Changes of Physical Properties of Rapeseed Oil with Added Vitamin A and E Studied Using the Differential Scanning Calorimetry Method (Dsc)

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Abstract
The Differential Scanning Calorimetry (DSC) method was used for the purpose of the studies to compare changes to physical properties of rapeseed oil with added vitamin A and E in the increasing temperature. Heating of oil samples with and without added anti-oxidants in the rate of 5°C/min. was performed for 50°C -300°C temperature range. It is stated that heating of rapeseed oil to the temperature of 200°C causes no changes to its chemical properties, whereas oxidation state changes appear in the temperature above 200°C. Adding of vitamin E to rapeseed oil decreases its susceptibility to oxidation state changes; no evident impact of the other vitamins in these changes was recorded.

Keywords: Edible oil; Vitamin A; Vitamin E; Differential scanning calorimetry; Oxidation; Physical properties

Introduction
The DSC method is used to measure changes in differences of the heating stream between the studied sample and the reference sample during the thermal transition [1-5]. Oxidative stability of the oils constitutes one of the key determinants of their quality and authorization on the food market. This is of particular importance since vegetable oils are willingly and frequently used for culinary purposes. They have a positive impact on numerous food products, however oils quality decreases due to their incorrect processing, which results in oxidation covering the basic decomposition reactions [6,7]. Long-term and multiple use of the same oil sample for frying purposes, causing changes in both chemical and physical properties of this compound may pose an example. The purpose of the study was to compare the changes in physical properties of rapeseed oil caused by temperature with added antioxidant vitamins A or/and E using the DSC method.

Materials and Methods
Study on changes in the oil sample induced by heating was performed for:
- rapeseed oil (sample a), rapeseed oil with added vitamin E (sample b), rapeseed oil with added vitamin A (sample c), rapeseed oil with added vitamin A and E (sample d).
- The studied material was the refined rapeseed oil of low erucic acid content (manufactured by F.H.P.U. 1MARLIBO; Batch No.: 302/08/10/3).

For the purposes of the study the differential scanning calorimeter (Perkin Elmer - Diamond DSC comp.) operating with the use of power compensation was used. Heating and cooling within the device may be performed at the rate between 0.01 and 500°C/min. Control, measurement and data analysis software operates under Windows XP OS. The analyses were performed in oxygen atmosphere (by Linde; O2: 99.92%; Humidity: 10.3 ppm; CO2: ≤ 5 ppm, CO2: ≤ 300 ppm).

Weights of the studied samples are: 3.410 mg (a), 3.430 mg (b), 3.400 mg (c) and 3.420 mg (d). The subsequent analyses were performed with the following additives: vitamin E (50 mg/100 g prod. Medana Pharma S.A.; Batch No.: 010510) or/and vitamin A (900 µg/100 g prod. Medana Pharma S.A.; Batch No.: 010610). Each sample was placed on aluminum plates covered with caps. Heating of oil samples at the rate of 5°C/min. was performed in the temperature range between 50°C -300°C. Each individual experiment in the above mentioned conditions lasted 51 min.

Results and Discussion
The DSC curves for all samples (a-d) are presented at Figure 1. When refined rapeseed oil (sample a) was subject to DSC no peaks which might indicate the changes in the oil were recorded to the temperature of 200°C. Upon exceeding of this temperature a low exothermic peak is noticeable, which shows the oxidation state changes. This peak is being observed after app. 32 minutes of experiment in the temperature of app. 205-210°C. For the following 14 minutes no visible changes were stated until the sample reached the temperature of app. 270°C, when a low endothermic peak is recorded followed by single sharp exothermic peak.

The endothermic peak proves the presence of polymerization process, whereas the exothermic peak on significant oxidation state changes (app. 47. minute of the experiment).

To the selected oil samples the antioxidant vitamins (A, E, A+E) were added to study their impact on physical and chemical properties of the heated oils. The rapeseed oils were heated to the temperature exceeding 300°C and therefore the polymorphic studies were applied. The samples were heated at the rate of 5°C/minute.

