Rapid Spectrophotometric Technique for Evaluation of Vanilla Extracts

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The importance of color in processed foods and the use of objective color evaluation techniques have frequently been stressed. Vanilla is often adulterated or substitutions are made, resulting in lower costs but loss of true flavor. The rapid scanning spectrophotometer makes possible the detection of adulteration by a simple and rapid method, and appears to offer significant promise for quality control.

THE MEASUREMENT OF COLOR in processed foods and the control of color during food processing are among the most important factors affecting grower-processor relationships and consumer acceptance. Only about 10% of government standards for grading employ objective color evaluation techniques.

Completely objective methods, which use physical instruments without the influence of the human element, under standardized conditions of observation provide the most reliable color analysis of foods.

Although many food products have been evaluated (*2*), this paper deals only with the possible application of a rapid scanning spectrophotometer to the vanilla processing industry as a quality control instrument for use by the food processor, to detect adulteration, and for potential use by federal agencies in grading and inspection. Vanilla, like many other flavoring extracts, is of extreme interest to the food technologist because of the ingenious substitutes and forms of adulteration that its relatively high price encourages. It is often skillfully adulterated, usually by the addition of caramel or coal-tar derivatives, to resemble the pure vanilla extract in odor and color. The adulterated products cost only a fraction of the price of pure vanilla, but they do not have the delicate flavor of the true extract.

Apparatus and Technique

The instrument used is a combination of an optical spectrophotometer and an electronic cathode-ray-tube indicating device. Complete spectrophotometric curves in the visible region (400 to 700 mμ) are instantaneously produced on the face of a cathode-ray tube. The coordinates for both the wave length and the percentage of transmittance are linear, and the illuminated grid (divided into wave length intervals of 20 mμ and photometric intervals of 5% transmittance) provides a convenient reference scale.

Detailed specifications for construction and operation have been published (*1*).

The data produced on the face of the cathode-ray tube may be observed visually or recorded photographically with a camera adaptable to a 5-inch cathode-ray oscilloscope. A Fairchild Polaroid Land Oscilloscope camera was utilized in this investigation.

The sample chamber, designed to accommodate absorption cells up to 100 mm. in length, has a hinged door at the front. The samples were placed directly in the cuvettes, which in turn were placed in the cell holder and inside the sample chamber. In this study cell holder No. 1076 was used to accommodate three Cenco No. 29340 absorption cells.

In operation, the instrument was set at the 0 and 100% transmittance lines; the wave length was then calibrated by means of a standard calibration curve published by the National Bureau of Standards as shown in Figure 1.

Observations

Spectral analyses were made on four commercial brands of pure vanilla and seven brands of imitation vanilla selected at random from shelves of food markets in the Boston-Cambridge area and not specially processed for this study. Because of the deep color of the vanilla samples, transmittance data were obtained for various dilutions in both water and 37% alcohol (the concentration in most commercial pure extracts).

The various brands of pure vanilla produced spectral curves of the same general character, but the percentage of transmittance at given wave lengths varied from brand to brand and, at times, even within the same brand when different bottles were used. This difference in intensity of transmittance is to be expected, because of differences in the grades and varieties of the vanilla bean, variations in the solvent employed in extraction, and the length of maceration in the solvent.

The general character of the spectral curve for imitation vanilla closely paralleled the curve for pure vanilla, and again the intensity of transmittance varied from brand to brand. As the greatest differences in percentage of transmittance occurred at 700 mμ, the following data are based on transmittance at this apparently critical wave length. Both pure and imitation vanillas, diluted in 37% alcohol, produce straight-line relations when the percentage of transmittance at 700 mμ is plotted against dilution. When water is used to dilute the samples, only the imitation vanillas produce a straight-line relation, identical with that of the alcohol dilution in both slope and intensity of transmittance. The pure vanilla curves are extremely distorted at the lower dilution (0 to 40% water), but a straight-line relation exists at the higher dilutions (40 to 80% water). Probably resins in true vanilla tend to precipitate at the lower water dilutions, causing turbidity; artificial vanilla remains perfectly clear.

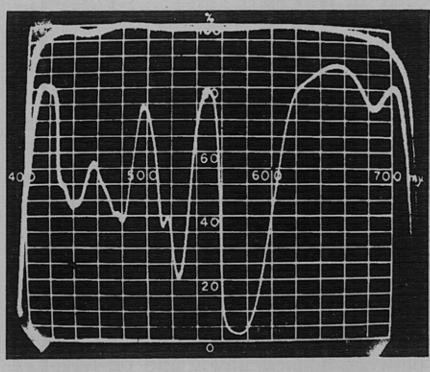

Figure 1. Didymium filter calibration curve also showing 100% and zero light curves

