

HYGROSCOPIC EQUILIBRIA OF ROUGH RICE¹

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ABSTRACT

Desorption and adsorption hygroscopic equilibria of rough rice of two nonwaxy (nonglutinous) and two waxy (glutinous) varieties at 27.5° and 32.5°C. were measured at relative humidities between 44 and 96.5%. At 75% r.h., rough rice had equilibrium moisture contents between 12.8 and 14.3% at 27.5°C. and between 12.6 and 14.2% at 32.5°C. Waxy rice had significantly higher moisture contents than nonwaxy rice at 75% and higher relative humidities for adsorption and at 84% and higher relative humidities for desorption.

Data on the hygroscopic equilibria of rough rice at temperatures above 25°C. are few (1,2), although most of the rice in Southeast Asia is harvested, conditioned, and stored at these temperatures. Studies show that, aside from temperature, moisture content critically affects the storage quality of rough rice (3,4). The current interest in unheated-air drying (5) raises the question as to whether this practice is feasible in humid areas of the tropics. Because such data are important in decisions relating to conditioning and storage, the adsorption-

¹Manuscript received June 13, 1963. From The International Rice Research Institute, Los Baños, Laguna, Philippines. Mailing address: Manila Hotel, Manila, Philippines. Issued as I.R.R.I. Journal Series No. 7.

desorption hygroscopic equilibria at 27.5° and 32.5°C., and at relative humidities (r.h.) between 44 and 96.5%, were measured for rough rice of four varieties of different starch composition.

Materials and Methods

The two nonwaxy (nonglutinous) varieties — Peta and Taichung 65 — and the two waxy (glutinous) varieties — Malagkit Sungsong and Taichung Glutinous 46 — were grown in the same plot at the Institute farm during the 1962 wet season. They were harvested at approximately the same time. The cleaned rough rice was artificially dried with a current of heated air (about 40°C.) to a moisture content of about 13%.

The dry weight of the starting and final samples was determined by the vacuum oven method (6).

A John Bass DB/TA Dry Box with interlock served as the chamber where the rough rice samples were equilibrated at various relative atmospheric humidities and weighed. Saturated solutions of reagent-grade salts provided the desired series of relative humidities (7). Relative humidity below 90% was checked with a Serdex HGS-HY-1 Laboratory Standard Model Hygrometer, accurate to $\pm 1.5\%$, and with a wet-and-dry-bulb thermometer above 90%. A centrifugal circulating fan connected to a variable transformer continuously blew air above the salt solution and over the surface of the samples, except during weighing. Weighings were made with a torsion balance accurate to ± 0.002 g. Thermostatic temperature control at either $27.5^\circ \pm 0.5^\circ\text{C.}$ or $32.5^\circ \pm 1.0^\circ\text{C.}$ was achieved with a heating tape attached to a Fenwal thermostwitch.

Three 10- to 12-g. samples of each variety were placed in cylindrical stainless-steel, 16-mesh-wire baskets, such that the seed layer was not more than 1 cm. thick. These were then soaked for 10 min. in distilled water and were equilibrated in the humidity chamber with a saturated potassium sulfate solution with an excess of the salt in a large crystallizing dish. The equilibrium relative humidity was 96.5% at both 27.5° and 32.5°C. After equilibration at 96.5% r.h., the potassium sulfate solution was replaced with the saturated solution of another salt. The salts successively introduced were potassium chloride, sodium chloride, sodium nitrite, and potassium carbonate. These correspond to corrected relative humidities of 84, 75, 64, and 44.5% at 27.5°C., and to 84, 75, 63.5, and 43.5%, respectively, at 32.5°C. A change of relative humidity treatment was made whenever the differences between the mean moisture contents of successive weighings of the sam-

ples at intervals of 10 hr. or longer were less than 0.1%. Moisture contents were expressed on the wet basis.

To prepare the samples for adsorption studies after the completion of the desorption isotherm, the samples in the dry box were dried with anhydrous calcium sulfate to moisture contents of about 5%. Then they were equilibrated in succession to the salt solutions mentioned above, in reverse order or in the order of increasing relative humidity up to 84%.

Amylose was determined by the method of Williams and co-workers (8). Crude protein ($N \times 5.95$) and starch were analyzed by standard methods (6). Amylopectin content was calculated by subtracting amylose content from starch, and starch composition was expressed as the amylose:amylopectin ratio.

