Oxygen Absorption Method for Assessing Oxidative Stability of Fats and Oils

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An oxygen absorption method was applicated for the evaluation of oxidative stability of fats and oils. The amount of oxygen absorbed by fats and oils was measured with a Warburg manometer at 70°C. The induction periods, that is, the time required for decreasing the pressure in the flask by 20 mmHg, showed excellent correlation with those obtained by an active oxygen method (A.O.M.) and the almost same precision as A.O.M. and weighing method.

1 Introduction

An active oxygen method (A.O.M.)¹⁾, weighing method^{2)~4)}, and organoleptic tests^{3),4)} are ordinarily used for assessing oxidative stability of fats and oils. These methods are time-consuming or complicated. The oxygen absorption methods have been applicated to the studies of autoxidation of fats and oils. There have been, however, few reports^{5),6)} on the evaluation of oxidative stability. The possibility of an oxygen absorption method with a Warburg manometer for assessing oxidative stability was investigated here, since this method seemed to be simple and rapid and further the apparatus was easily obtainable.

2 Materials and Methods

Fresh fats and oils: lard, safflower oil, soybean oil, rapeseed oil, and rice-bran oil were obtained from Tsukishima Food Co., Ltd.,

Oxygen absorption was measured with a Warburg manometer (Mitamura Riken Kogyo Co., Ltd.). Mercury was used in the place of Brodie's solution as a manometer fluid. The average volume of the flasks was about 25.8 ml.

About 100 mg ($\pm$ 5 mg) of fats and oils was spread on the bottom of main compartment in the flask. The flasks were attached to the manometer and then the air over the sample was fully replaced with oxygen. To avoid the effect of light on the autoxidation, the flasks

were covered with aluminium foil. They were incubated in a water bath controlled at $70 \pm 0.1^\circ\text{C}$. The decrease of pressure in the flask was measured with manometer at intervals.

Weighing method was carried out at 60°C and the induction period was estimated from the time required for increasing the weight of sample (1 g) by 10 mg.

3 Results and Discussions

3.1 Experimental conditions

Brodie's solution was ordinarily used as a manometer fluid. However, the amount of oxygen absorbed by the sample was too large to measure the induction period with Brodie's solution under the conditions as above. The incubation temperature controlled at $70 \pm 0.1^\circ\text{C}$ resulted in the disturbance of level of Brodie's solution. Therefore, mercury was used as a manometer fluid. The test time could be shortened at the high temperature, but it became difficult to control the water bath at such temperature precisely. The incubation temperature was set at 70°C.

Fig.-1 shows the decrease of pressure in the flask caused by the oxygen absorption of safflower oil. The induction period was shortened when the air over the sample was replaced with oxygen (Curve 1). The time required for decreasing pressure by 20 mmHg was 40 h under oxygen and this value was about two-thirds of that required under the air. The air

(1): under oxygen (2): under the air

Fig.-1 Oxygen absorption of safflower oil.

over the sample was replaced with oxygen in the following experiments.

3.2 Oxygen absorption curves

Characteristics of various fats and oils used in this experiment are shown in **Table-1** and fatty acid compositions in **Table-2**.

Table-1 Characteristics of fats and oils.

Fats and oils	POV	AV	IV
Lard	0.41	0.13	64.9
Safflower	1.13	0.23	143.4
Rapeseed	0.12	0.16	117.8
Soybean	0.84	0.11	142.3
Rice-bran	1.05	0.28	107.0

Table-2 Fatty acid compositions of fats and oils.

Fats and oils	Fatty acids (%)							
	14:0	16:0	16:1	18:0	18:1	18:2	18:3	
Lard	1.7	24.7	4.0	11.9	45.8	10.5	1.5	
Safflower	0	8.3	0	2.6	13.0	74.9	1.2	
Rapeseed	0	3.8	0	1.8	62.4	20.9	11.1	
Soybean	0	11.9	0	4.2	25.2	52.0	6.9	
Rice-bran	0	19.5	0	1.8	41.5	35.0	1.9	

Fig.-2 shows the decrease of pressure with the oxidation of fats and oils at 70°C. Little oxygen absorption was observed during the induction period followed by very rapid decrease of pressure. The time required for decreasing pressure by 20 mmHg under the conditions as above was used as the index of oxidative stability. It corresponded to oxygen absorption of

Fig.-2 Oxygen absorption curves.

about 0.54 ml on the assumption that gravity of sample and Bunsen coefficient^{7),8)} were 0.9 and 0.1, respectively.

3.3 Comparison of three methods

The values obtained by three methods were compared using various fats and oils (**Table-3**). The values of rice-bran oil were 22 h by A.O.M., 21.4 d by weighing method and 149 h by the oxygen absorption method (O.A.M.). The time required for assessing oxidative stability could be shortened to about one-thirds of that required in weighing method.

Table-3 Comparison among the values by three methods.

Fats and oils	A.O.M. (h)	Weighing method (d)	O.A.M. (h)
Lard	7.4	5.6	35.0
Safflower	7.7	8.3	40.0
Soybean-Safflower*	11.6	12.1	66.0
Rapeseed	15.5	11.4	88.6
Soybean	15.9	15.3	93.0
Soybean-Rice-bran*	17.8	17.2	114.0
Rice-bran	22.0	21.4	149.0

* Mixture of two oils by the ratio of 1:1 (wt/wt)

Fig.-3 shows the correlation between these methods, and it became apparent that O.A.M. showed excellent correlation with A.O.M. ($r=0.995$). **Table-4** shows the values of rapeseed oil obtained in several experiments. The ratio of standard deviation to average was smaller in O.A.M. than in weighing method.

These results indicated that the oxygen ab-

Fig.-3 Correlation between three methods.

Table-4 Values of rapeseed oil by three methods.

Number of experiment	A.O.M. (h)	Weighing method (d)	O.A.M. (h)
1	16.0	11.4	86
2	15.8	11.7	89
3	15.7	10.6	88
4	15.4	11.6	89
5	15.4	11.8	91
6	15.4	11.7	—
7	15.2	11.1	—
8	15.4	11.7	—
Average (x)	15.5	11.5	89
Standard	0.24	0.37	1.9
Deviation (s)			
(s/x) 100	1.5	3.2	2.1

sorption method investigated in this experiment with a commercial Warburg manometer showed excellent correlation with A.O.M. and the almost same precision as A.O.M. and weighing method. Furthermore, the amount of sample required in O.A.M. was very small (100 mg) compared with A.O.M. (20 ml) and weighing method (1 g).

O.A.M. was the most simple of these methods and not so time-consuming as weighing method.

Further improvement of the apparatus, for example, incubator and manometer, would be expected to shorten the test time and to record the oxygen absorption automatically.

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酸素吸収法による油脂の酸化安定性試験法

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酸素吸収法による油脂の酸化安定性試験法を検討した。70°C で油脂に吸収される酸素量をワールブルグ検圧計を用い測定した。フラスコ内の圧力が 20 mmHg だけ減少するのに要する時間を誘導期の長さとした。本法によって得た値は、A.O.M. の値と極めてよい相関性を示し、また A.O.M. や重量法と同程度の精度を示した。