Turbidimetric Measurement of Wax in Sunflower Oil

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Wax content in sunflower oil may be estimated quickly at room temperature by a turbidimetric method. Five ml of a thoroughly mixed oil/solvent mixture is transferred to a 13-mm sample vial and inserted in a turbidimeter. Instrument readings are converted directly to wax content in the original sunflower oil by means of a correlation equation. This rapid method can be used over a wider range of wax values than previous methods.

In this decade the U.S. is expected to become a leading producer of sunflowerseed. It would be beneficial for growers and oil processors to have an early measure of the wax content of sunflower oil. The wax content in processed oil may vary with seed variety, geographical growing area and seasonal growing conditions, and will affect processing conditions and economics. Crude sunflower oil has been reported to have a range of wax content from 200 to 3500 ppm (1) and commercial "dewaxed" sunflower oil to contain less than 80 ppm wax (2).

Turbidity methods to measure wax content in sunflower oil have been reviewed by Sleeter (3) and reported by Brimberg and Wretensjo (4) and Morrison (2). The Brimberg method measures wax content in neat oil without solvent, but is limited to low-wax winterized oils. The Morrison method measures wax content of "dewaxed" and crude oils on a 50/50 (v/v) blend of sunflower oil/cetone. These turbidity methods require measurements at a temperature of 0 C.

This paper describes a rapid and improved method in which weighed crude or processed oil samples are blended with solvent at room temperature and the turbidity conveniently measured within one min. The present method eliminates cooling to room temperature of hot filtered sunflower oil and the subsequent heating and cooling steps of 50/50 (v/v) oil/solvent samples described in the Morrison method.

MATERIALS AND METHODS

Wax-free sunflower oil was prepared from commercial Sunlite Wesson sunflowerseed oil (Beatrice Companies, Inc., Fullerton, California) by cooling the oil to 34 F (1 C) for three days and then vacuum filtering at 34 F (1 C) through Hyflo Super-cel filter aid (Johns-Manville Products Corp., Lempoc, California).

A standard oil containing 4800 ppm wax was prepared by dissolving 0.119 g sunflower wax (mp 75–78 C) in wax-free oil at 85 C to give 24.9 g oil. A series of standard oils containing 2400, 1200, 600 and 300 ppm wax was prepared by successive 50/50 dilutions of the standard oil containing 4800 ppm wax with wax-free oil.

In preliminary experiments, neat oil and oil/solvent mixtures were used to determine the wax content in sunflower oil as a function of turbidity with a Hach Model 18900 Ratio Turbidimeter® (Hach Chemical Co., Loveland, Colorado). Neat crude sunflower oil was found to be too cloudy to transmit light from the source to the detector. Various levels of oil with acetone and oils with acetone/hexane (85/15) gave values within the instrument range of 0–200 NTU, but turbidimeter readings frequently were unstable at high wax concentration because of nonuniform settling of wax particles in the recommended 25-mm sample vial. A prepared solvent of 120 g wax-free sunflower oil made up to one liter with acetone was found to give more stable readings, apparently due to delayed settling of the resulting wax particles. This solvent is designated as "WS solvent" in this paper.

Although turbidity readings of test oil/solvent samples were relatively stable with the 25-mm sample vial, the range of the instrument was exceeded for crude oils with high wax content. A 13-mm vial inserted in a special adapter (Hach No. 42010-00) gave stable readings for a one-min reading time. Culture tubes (13 mm with screw caps) were found to be satisfactory as sample vials. However, due to variable properties in the glass, the point of lowest NTU value was located beforehand and the tube marked to assure tube alignment for reproducible and accurate readings.

RESULTS AND DISCUSSION

The most effective sample size was determined by measuring the turbidity of oil/WS solvent mixtures at various oil concentrations. Turbidity (in nephelometric turbidity units, NTU) of each standard oil was first measured neat (without solvent), then, diluted 50/50 with WS solvent to obtain 50%, 25%, 12.5%, 6.25% and 3.125% oil-in-solvent successively. These test oil blends were prepared from the oils containing 4800, 2400, 1200, 600 and 300 ppm wax. The resulting data are plotted in Figure 1.

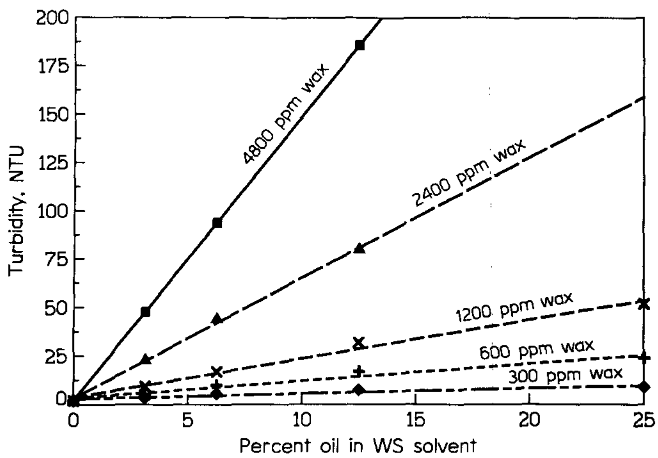

FIG. 1. Effect of oil concentration in WS solvent on turbidity (NTU) of samples for standard sunflower oils containing 4800, 2400, 1200, 600 and 300 ppm wax. (WS solvent = 120 g wax-free oil made up to one l with acetone).

