# Varietal Differences in Properties of Extrusion-cooked Rice Flour

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#### ABSTRACT

Ten milled rices differing in starch properties were extrusion cooked through a twin screw extruder at 15% moisture and 120, 135 and 150°C. Instron hardness values of extrudate were lowest at 150°C. Extrusion cooking drastically reduced viscosity in 0.2N KOH and in water without any decrease in amylose content and with little increase in reducing sugars. Spherical protein bodies were intact in the protein masses

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uniformly dispersed in the gelatinized starch matrix. Amylose content and gel consistency of raw rice flour were significantly correlated with expansion ratio only at  $135 \,^{\circ}$ C, water absorption and solubility indexes at all temperatures and with gel viscosity of extrudates. Only gel consistency of raw flour correlated with cold paste viscosity of extrudate. Final gelatinization temperature of starch was not significantly correlated with any extrudate property.

### INTRODUCTION

There has been considerable interest in high-temperature short-time (HTST) extrusion cooking in food processing because of the flexibility and ease of obtaining products which require minimum drying (Wilson & Tribelhorn, 1979; Linko *et al.*, 1981). The process has been recommended for the production of ready-to-eat weaning foods in developing countries (Wilson & Tribelhorn, 1979).

Although HTST extrusion cooking technology has advanced considerably, little is known about its effect on properties of the starch and protein (Linko *et al.*, 1981). Recent results suggest that some starch degradation occurs with negligible production of reducing sugars (Mercier & Feillet, 1975; Launay & Lisch, 1983; Colonna & Mercier, 1983; Colonna *et al.*, 1984; Davidson *et al.*, 1984). Extrusion cooking reduced rice protein solubility with no change in the electrophoretic pattern of its protein subunits (Noguchi *et al.*, 1982).

Using a mixture of waxy and high-amylose (61%) corn starches, Mercier & Feillet (1975) reported decreased expansion at 225 °C with increasing amylose content of 22%-moisture flours, in contrast to results from different corn starch samples which showed increased expansion between 1% and 52% amylose. High-amylose corn starch, however, differs considerably from waxy and normal corn starch in granule morphology and gelatinization temperature (Kramer *et al.*, 1958).

Extrusion cooking of rice flour has been reported in the US since 1969 (Mottern *et al.*, 1969; Spadaro *et al.*, 1971). Because of the increasing use of rice in extrusion-cooked weaning foods in Southeast Asia (Payumo *et al.*, 1979; Bhumiratana, 1983) and the wide range in amylose content and gelatinization temperature in rice starch (Juliano, 1979), this co-operative study was undertaken to determine the possible effect of differing starch properties on the properties of extrusion-cooked rice flour.

## MATERIALS AND METHODS

#### Materials

Ten aged rough rice samples, differing widely in starch properties, obtained from the IRRI farm in 1982, were dehulled with a Satake THU-35 testing dehusker, milled with a Satake TM-05 testing mill and stored at 4°C until use. The larger samples of IR43 and IR45 were dehulled and milled with a Satake SB-2B one-pass pearler. The milled rice was ground into flour with a disc pin mill and stored at 4°C until use. Crude  $\beta$ -amylase (12  $\mu$ g per milligram of solids) and pullulanase (39  $\mu$ g per milligram of protein) were from Sigma Chemical Co.

### Methods

Extrusion cooking was carried out in a Werner & Pfleiderer twin-screw extruder Model 37 (480 mm barrel length, 37.7 mm barrel diameter, 37.4 mm screw diameter, 4 mm diameter die hole, 140 rpm screw speed and 45 bars barrel pressure). The rice flour samples were blended with water up to the desired moisture content in a Hobart mixer. The premoistened sample was then uniformly fed into the extruder with a feeder at 160 g/min. This optimum feed rate was lower than the manufacturer's rate of 250 g/min. In preliminary trials to determine optimum extrusion moisture, IR43 (low amylose) and IR45 (high amylose) batters were extruded at 25%, 20% and 15% moisture wet basis and at temperatures of 120, 135 and 150°C. Based on the preliminary results, the other rices were extruded at 15% moisture only.

Extruded samples were stored in sealed polyethylene bags at 4 °C until analysis. Extrudates were analysed for mean of 10 diameter readings and expansion degree was calculated from the ratio of diameter of extrudate and die diameter (4 mm). Hardness was measured with an Instron Model 1140 Food Tester with the OTMS bite tester test cell with a 6.0 mm thick  $\times$  5.1 cm wide rounded-edge upper blade and corresponding to a 6.5 mm wide clearance below (lower adapter without the cutting blade). The extrudate was pressed at a crosshead speed of 10 cm/min and a chart speed of 20 cm/min until crushed. Hardness was the maximum pressure, in kilograms, averaged for ten determinations using the 50-kg load cell.

