

## INFLUENCE OF MATURITY ON PROPERTIES OF WESTERN RICES<sup>1</sup>

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### ABSTRACT

Chemical constituents and physical properties of rice change as the rice matures. Measurements made on Calrose, Caloro, and Colusa varieties revealed that maximum yields of head rice and minimum hot paste viscosity and water absorption occurred early in the maturation process, and then the trends reversed. Amylolytic activity was highest at the point of trend reversal. Reflectance properties shifted direction simultaneously. Rice harvested at mid-season showed the most yellow color, the lowest lightness and whiteness values, and the least chalkiness. Chlorophyll was detectable in the mid-harvest season, but disappeared several days later when translucency, as measured by light transmission, reached near maximum values. The late stages of maturity were characterized by loss of yellow color, increasing lightness and whiteness values, but little change in chalkiness. Other properties trended upward or downward to plateaus, or showed little change.

Physical and chemical properties characterizing varieties of rice have been reported extensively (5,10,11,16,19,20,21,22,24,32), but little information is available on changes that occur as a variety matures. In one-year studies of Caloro and Edith varieties in Arkansas and Shoemed variety in Louisiana, Smith *et al.* (26) found that maximum yields of whole kernels were obtained when harvest moisture was respectively, 26.4, 24.6, and 22.1%. In field trials of Blue Rose variety, head yields dropped from 73.4% at a harvest moisture of 21.7% to

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59.5% at 15% harvest moisture. These investigators observed that kernel weight increased as harvest moistures declined; they recorded a maximum of 2.30 g. per 100 kernels for shelled Caloro variety at a field moisture content of 24%, beyond which the determinations were not continued. Langfeld (18), in experiments with two rice varieties in Australia, demonstrated that yields of whole kernels in milled samples were progressively smaller as harvest moisture content decreased from initial values of 20.8 and 26.2%. Ten Have (27), in a study of four varieties in Surinam, found that least kernel breakage occurred when rice was harvested at field moisture contents between 19 and 21%.

Studies at the Western Regional Research Laboratory (28,31) suggested that properties other than milling yields might change during maturation. Therefore, a co-operative program was undertaken in 1957 and 1958 with the California Rice Experiment Station at Biggs, California, to study the effects of rice maturity on composition, milling yields, kernel weight, viscosity of heated flour pastes, gelatinization ranges, water uptake at different temperatures, chalkiness, and light transmission and reflectance.

### Materials and Methods

Fourteen samples each of Caloro and Calrose varieties grown at the Biggs Rice Station were harvested between September 26 and November 7, 1957. Field moisture contents were, respectively, 36.5 and 33.3%, at the start of sampling. Field samples of 4 to 10 lb. were taken at intervals of 2 to 5 days until the moisture of the grain at time of harvest was 15 to 17%.

In 1958, three varieties — Caloro, Colusa, and Calrose — were grown at the Biggs Station and harvested daily during maturation. The initial sample of Colusa, an early-maturing variety, was obtained on September 8 when harvest moisture content was 34.6%. First samples of Caloro and Calrose were obtained on September 10 when moisture contents were 58.0 and 53.9%, respectively. Samples were collected until moisture of field samples had dropped to about 13 to 15%.

All samples were shipped immediately (by bus) to the Western Regional Research Laboratory. They were dried at 80°F. in through-flow air dryers to about 13.5% moisture.

*Milling Tests.* Milling tests were performed as follows: Rough rice (1,000-g. samples) was cleaned in a dockage tester and dehulled in a McGill Sheller. Small samples of brown rice were reserved for kernel weight determination, and 800 g. of brown rice were charged to the No. 3 McGill sample mill. The 12-lb. weight was allowed to drop 2 in.,

after which the weights were removed and the rice given a 30-sec. polishing, for which only the weight carrier was used on the lever arm. Weight of total milled rice was recorded. The milled sample was split four times in a Boerner seed sampler to obtain a subsample weighing about 50 g., which was treated in a small disk separator to remove broken kernels. Weight of head rice from the subsample was used to calculate head rice yield.

*Kernel Weight.* Five 100-kernel samples of brown rice were weighed, and the average weight of 100 kernels reported as kernel weight. (Attempts to determine kernel weight of rough rice were unsatisfactory, because shriveled or undeveloped kernels inside hulls could not be easily detected and they seriously affected the accuracy of the determination.)

