has been mentioned as well as kinetic and thermodynamic comprehension in oxidation process. Additionally, all the mentioned stability measuring methods have been discussed, respectively.

1. Introduction

Consumption of foodstuffs keeps increasing as a result of the rise in human population of the world. So, consumption of vegetable oil, which is one of the basic components of foodstuffs has been rising rapidly. With the increase in the need for vegetable oil in the food industry, it has been emerged to carry out operations in this direction with the aim of preserving the existing quality without deterioration until the vegetable oil reaches the consumers from the production stage. The big quality problem in vegetable oil industry is *Lipid Oxidation* (LO), which gives rise to the existence of unsatisfactory and toxic compounds in the relevant products [1].

Some tests have been arisen for the determination of lipid oxidation in the selected oil at accelerated conditions [2, 3, 4]. A thermal analysis method known as *Differential Scanning Calorimetry* (DSC) is a more than fifty year-old method to assess the thermal oxidation process in lipid containing products [5, 6]. *Active Oxygen Method* (AOM) is another way for measuring the resistance of the fat-containing sample to oxidation [7, 8]. Oxygen uptake method (oxydograph) method has been also applied to anlayse the oil stability [9]. *Thermogravimetric analysis* (TGA) might be utilized to evaluate the oxidative stability in the fats and oils by detecting the related sample's mass change through thermal degradation [10]. Fourier transform infrared spectra (FTIR) might also be evaluated for detection of the decomposition of the oxidative stability in fats and oils [11]. Schaal oven is also known as another accelerated assay for measuring the stability of oils [12]. Rancimat is an easy test method, including parameters such as amount of sample, air flow rate and temperature [13]. It requires no extra analyses such as titrations with too much time and chemical consumptions [14].

2. Lipid Oxidation

Deteriorative intermediates of lipid oxidation has adverse effect on shelf-life and characteristics (deterioration some physical properties such as of taste, color and odour) of lipid-containing food products [15]. Formation of this hazardous intermediate products in the concerning food is also inconvenient for the health of consumers. The relevant oxidation process is attributable to 2 structures of oxygen, which are singlet ($^1O_2$) and triplet ($^3O_2$) oxygens [16]. Those species are also known as *Reactive Oxygen Species* [17].

Figure 1 represent the 3 stages (initiation, propagation and termination) of LO process. *RH* represents the fatty acids/acylglycerols in fat-containing food product oil, while *R* is the lipid alkyl occured in the initiation stage depending on the food processing, transportation and preservation conditions [18]. Later, *R·* forms *ROO·* (lipid peroxy radical) by means of the reaction with triplet oxygen. This reactive species also reacts with the hydrohen of another *RH*, leading to generation of anaother *R·*. In the end of the related chain reactions, undesired and toxic compounds such as aldehydes and ketones, hydrocarbons, organic acids, volatile and polymeric compounds [3]. In the termination step, the reaction is terminated after lipid alkyl radicals react with each other [16].

3. Prediction of Shelf-life

It is well known that *peroxide value* (PV) is usually used as quality parameter for primary lipid oxidation, but if oil goes rancid further to secondary oxidation, PV is not necessary indicating oxidation status. Actually, PV is a measure of the extent of primary oxidation reactions but primary reactions do not prevent rancidity development but they are just the early reactions of lipids oxidation [19]. In order to avoid such inconsistencies, several oxidation tests have been emerged to comprehend the oxidation in the relevant

products under several conditions. On the other hand, kinetics and thermodynamic factors are necessary for prediction of the lipid oxidation in oils under several conditions for composing better preparations regarding quality indicators [20]. The time for the resistance of the oil againt to oxidation is expressed as induction time (*IT*), which is utilized as an indicator for oxidative stability of fat-containing foods [21].

