RapidOxy 100: Oxidation Stability of Cocoa Mass and Vanilla Extracts

Relevant for: Food Industry

Autoxidation is a major cause of food quality deterioration, causing for example the formation of undesirable off-flavors and degradation of essential nutrients. The oxidation process of a product is affected by various parameters such as processing, storage, and product ingredients. For a positive impact on shelf-life, however, a solution for each product needs to be developed. RapidOxy 100 can be a powerful investigation tool to guarantee the success of this process. The benefits and possible sample variety of the fully automatic Rapid Small Scale Oxidation Test (RSSOT) was demonstrated on semi-solid cocoa mass samples and on liquid vanilla extract samples.

1 Introduction

Foods, ingredients, and supplements can suffer from autoxidation leading to deteriorative changes affecting their nutritional, sensory, and chemical properties. Therefore, one of the most important considerations during product development or reformulation of a product is to ensure the exclusion of oxidative degradation during shelf-life. To guarantee the quality and maintain desired attributes, manufacturers need to consider several aspects influencing the oxidation stability of their food product. For that purpose the crucial starting point is a suitable product formulation. Typically, certain desired product properties are realized by specific ingredients which can have a significant impact on oxidation stability and as consequence on shelf-life. However, two main parameters are typically optimized during formulation to enhance the lifetime of the finished product: One is processing either for ingredients or final products suffering from poor oxidation stability, the second is the addition of a suitable antioxidant or antioxidant system. Usually, both are investigated during development or reformulation of food products. To determine appropriate measures to enhance the oxidation stability the RapidOxy 100 can be a highly effective tool.

One major benefit for the customer is the fast measurement to get results in a fraction of time compared to other methods. Additionally, development costs can be significantly lowered with the fully automatic measurement (saves time, no laboratory staff required after start) and the small required sample amount (antioxidants are very expensive).

2 Instrument

During a RapidOxy 100 measurement 4 g or 5 mL of a sample is exposed to an excess of pure oxygen (up to 700 kPa) and elevated temperature (up to 180 °C).

Typical conditions of the method are temperatures between 80 °C to 140 °C and an initial oxygen pressure of 700 kPa. These conditions initiate a rapid oxidation process, which is monitored by recording the pressure until a predefined pressure drop. The elapsed time until the pressure drop is directly related to the total oxygen consumption and therefore to the oxidation stability of the sample.

As a benefit, the oxidation stability of complex food products can be investigated since even solid samples can be measured without prior sample preparation. The significantly reduced measurement time and the high precision of the method represent its major advantages, allowing for quick and direct measurement of the oxidation stability for research, process and test bench control.

Figure 1: RapidOxy 100

3 Influence of Processing on the Oxidation Stability of Cocoa Mass

Cocoa beans and in the later stage derived Cocoa mass is the origin for chocolate products we know as a consumer. After the harvest the beans are usually fermented, dried, winnowed, roasted, and ground. Those five steps as well as the beans themselves influence the properties of the produced cocoa mass. At each of the production stages the RapidOxy 100 can be successfully employed.

Usually the raw beans are transported after harvest to a different location for processing. Since the raw beans are especially susceptible to spoilage from heat (risk of fermentation and oxidation) and contamination, the transportation stage is crucial for the later shelf-life of the cocoa mass. At this point the RapidOxy 100 is the perfect choice for checking the quality of incoming raw material (= cocoa beans).

Especially roasting has a significant influence on the oxidation stability of cocoa mass which is starting material for cocoa butter and cocoa powder. The cocoa beans can be tested before and after roasting with the RapidOxy 100.

This report, however, describes the investigation of the applicability of the RSSOT at a later production stage. During this investigation, the conventional methods of testing the oxidation stability of the customer's cocoa mass were compared with the RapidOxy 100. Therefore, measurements were conducted with four cocoa mass samples which slightly differed in their processing.

3.1 Measurement of Cocoa Mass Samples

3.1.1 Accessories

For cocoa mass measurements the following accessories were used:

- **107296** O-ring set, Viton®, pack of 100
- **107138** Cleaning tissues, 150 sheets
- **106381** Glass dish, large

3.1.2 Settings

The parameters for the cocoa mass measurements are listed in Table 1.

