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Determination of the stability index of solid foodstuffs by the Rancimat method

Branch

Food, stimulants, beverages, flavors

Keywords

Oxidative stability; oxidation stability; rancidity; 892; solid food; bread crumbs; cookies; wheat; wheat flour; coconut; cereals; instant drink powder; polenta; corn; sample preparation; stability index; branch 7

Summary

The oxidative stability of solid foodstuffs can be determined by the Rancimat method. This can be done directly with the solid foodstuffs in some cases, but this is often impossible due to a low fat content. A further method is to determine the stability index, which is a measure for the antioxidant capability of a product. In this method, the sample is mixed with a reference of antioxidant-free fat (e.g., pure lard) and the determined induction time is compared to the reference alone as a control. Due to its properties the product slows down the oxidation of the reference. In this work, pure lard was used as the reference. Using this approach the oxidative stability of the sample is not measured directly, but its influence on the oxidative stability of the reference.

In the Rancimat method the lard/sample mixture is heated precisely to a specified temperature (here 110 °C are used) while a constant stream of air flows through the sample. The lard is oxidized during this process and the decomposition products are transferred into a measuring cell. Therein, some of the oxidation products such as low-molecular weight organic acids increase the electrical conductivity. This serves as a measure for the oxidation progress. A sharp increase in the conductivity indicates the point where the sample is oxidized at a fast rate. The time after which this process starts is defined as the induction time.

The oxidative stability of the samples can be measured by the stability index which is defined as the induction time of the sample/lard mixture divided by the induction time of pure lard.

Sample

Foodstuffs: Bread crumbs, butter cookies, chocolate sprinkles, durum wheat semolina, grated dried coconut, breakfast cereals (two different kinds, named I & II), hazelnut cookies, instant malt dairy drink powder, polenta (cornmeal), wheat flour.

Reference/control: Pure lard

Instruments

892 Professional Rancimat	2.892.0010
Equipment for determination of temperature correction	6.5616.100
StabNet PC software	6.6068.xxx
Auxiliary instruments for sample preparation • Thermostat bath (or oven) • Mortar and pestle • Laboratory balance (resolution ± 0.01 g)	

Reagents

- Deionized water (ISO 3696 Type II)
- Pure lard

Sample preparation

Samples that have a grainy consistency were used directly. Samples such as butter cookies, hazelnut cookies, and breakfast cereals had to be crushed with a mortar and pestle to a grain size of less than 1 mm.

Pure lard is heated to 50 °C in a closed flask in a thermostat bath for 30 min to melt. The lard was melted to facilitate mixing with the sample in the measuring vessel. The same procedure was used for the control as well.

Analysis

Preparation of the Rancimat

The heating block is heated up to the respective temperature.

Preparation of the measuring vessel

The measuring vessel is filled with 60 mL deionized water and placed on the Rancimat together with the measuring vessel cover.

Preparation of the reaction vessel

For each determination, a new reaction vessel is used. To remove particles (e.g., from the cardboard box) the reaction vessel is air-cleaned inside and outside by a sharp stream of nitrogen. Then, the sample is weighed directly into the reaction vessel. In case of the control, 3.0 ± 0.1 g of melted pure lard were used. For the samples, 0.5 ± 0.05 g of sample and 2.5 ± 0.1 g of melted lard were used. During the sample addition, it must be avoided that the sample gets stuck at the side walls. The reaction vessel is closed with a reaction vessel cover assembled with an air inlet tube.

Determination

Before the determination can be started, the temperature of the heating block has to be stable. The two tubings between Rancimat and reaction vessel and between reaction vessel and measuring vessel are connected. Then, the reaction vessel is placed in the heating block and the measurement is started immediately.