The following model describes the relationship between the induction time (stability of the oil against oxidation) and temperature:

$$\log IT = a(t) + b \tag{1}$$

t= Temperature (°C)
a, b= Coefficients of the Eq.(1)

Arrhenius equation is used to define the robust relationship between the reaction rate constant (*k*) and the temperature in lipids:

$$\ln k = \ln A - \frac{E_a}{R}\frac{1}{T} \tag{2}$$

k= Reaction rate constant (h^{-1})
E_a= Energy for activation (kJ mol^{-1})
A= Frequency factor
R= Universal gas constant (J mol^{-1} K^{-1})
T= Absolute temperature (K)

Actually, *k* is stated with the reciprocal of the induction time, since lipid oxidation in fats is assumed as first-order kinetic reaction [20]. Thermodynamics of a chemical process is a must to have knowledge about the nature of a system. Using Activated Complex approach, enthalpy (ΔH^o) and entropy (ΔS^o) were derived from the Eyring equation:

$$\ln \frac{k}{T} = \left[\left(\frac{k_B}{h}\right) + \left(\frac{\Delta S^\circ}{R}\right)\right] - \left(\frac{\Delta H^\circ}{R}\right)\left(\frac{1}{T}\right) \tag{3}$$

Where k_B is Boltzmann (1.38065×10^{-23} J K^{-1}) constant, and and *h* is known as Planck's (6.62608×10^{34} J s) constant. One of the fundamental equations of thermodynamics is applied to calculate the change of Gibbs free energy (ΔG^o, kJ mol^{-1}) through enthalpy change, the universal gas constant and absolute temperature:

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \tag{4}$$

Thermodynamic parameters obtained from Eqs.(3) and (4) are necessary in order to decide if the chemical reaction will be spontaneous or non-spontaneous, exergonic or endergonic, and exothermic or endothermic in the future.

3.1 Active oxygen method

Active oxygen method (AOM) is an option to identify the oil oxidation stability. A specific amount of sample is exposed to air at an elevated temperature. The sample is received regularly from the analyzed substance to measure the peroxide value. The time required to attain a certain amount of peroxide value is accepted as an indicator for the oxidative stability [9]. However, it has many disadvantageous such as being labor-intensive and costly due to the fact that AOM contains analyses such as the peroxide titration with too much time and chemical consumptions [8]. Therefore, the relevant method has not been applied into the fats and oils recently, although the literature has been reviewed thoroughly for the last 5 years.

3.2 Schaal oven test

Schaal oven test is accepted as the easiest method among the accelerated oxidation tests since certain

amount of oil sample is heated in an oven at aroun 70 °C [9]. However, it requires extra analytical methods such as PV, p-anisidine value and iodine value determinations [22]. After treated with rosemary extract, cottonseed, soybean and rice bran oils were analysed with Schaal oven test at 62 °C by measuring their PV, total phenolic and tocopherol contents, antioxidant activity and fatty acid concentration [1]. Thermal oxidative stability of refined palm olein enrihed with natural antioxidants was also assessed by Schaal oven test by measuring its total oxidation, peroxide, iodine, p-anisidine and thiobarbituric acid values for 30 days [22, 23]. Schaal oven test was also performed by Souza et al. , where chia oil was monitored with respect to thermal oxidation according to its fatty acid and α-linolenic change [24]. Oxidation process of sunflower oil enriched by cold-pressed black cumin oil was investigated by variation in PV, conjugated dienes and trienes, tocopherols, tyhymoquinone and volatile compounds [25]. Moringa oil was monitored depending on its structure variation due to the thermal oxidative degradation through infrared spectra [26]. Several edible oils were compared depending on their thermal oxidative stability by measuring their peroxide and anisidine values under Schaal oven test conditions [27]. In order to see the effect of lycopene on the quality of walnut oil, Schaal ovent test was applied by measuring its antioxidant activity, total phenolic ingredient, PV, acid value and fatty acid [28]. Recently, Kiralan et al. used this method for the observation of thermal oxidative stability of grape, flax and black cumin seed oils by measuring the PV and conjugated dienes [29].