Parameter	Setting
Program	User-defined program
Test temperature	120 °C
Filling pressure	700 kPa
Stop by pressure drop below p_{max}	10 %
Sample amount	5 g

Table 1: Instrument settings for measuring cocoa mass

3.1.3 Test Procedure

The samples were tested with a user-defined program using 5 g of the sample in a glass dish at a test temperature of 120 °C and 700 kPa filling pressure until a pressure drop of 10 % below maximum pressure (p_{max}) is detected.

3.1.4 Results

The following table displays the results of the cocoa mass samples measured with the RapidOxy 100:

Sample	Date	Mean [decimal minutes]
A	25.01.2018	356.32
B	26.01.2018	343.78
C	26.01.2018	318.49
D	27.01.2018	303.27

Table 2: Results for cocoa mass samples

3.1.5 Discussion

The measurement confirmed the stability order expected by the customer and corresponds to the results of the conventional testing.

Sample A → Sample B → Sample C → Sample D

The investigations at the customer's site included oven storage of cocoa mass samples at 40 °C for a period of three months. Afterwards, a sensory panel evaluated the oven-aged samples. The fully automatic measurement of RapidOxy 100 allows the customer to get results within few hours instead of months by just the push of a button. At the same time the customer appreciates the objectivity of results in comparison to the very subjective sensory evaluation that had to be carried out in the past.

4 Influence of Antioxidants on the Oxidation Stability of Vanilla Extracts

Vanilla extract is the most common form of vanilla used worldwide and often is an essential part of various desserts and pastries. Its main flavor is vanilla, but vanilla extract additionally contains several hundred flavoring compounds which add up to its complex flavor. The final flavoring experience is based on molecular structures that are prone to oxidation due to certain chemical functionalities (e.g. double bonds, aldehydes). To prevent the flavoring degradation of vanilla extract antioxidants have to be included in the final formulation. The high amount of compounds and chemical complexity of antioxidant interaction in certain formulations lead to extensive investigations. Additionally, the high costs for small quantities of antioxidants prohibits the industry to employ them in excess amount.

4.1 Measuring Vanilla Extracts

4.1.1 Accessories

For vanilla extract measurements the following accessories were used:

- **107296** O-ring set, Viton®, pack of 100
- **107138** Cleaning tissues, 150 sheets

4.1.2 Settings

The parameters for the vanilla extract measurements are listed in Table 3.

Parameter	Setting
Program	User-defined program
Test temperature	100 °C and 140 °C
Filling pressure	700 kPa
Stop by pressure drop below p_{max}	10 %
Sample amount	5 mL

Table 3: Instrument settings for measuring vanilla extracts

Tip: Keep in mind that there is a linear dependency of induction period and test temperature.

4.2 Test Procedure

All it takes on preparation is to fill the fluid sample directly into the test chamber, tighten the screw cap and close the instrument. The test chamber is automatically charged with oxygen pressure and heated until it reaches the test temperature (e.g. 140 °C in a standard program). The pressure is monitored until a pressure drop of 10 % below maximum pressure (p_{max}) is detected.

4.2.1 Results

The following table provides the results for the oxidation stability determination of four vanilla extracts varying only in the type of antioxidant added to the formulation. Each test was carried out using temperatures of 100 °C and 140 °C.

Sample	t [min] at 100 °C	t [min] at 140 °C
A	1139	69.55
B	1065	60.66
C	1228	73.01
D	1091	60.91

Table 4: Results for vanilla extract samples

4.2.2 Discussion

The least stable sample was determined to be vanilla extract sample D with the shortest induction period at both investigated temperatures. The general stability order revealed with the RapidOxy 100 matches the expectation of the customer with many years of experience in flavor formulation. UV testing, and oven storage at elevated temperatures with subsequent GC/MS analysis was implemented to determine the oxidative stabilities. However, the ease of use and accurate measuring results could convince the customer to substitute their basic testing with the RSSOT. As a further benefit it was possible to increase the measuring temperature to 140 °C to significantly shorten the RapidOxy 100 test times even more and at the same time achieve reliable results.

5 Summary

The measurements of the cocoa mass and of the vanilla extract samples demonstrated how RapidOxy 100 is highly suitable for formulation development of a wide variety of samples independent from aggregation stage or texture. The RapidOxy 100 offers an easy and reliable method for manufacturers to investigate formulas and products with regards to shelf life and product quality. The unique measuring principle according to ASTM D8206 leads to a significantly reduced measuring time for the describe investigations. The high ease of use as well as the fully automatic measurement are one of the major benefits for the customers.

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