*Analyses of Milled Rice.* Published procedures were used to determine: moisture (30), total nitrogen (4; Sec. 22.11, modified), nonprotein nitrogen (3; Sec. 20.34), crude fat<sup>4</sup>, crude fiber<sup>4</sup>, ash<sup>4</sup>, total starch (7), and amylose (32). The method of Halick and Kelly (10) was used to determine water absorption at 77° and 82°C. Alpha- and beta-amylase were determined as mg. of maltose per g. of rice, according to Bernfeld's 3,5-dinitrosalicylic acid method (6).

*Amylograph Viscosity.* Milled rice was ground in a Wiley mill to pass a 40-mesh screen. The equivalent of 50 g. of 13.5%-moisture material was stirred 1.5 min. with 300 ml. of distilled water in a Waring Blendor, then transferred immediately to the bowl of a Brabender Amylograph with 150 ml. of water. Temperature was adjusted to 30°C., then raised 1.5°C. per min. to 95°C. Viscosity in Brabender units and temperature at the peak of the curve were recorded. At 95°C., temperature was held constant for 20 min., then lowered 1.5°C. per min. The final viscosity reading, designated as "set-back on cooling," was made at 50°C. Gelatinization range was computed from the point of departure of the pen from the base line on the kymograph chart to the intersection of the extended base line with the tangent to the rapidly rising viscosity curve.

Data of principal significance obtained from the amylograph curves are the gelatinization range, peak viscosity values, and the positive or negative differences between values for peak viscosity and set-back on cooling.

*Light Transmittance.* The percentage of light transmitted through rice was determined with an instrument similar to the Smut meter (15) but designed for sensitivity to the entire range of visible light. The sample was contained in a glass cell 10 × 10 cm. square and 1 cm.

<sup>4</sup>See reference 3: crude fat, Sec. 22.26; crude fiber, Sec. 13.20 (modified); ash, Sec. 22 (modified).

deep. Uniform packing of the cell was obtained by agitating it with a Foster vibrator while the cell was being filled. The photovolt model 511 photometer was adjusted to read nearly full scale when rice with high light-transmitting ability was used as a standard of reference. The zero point was reset before each reading and the reference sample reread after a series of measurements. A standard deviation of 2.4 was obtained for ten readings on each of twelve samples.

*Chalky Kernels.* Percent of nonchalky kernels was determined by hand-separating from 35 g. of rice the kernels estimated to be at least half chalky and then weighing the remainder.

*Reflectance Spectrophotometry.* The ability of samples to reflect light of each wave length in the visible spectrum was measured with a General Electric Recording Spectrophotometer. A measure of the relative chlorophyll content in a sample was derived by computing the ratio of percent reflectance at 720  $m\mu$  where absorption is similar for all samples to that at 676  $m\mu$  where it reaches a maximum in the presence of chlorophyll.

*Reflectance Color.* The color of the rice was measured with the Gardner Automatic Color Difference Meter. Color values are read from three scales,  $L$ ,  $a$ , and  $b$ , which form three dimensions of a rectangular color solid (25).

In practice, the color meter scales were first adjusted to read the values assigned to a standard<sup>5</sup> chosen for the similarity of its color to that of rice. Then each sample was poured into a glass container<sup>6</sup> and placed on the instrument over its aperture (2¼ in. diameter). Four readings at different orientations were obtained. The average value for each sample was computed.

The data can be usefully interpreted in terms descriptive of normal visual color experience: lightness, hue, and saturation. These are often represented by cylindrical co-ordinates in which the central axis denotes lightness and a radial vector indicates hue by its angle of orientation and saturation by its length. The hue angle and saturation index were computed from the equations:

$$\text{H.A.} = \arctan \frac{b}{a}, \quad \text{S.I.} = (a^2 + b^2)^{1/2}.$$

Croes has derived (8) and successfully used for wheat flour white-

<sup>5</sup>The ceramic standard, supplied by the instrument manufacturer, has the values:  $L = 78.2$ ;  $a = -1.7$ ;  $b = +22.9$ .