Results and Discussion

The hygroscopic equilibria data of the four varieties (Table I) were comparable to reported data at 25° to 34.4°C., summarized in Table II. At 75% r.h. and below, the moisture contents of rice were lower at 32.5°C. than at 27.5°C. for all varieties. The observed moisture contents at 96.5% r.h. of 19.6 to 22.3% are lower than the 25% moisture content reported (9) for freshly combined rough rice. The equilibrium moisture contents of rough rice at ambient temperatures are lower than those of brown rice (1,10), polished rice (11), and rice starch (12).

At 75% relative humidity, rough rice moisture contents ranged from 12.8 to 14.3% at 27.5°C. and from 12.6 to 14.2% at 32.5°C. These data are important, as others (3,4) have observed that rough rice stored at moisture contents more than 14%, or at higher than 75% r.h., exhibited accelerated loss of viability and increased mold growth at temperatures between 60° and 100°F. (15.6° to 37.8°C.). The rate of mold growth was greater at higher temperatures, and sourness developed at high moisture contents and temperatures (4).

This study produced differences in the order of 0.9% and higher between final adsorption and desorption moisture contents of rough rice at 75% r.h. and lower. These differences reflect hysteresis, although they may in part result from insufficient time of equilibration. Such differences are lower than the hysteresis noted by Breese (13) for a short-grain rough rice which exceeded 1.5% from 50 to 70% r.h.

The waxy rice varieties were observed to be more sensitive to humidity changes and achieved final moisture values faster than non-waxy rice in the relative humidity range studied. For all varieties, constant weight was achieved within 2 to 4 days, the longer periods being required at 84% r.h. and higher. Babbitt (14) reported an adsorption

equilibration time for dried wheat of 2 to 4 days in a similar chamber at 75% r.h. The relatively rapid rate at which final moisture content of rice was attained in the humidity chamber with fan is one reason why no mold growth was noted in the humid atmospheres. The mean change in dry matter during the course of the desorption-adsorption cycle covering a period of 7 weeks was not more than 0.1%.

The use of bigger samples resulted in moisture gradients in the samples, since air circulation between the grains was impaired.

TABLE I
CHEMICAL COMPOSITION OF ROUGH RICE AND ITS EQUILIBRIUM MOISTURE CONTENTS^a
ATTAINED BY DESORPTION AND ADSORPTION AT DIFFERENT RELATIVE
HUMIDITIES AT 27.5° AND 32.5°C.

RELATIVE HUMIDITY	NONWAXY		WAXY		STANDARD ERROR	
	Peta (<i>indica</i>)	Taichung 65 (<i>japonica</i>)	Taichung Glu. 46 (<i>japonica</i>)	Malagkit Sungsong (<i>indica</i>)	Variety (d.f. = 3)	Waxy vs. Nonwaxy Variety (d.f. = 1) ^b
%	Desorption at 27.5 ± 0.5°C.					
44.5	10.2	10.2	10.2	10.3	0.06	0.04
64	12.4	12.2	12.4	12.5	0.08	0.05
75	14.0	14.1	14.1	14.3	0.08	0.06
84	15.8	15.8	16.4	16.3	0.13*	0.09**
96.5	19.6	20.4	21.7	22.3	0.18**	0.13**
	Adsorption at 27.5 ± 0.5°C.					
44.5	8.7	8.8	8.8	8.9	0.10	0.07
64	11.3	11.3	11.3	11.4	0.07	0.05
75	12.8	13.0	13.2	13.3	0.07**	0.05**
84	14.8	15.0	15.6	15.4	0.09**	0.06**
	Desorption at 32.5 ± 1.0°C.					
43.5	9.7	9.7	9.7	9.6	0.08	0.05
63	12.1	11.8	11.9	12.0	0.13	0.09
75	13.9	13.8	13.9	14.2	0.18	0.12
84	15.4	15.4	16.5	16.2	0.10**	0.07**
96.5	19.7	20.3	21.8	22.3	0.27**	0.19**
	Adsorption at 32.5 ± 1.0°C.					
43.5	8.3	8.3	8.3	8.4	0.10	0.07
63	10.7	10.8	10.9	10.9	0.10	0.07
75	12.6	12.9	13.0	13.1	0.07**	0.05**
84	14.6	14.9	15.4	15.3	0.08**	0.06**
Chemical composition						
Amylose:						
amylopectin ratio	0.47	0.34	0.08	0.05		
Protein, % dry basis	10.66	9.22	9.48	11.88		

^a % wet-weight basis for four varieties. Mean of three replications.

^b Based on six replications.