Parameters

Sample size	3.0 ± 0.1 g
Measuring solution	60 mL deionized water
Gas flow	20 L/h
Temperature	110 °C
Temperature correction	Automatic
Stop criteria	Endpoints Conductivity: 100 µS/cm
Evaluation	Induction time automatic
Evaluation sensitivity	1.0

Calculation

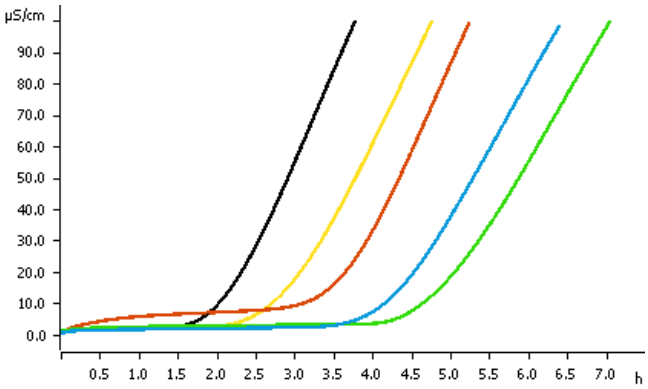
The stability index (SI) is defined as

$$SI = \frac{\text{Induction time of sample/lard mixture}}{\text{Induction time of pure lard}}$$

Results

Product	Induction time / h	SI
Breadcrumbs	2.16	1.10
Instant malt dairy drink powder	2.37	1.21
Breakfast cereal I	2.55	1.30
Polenta	2.87	1.47
Durum wheat semolina	2.98	1.52
Grated dried coconut	3.17	1.62
Wheat flour	3.38	1.73
Chocolate sprinkles	3.46	1.77
Breakfast cereal II	4.05	2.07
Butter cookies	4.33	2.21
Hazelnut cookies	4.52	2.31
Control	1.96	

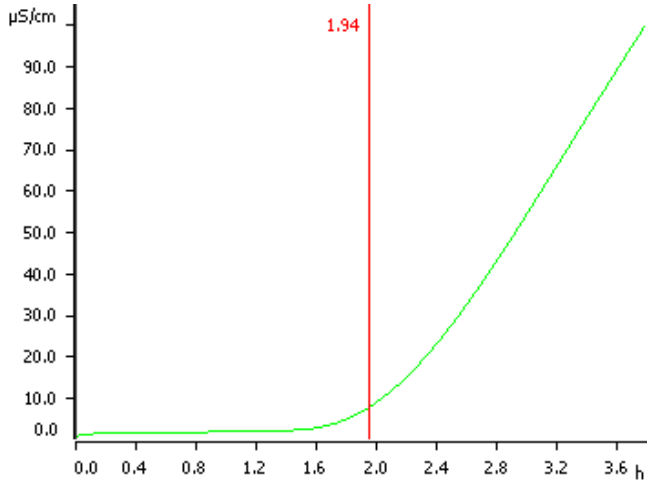
Exemplary curves are displayed in the following figure.



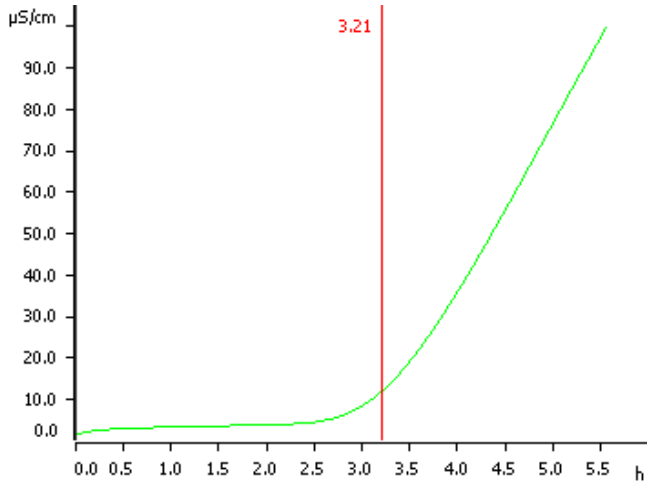
Color	Sample
	Control
	Polenta
	Chocolate sprinkles
	Breakfast cereal II
	Butter cookies

