# Removal of Waxes from Sunflower Seed Oil by Miscella Refining and Winterization

W. HERBERT MORRISON III, and J.K. THOMAS, Field Crops Research Laboratory, Richard B. Russell Agricultural Research Center, ARS, USDA, PO Box 5677, Athens, Georgia 30604

### ABSTRACT

Miscella refining and winterization produced a sunflower seed salad oil that did not cloud on refrigeration for 7 days. Refining reduced phospholipid content, and this facilitated wax removal during winterization.

# INTRODUCTION

Cloud formation during low temperature storage is a problem in salad oils prepared from sunflower seed oil and other vegetable oils (1). With most vegetable oils, this problem is taken care of by winterization. However, winterization is difficult to do with sunflower seed oil owing to the low concentration of waxes and the mucilaginous materials which coat crystals and retard filtration of the cold, viscous oil (2).

Solvent winterization, with a hexane-acetone mixture, was used to reduce viscosity and facilitate crystallization of the waxes (3). Were this procedure to be used commercially, a separation step would be necessary after removal of solvent from the winterized oil to separate acetone and hexane. We now have investigated ways to process the miscella that would keep the desirable features of solvent winterization but eliminate extra separations.

Miscella refining is carried out most economically by incorporation into an existing solvent extraction process of an oilseed crushing mill (4-6) and was the basis of this study.

We compared wax removal from the miscella by two sequences. First, the miscella was winterized then refined, and secondly refined then winterized.

## MATERIALS AND METHODS

Sunflower seeds grown in Georgia were ground in a Wiley mill and extracted for 6 hr with hexane. The oil was prepared for refining and winterization as described (3).

Miscella refining and winterization were done with reagent grade hexane with oil in solvent concentrations of 40, 50, and 60% by wt.

One portion of each mixture was added to centrifuge bottles to provide ca. 100 g oil after evaporation of solvent. The samples, in triplicate for each concentration, were winterized at 0 C for 5 hr and centrifuged at 9,000 x g for 20 min in a refrigerated centrifuge maintained at 0 C. Half of each winterized sample was frozen for chemical evaluation. The remainder was refined by stirring at 40 C for 15 min with 18° Bé NaOH with 0.2% excess and allowed to settle for 30 min. The oil-solvent layer was decanted and filtered through anhydrous sodium sulfate. Solvent was removed, and the oil was prepared for cold testing and evaluation as reported (3).

A second portion of the oil-hexane mixture was first refined as above and the oil-solvent mixture decanted. Half of the sample was frozen for chemical evaluation; the other half was winterized as described, centrifuged, prepared for cold testing, refrigerated, and evaluated for clouding.

Unsaponifiable materials and phosphatides were determined according to standard AOAC (7) and AOCS (8) methods. Wax contents were measured as reported (3). All values given are the averages of triplicate analyses.

				Sequence, winte	rized-refined					Sequence, refine	d-winterized		
				Stel	<b>35</b>					Step	8		
			Winterized			Refined			Refined			Vinterized	_
				% Oil in s	olvent					% Oil in s	olvent		
Evaluations	Initial <sup>a</sup> values	40	50	60	40	50	60	40	50	60	40	50	9
Phosphatides (%) <sup>b</sup>	.345	.315	1331	.331	039	.036	.061	.056	.036	015	046	0.7.6	6
Unsaponifiables (%)	.73	.72	.51	.63	.56	.64	.60	.55	48	.60	909	202	ο α
Waxes (%)	-021	.019	.020	.017	.020	.015	.017	910	.017	.016	007	000	
Clouding time (days)	I		•		-	-	1	•		•	3.3	~	22

#### RESULTS AND DISCUSSION

Table I shows the amounts of phosphatides, unsaponifiables, and waxes remaining in the oil after each step of the two sequences. In addition, the refrigeration time necessary for cloud formation is indicated.

In the first sequence, where miscella was winterized before it was refined, only small amounts of phosphatides, unsaponifiables, and waxes were removed by winterization. After refining, however, unsaponifiables in general decreased and phosphatides decreased markedly. Wax content showed little change with refining, and the oil clouded after only one day's refrigeration.

In the second sequence, miscella was refined before it was winterized. Refining reduced the phosphatide content to ca. 10% of the initial value, unsaponifiables by ca. 50%, and waxes by an average of 22%. The removal of phosphatides prior to winterization apparently facilitated removal of waxes in the winterization step. The oil's refrigeration characteristics improved markedly; two of the three samples remained free of cloud for longer than 7 days.

Bailey reported that lecithin acts as a crystal inhibitor in cottonseed oil (9). Similarly, Rac (2) has shown that the addition of 1% lecithin to an oil containing 1% pure wax reduced the range of crystal size on refrigeration from 50-90 $\mu$  to 20-30 $\mu$ . In sunflower seed oil, phosphatidyl cholines represent 51% of the phospholipids in the oil (10). Removal of phospholipids should favor crystallization and facilitate crystal formation.

Our data indicate that miscella refining removes sub-

stantial amounts of the phospholipids which retard crystal formation on winterization. When the oil is miscella refined and winterized, crystallization appears to be more complete than when the oil is merely winterized. Viscosity and entrained oil are reduced, resulting in lower oil loss (11). The final oil then has good refrigeration characteristics which should be acceptable to the consumer.

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