Oxidation Stability on Biscuits

Reference: **EURACHEM GUIDELINES**

Tested with **VELP Scientifica OXITEST Oxidation Stability Reactor** (Code F30900248)

Introduction

Britain has been the first biscuits producers in history, during the 10th century.
It was the bread of the sailors who needed durable bread to eat during their long voyages: made without moisture, it was a hard dough bread, flat shape with all holes in the surface and it was cooked twice, "bis-cooked".
During the 19th century, the formula started to include also butter and sugar to make them delightful, becoming a sort of luxury food.
Today the consumption of biscuits is widely spread because they include a range of different shapes and flavors, ingredients and formulas, that make them suitable for all the consumers' needs.

Oxidation Stability in Food

One of the most important quality alteration of food is due to oxygen absorption by the unsaturated fatty acids, free or esterified. The auto-oxidation of fats is a chemical reaction promoted by oxygen, light, high temperatures, metal traces and, sometimes, enzymes.
OXITEST can determine the oxidation stability on various sample types, without the need for preliminary fat separation.

OXITEST Principle

OXITEST speeds up the oxidation process because of the two accelerating factors, temperature and oxygen pressure, according to the most common applications.
The instrument measures the absolute pressure change inside the two chambers, monitoring the oxygen uptake by reactive components in the sample and automatically generates an IP value.

IP Definition: IP stands for Induction Period and it is the time required to reach the starting point of oxidation, corresponding to either a level of detectable rancidity or a sudden change in the rate of oxidation. The longer the Induction Period, the higher the stability against oxidation over time.

Sample

Biscuits formula F1
Fat labeled value: 18.1 g / 100 g, composed by 12.1 g of unsaturated fatty acids and 6.0 g of saturated fatty acids

Biscuits formula F2
Fat labeled value: 18.3 g / 100 g composed by 9.3 g of unsaturated fatty acids and 9.0 of saturated fatty acids

Equipment and Chemicals

- Analytical balance, 3 decimals
- Grinder
- Silicone grease
- Oxygen, purity grade 5.0

Sample Preparation

Store the samples at room temperature.
Grind the biscuits to a suitable finesse and put 10 grams of homogeneous sample directly on the surface of the titanium sample holder, by using a spatula.
In each reaction chamber (A and B), place 3 sample holders containing the sample for a total of 30 g, grinded biscuits.

Analysis Procedure

Grease the O-rings with silicon grease and set them in their position. Close the chambers with the titanium covers and turn the discharge valves in open position. Set the following conditions on the OXISoft™ software:
Temperature: 100 °C
Oxygen Pressure: 6 bars

When the temperature set is reached inside the chambers, close the discharge valves and start loading oxygen.
Data acquisition is automatically started by the software.

Typical Results on Biscuits

Each biscuit sample has been monitored four times. At the end of the oxidation tests, the IP of every run is calculated by the software OXISoft™.

It is possible to elaborate the oxidation curves obtained for each kind of biscuit.

Sample	Weight (g)	Set Point (bars)	Set Point (°C)	IP (hh:mm)	Line
Biscuits F1	30.000	6.00	100.0	54:41	
Biscuits F1	30.000	6.00	100.0	56:40	
Biscuits F1	30.000	6.00	100.0	51:50	
Biscuits F1	30.000	6.00	100.0	57:52	
Biscuits F2	30.000	6.00	100.0	100:09	
Biscuits F2	30.000	6.00	100.0	96:31	
Biscuits F2	30.000	6.00	100.0	92:35	
Biscuits F2	30.000	6.00	100.0	92:15	
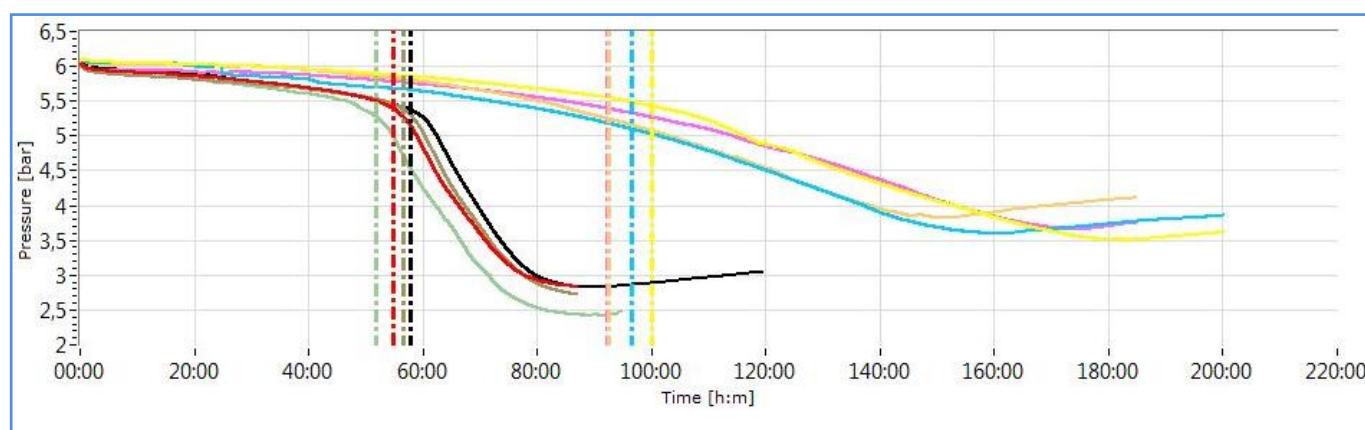
Repeatability Test

