Determination of Oxidation Stability of Soybean Oil with the Oxidative Stability Instrument: Operation Parameter Effects[1]

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ABSTRACT: Operation parameters of the oil stability index instrument were evaluated to determine their effect on the oxidative stability of commercial soybean oil. A factorial design was developed to evaluate the following three parameters, each at two levels, sample weight (2.5 or 5.0 g), conductivity tube temperature (20 or 30°C), and air flow rate (12 or 20 L/h), for a total of eight observations. Significance testing indicated that sample size and air flow rate affected oil oxidative stability independently ($P < 0.001$), but not in combination. The conductivity tube temperature did not affect the oxidation stability index. *JAOCS 72,* 741–743 (1995).

KEY WORDS: Oil stability index, oxidation stability, Rancimat, soybean oil.

Oil and food processors are always seeking methods to rapidly assess the oxidative stability of edible oils. Historically, the Schaal Oven Method and the Active Oxygen Method [AOM; the American Oil Chemists' Society (AOCS), Cd 12-57] (1) have been the most widely used tests to evaluate oil stability (2–4). Both involve the use of elevated temperatures to increase the reaction rate of lipid oxidation. The Schaal Oven Method typically produces results in days, whereas the AOM yields data in hours. Consequently, the primary method for accelerated oil stability analysis has been the AOM. Even with its widespread use, AOM analysis has many deficiencies and difficulties (5). An alternative method for the determination of fat and oil stability, developed over the past two decades, is based on the measurement of conductance produced by volatile organic acids collected in deionized water. The organic acids are stable secondary reaction products from heated oil that is oxidized by air bubbling through it (9).

Two oil stability index (OSI) instruments are commercially available, the Rancimat (Brinkmann Instruments, Inc., Westbury, NY) and the OSI Instrument (Omnion, Inc., Rockland, MA). Previous efforts have established the OSI as a potential alternative for the AOM. Investigations by deMan *et al.* (10) determined that the relationship between Rancimat and AOM times from various fats and oils was linear ($r = 0.997$). Läubli and Bruttel (11) also found results from the Rancimat and AOM to correlate highly ($r = 0.987$, 0.976, and 0.905 at 100, 110, and 120°C, respectively). Hasenhuettl and Wan (12) found OSI time reproducibility (standard deviation) within and between experiments to be 0.066 and 0.25 h, respectively, with soybean oil in the Rancimat.

In 1991, a collaborative study was conducted by the AOCS to establish relative error statistics, confidence limits, and effective temperature conditions for the OSI analysis (5). Fifteen laboratories, using either Rancimat or OSI instruments, were included in the study of vegetable oils at two operation temperatures, 110 and 130°C. The overall coefficient of variation (CV) was 11.3%. This study resulted in the approval of the OSI method as a new AOCS standard method, Cd 12b-92 (13).

Conductivity measurements of water, which has volatile oxidation products passing through, is the basis of the OSI method. Temperature is known to have an impact on conductivity measurement (14), yet how it affects the OSI time determination is unknown. Oil sample size and air flow rate are other parameters that can be adjusted easily in the OSI method and may affect OSI time. The present investigation was initiated to determine the effects of different operating parameters, air flow rate, conductivity tube temperature, and oil sample size, of the OSI instrument on the OSI time for edible soybean oil (SBO) at 100°C.

MATERIALS AND METHODS

Experimental design. An experimental design was developed (15) to evaluate the effects of three parameters, each at two levels: sample weight (2.5 or 5.0 g), conductivity tube temperature (20 or 30°C), and air flow rate (12 or 20 L/h), for a total of eight observations (Table 1). Three replications were run, each in duplicate. This experiment was run in a randomized order to assure that any treatment combination had an equal chance of being run first or last. Data were analyzed by analysis of variance and the Student's *t*-test.

Materials. Samples of three different lots of a commer-

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TABLE 1
Experimental Design and Corresponding Oil Stability Index (OSI) Time Means and Standard Deviations

Treatment	Flow rate[a]	Conductivity tube temperature[b]	Sample size[c]	OSI time (h)	SD (h)
1	–	–	–	15.75	0.91
2	+	–	–	14.83	0.83
3	–	+	–	15.35	0.17
4	+	+	–	15.10	0.18
5	–	–	+	14.78	0.08
6	+	–	+	13.87	0.06
7	–	+	+	14.83	0.16
8	+	+	+	13.75	0.22

[a]+ = 20 L/h, - = 12 L/h.
[b]+ = 20°C, - = 30°C.
[c]+ = 5 g, - = 2.5 g.

cially available SBO were obtained, with each treated as a different replicate. Peroxide values were determined by AOCS method Cd 8-53 (12). The air was generated in the laboratory and was purified by commercially available drying traps. A circulating tap-water bath produced the 20°C conductivity tube temperature condition. The 30°C condition was attained by heat from the 100°C block.