<sup>6</sup>A convenient sample container was constructed from a 1½-in. section of glass tubing with an inside diameter of about 3¼ in. on which was cemented a glass bottom cut from a plate 1/32 in. thick. Readings obtained through the nearly clear and colorless glass bottom were adjusted for the effect of the glass due to surface reflections by adding 4.0, determined by experiment, to the  $L$  scale value as read.

<sup>7</sup>Color terminology and measurements are discussed more fully by Hunter (13), Judd (17), and others (23).

ness measurements (9) a formula based on lightness and yellowness as the important determining factors for whiteness of many near-white organic substances, such as rice, which have practically constant dominant wave lengths near  $575 \text{ m}\mu$ . It seems justifiable, therefore, to use an appropriate lightness-yellowness formula to provide an index of whiteness, and for this study we used one proposed by Hunter (14):

$$W = 100 - [(100 - L)^2 + 10 b^2]^{1/2}$$

In this formula, the yellowness or  $b$  factor has about three times the weight of the lightness or  $L$  factor. The greenness or  $a$  factor, which changes by only small amounts, is ignored.

### Results and Discussion

The 1957 tests were mainly exploratory. Sampling started too late and was not frequent enough to establish reliable trends. Conclusions are therefore based principally on 1958 data.

*Chemical Composition.* Crude fat, fiber, and ash contents decreased as the harvest season progressed (Fig. 1). Total nitrogen fell within

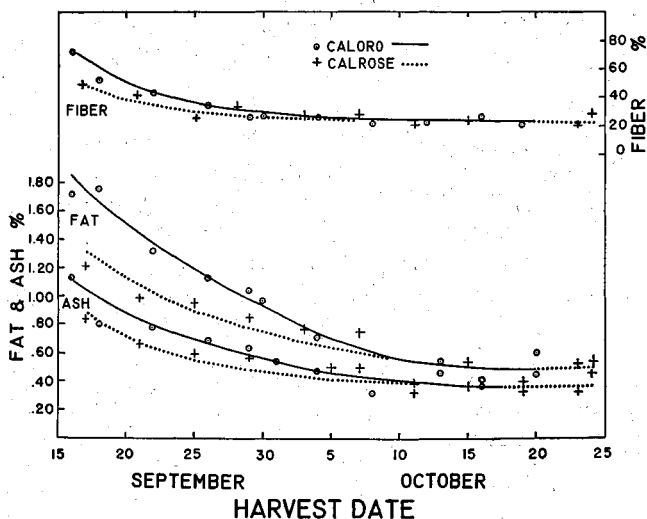


Fig. 1. Changes in percentage of fiber, fat, and ash.

narrow ranges (Table I). Salt-soluble nitrogen (representing albumins, globulins, and nonprotein nitrogen compounds) decreased (Fig. 2).

In the fully mature samples, the salt-soluble nitrogen was 9–11% of total nitrogen, and the nonprotein nitrogen less than 2%. About 1% of the total nitrogen in the first sample of 1957 Calrose rice was

TABLE I  
NITROGEN CONTENT OF RICE DURING MATURATION

VARIETY	YEAR	No. OF OBSERVATIONS	STANDARD DEVIATION	TOTAL N, % MEAN
Caloro	57	14	0.09	1.03
Calrose	57	14	0.07	0.82
Caloro	58	20	0.07	1.08
Calrose	58	22	0.07	1.09
Colusa	58	16	0.05	1.14

water-extractable amino nitrogen (probably in the form of amino acids), but as maturity proceeded, the value dropped to about 0.2%. Neither starch nor amylose concentration changed much during maturation (Table II). The small increases undoubtedly reflect decreases in other constituents.

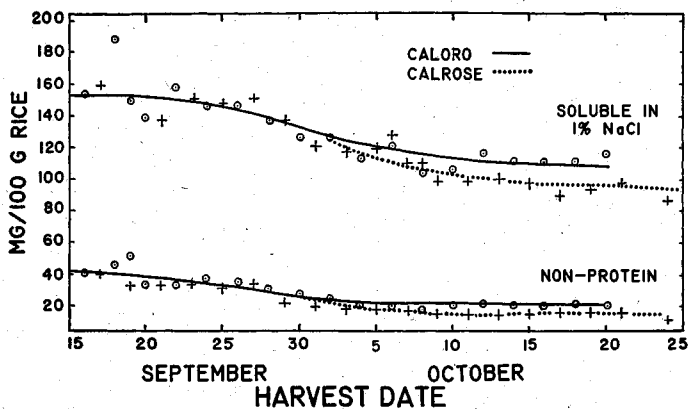


Fig. 2. Changes in percentage of saline-soluble and nonprotein nitrogen.