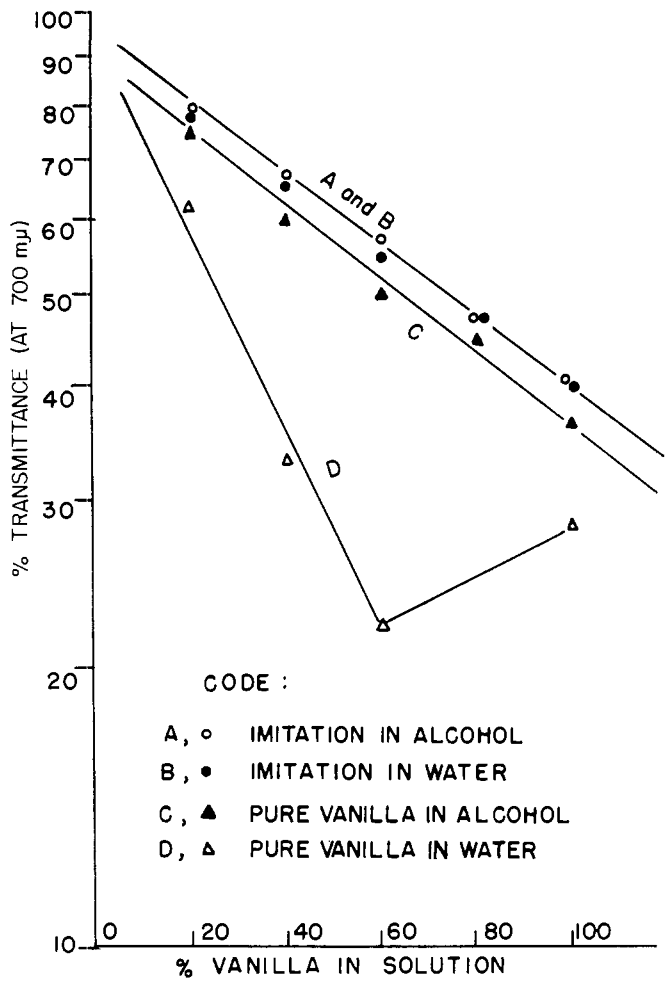

Figure 2. Transmittance of light through varying concentrations of pure and imitation vanilla in aqueous and alcohol solutions

as it contains no resins. This relationship is portrayed graphically in Figure 2.

The most sensitive dilution appears to be 60% vanilla and 40% water. Figure 3 indicates the typical difference in the transmittance curves of pure and imitation vanilla at this critical dilution.

Conclusions

The rapid scanning spectrophotometer can be used to differentiate between pure and artificial vanillas of the 60% vanilla solutions in water. The difference between pure and imitation vanillas should be readily apparent from the plot of percentage of transmittance (at 700 mμ) against water dilutions. The instrument should also be a distinct asset in quality control to assure uniformity of product.

The primary objectives of a physical instrument are to eliminate the variable factors prevalent in visual grading and to make possible direct readings by non-technical personnel. The instrument studied appears to possess the following desirable characteristics:

Complete spectrophotometric curves in the visible spectral range are instantaneously recorded on the face of the instrument.

Readings can be interpreted by simple ratios or other similar indices—a convenient numerical form suitable for tabulation and correlation studies—rather than by calculations of the tristimulus values.

Highly trained personnel are not required for the method of presentation of the samples to the instrument.

The use of an oscilloscope camera attachment makes it possible to procure permanent records of the colors analyzed for use as standards at some future date, or as pertinent records to aid in controversies that may arise over color grading.

This study was in the nature of an exploratory and preliminary evaluation. Much more extensive sampling and investigation must be accomplished under various conditions before results and conclusions can be recommended as being absolute tests for objective physical-color determinations. The preliminary investigation of the rapid scanning spectrophotometer resulted in the following conclusions:

It can be used successfully for color quality control work. The rapidity of operation makes it possible to examine every batch if necessary, and the required adjustments in the production line can be handled on a preventive rather than a "post-mortem" basis.

It may be useful in the detection of adulteration and mislabeling if spectral curves of samples of known purity are available, as the methods of adulteration are difficult to detect and the present analytical methods for examination are lengthy and complex.

It appears that this instrument possesses potential as a research tool in addition to its more direct practical applications and should stimulate further interest in undertaking investigations of the fundamental causes for changes in food commodities before, during, and after processing.

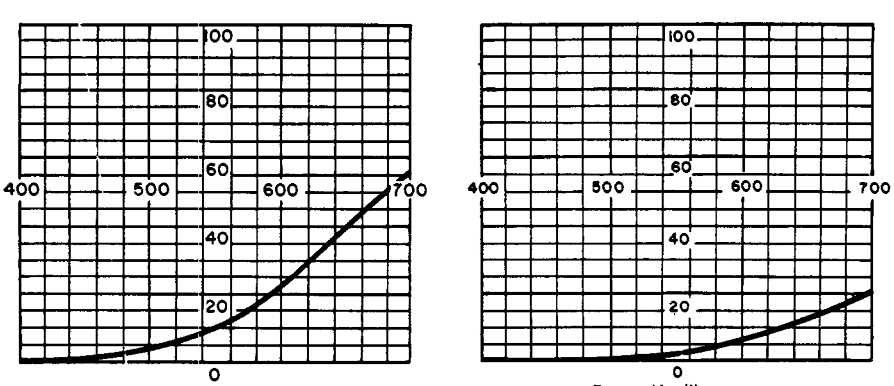

Figure 3. Typical transmittance curves of pure and imitation vanilla in 60% vanilla solution in water

Acknowledgment

The authors express appreciation to the American Optical Co. which kindly loaned a rapid scanning spectrophotometer.

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(2) Pomerantz, R., Goldblith, S. A., Proctor, B. E., *Food Technol.* **9**, 478–82 (1955).

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