3.3 Fourier transform infrared spectroscopy

Fourier transform infrared (FTIR) spectroscopy has been proposed to be an alternative in deteriation studies of fat-containing food products with its advantegous as an easy, rapid and precise [30]. Some specific peaks can be examined during thermal oxidation process. Hu et al. used FTIR spectroscopy combined with infrared quartz cuvette to observe the oxidation in several edible oils (rapeseed, cottonseed, walnut, sesame, linseed, sunflower and soybean) by determination of acid value [31]. Shang et al. also used FTIR with infrared quartz cuvette to detect the oxidation process in several oil samples by identification of PV [32]. Canola oil was also investigated with FTIR regarding oxidative stability to comprehend the effect of heating and frying [33]. Heating (conventional and microwave) effect on the thermal deterioration of corn and soybean oils was monitored by peak changes infrared spectra of FTIR, proving the degradation of the products due to the secondary oxidation compounds [34]. Corn, sunflower, colza and a mixture of frying oils were studied by FTIR coupled with attenuated total reflectance (ATR) with the assistance of multivariate curve resolutionalternative least square (MCR-ALS) [35]. It was also proposed as a cost effective method as well as including no time-consuming sample preparation stage. FTIR technique together with a mesh cell was applied to determine the change of some functional groups during the lipid oxidation in some oils under ambient storage conditions [36]. Tena et al. also used similar technique to study the stability of virgin olive oil [37].

3.4 Thermogravimetric analysis

Thermal deterioration of the oils due to the lipid oxidation can be also examined by thermogravimetric analysis (TGA). Oxidation process in the product is monitored by means of weight change according to the oxygen taking and thermally deterioration [10, 38]. Gao and Birch suggested TGA as a relatively simple and time-saving method for the prediction of oxidation initiation in flax, hemp, and canola seed oils [39]. Shelf-life of hoki oil was estimated as 0.56 by using TGA through Arrhenius extrapolation, while it was predicted as 1.39 years by DSC [10]. Li et al. exploited TGA to compare the thermal oxidation stabilities of several

vegetable oils such as palm, rapeseed, sunflower and linseed oils [40]. Alzate Arbeláez et al. studied the thermal stability of Lecythis tuyrana oil by means of TGA [41].

3.5 Differential scanning calorimetry

Differential scanning calorimetry (DSC) has been emerged to measure the stability of the fat-containing food against oxidation. It is a thermal analysis method with advantages such as efficient sensitivity, fastness and low sample requirements [21]. This accelerated method also allows to track the thermal activity in oxidation process continuously consideering the other methods [42].

It also required less stability measurement period comparing to Rancimat method. Similarly, Ramezan et al. compared the findings of Rancimat with that of DSC for 8 oil types such as sunflower, canola, palm, soybean, maize, peanut, sesame and coconut oils oxidized at 110, 120, 130 and 140 C [43]. They also recommended DSC as an alternative accelerated method with its advantegous (as mentioned above) over Rancimat. By using different heating rates (5, 7.5, 10, 12.5 and 15 C per minute), refined palm, olive, grapeseed, sunflower, corn, soybean, safflower and sesame oils were heated at 100-400 C by means of DSC [6]. Kinetics of the lipid oxidation occured in the selected oils were investigated by means of reaction rate constant and activation energy parameters. Activation energy changed depending on the compositon of the oils Tengku-Rozaina and Birch measured the stabilitiesof hoki and tuna oils against oxidation at 80 C [10]. Prediction of shelf-lifes of the products were conducted through Arhenius model. Srivastava et al. reported the thermodynamic structure of virgin coconut oil blended with different oils (refined soyabean and refined safflower oils) depending on the findings of DSC [44]. Belayneh et al. investigated Camelina seed oil to comprehend the effect of

extraction method (cold press, soxhlet and supercritical CO_2 extractions) on oxidative stability of the oil [45]. Symoniuk et al. monitored the thermal oxidation of some selected cold-pressed oils by DSC [46]. Recently, Echium oil has been studied to observe the effect of rosemary extract and hydroxytyrosol on the stability of the oil towards oxidation [47].