The data indicate that in areas with relative humidity higher than 75%, drying of rough rice with unheated air may be used only on wet grain. Final supplemental heating is required; otherwise, the equilibrium moisture contents of the rice will be more than 14%.

Differences in moisture contents were evident among the varieties at 84% r.h. and higher for desorption and at 75% r.h. and higher for adsorption (Table I). By partitioning the three degrees of freedom (d.f.) of the varietal comparison into individual d.f. (one each for waxy against nonwaxy, between waxy and between nonwaxy varieties), it was noted that the varietal differences evidenced were mainly between waxy and nonwaxy varieties. The waxy rice samples, Malagkit Sungsong and Taichung Glu. 46, the starch of which is essentially amylopectin, had significantly higher moisture contents at the higher relative humidities than the nonwaxy varieties, Peta and Taichung 65. The differences in moisture content among the waxy rice samples were not significant. Taichung 65 had significantly higher moisture contents than Peta only at 75% r.h. for adsorption at 27.5° and 32.5°C. and at 96.5% r.h. at 27.5°C. Peta and Taichung 65 had amylose:amylopectin ratios of 0.47 and 0.34, respectively. Amylopectin is more hygroscopic than amylose in fractionated starches (15), and the data indicate that this also may be true for native starch granules. In fact, *japonica* rice has been reported (16) to be generally higher in moisture content than *indica* rice at high relative humidities.

The protein contents of the samples were not correlated with

TABLE II
EQUILIBRIUM MOISTURE CONTENTS^a OF ROUGH RICE AT VARIOUS RELATIVE HUMIDITIES
AT TEMPERATURES BETWEEN 25° AND 34.4°C., AS REPORTED BY
VARIOUS INVESTIGATORS

INVESTIGATORS ^b	TEMPERATURE	RELATIVE HUMIDITY					
		40%	50%	60%	70%	80%	90%
	°C.						
Desorption							
Karon and Adams (11)	25	9.2	10.5	11.7	13.2	14.8	...
Breese (13)	25	9.4	10.8	12.2	13.4	14.8	16.7
Hogan and Karon (2)	26.7		10.2	11.7	13.2	14.7	16.9
Table I	27.5	9.8	10.8	11.9	13.3	15.1	17.8
Gerzhoi and Samochetov (1)	30	10.00	10.88	11.93	13.12	14.66	17.13
Table I	32.5	9.3	10.4	11.5	13.0	14.8	17.6
Hogan and Karon (2)	34.4		9.4	11.3	12.9	14.6	16.8
Adsorption							
Breese (13)	25	7.9	9.2	10.4	11.8	13.6	16.6
Karon and Adams (11)	25				12.6	13.8	17.0
Table I	27.5	8.3	9.5	10.7	12.2	14.2	17.1
Table I	32.5	7.9	9.1	10.4	12.0	14.0	17.0

^a Mean values, % wet-weight basis.

^b Reference numbers in parentheses.

varietal differences in moisture content. Coleman and Fellows (17) observed a similar lack of correlation between protein and moisture contents in wheat.

Since these varieties were grown simultaneously, environmental influences in these samples were minimized. However, the reported² differences in equilibrium moisture content of samples of the same variety grown during different years when stored under identical conditions may be attributed to environmental influences. Temperature during ripening of rice has been reported to affect the chemical composition and physical properties of the starch (18,19,20). Hofstee (21) noted that the equilibrium moisture content and the type of X-ray diagram of various native starches are closely correlated. Nikuni and co-workers (19,20) found that season of planting of Japanese rice affected the X-ray pattern of rice kernel starch. These workers (22) earlier demonstrated that temperature plays a dominant role in determining the type of X-ray pattern of synthesized starch in germinating soybean seeds.

Presumably, the complex influence of variety and environment on the physicochemical properties of the starch granules is reflected in the equilibrium moisture content of rough rice.

Acknowledgments

The assistance of Miss Remedios Santiago, Mrs. Gloria B. Cagampang, and J. Lugal in conducting the work is acknowledged. Seeds of Taichung 65 and Taichung Glu. 46 were obtained from the Plant Industry Division, Joint Commission on Rural Reconstruction, Taiwan.

