# Technical



# Oat Oil: Refining and Stability<sup>1</sup>

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### ABSTRACT AND SUMMARY

Oil obtained by petroleum ether extraction of Dal oats was refined by conventional methods. Degumming loss was reduced to 15% by degumming in hexane solution and partially neutralizing the oil with sodium hydroxide. The free fatty acid was 6-8%, and alkali refining losses were 25-30%. Oat oil was bleached successfully with charcoal and deodorized. Stability of the refined oil was compared with soybean oil at 25 and 55 C by peroxide values and organoleptic tests. Stability of oat oil was increased by the addition of citric acid and was significantly greater than that of soybean oil, especially at 25 C. Oat oil contained significant amounts of  $\alpha$ -tocopherol, but ferulic and caffeic acids, antioxidants important in whole oats, were not extracted by hexane.

#### INTRODUCTION

Oats seldom have been considered a potential source of edible oil because the amount of oil found in most oats [3.8 to 8.5%(1)] is too low to make extraction profitable. But recently, wild oat (*Avena sterilis*) varieties have been discovered with 11-12% oil (2). These lines can be crossed freely with cultivated oats (*Avena sativa*), and it seems that oats with this much or more oil probably could be produced. The composition of oat oil also could be varied over a considerable range, and some compositions would be expected to make a good stable vegetable oil.

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FIG. 1. The peroxide values of oat oils and soybean oil during storage at 25 C.

This investigation was to see how well conventional refining procedures worked on oat oil and how stable the resulting oil would be.

#### METHODS

Two lots of crude oat oil were obtained through the courtesy of the Quaker Oat Company (Barrington, IL). The oil had been extracted from dehulled Dal groats by steaming, tempering to 12% moisture, flaking, and extracting at 55 C with hexane in a bucket-type extractor. Solvent was removed from the resulting miscella. One lot, designated 1975, had been stored for about 1 yr at 25 C. A second lot, designated 1972, had been stored for 3 yr at 4 C.

Dal oats obtained from the Iowa State University Agronomy Department were ground in a Wiley mill and extracted immediately with excess hexane. Starch and debris were removed by centrifuging for 5 min in a clinical centrifuge at 2,500 rpm. The solvent was removed in a rotary evaporator. This oil was designated 1976.

Triglyceride in crude oil was determined by thin layer chromatography on silica gel plates 0.75 mm thick developed in hexane-ether 85:15 (v/v). The triglyceride was extracted and weighed.

Refined soybean oil was obtained from the Anderson Clayton Company.

To degum the 1972 and 1975 oat oil, about 600 g was heated to 60 C, diluted with 480 to 600 ml of hexane, mixed with 1-2% (wt/vol) of 23.5% NaOH solution and centrifuged in a Sorvall RC2B centrifuge at 4,860 x G and 40 C. The hexane was removed in a rotary evaporator. Free fatty acids were determined (3), and 0.3 to 0.5% excess 23.5% NaOH was added to 100-g batches of the oil at 40 C



FIG. 2. The peroxide values of oat oils and soybean oil during storage at 55 C.



FIG. 3. The flavor scores of oat oils and soybean oil during storage at 25 C.



FIG. 4. The flavor scores of oat oil 1972 plus citric acid and soybean oil stored at 55 C.

with vigorous mixing. The mixture was held at 80 C for 3 min and then centrifuged as before for 1 hr. The oil was next bleached with 2% carbon (Darco G-60) at 80 C for 15 min, diluted to 30% (wt/vol) with hexane to decrease viscosity, and filtered. Solvent was removed with a rotary evaporator, and the oil was deodorized at 200 C for 2 hr in an all-glass apparatus (4). In some instances, 0.2% citric acid was added before deodorization. Refined soybean oil used in stability comparisons was freshly deodorized in the same apparatus.

Photometric oil color was determined by the AOCS Official Method (3).

For stability tests, about 1 liter of oil was stored in a closed 2-liter Erlenmeyer flask at 25 and 55 C. Peroxide values were determined by the Stamm method (5). Oils were tasted by a panel of 9-12 members using a scale on which 10 was bland and 1 was very strong. Samples were tasted at 25 C in semiprivate flavor booths under lights that minimized color differences in the samples. Results were examined by an analysis of variance (6).

Tocopherol was determined by the methods of Chow et al. (7) and Tsen (8). To determine caffeic and ferulic acids,

TABLE	I
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Free Fatty Acid and Triglyceride Percentage in Dal Oat Oils

Oil	% Free fatty acid	% Triglyceride	
1972	6.0	69.0	
1975	8.0	61.5	
1976	2.6	79.5	

#### TABLE II

The Fatty Acid Composition of Oat and Soybean Oils Used in These Studies

Range for oats <sup>a</sup>	Dal oat oils				
	1976	1975	1972	Soybean oil	Fatty acid
14-23	16.4	16.1	15.0	10.5	16:0
<1-4	2.7	1.9	2.8	4.4	18:0
29-53	41.0	39.5	43.6	25.7	18:1
24-48	37.9	39.8	36.2	52.8	18:2
<1-5	2.0	2.7	2.4	6.5	18:3

<sup>a</sup>Reported by Frey and Hammond (1).

oats were extracted with chloroform-methanol 2:1 (v/v), and the lipid recovered from the washed chloroform layer was saponified with alcoholic potassium hydroxide under a nitrogen atmosphere. The fatty acids recovered were crystallized from a 2% acetone solution according to Chow et al. (7). Acetone-soluble material was separated by thin layer chromatography on silica gel plates, 0.75 mm thick, with chloroform-methanol 90:10 (v/v). Guide spots were used to identify the areas containing caffeic and ferulic acids. Guide spots sprayed with 0.2% dichlorofluorescein solution were viewed under ultraviolet light. Areas containing the aromatic acids were scraped off and eluted with methanol. Absorbency of methanol extracts was determined with a Beckman DU spectrophotometer.

