Note on Comparison of Pigment Extraction and Reflectance Colorimeter Methods for Evaluating Semolina Color[1]

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Product color is one of the most important criteria for determining pasta quality. Domestic and foreign consumers prefer bright yellow, translucent pasta products. Thus, cereal technologists and durum breeders have attempted to develop techniques to rapidly and accurately evaluate semolina and pasta color. Over the past several years, three major categories of pasta color prediction and evaluation have evolved; visual comparisons with standard samples, chemical pigment extractions, and light reflectance measurements.

The relationships between these different methods have been reported by many workers. Fifield et al (1937), using a disk colorimeter, determined the percent yellow color of pressed disks of moist durum semolina. Using their data, we calculated a correlation coefficient of 0.67 (n = 11) between percent yellow color of macaroni and percent yellow color in the disks. Irvine and Anderson (1953), using the standard water-saturated butanol method, reported a correlation between semolina pigment and macaroni pigment of 0.89 (n = 138) and later (1955) reported a correlation between whole kernel pigment and macaroni pigment of 0.89 (n = 137). Matz and Larsen (1954) compared four reflectance colorimeters to visual ranking and obtained correlation coefficients ranging from 0.77 to 0.89. Walsh (1970), in similar experiments, reported correlations of 0.91 and 0.96 between colorimeter and visual ratings.

Although the visual rating method has shown high correlations with reflectance colorimeters, the potential for subjective error in its practical laboratory usage has caused it to be replaced by pigment extraction and reflectance colorimeters that offer greater precision and objectivity. The relationship between these two methods has not been reported. Therefore, the objective of this study was to determine the relationship between the American Association of Cereal Chemists' water-saturated butanol (WSB) method of pigment extraction (AACC 1961) and readings from a reflectance colorimeter.

MATERIALS AND METHODS

Ten durum cultivars and selections were grown at Langdon, ND, in 1978 in a randomized complete block design with three replications. The entries, selected to provide a range in semolina pigment content, were Rolette, Rugby, D7429, D73105, D6750, and D7099 (North Dakota); Capeiti (Italy); Cocorit 71 (Mexico); Alifen (Chile); and Wakooma (Canada). The entries were harvested, cleaned, tempered to 15.0% moisture, and milled to semolina in a Brabender Quadramat Jr. mill (Vasiljevic et al 1977). Each milled sample was separated into two lots: twenty grams for evaluation on the reflectance colorimeter and eight grams for the pigment extractions. The reflectance colorimeter was the Hunter Color Difference Meter equipped with a D25A optical unit. Only the "b" values, which measure the degree of yellowness, were recorded on this unit, as recommended by Matz and Larsen (1954). The predominant pigments in durum semolina are xanthophylls or luteins (Sims and Lepage 1968) and not carotenes. However, because the wavelength for β-carotene is used in the AACC method, pigment content was determined using the wavelength for free lutein and β-carotene on each sample. The values, reported as μg of β-carotene, are not true measures of the amount of carotene present. Pigment extractions followed the WSB method with the exception of extraction time, which was 18 hr in this experiment. (The normal time is 16 hr.) A Bausch and Lomb Spectronic 20 spectrophotometer was used to obtain percent absorption, and instrument calibration was checked between each sample with a pure WSB solution. The spectrophotometer was set at 449 nm for determining free lutein. The absorptivities used in calculating pigment content (micrograms of pigment per gram of sample or ppm) were 1.663 for carotene and 2.336 for free lutein. The wavelength and absorptivity of free lutein are those reported by Sims and Lepage (1968).

The formula used to calculate pigment concentration was:

$$\frac{\frac{A_s}{A_{st}} \times \frac{V}{100} \times 1{,}000}{\text{dry weight of sample}} = \text{pigment, } \mu g/g$$

where A_s = sample absorbance.
A_{st} = standard absorbance of one milligram of pigment per 100 milligrams of solution.
V = volume of the extraction solution.

RESULTS AND DISCUSSION

The entry mean and rankings for carotene content, free lutein content, and Hunter reflectance values are presented in Table I. Significant differences among entries occurred within all methods, and in most cases the reflectance values corresponded well with the spectrophotometer values for pigment content. The only exceptions were D6750 and D7099, which had lower reflectance values than their pigment content would indicate. The relative entry ranking, compared across methods, reveals that all methods are effective in distinguishing the high from the low entries. The Hunter reflectance values were highly correlated ($\alpha = 0.01$, n = 30) with the lutein (r = 0.85) and carotene pigment (r = 0.83) contents. As expected, a high correlation (r = 0.99) was found between the values obtained from the β-carotene wavelength and the free lutein wavelength.

TABLE I
Means and Rankings Obtained from Three Methods of Semolina Color Evaluation

	Method[a]					
	Free Lutein		Carotene[b]		Hunter	
Entry	ppm	Rank	ppm	Rank	b Value	Rank
D7429	8.59	2	9.87	2	25.36	1
D73105	6.66	3	7.90	3	23.66	2
D6750	8.73	1	10.26	1	23.66	2
Rugby	5.17	4	6.00	4	22.20	4
Wakooma	4.45	7	5.05	7	21.40	5
Capeiti	5.12	5	5.85	5	20.73	6
Rolette	3.62	8	4.21	8	20.46	7
D7099	5.06	6	5.77	6	20.33	8
Alifen	3.17	9	3.89	9	16.76	9
Cocorit 71	2.03	10	2.48	10	16.30	10
LSD ($\alpha = 0.05$)	0.53		0.32		0.33	

[a] Mean of three replications.
[b] Not a true measure of carotene content, as explained in text.

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These data indicate that semolina color values obtained from the Hunter Color Difference Meter and the WSB pigment extraction are comparable. When selecting the proper method, factors that should be considered are objectives of the experiment, time required, equipment available, and number of samples to be evaluated. When the objective is to determine the actual amount of pigment present, the WSB method should be used, with the spectrophotometer wavelength set at the peak absorption of the pigment under study (Sims and Lepage 1968). For durum semolina, the pigment is free lutein. When the objective is to distinguish the high from the low entries of large sample numbers (breeding populations), the Hunter Color Difference Meter or an equivalent reflectance meter is recommended. To use reflectance colorimeters, tempering and milling are the only sample modifications necessary, eliminating timely pigment extractions (including the use of potentially harmful organic solvents) and offering considerable savings in laboratory space and equipment. This method should result in an overall increase in efficiency. These data will also facilitate better communication between laboratories that use different means of semolina color evaluation.

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