Example determinations

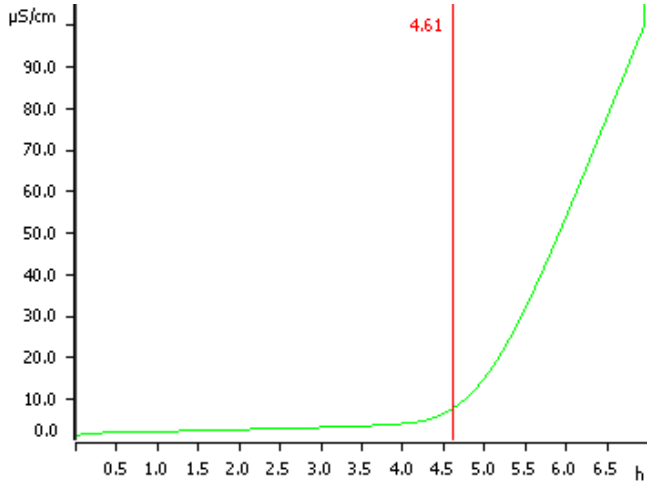
Pure lard



Grated coconut



Hazelnut cookies



Detailed results

Bread crumbs

Sample	Induction time / h
1	2.09
2	2.13
3	2.16
4	2.27
Mean	2.16
RSD	3.6%

Instant malt dairy drink powder

Sample	Induction time / h
1	2.50
2	2.22
3	2.32
4	2.42
Mean	2.37
RSD	5.1%

Breakfast cereal I

Sample	Induction time / h
1	2.61
2	2.52
3	2.54
4	2.52
Mean	2.55
RSD	1.7%

Polenta

Sample	Induction time / h
1	2.79
2	2.85
3	2.82
4	3.02
Mean	2.87
RSD	3.6%

Durum wheat semolina

Sample	Induction time / h
1	2.98
2	2.95
3	3.06
4	2.91
Mean	2.98
RSD	2.1%

Breakfast cereal II

Sample	Induction time / h
1	4.34
2	4.02
3	3.89
4	3.94
Mean	4.05
RSD	5.0%

Grated dried coconut

Sample	Induction time / h
1	3.13
2	3.21
3	3.12
4	3.22
Mean	3.17
RSD	1.6%

Butter cookies

Sample	Induction time / h
1	4.53
2	4.25
3	4.29
4	4.25
Mean	4.33
RSD	3.1%

Wheat flour

Sample	Induction time / h
1	3.27
2	3.46
3	3.38
4	3.40
Mean	3.38
RSD	2.3%

Hazelnut cookies

Sample	Induction time / h
1	4.73
2	4.61
3	4.25
4	4.49
Mean	4.52
RSD	4.5%

Chocolate sprinkles

Sample	Induction time / h
1	3.43
2	3.43
3	3.44
4	3.53
Mean	3.46
RSD	1.4%

Control

Sample	Induction time / h
1	1.89
2	1.94
3	1.96
4	2.04
Mean	1.96
RSD	3.2%

Comments

- The induction time of the reference (pure lard) should be determined frequently, i.e., once per week. Also care has to be taken that the lard from the sample/lard mixture is always from the same batch (or package) as the pure lard reference since the induction time might be different between batches.
- Pure lard should always be used fresh and stored in a refrigerator at approx. 5 °C.
- The melting of lard does not have an influence on the induction time of pure lard. This was tested with pure lard that was not heated before the measurement. Without melting, pure lard showed an induction time of 2.55 h (mean of 4 samples, standard deviation = 0.08 h), which was not significantly higher than with melting (2.49 h, standard deviation = 0.06 h).
- The melting of lard can also be carried out in a closed bottle in a heating oven. In this case, 20 g of pure lard are weighed in a well-cleaned glass bottle and closed with a screw cap. The glass bottle is put in the heating oven for 30 min at 50 °C. Thereafter, the lard is a transparent liquid which can easily be mixed with the product. This melting procedure also does not have an influence on the induction time. 30 min is the maximum time for lard to be liquefied at 50 °C. With longer treatment or when melting at higher temperatures, the induction time is decreased with respect to the untreated reference sample.