An Evaluation of the Efficacy of Antioxidants in Soap by Differential Scanning Calorimetry (DSC)

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A simple, rapid method for the evaluation of the efficacy of commercial antioxidants in soap products has been developed. The procedure involves forced oxidation of a sample in an oxygen-pressurized differential scanning calorimetry (DSC) cell. The analysis is complete in 30 min and requires 5-10 mg of sample. The forced oxidation DSC methodology described in this work may be a useful tool in the development and optimization of an antioxidant system for bar soap products and for fatty materials in general.

KEY WORDS: Analysis, antioxidant, BHT, DSC, oxidation, rancidity, shelf-life, soap, stability, thermal.

The determination of the oxidative stability of fats, fatty acids, and soaps has been a vigorously studied field. Although a number of methods have been utilized, the search for a simple procedure which also correlates with observed sensory attributes, such as odor, flavor, and color of the fatty materials, is still in active pursuit (1).

In the course of our work on the thermo-analytical study of bar soap products (S. Gupta, submitted for publication), we found that the oxidation of fatty materials in a differential scanning calorimetry (DSC) cell pressurized with oxygen proceeded rapidly with the appearance of sharp exothermic heat flow. We also noted an increase in both the induction and the initiation periods, relative to a control, in the soap samples which contained small quantities of antioxidants. This observation led to the development of the present methodology.

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EXPERIMENTAL PROCEDURES

A DuPont Thermal Analysis 1090 instrument (DuPont, Boston, MA) equipped with a pressure DSC cell was used. All calculations were performed by a DuPont Oxidation Analysis (V 0.1) program. The onset of induction and initiation times were automatically selected by this computer program.

The soap samples (5-10 mg) were placed in an aluminum DSC pan, the DSC cell was pressurized (500 psig) with oxygen, and analysis conducted starting at 100°C with a program rate of 5°C/min to a final temperature of 250°C. A typical analysis was complete in 30 min.

RESULTS AND DISCUSSION

A typical DSC oxidation curve is illustrated in Figure 1.

In our study, soap pellets containing only the antioxidant as an additive were prepared in the standard manner from the neutralization of fatty acid blends followed by drying them to a specified moisture content (S. Gupta, submitted for publication). The soap pellet samples were analyzed by the forced oxidation procedure. The samples of these soap pellets were also stored in sealed bottles under ambient conditions for a period of 30 days, following which an odor evaluation for rancidity development was conducted by trained personnel.

The DSC oxidation and odor evaluation data are summarized in Table 1.

Although no attempt was made in the present study to optimize the level of antioxidants in soap pellets for oxidative stability, the data in Table 1 illustrate that the present methodology correlates with observed sensory odor evaluations for rancidity.

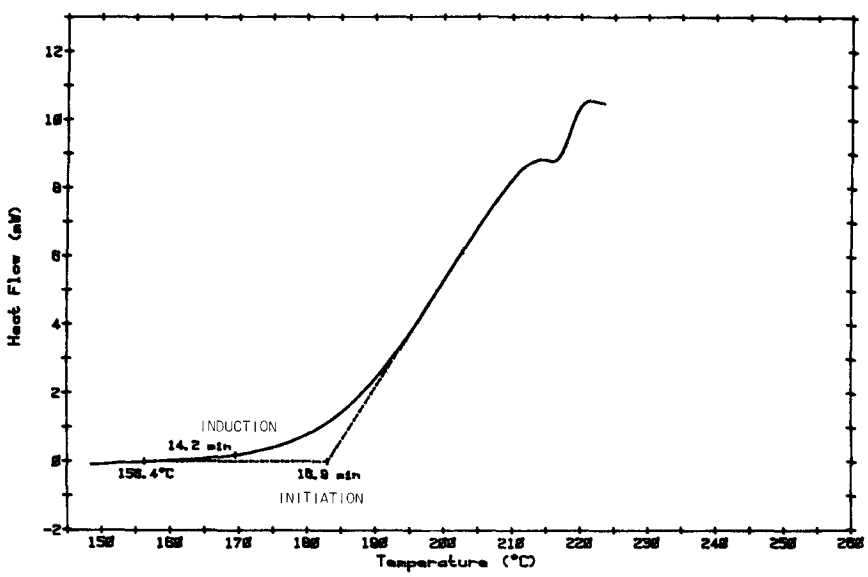

FIG. 1. DSC oxidation data of a soap sample.

TABLE 1

The Forced Oxidation DSC and Odor Evaluation Data of Soap Pellet Samples

Antioxidant (% in soap[a])	Forced oxidation time (min)		Odor scores[b]
	Induction	Initiation	
None	14.2	16.9	5.0
BHT[c] (0.03)	15.0	16.4	5.5
DTPA[d] (0.2)	16.3	18.7	6.0
HEDP[e] (0.2)	17.5	18.7	7.0
BHT/DTPA (0.03/0.2)	17.3	19.7	7.0
BHT/DTPA/HEDP (0.03/0.1/0.1)	17.9	19.2	7.5

[a] Soap from 85% tallow fatty acid/15% coconut fatty acid blend.

[b] The odor evaluation was performed on a scale of 1-10; a value of 10 denotes total absence of rancid odor formation.

[c] Butylated hydroxytoluene (2,6-di-*t*-butyl-4-methyl phenol).

[d] Diethylenetriaminepentaacetic acid.

[e] Hydroxyethylidene-1, 1-diphosphonic acid.

TABLE 2

Forced Oxidation Data of Soap Bar Samples

Antioxidant[a]	Forced oxidation time (min)	
	Induction	Initiation
None	5.4	5.7
BHT	13.2	14.8
DTPA	14.8	18.0
HEDP	17.5	18.7
BHT/DTPA	16.7	18.5
BHT/DTPA/HEDP	17.9	19.0

[a] See Table 1 for the identification of antioxidants, their quantity in soap pellets, and composition of soap.

In a study of the evaluation of bar soap oxidation stability by this methodology, a number of soap bars were prepared from the soap pellets containing various antioxidants in Table 1. All those bars also contained a small quantity of an opacifier (TiO_2) and a perfume (1%). The forced oxidation data (Table 2) reveal that these soap bars also showed distinct oxidation induction and initiation points. A long-term stability study showed the concordance of oxidation data noted in Table 2 with observed sensory odor profile of those soap bar samples.

REFERENCE

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[Received July 6, 1990; accepted January 23, 1991]