OSI evaluation. The OSI instrument, an automated apparatus based on conductimetric measurements, operates by a stream of purified air passing through a sample of fat or oil that is held in a thermostated aluminum block (5). The air distribution system does not heat the air prior to being bubbled through the oil. The air manifold has off/on valves, while the incoming dry air is regulated with a needle valve to vary the flow rate. After bubbling through the oil, the effluent air is bubbled through a tube that contains deionized water. The effluent air contains volatile organic acids from oil oxidation, which increases the conductivity of the water. The water conductivity is monitored with a nickel conductivity probe, which in turn is monitored and controlled by computer software. A mathematical equation, incorporating the conductivity signal and run time, is used to define the OSI time. The induction period, the length of time before the production of organic acids, is assumed to be a measure of the resistance of the oil toward oxidation.

To set up a run, 2.5 g or 5 g soybean oil was weighed into a 25 mm × 200 mm test tube and capped with a two-holed #4 silicon rubber stopper. A 9.5-inch Pasteur pipette was inserted in one hole with the tapered end in the oil; in the other hole, a 5–10 cm × 8 mm glass tube was inserted. Silicone tubing, 5-mm i.d., was used to connect the pipette to the air source and to transfer the effluent air from the oxidized oil tube to the conductivity tube.

The conductivity tube was a 32 mm × 162 mm polycarbonate centrifuge tube. Deionized water (50 mL) was added, and the tube was capped with a three-holed #6 stopper. A 9.5-inch Pasteur pipette was inserted through one hole, and a nickel conductivity probe through another. The last hole was left open to prevent pressure buildup. The pipette was placed at the bottom of the tube while the probe was left approximately 0.75 inch from the bottom. The probe was connected directly to the computer, which received the conductivity signal and processed it to generate the OSI time.

RESULTS AND DISCUSSION

Mean OSI time and standard deviation results (n = 3) of treatment combinations are presented in Table 1. The OSI times ranged from 13.75 to 15.75 h, while standard deviations ranged from 0.08 to 0.91 h. All SBO samples contained peroxide levels under 1 meq/kg oil, prior to the start of OSI analysis.

Results of parameter significance testing showed that sample size and air flow rate each had a significant effect ($P <$ 0.001) on the OSI time, while the conductivity tube temperature did not have a significant effect (Table 2). Two- and three-way interactions had no significant effect on OSI time.

Two-way interaction plots are displayed in Figure 1. While the interactions did not produce significant effects on OSI time, the interaction between flow rate and sample size merits

TABLE 2
Analysis of Variance on the Effect of OSI Instrument Parameter Treatments on OSI Time (F-values statistical significance for independent and variables interactions)[a]

Parameter	F-Ratio	P < \F\
Flow rate (FR)	4.25	0.001
Conductivity tube temperature (CT)	0.27	0.79
Sample size (SS)	5.10	0.001
FR × CT	0.67	0.51
FR × SS	−1.10	0.28
CT × SS	0.08	0.93
FR × CT SS	1.13	0.28

[a]See Table 1 for abbreviation.

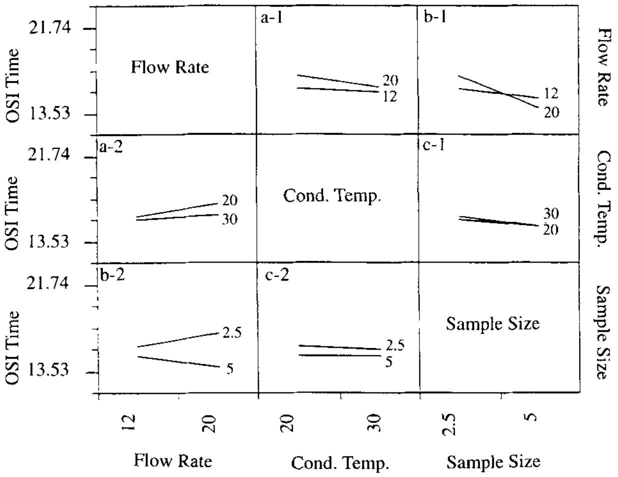

FIG. 1. Two-way interaction plots; a-1 and a-2 represent flow rate—conductivity temperature (cond. temp.) interaction; b-1 and b-2 represent flow rate—sample size interactions; c-1 and c-2 represent sample size—conductivity temperature interaction. OSI, oil stability index.

further attention (Fig. 1, plot b-2). At the low flow rate, the two sample sizes have similar OSI times, yet at the higher flow rate, the two sample sizes have significantly different OSI times. Jebe *et al.* (5) reported that a flow rate of 9 L/h was sufficient to continually saturate the oil sample with oxygen during the induction phase. However, results presented here indicate that air flow rates above 9 L/h do affect the OSI time. Jebe *et al.* (5) reported that temperature stabilization at a sample size of 2.5 g was difficult when using the Rancimat and that a 5-g sample size was recommended. A large standard deviation resulted in our test when the 2.5-g sample size was part of the treatment combination 30°C, as compared to the standard deviation resulting from the 5.0-g sample (Table 1). This indicates that the conditions under which the 2.5-g sample size oxidizes vary more than the 5.0-g conditions. These flow rate and sample size data results indicate that preferred conditions may be a flow rate of either 12 or 20 L/h and a sample size of 5.0 g.

While the conductivity tube temperatures used in the study did not affect the OSI time, a lower temperature (20°C) may be desirable if a high stability oil is used. Oils and fats with high stability, such as a frying oil, analyzed near 100°C, may result in conductivity tube water loss due to evaporation. This could create erroneous results because the organic acids may concentrate in the water as it evaporates.

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