3.6 Rancimat test

Rancimat is an easy test method, including parameters such as amount of sample, air flow rate and temperature [13]. It requires no extra analyses such as titrations with too much time and chemical consumptions [14]. Şahin et al. performed Rancimat test to define the influences of olive leaf and lemon balm extracts on the shelf-life of corn oil [48]. Şahin et al. also reported the stability of virgin olive oil against oxidation after they enriched the oil with olive leaf extract through different methods [49, 50]. Similarly, they exposed the oil samples to air at a flow rate of 20 L/h for the Rancimat accelerated conditions (130°C). The same conditions were also applied to sunflower oil to assess the effect of olive leaf extract on the oil stability to oxidation [51].

Kinetics and thermodynamics factors are necessary for prediction of the lipid oxidation in oils under several conditions for composing better preparations regarding quality indicators [20]. However, the kinetics data obtained by rancimat method to assess the oxidative stability in vegetable oils are scarce. Farhoosh and Hoseini-Yazdi reported the olive oil oxidation process regarding kinetics studies achieved by accelerated rancimat conditions (100-130°C) [4]. Upadhyay and Mishra enriched the sunflower oil with sage extract, and investigated the kinetic and thermodynamic parameters of the lipid oxidation to comprehend the nature of the process [52]. The fact of lipid oxidation occured in vegetable oils is distinctive for each system. Therefore, Arrhenius equation has to be reproduced for every oil

system to discriminate the characteristics of the related products [20]. Elhussein et al. identified the kinetic and thermodynamic parameters of sesame oils of different origins (Turkey, Yemen and Sudan) by using Rancimat test under accelaretad conditions such as 110, 120, 130 and 140ºC [53]. Kurtulbaş et al. also carried out similar study to evaluate the nature of the lipid oxidation process in cottonseed oil treated with phytonutrients (gallic acid, rutin and carotenoid) [54]. Recently, kinetics of the lipid oxidation in sunflower and sesame oils were calculated through Arrhenius model under Rancimat accelerated conditions [55].

the proposed methods produced from the relevant tests. However, it is not a good way to state which is the best method after mentioning the advantages and disadvantages of these measurement methods. Therefore, the results of multiple test methods should be given comparatively in an investigation of thermal oxidative stability. Kinetic and thermodynamic information should be calculated in the light of data produced from the selected methods. In this way, formulation of the fat-containing food product should be improved after having a provision on the nature of the complex lipid oxidation.

4. Concluding Remarks

Table 1 summarizes the stability measurement tests of several edible oils and fats against oxidation. Quality of the fat-containing food product might be monitored by
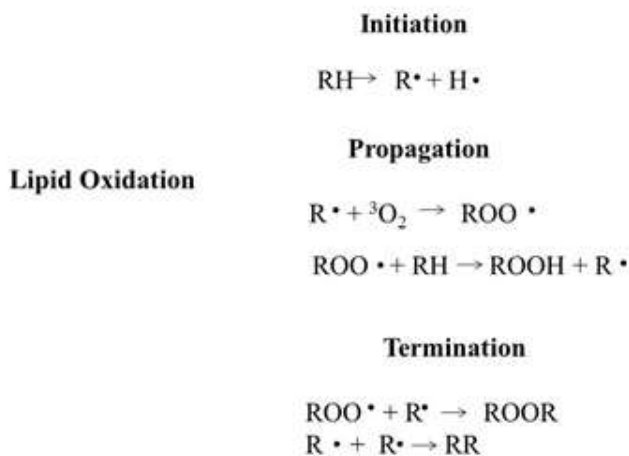
Initiation

$$RH \rightarrow R^\bullet + H^\bullet$$

Propagation

$$R^\bullet + {}^3O_2 \rightarrow ROO^\bullet$$

$$ROO^\bullet + RH \rightarrow ROOH + R^\bullet$$

Lipid Oxidation

Termination

$$ROO^\bullet + R^\bullet \rightarrow ROOR$$
$$R^\bullet + R^\bullet \rightarrow RR$$

Figure 1: Stages of lipid oxidation process occured in fat-containin food products.