#### **RESULTS AND DISCUSSION**

When the crude 1972 and 1975 oat oil was degummed like soybean oil, by adding 3-5% hot water and centrifuging, 25% of the weight was lost to the gum fraction. This was not reduced appreciably by adding ions to the water. If the oil were diluted with hexane to decrease its viscosity and centrifuged for 2 hr, the degumming loss could be reduced to 16%, but the oil was still slightly cloudy. The clearest oils were obtained at dilutions of 80 to 120 ml of hexane/100 g of oil. Residual cloudiness could be removed by adding concentrated NaOH along with the hexane. In this way, the degumming loss was reduced to 15%, and a clear, dark brown oil was obtained. It was not necessary to add enough alkali to neutralize all the free fatty acids of the oil to achieve clarity. In practice, it would probably be best to combine the degumming and alkali-refining steps, but we did them separately to measure the losses at each stage, adding only enough alkali in the degumming step to achieve oil clarity.

The alkali-refining loss was 25-30% even though the usual washing and drying steps were omitted. This is about four times the free fatty acid content.

Good bleaching was achieved with charcoal, which resulted in a pale yellow oil. The photometric color index for the oil was 2.1. The loss in bleaching was less than 1%. Bleaching earth was not very effective in decolorizing oat oil.

Standard deodorization gave a bland oil with negligible loss.

Oats contain a potent lipase that can act at very low moisture levels (2) Table I shows that if Dal oats are ex-

tracted quickly before the lipase can act, the free fatty acid is 2.6%. The oats used for the large scale production of oil (1972 and 1975) were steamed, but it is not certain that the lipase was destroyed. The 1972 and 1975 oils contained significantly more free fatty acid. Possibly the free fatty acids increased during storage of the crude oil, but crude cottonseed and soybean oil can be stored for up to 4 yr with only small increases in refining loss and negligible deterioration in stability after refining (9-11).

The data on triglyceride percentage in Table I suggests that the 15% degumming loss observed may be near the minimum possible. It is probable that, if the oil percentage of oats were increased by breeding, the amounts of phosphatides and other gummy substances would not increase. If the oil percentage of Dal oats were doubled from the present value of 7% or 8% to 16%, then the refining loss might be only half of the present 15%.

Figures 1 and 2 show plots of peroxide values of oat oils against time at 25 C and 55 C. A soybean oil sample is included as a comparison. These results show that, at both temperatures, inclusion of 0.02% citric acid improved the stability of the oil markedly. Oat oils in which citric acid was included maintained lower peroxide values than soybean oil (which also contained citric acid), and these differences were statistically significant.

Figure 3 shows flavor scores of oat oils stored at 25 C, with soybean oil included as a comparison. Flavor scores for the oat oil were significantly increased by inclusion of citric acid, and all oat oils, even those without citric acid, scored significantly higher than the soybean oil.

Figure 4 shows flavor scores of an oat oil (1972 + citric acid) compared with soybean oil at 55 C. The 1975 oil with and without citric acid showed very similar results. None of them were statistically different from each other; in all instances, however, oat oil had a higher flavor score than soybean oil for 2 or 3 days and then had a generally similar score.

The relatively poor flavor stability of soybean oil has been blamed on its linolenic acid content (12,13). Table II compares the fatty acid composition of soybean oil used in these experiments with that of Dal oat oil. Linolenic and linoleic acid content in soybean oil is significantly higher. The range of fatty acid composition shown in oats (2) indicates that oats with even less linolenic acid than the Dal cultivar should be possible.

Oat oil was tested for antioxidants. a-Tocopherol was the main component, although in crude oil, traces of  $\beta$ - and  $\gamma$ - tocopherol also were detected. After degumming, only  $\alpha$ -tocopherol was detected. Tocopherol content at various stages of refining in  $\mu g/g$  was: crude oil 105, degummed oil 67, alkali refined oil 40, bleached oil 38. Novzhilova et al. (14) reported that tocopherol in oat lipids varied from 100 to 740  $\mu$ g/g and was composed of  $\alpha$ ,  $\beta$ - and  $\gamma$ -isomers. Chow

et al. (7) reported 175  $\mu g/g$  in oat oil, of which 70  $\mu g/g$ was  $\alpha$ -tocopherol.

Oats are known to contain esters of ferulic and caffeic acids, which act as antioxidants in oat products (15,16). We were not able to detect either of these acids in the crude oat oil. The exact combinations in which esters of ferulic and caffeic acids exist are unknown, but they are not soluble in petroleum ethers; thus, it would not be expected that they would be included in oat oil extracted with hexane. In chloroform-methanol extracts of Dal oats, we were able to detect material that migrated with these acids on thin layer chromatograms and that had absorption spectra resembling impure ferulic and caffeic acids. From the spectra, we estimate that Dal oats contain 22  $\mu g/g$  of ferulic and 34  $\mu g/g$ of caffeic acid. Extraction and refining methods that included these antioxidants in the oat oil might enhance its stability considerably.

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