TABLE II  
STARCH AND AMYLOSE CONTENT OF RICE HARVESTED IN 1958

		CALORO	CALROSE	COLUSA
		%	%	%
Total starch	Initial	87.7	87.8	88.2
	Final	89.3	90.8	88.5
Amylose	Initial	17.5	15.1	17.6
	Final	18.7	16.1	18.4

*Physical Characteristics and Properties.* The first six or seven Caloro and Calrose samples taken in 1958 were so high in moisture content and so immature that the kernels disintegrated badly when the rice was dried and milled. Examination therefore began when field mois-

tures were 46.7% for Caloro and 47.7% for Calrose. These yielded, respectively, 49.0 and 49.6% total milled rice and 27.0 and 23.3% head rice. Milling yields of subsequent samples (Fig. 3) increased rapidly to maxima, after which the yields of head rice declined, while those of total milled rice remained constant. All Colusa samples (Fig. 4) and all 1957 Caloro and Calrose samples (Fig. 5) could be milled

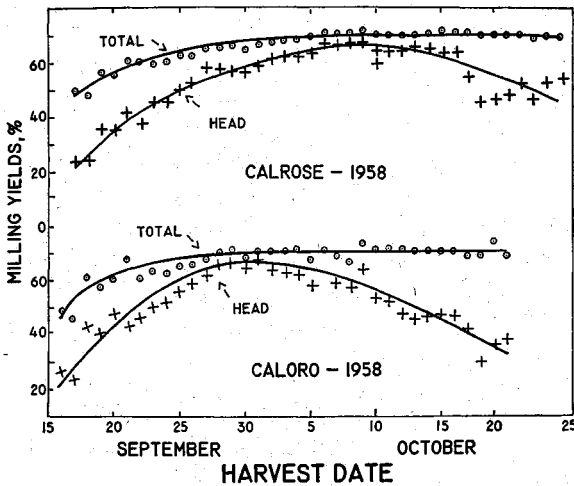


Fig. 3. Total and head rice yields, Calrose and Caloro, 1958.

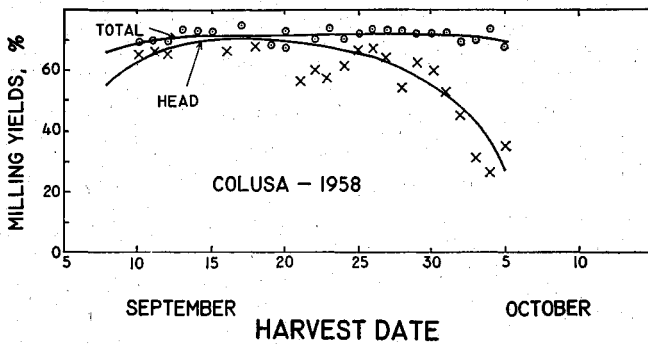


Fig. 4. Total and head rice yields, Colusa, 1958.

readily and reached maximum total and head rice yields in the same moisture range as the 1958 Caloro and Calrose series (Table III). Rate of field drying was considerably more rapid in 1958 than in 1957; this factor doubtless accounts for the more rapid loss of head yields in 1958 after maxima had been reached.

Kernel weight rose rapidly in the early stages of maturation and reached maxima only after field moisture had dropped to relatively low levels (Table IV).