Test	Conditions	Sample	Purpose of the study	Reference
Schaal oven	• 62°C • 24 days • Sampling every 6 days	• Soybean oil • Rice bran oil • Cottonseed oil	To investigate the effect of rosemary extract on the enhancement of the oil stability	[1]
	• 70°C • 16 days • Sampling every 10 days	• Palm olein	To investigate the effect of soursop flower extract on the enhancement of the oil stability	[23]

	• 60°C • 16 days • Sampling every 2 days	• Sunflower oil	To investigate the effect of black cumin oil on the enhancement of the oil stability	[25]
	• 60°C • 30 days • Sampling after 1, 2, 5 and 16 days	• Chia oil	To monitor the thermal oxidative stability of the oil	[24]
	• 70°C • 30 days • Sampling every 10 days	• Refined palm olein	To investigate the effect of ginger root extract on the enhancement of the oil stability	[22]
	• 60°C • 16 days • Sampling every day	• Moringa oil • Olive oil • Canola oil	To monitor the comparative results of thermal oxidative stability of the oils	[26]
	• 63°C	• Peanut oil • Corn oil • Rice bran oil • Grapeseed oil • Rapeseed oil	To monitor the comparative results of thermal oxidative stability of the oils during 12 months of storage	[27]
	• 60°C • 45 days • Sampling every 3 days	• Walnut oil	To investigate the effect of lycopene on the enhancement of the oil stability	[28]
	• 60°C • 6 days • Sampling every day	• Grapeseed oil • Flaxseed oil • Black cumin seed oil	To monitor the comparative results of thermal oxidative stability of the oils	[29]
FTIR	• Together with ATR	• Virgin coconut oil • A mixture including virgin coconut and refined soyabean/ refined safflower oil oils	To determine the oxidative deterioration	[44]
	• Heating at 30,	• Canola oil	To comprehend the effect of	[33]

		heating and frying on oxidative stability of the oil	
• 40, 50 and 60°C • Exposure to frying 5 times			
• Combined with infrared quartz cuvette	• Rapeseed oil • Soybean oil • Peanut oil • Sunflower oil • Corn oil • Linseed oil • Sesame oil • Walnut oil • Blend oil • Peony seed oil	To observe the oxidation in the oils by determination of acid value	[32]
• Including a chemometric method • 170°C • 36 h • Sampling every 3 h	• Corn oil • Sunflower oil • Colza oil • A mixture including palm, sunflower and soybean/cottonsee d oils	To monitor the heating kinetics	[35]
• Together with a mesh cell • Exposure to visible light • Ambient conditions	• Peanut oil • Soybean oil • Rapeseed oil • Linseed oil	To define the oxidative stability at ambient storage	[36]
• Together with a mesh cell • 23, 35, 65°C • Exposure to different light intensities (400, 1000 and 7000 lx)	• Virgin olive oil	To study the resistance of the oil against oxidation and photooxidation	[37]
• Combined with infrared quartz	• Rapeseed oil • Linseed oil	To observe the oxidation in the oils by determination of acid	[31]

	cuvette	• Cottonseed oil • Peanut oil • Soybean oil • Sesame oil • Sunflower oil • Walnut oil • Silybum marianum seed oil • A mixture of all oils	value	
	• Exposure to conventional and microwave heating	• Soybean oil • Corn oil	To monitor the peak changes for proving the degradation of the oils	[34]
TGA	• Heating from 25 to 700°C • 2°C/min • At air atmosphere	• Hoki oil • Tuna oil	To estimate the shelf-life of the oils by Arrhenius extrapolation	[10]
	• Heating from 25 to 700°C • 2°C/min • At air atmosphere	• Carrot seed oil • Flax seed oil • Hemp seed oil • Canola seed oil	To predict the initiation of the thermal decomposition of the oils	[39]
	• Heating from 50 to 620°C • 1, 5, 7.5, 10, 15, 20°C/min • With a constant oxygen flow rate of 30 mL per minute • At atmospheric pressure	• Palm oil • Rapeseed oil • Sunflower oil • Linseed oil	To compare the thermal oxidation stabilities of the oils	[40]
	• Heating from 100 to 800°C • 20°C/min	• Lecythis tuyrana oil	To identify the thermal stability of the oil	[41]
	• Heating at 100,	• Sunflower oil	To compare the oxidative	[43]