With OXISoft™, it is possible to create a repeatability test for each analysis, in order to obtain the average, standard deviation and relative standard deviation of the results.
For repeatability test, it is necessary to analyze the same quantity of the sample in duplicate or more, at the same values of temperature and pressure. In the table below the results are summarized:

Sample	IP average (hh:mm)	SD (hh:mm)	RSD %
Biscuits F1	55:15	2:38	4.8
Biscuits F2	95:22	3:43	3.9

RSD value must be < 5% in order to obtain good results.

Formula Comparison

With OXISoft™, it is also possible to easily compare the obtained IP values, of different formulations but tested at the same condition, and identify the most stable one.
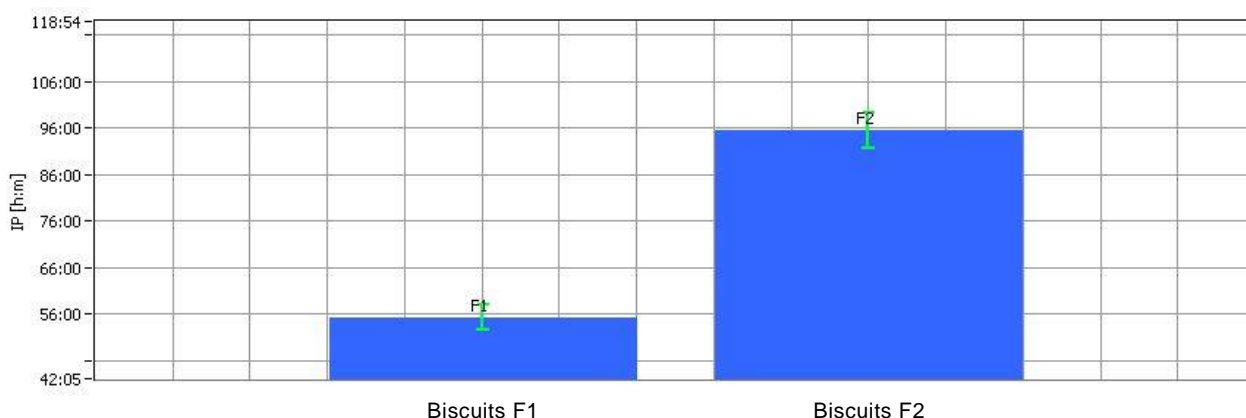
Conclusion

The results obtained by OXISoft™ and the formula comparison function clearly discriminate the biscuits' resistance to oxidation, although the total fats percentage of their formulations is very similar.
Biscuits F1 have a shorter IP value, hence a lower oxidation stability.
This is related to their higher content of unsaturated fatty acids that have a higher oxidation kinetic compared to saturated fatty acids.
In conclusion, biscuit F2 results significantly more stable against oxidation.

Benefits of OXITEST are:

- Test is made directly on the whole sample
- No need for preliminary fat separation of the sample
- Resistant titanium chamber
- Time saving analysis, if compared to the traditional methods
- Especially designed for R&D, Product Development and Quality Control labs
- Many investigations available through the software OXISoft™:
  1. Repeatability test: a series of tests run on the same sample or standard to verify its IP period, to calculate accuracy and repeatability of the data
  2. Freshness test: to verify the quality of different lots, for example of the same raw material, and compare them
  3. Formula comparison: to identify the most stable formula of a finished product, under the same conditions
  4. Packaging comparison: for testing which packaging maintains the product in the freshest condition
  5. IP during ageing: to obtain a graph of the decrease of the product IP during the shelf-life period
  6. Estimated shelf life: to have a prediction of oxidation stability during the shelf life.