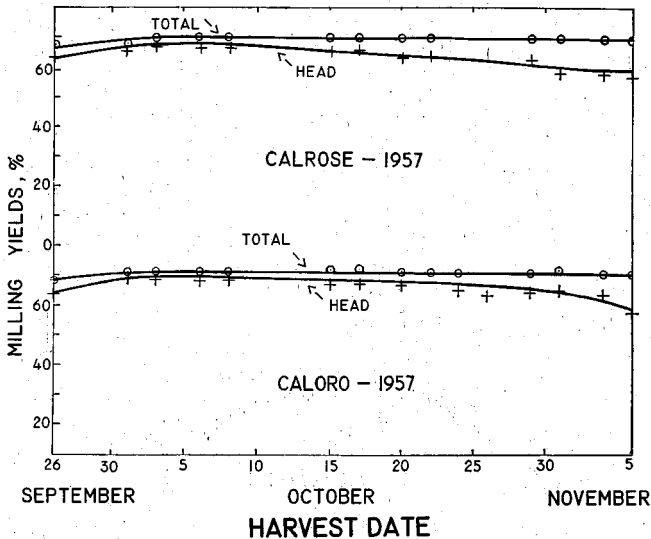


Fig. 5. Total and head rice yields, Calrose and Caloro, 1957.

TABLE III  
HARVEST MOISTURE AT TIME OF MAXIMUM OR MINIMUM  
VALUES OF RICE PROPERTIES

	CALORO		CALROSE		COLUSA
	1957	1958	1957	1958	1958
	%	%	%	%	%
Maximum milling yields					
Total	31.8	31.4 <sup>a</sup>	27.1 <sup>a</sup>	27.0 <sup>a</sup>	31.3
Head	28.3	27.6-29.5	27.0	21.6-27.0	21.7-28.2
Minimum peak viscosity	20.7	24.5-30.0	30.6	30.4	25.2
Minimum water uptake					
77°C.	...	30.0	...	30.4	...
82°C.	...	24.8-34.4	...	30.4	28.8
Minimum lightness	...	27.6	...	27.8	...
Minimum whiteness	...	30.4	...	27.8	...
Maximum nonchalky kernels, %	...	24.5 <sup>a</sup>	...	28.0 <sup>a</sup>	...

<sup>a</sup>Plateau reached at this point.



TABLE IV  
 KERNEL WEIGHT, GRAMS PER 100 KERNELS  
 (Moisture-free)

	1957		1958		
	Caloro	Calrose	Caloro	Calrose	Colusa
Maximum Harvest moisture, %	2.55	2.30	2.54	2.22	2.46
	15.4	15.9	13.4	18.2	19.2

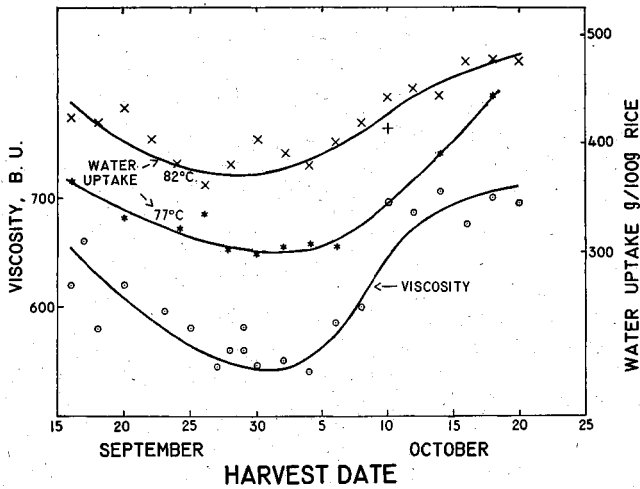


Fig. 6. Changes in peak viscosity (Brabender units) and water uptake with maturity, Caloro.

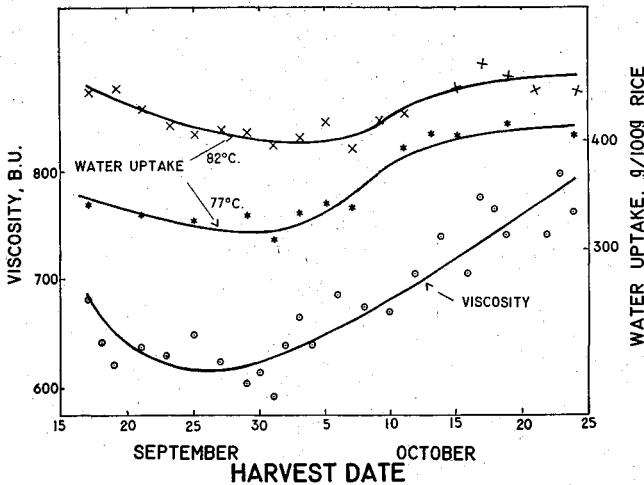


Fig. 7. Changes in peak viscosity (Brabender units) and water uptake with maturity, Calrose.