DSC	110, 120 and 130°C • With a constant oxygen flow rate of 50 mL per minute	• Canola oil • Refined-bleached-deodorized palm oil • Soybean oil • Maize oil • Peanut oil • Coconut oil • Sesame oil	stabilities of the oils	
	• Heating from 50 to 250°C • 2.5, 5, 10 and 15°C/min • At 20 psi	• Camelina seed oil	To comprehend the effect of extraction method on oxidative stability of the oil	[45]
	• Heating from 30 to 400°C • 5, 7.5, 10, 12.5 and 15°C/min	• Refined palm oil • Olive oil • Grapeseed oil • Sunflower oil • Corn oil • Soybean oil • Safflower oil • Sesame oil	To observe the effect of oil composition on the lipid oxidation	[6]
	• Heating at 80°C • At air atmosphere	• Hoki oil • Tuna oil	To estimate the shelf-life of the oils by Arrhenius extrapolation	[10]
	• Heating from -60 to 25°C • 10°C/min	• Virgin coconut oil • A mixture including virgin coconut and refined soyabean/ refined safflower oil oils	To define the thermodynamic nature of the oil	[44]
	• Heating at 120°C • 5°C/min • At 1380-1400 kPa	• Camelina oil • Rapeseed oil • Sunflower oil • Linseed oil • Black cumin oil	To compare the thermal oxidation stabilities of the oils	[46]

	• Evening primrose oil • Hempseed oil • Milk thistle oil • Poppy oil • Pumpkin oil			
	• Heating at 50, 60, 70, 80, 90, 100 and 110°C • With a constant oxygen flow rate of 60 mL per minute	• Echium oil	To observe the effect of hydroxytyrosol and rosemary extract on the lipid oxidation	[47]
Rancimat	• Heating at 100, 110, 120 and 130°C • With a constant air flow rate of 25 L per hour	• Olive oil	To calculate the kinetic and thermodynamic values of the lipid oxidation in the oil	[4]
	• Heating at 100, 110, 120 and 130°C • With a constant air flow rate of 20 L per hour	• Sunflower oil	To calculate the kinetic and thermodynamic values of the lipid oxidation in the oil enriched with natural and synthetic antioxidants	[52]
	• Heating at 130°C • With a constant air flow rate of 20 L per hour	• Corn oil	To observe the effect of olive leaf and lemon balm extracts on the lipid oxidation	[48]
	• Heating at 130°C • With a constant	• Olive oil	To observe the effect of olive leaf extract on the lipid oxidation	[49]

	air flow rate of 20 L per hour		
• Heating at 130°C • With a constant air flow rate of 20 L per hour	• Sunflower oil	To observe the effect of olive leaf extract on the lipid oxidation	[51]
• Heating at 110, 120, 130 and 140°C • With a constant air flow rate of 20 L per hour	• Sesame oil	To calculate the kinetic and thermodynamic values of the lipid oxidation in the oils from different origins	[53]
• Heating at 110, 120, 130 and 140°C • With a constant air flow rate of 20 L per hour	• Cottonseed oil	To calculate the kinetic and thermodynamic values of the lipid oxidation in the oil enriched with phytonutrients	[54]
• Heating at 100, 110, 120 and 130°C • With a constant air flow rate of 20 L per hour	• Sesame oil • Sunflower oil	To calculate the kinetic parameters of the lipid oxidation in the oil	[55]
• Heating at 130°C • With a constant air flow rate of 20 L per hour	• Olive oil	To observe the effect of olive leaf extract on the lipid oxidation	[50]

Table 1: Summary of the reported studies on oxidative stability of several edible oils and fats.

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