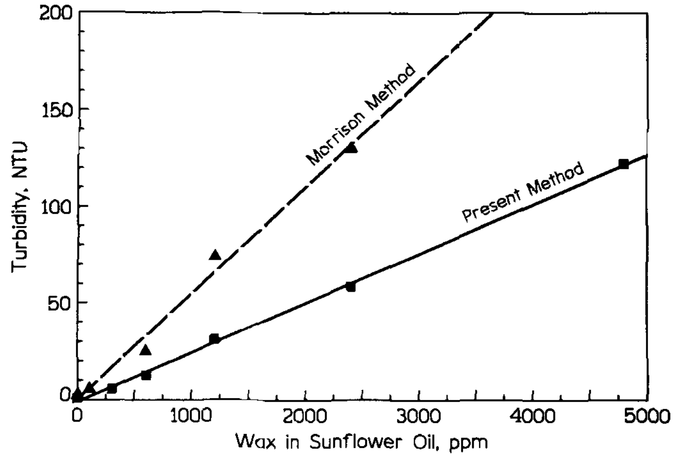

FIG. 2. Correlation of wax content in sunflower oil with turbidity (NTU) of oil/WS solvent samples. Present method (wax in sunflower oil, ppm = 40.1 + 39.2 × NTU; correlation coefficient = 0.999). Morrison's method based on graph data (2) (wax in sunflower oil, ppm = 3.2 + 18 × NTU; correlation coefficient = 0.994).

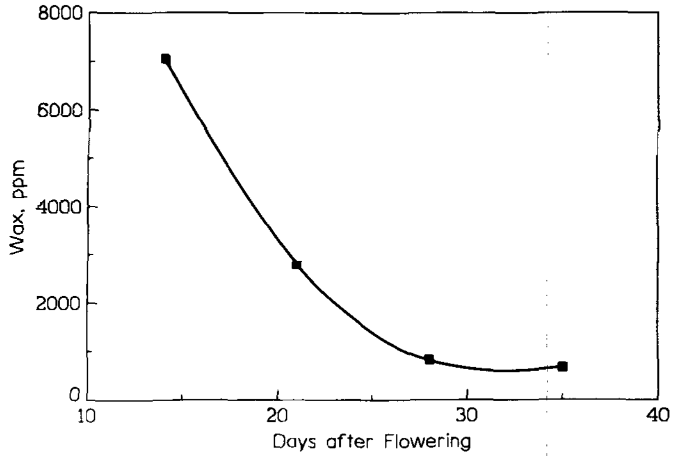

FIG. 3. Effect of sunflowerseed maturity on the wax content in the extracted sunflowerseed oil.

Samples with 6.25% and 12.5% oil in WS solvent could be measured within the instrument range of 0-200 NTU. On this basis a simplified rapid method to measure wax in sunflower oil was developed by using 6.25% oil in WS solvent as follows: Heat test oil to 130 C; filter hot oil through Whatman #4 filter paper and weigh 1.50 g clear oil into a 25-ml volumetric flask; add WS solvent to the mark; mix well by shaking; transfer ca. five ml of this solution to 13-mm screw top vial; insert in Hach Turbidimeter® equipped with 13-mm adapter; choose proper NTU range; record instrument NTU reading after it is stabilized for one min; calculate wax content in the oil sample using the linear regression equation: wax in sunflower oil, ppm = 40.1 + 39.2 × NTU. A calibration curve as shown in Figure 2 (present method), or a table, can be used in the plant if preferred.

Our present method was used to measure wax in sunflower oil by determining NTU values for standard oils containing 4800 ppm, 2400 ppm, 1200 ppm, 600 ppm, 300 ppm and 0 ppm wax. Multiple analyses of oil/solvent samples followed the linear curve $Y = -0.95 + 0.025 X$, where X = wax content, ppm and Y = turbidity, NTU, with a correlation coefficient of 0.999 (Fig. 2). This method was compared to that of Morrison to measure wax in sunflower oil (2) on the same series of wax oils with the Hach Model 18900 Ratio Turbidimeter®. NTU values of standard oils by Morrison's method correlated with wax content according to the curve $Y = 0.39 + 0.055 X$ with a coefficient of 0.994 (Fig. 2). Morrison's method required more steps than the present method and exceeded the limit of the instrument with oils containing more than 3500 ppm wax.

Our present method to measure wax in sunflower oil was tested for repeatability. After a crude oil was dried and filtered, WS solvent was added to 10 individual 1.5-g oil samples, the turbidity measured and the wax content in each sample calculated. Average of the 10 measurements was 2600 ppm wax with a coefficient of variation of 3.8%.

Commercially processed crude sunflower oils were analyzed for wax content. Two oils from one supplier contained 2200 and 2425 ppm wax; one from a second, 1375 ppm wax; and two from a third supplier 4050 and 5250 ppm. A commercial winterized sunflower salad oil analyzed at 48 ppm wax by this turbidimetric measurement method. The wax content in crude oil from immature sunflowerseed was found to be higher than in oil from mature seed. For this latter determination, the oils were extracted with hexane from seeds taken from relative locations in the same flower head at 14, 21, 28 and 35 days after flowering (DAF). Wax in the oil diminished from 7051 ppm at 14 DAF to a near constant value of 667 ppm at 35 DAF (Fig. 3). Oil/WS solvent mixtures of oils with native wax appeared to be more stable for a longer period in the turbidimeter than the wax-oil mixtures prepared to establish the correlation data.

This rapid procedure should have application for oil processors to predict condition of the seed and expected processing losses due to the wax content in crude sunflower oils. If high losses are predicted, further evaluation of an oil may be required to compensate during processing for the effects of immature, damaged or abused seed which lower the quality of the finished oil.

ACKNOWLEDGMENT

Sunflower wax, used to prepare oils of known wax content, was obtained from W. H. Morrison III, Richard Russell Agricultural Research Center, Athens, Georgia. Sunflowerseeds of varied known maturity were furnished by Don Zimmerman, North Dakota State University, Fargo, North Dakota. Ray Holloway extracted sunflowerseeds for crude oil evaluations.

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