Water absorption at 77° and at 82°C. and peak Brabender viscosity values (Figs. 6 and 7) usually reached minima in the range of about 25 to 32% harvest moisture, then increased during the later part of the harvest season. (A reversal of trend was noted also in connection with head rice yield.)

The fall and rise of peak viscosity values are probably due to changes in amylase activity. Highest amylase values were found at points of lowest viscosity, as would be expected if this reasoning is correct. The reversal of peak hot-paste viscosity values and amylase activities during maturation, as found in this investigation, does not agree with the findings of Aimi and Murakami (2), who reported that amylase activity in the rice kernel reached a maximum 20 days after pollination and remained at this level to complete maturity. Ueda *et al.* (29), who investigated physicochemical properties of rice starch during maturation of the grain, found that the kinematic viscosity of starch pastes increased continually until the grain was fully mature. This observation suggests that amylase activity may affect the viscosity behavior of rice flour pastes.

Positive or negative differences between set-back on cooling and peak viscosity are being used currently for rice quality evaluation in rice-breeding work. Mature rice of varieties that cook dry and flaky and show little splitting and fraying of kernels in parboiling—the type most suitable for certain kinds of processing—has a set-back higher than peak viscosity (1). In the present study these viscosity differences changed either from positive to negative or from negative to more negative values as the season advanced.

Because most samples showed a gelatinization range of about 67° to 78°C. in the amylograph tests, it was concluded that stage of maturity has no influence on this property.

*Light Transmission and Reflectance.* Caloro and Calrose sampled early in 1958 yielded spectrophotometric reflectance curves showing absorption in the region of 676  $m\mu$  characteristic of chlorophyll. Reflectance ratios (720  $m\mu$ /676  $m\mu$ ) plotted against date of harvest show that chlorophyll content diminished as maturity progressed and was undetectable by this method after about October 10 (Fig. 8). Light transmittance (Fig. 9) gradually increased with the disappearance of chlorophyll and attained maximum values after chlorophyll was no longer detectable. Light transmittance and reflectance ratio were negatively correlated (Caloro,  $b = -0.935$ ; Calrose,  $b = -0.917$ ); the variation in chlorophyll content accounted for about 85% of the variation in light transmittance (12).

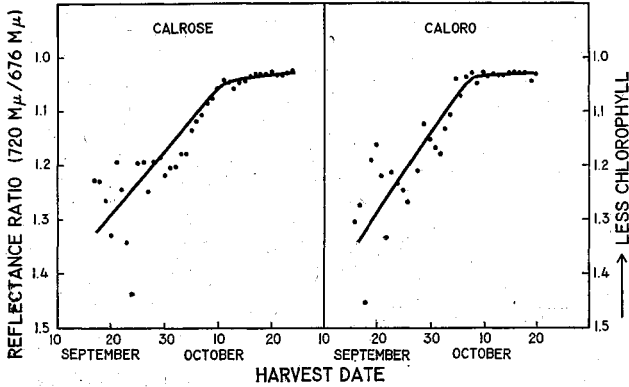


Fig. 8. Change in chlorophyll content during maturation. Ratio of reflectance readings from spectrophotometric curves at wave lengths of 720 and 676 m $\mu$ .

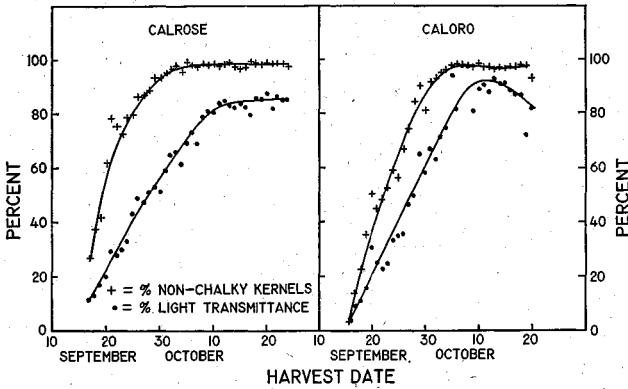


Fig. 9. Effect of maturity on light transmittance of rice and proportion of non-chalky grains.

