Short Communication

Oxidative Oil Stability Using Volatiles Methodology

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An accelerated oil oxidation procedure was used with the assessment of volatiles via gas chromatography to measure oil stability. This procedure utilized a mixture of oxygen and nitrogen as the purge gas. Preliminary results have shown good agreement with the standard AOM oxidative stability test.

Gas liquid chromatographic measurements of volatiles resulting from oil and fat autoxidation have shown good correlations with flavor scores (1-3). The necessity for predicitive or stability tests with more specificity and sensitivity and shorter analysis times still exists.

We have developed an accelerated oxidation procedure directly linked to a volatile method which should be of value as a rapid and sensitive oxidative stability method.

EXPERIMENTAL METHODS

The GLC volatiles procedure used was described previously (2, 4).

The modified method for assessing the stability of oil incorporates the following change: after the initial five-min purge with helium at room temperature to remove oxygen from the sample and U-tube, the purge gas was switched to a 250 ppm oxygen in nitrogen mixture. The sample tube was then placed in the oven at 170 C for 20 min while being purged with the gas mixture, and the volatiles produced from this accelerated oxidation were collected as usual on a Porapak P column.

Samples were analyzed to establish possible correlations between accelerated volatiles and the AOM stability test (5).

The first set of samples consisted of a freshly deodorized soybean oil, a partially hydrogenated soybean oil and a purchased sample consisting of partially hydrogenated soybean and palm oils. Samples were analyzed for volatiles using the previous procedure (2), and with the accelerated oxidation procedure presented here. The same samples also were run using the AOM method (5).

The second set of samples consisted of a freshly deodorized soybean oil to which 0.02% of the antioxidants BHA, BHT and TBHQ were added, along with a control. These samples were then aged 28 days at room temperature under normal fluorescent lighting. Again, the samples were analyzed by AOM and the accelerated oxidative volatiles procedure.

A third set of samples consisted of soybean, cottonseed, peanut and corn oils, which were analyzed as such and with BHA, BHT and TBHQ added separately at the 0.02% level without storage. Both accelerated volatiles and AOM were run on each sample.

RESULTS AND DISCUSSION

The results on the first set of samples are presented in Table 1. The ranking of samples is in good agreement, and the magnitude of differences is reasonable.

The results on the second set of samples are shown in Table 2. With this set of samples we compare soybean oil which has been aged (28 days) with and without antioxidants to the accelerated oxidative volatiles procedure and the AOM, both of which were run on the oils before storage. Again, the relative comparisons of the AOM and accelerated volatiles are reasonable, and both agree with the relative results, using the normal volatiles procedure (2) after aging of the oils.

The results of the third set of samples are shown in Table 3. This set reflects results on four different oils with no treatment, and the same four oils with BHA at 0.02% and with TBHQ at 0.02%. AOM comparisons with the accelerated volatiles method again show good agreement.

These preliminary results using a modified volatiles procedure suggest that it performs similarly to the standard AOM procedure in determining oxidative stability. The advantages of this procedure are greater sensitivity and a shorter analysis time.

The overall correlation between the accelerated volatiles procedure and the AOM results from all three tables is 'r' equals −.879. This correlation is significant at the 99% confidence level.

TABLE 1

AOM Volatiles Comparison

Sample	Initial Volatiles	Accelerated Volatiles	AOM[a] Hours
Soybean oil	4.6 ppm	54 ppm	8
Soybean oil (partially hydrogenated)	2.9 ppm	18 ppm	27
Soybean, palm oil blend (partially hydrogenated)	1.1 ppm	7 ppm	40

[a] Hours necessary to reach a peroxide value of 100.

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TABLE 2

AOM Volatiles Comparison

Sample[c]	Initial Volatiles PPM	Volatiles 28 days Storage PPM	Accelerated[a] Volatiles PPM	AOM[a,b] Hours
Soybean oil + BHA	—	28.4	36.4	12.25
Soybean oil + BHT	—	17.8	29.3	13.75
Soybean oil + TBHQ	—	8.7	18.9	30.0
Control soybean oil	4.6	41.6	57.8	8.0

[a] These values were run on the samples prior to storage.

[b] Hours necessary to reach a peroxide value of 100.

[c] BHA, BHT, TBHQ added at 0.02% by weight of sample.

TABLE 3

AOM Volatiles Comparison

Sample[a]	Initial Volatiles PPM	Accelerated Volatiles PPM	AOM[b] Hours
Soybean oil	3.2	79.0	8.75
Cottonseed oil	7.9	59.0	11.75
Peanut oil	6.6	45.1	15.75
Corn oil	2.5	30.7	22.5
Soybean oil + BHA	—	55.0	11.25
Cottonseed oil + BHA	—	45.0	14.25
Peanut oil + BHA	—	24.8	27.0
Corn oil + BHA	—	26.1	25.0
Soybean oil + TBHQ	—	28.1	23.0
Cottonseed oil + TBHQ	—	18.6	32.25
Peanut oil + TBHQ	—	10.4	46.0
Corn oil + TBHQ	—	9.3	47.25

[a] BHA and TBHQ added at 0.02% by weight of sample.

[b] Hours necessary to reach a peroxide value of 100.

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