The DSC thermograms recorded no changes to the temperature threshold of 200°C. Upon exceeding of this temperature the exothermic

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peaks appeared, which proved oxidation state changes in the oils. Also the endothermic peaks were observed on the DSC thermograms, of high possibility of polymerization transitions in the oils.

For the oil sample with added vitamin E (sample b) the following regularities were recorded. The exothermic peak recorded at the graphs of oil samples with no additives (temperature range of app. 200°C) becomes unnoticeable for the studies on oil with added vitamin E. This may prove an antioxidant impact of vitamin E, addition of which was inhibited by oxidation process. The following two peaks observed on the graphs of oil with no additives are present here. Upon app. 45 minutes of the experiment (as in the studies with no vitamin E) the endothermic peak is recorded changing over into sharp exothermic peak appears. This peak is of noticeably lower enthalpy comparing to the samples with no added vitamin E. Decrease in enthalpy proves most probably lower oxidation state changes in the oil upon using of tocopherol.

Adding of vitamin E to the selected samples of the heated oils caused decrease in the enthalpy of exothermic peaks. No differences in time of appearance of exothermic peaks on the DSC thermograms of oil samples with vitamin E comparing to the thermograms of samples with no vitamin were recorded.

The DSC curve of oil samples with vitamin A (sample c) are different. Upon app. 26 min. of the study a relatively wide exothermic peak appears, resulting most probably from the changes in the vitamin A itself.

A specific endothermic peak (probably polymerization process) recorded at the DSC graph of the oil sample with no additive remains unnoticeable on the sample graph, whereas the exothermic peak appears in app. 47 min. of the experiment in the temperature of app. 285°C i.e. in similar conditions as in the phase without vitamin. Opposite to the peaks for the oil alone this peak is of significantly lower enthalpy.

The DSC curve for sample oil with added vitamin A and E (sample d) present the endothermic peak at the 45. minute of the study in the temperature of app. 265°C. This peak is slightly higher comparing to the experiments for the oil alone.

The exothermic peak specific for the sample of the oil with no additives is unnoticeable on these curve, which may result from decrease in the oxidation processes due to adding of vitamins A and E. In contrary to the previous repetition, we may observe the low exothermic peak between 25. and 30 min. of the study.

The scientists from the University in Parma have examined the transitions in the 'extra virgin' olive oil. On the basis of analysis of thermograms they describe repeating regularity of appearance of very low exothermic peak followed by major endothermic peak [8].

Thermoanalytical studies on oils have been also performed in Brazil. The authors stated that sunflower oil shows oxidative stability to the temperature of app. 200°C. The similar regularity was observed in own studies on rapeseed oil, in which the first deviation from the DSC curve is noticeable upon exceeding the threshold of 200°C. The authors have also evaluated the impact of synthetic antioxidants (among others vitamin E) for the occurring processes. They confirmed their participation by change of shape of the DSC curve decreasing the enthalpy of the peaks of both polymerization reaction and decomposition-related (oxidation) processes. In case of own studies, the exothermic peak causing possibly the oxidation state changes was decreased due to adding of synthetic vitamin E, whereas the endothermic peak related to polymerization reactions failed to change its parameters in observable manner. Oil samples to which the antioxidants were added, were of higher oxidative stability comparing to the samples alone – which was reflected by increase of average activation energy for fatty acids decomposition processes in the samples containing antioxidants [9]. According to Gouvei de Souza A [9] increased rate of oils heating results in greater fatty acids decomposition. According to the same scientists, the peaks enthalpies are strictly related to the composition of the fatty acids of the studied oils. We conclude that: a) heating of rapeseed oil to the temperature of 200°C results in to chemical transitions, b) oxidation processes in rapeseed oil initiate upon exceeding the temperature of
200°C, c) adding of vitamin E to rapeseed oil lowers susceptibility to oxidation transitions [10].

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