The more immature rice samples contained a high percentage of chalky grains. The proportion of nonchalky kernels increased with increasing maturity (Fig. 9) until about October 3, which was several days before the harvest of samples with the highest light transmittance. Highly significant correlations between the above two factors are indicated by the correlation coefficients of 0.870 for Calrose and 0.851 for Caloro rice. Chalkiness, therefore, has a pronounced effect on the translucent appearance of these samples as measured by light transmittance.

The immature rice is light, pale, greenish yellow. Caloro is lighter and paler than Calrose. The effect of the chalkiness on color is small but is such as to increase the lightness of the less mature samples. During the first part of the harvest period, the color changed toward

a darker and stronger yellowish hue which is slightly less green. The change was accompanied by decreases in chlorophyll and chalky content of the rice. This trend continued until about October 3, then reversed abruptly, and samples harvested later were progressively whiter, lighter, paler yellow, and slightly greener than those of mid-harvest (Fig. 10). Saturation index, hue angle, and lightness also exhibited reversals at approximately the same time. The intermediate samples, which had the most yellow color and lowest lightness, showed

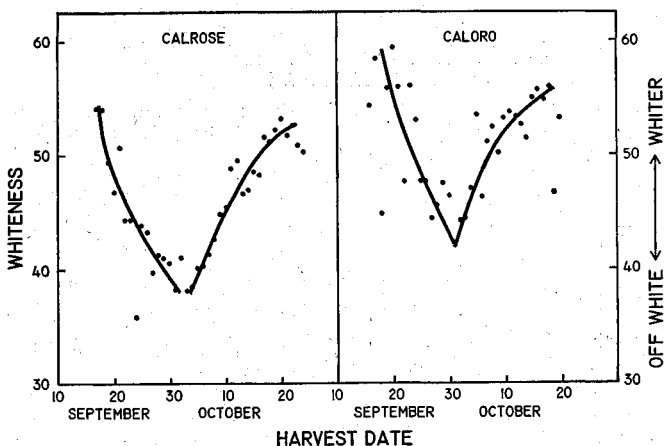


Fig. 10. Changes in whiteness of rice during maturation.

TABLE V  
CORRELATION OF PERCENT NONCHALKY KERNELS WITH COLOR FACTORS

COLOR FACTORS	CALROSE		CALORO	
	Before Color Reversal	After Color Reversal	Before Color Reversal	After Color Reversal
	%	%	%	%
Whiteness	-0.852	+0.399	-0.673	-0.040
Lightness	-0.861	+0.486	-0.790	-0.049
Hue angle	-0.449	+0.305	-0.645	+0.154
Saturation index	+0.779	+0.375	+0.562	+0.047

the lowest chalkiness content, 2 to 3%. Before color reversal, the percent nonchalky kernels is correlated with color factors; after color reversal the chalkiness content remained nearly constant and essentially no significant relationship with color was found (Table V). It seems reasonable to interpret these results as showing that before color reversal the kernel undergoes development climaxed by maximum color and minimum chalkiness. After color reversal the fully

mature kernel whitens by decreased selective absorption of light by the constituents, and by increased over-all reflectance.

The presence of surface lipids may affect color measurements. If the amount of lipids present depends on the efficiency of milling, color readings might not be realistic. Thirteen samples of the Caloro series were analyzed, and it was found that the variation of surface lipids was not more than 0.11% in each phase.

*General Comment.* These observations made during two crop years show that various properties of rice reach maximum or minimum values at about the same point of maturation (Table III), after which either a reversal of trends occurs or a plateau is reached. This is a major point of interest. Water absorption and peak hot-paste viscosity may be related because of enzyme activity, which, in a water system, conceivably affects both. On the other hand, it is not probable that an enzyme factor has a bearing on yield of head rice in a milling test.

The optimal time for harvesting the short-grain varieties to obtain maximum milling yields occurred when field moisture content was between about 25 and 32%. The fact that hot-paste viscosity and water absorption reach a low point at this field-moisture range is not important, because both improve after the rice is harvested, milled, and stored at ambient temperatures.

Reference to a company or product name does not imply approval or recommendation of the product by the U.S. Department of Agriculture to the exclusion of others that may be suitable.

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