Literature Cited

1. GERZHOI, A. P., and SAMOCHETOV, V. F. Grain drying and grain driers (3rd rev. enl. ed.), ed. by A. S. Ginzburg. Khleboizdat: Moscow (1958). Translation by B. Shapiro, pp. 36-38. Israel Program for Scientific Translations: Jerusalem (1960).
2. HOGAN, J. T., and KARON, M. L. Hygroscopic equilibria of rough rice at elevated temperatures. *J. Agr. Food Chem.* **3**: 855-860 (1955).
3. DEL PRADO, F. A., and CHRISTENSEN, C. M. Grain storage studies. XII. The fungus flora of stored rice seed. *Cereal Chem.* **29**: 456-462 (1952).
4. HOUSTON, D. F., STRAKA, R. P., HUNTER, I. R., ROBERTS, R. L., and KESTER, E. B. Changes in rough rice of different moisture content during storage at controlled temperatures. *Cereal Chem.* **34**: 444-456 (1957).
5. U.S. DEPARTMENT OF AGRICULTURE. Research on conditioning and storage of rough and milled rice. A review through 1958, ed. by W. C. Dachtler. *Agr. Research Service Rept.* **20-7** (1959).
6. ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS. Official methods of analysis (9th ed.); Secs. 2.036, 13.003, and 22.045. The Association: Washington, D.C. (1960).
7. SPENCER, H. M. Laboratory methods for maintaining constant humidity. *In International Critical Tables*, vol. 1, pp. 67-68. McGraw-Hill: New York (1926).
8. WILLIAMS, VIRGINIA R., WU, WEI-TING, TSAI, HSU YING, and BATES, H. G. Varietal differences in amylose content of rice starch. *J. Agr. Food Chem.* **6**: 47-48 (1958).

²Unpublished data, H. A. Kramer; cited in ref. 5.

9. McFARLANE, V. H., HOGAN, J. T., and McLEMORE, T. A. Effects of heat treatment on the viability of rice. A report of research and a literature survey. U.S. Dept. Agr. Tech. Bull. 1129 (1955).
10. HOUSTON, D. F. Hygroscopic equilibrium of brown rice. *Cereal Chem.* **29**: 71-76 (1952).
11. KARON, M. L., and ADAMS, M. E. Hygroscopic equilibrium of rice and rice fractions. *Cereal Chem.* **26**: 1-12 (1949).
12. SCHIERBAUM, F. Die Hydratation der Stärke. II. Tensiometrische Untersuchungen der Ad- und Desorption von Wasser durch Stärke. *Stärke* **12**: 257-265 (1960).
13. BREESE, M. H. Hysteresis in the hygroscopic equilibria of rough rice at 25°C. *Cereal Chem.* **32**: 481-487 (1955).
14. BABBITT, J. D. Observations on the adsorption of water vapour by wheat. *Can. J. Research* **27F**: 55-72 (1949).
15. WOLFF, I. A., DAVIS, H. A., CLUSKEY, J. E., GUNDRUM, L. J., and RIST, C. E. Preparation of films from amylose. *Ind. Eng. Chem.* **43**: 915-919 (1951).
16. ISO, E. Rice and crops in its rotation in subtropical zones, p. 136. Japan FAO Association: Tokyo (1954).
17. COLEMAN, D. A., and FELLOWS, H. C. Hygroscopic moisture of cereal grains and flaxseed exposed to atmospheres of different relative humidity. *Cereal Chem.* **2**: 275-287 (1925).
18. HALICK, J. V. Effect of temperature during ripening on quality characteristics of rice. Proc. 9th Meeting Rice Tech. Working Group, 1960, p. 14 (1961).
19. SUZUKI, H., HIZUKURI, S., and NIKUNI, Z. Studies on the rices cultured by the early and late season growing. II. Physical and chemical properties of glutinous rice and their starches. *Nippon Nogeikagaku Kaishi* **37**: 63-66 (1963); *in Agr. Biol. Chem. (Tokyo)* **27**: A9 (1963).
20. SUZUKI, H., MORI, T., DOI, K., and NIKUNI, Z. Studies on the rices cultured by the early and late season rice growing. III. On the X-ray diffraction and blue color of non-glutinous rice starches. *Nippon Nogeikagaku Kaishi* **37**: 112-115 (1963); *in Agr. Biol. Chem. (Tokyo)* **27**: A13 (1963). [English translation by Takehiko Yoshida; typescript.]
21. HOFSTEE, J. Spezielle Rheologie der Stärke. *Stärke* **14**: 318-324 (1962).
22. HIZUKURI, S., FUJII, MICHIKO, and NIKUNI, Z. Effect of temperature during germination on the crystalline type of starch in soybean seedlings. *Nature* **192**: 239-240 (1961).