Whole milled rice was analyzed for alkali spreading value (Little *et al.*, 1958) and rice flour was analyzed for moisture by loss of weight 2 h at

135 °C (AACC, 1983), Kjeldahl protein (N × 5.95) (Juliano & Pascual, 1980), amylose (Juliano *et al.*, 1981), final gelatinization temperature (GT, Ignacio & Juliano, 1968), gel consistency (Cagampang *et al.*, 1973), water absorption index and water solubility index (Anderson *et al.*, 1969), and degree of gelatinization of starch by the  $\beta$ -amylase-pullulanase method of Kainuma *et al.* (1981), modified by dispersing the sample in 0.1N NaOH, and neutralized with 0.1N acetic acid instead of direct dispersion in sodium acetate buffer. Similarly, extrudates were analyzed for amylose, water absorption index and water solubility index, and degree of gelatinization of starch. A 1-ml portion of the gel in the consistency test was measured for gel viscosity in a Wells-Brookfield 1.565° cone-plate microviscometer RVT-C/P at 2.5 rpm at 25°C.

Duplicate flour samples (60 mesh, 2.00 g) were suspended in 30 ml water in a tared centrifuge tube, kept for 30 min in a 50 °C water bath, cooled and centrifuged at  $390 \times g$  for 10 min (Anderson *et al.*, 1969). The supernatant was decanted carefully, transferred to a tared dish and dried to constant weight to measure solubilized solids. The water absorbed was calculated from the weight of the gel.

Non-starch lipids were extracted with 5 vol/wt of chloroform:methanol (2:1 v/v) for  $2 \times 4 h$  and with water-saturated butanol for  $2 \times 30 min$ , including 10 minutes' centrifugation. The partially defatted milled rice flour was extracted for starch lipids with refluxing water-saturated butanol (37:63 v/v) at 92 °C thrice for 2 h each (Maniñgat & Juliano, 1980). The lipid extracts were evaporated to dryness, dissolved in chloroform, dried with anhydrous sodium sulfate, filtered, evaporated to dryness and weighed.

Cold paste relative viscosity was determined with a Brookfield digital viscometer Model LVTD using spindle No. 34 at 12 rpm with the small sample adapter at 29 °C. Raw flour was prepared as 6% paste (0.84 g/13.16 ml water) by cooking in a boiling water bath for 10 min with stirring by a glass rod, cooled 1 h and stirred before 10 ml was transferred into the viscometer chamber. Extrudate was prepared as 12% paste (1.68 g/12.32 ml water) at room temperature by suspending for 30 min, with stirring using a glass rod. Reading was converted to centipoise by multiplying by the factor 50.

Samples of extrudates were sealed in polyethylene bags and sent by air to the US Grain Marketing Research Laboratory, Manhattan, Kansas, for photomicrography. Samples for light microscopy were fixed in glutaraldehyde and stained with Coomassie Brilliant Blue (CBB) R-250 for protein or with periodic acid-Schiff's reagent (PAS) for carbohydrate (Bechtel & Pomeranz, 1978). Sections of air-dried extrudate could not be obtained. Samples for transmission electron microscopy were prepared by previously described methods (Bechtel & Pomeranz, 1978; Bechtel & Juliano, 1980).

# **RESULTS AND DISCUSSION**

## **Raw flour properties**

The ten milled rice flour samples consisted of one waxy rice (IR29), three low amylose (12-20%) rices (IR24 and two samples of IR43), two intermediate amylose (20-25%) rices (IR48 and IR29708-41-2) and four high amylose (>25%) rices (IR32, IR36, IR42 and IR45). They were selected mainly for differences in starch amylose content and GT. No high GT sample could be included. The ten samples also showed a wide range in gel consistency and viscosity, degree of gelatinization, water absorption and solubility indexes, cooked paste viscosity and starch and non-starch lipids (Table 1).

Among the samples, amylose content correlated with gel viscosity, water-solubility index (WSI) and starch and non-starch lipids. Starch final GT correlated significantly with alkali spreading value, gel viscosity, degree of gelatinization and non-starch lipids. Gel consistency did not correlate significantly with any milled rice property, not even gel viscosity, because IR29 (waxy) had the highest gel viscosity of 1299 cps, followed by IR42 with 1130 cps. The lowest gel viscosity was obtained for the high amylose IR32 with soft gel consistency (92 mm). In contrast to gel viscosity in 0.2N KOH, the relative viscosity of a cooked and cooled 6% paste from raw flour was highest for low-amylose rices (4380–4890 cps) and lowest for IR32 (1500 cps). Waxy rice (IR29) had a paste viscosity of 3280 cps.

### **Optimum moisture for extrusion cooking**

Preliminary runs using IR43 (16 % amylose, GT 65.5 °C) and IR45 (25 % amylose, GT 72 °C) showed maximum expansion ratio and least Instron

Property	Values		Correlation coefficients <sup>b</sup> with		
	Range	Mean	Amylose	GT	Gel consistency
Moisture (% wet basis)	10.8-11.9	11.4			
Protein (% wet basis)	7.2-10.4	8.6	-0.09	0.33	-0.11
Amylose (% dry basis)	1.5-31.4	21.5	1.00	0.49	-0.56
Alkali spreading value	3.87.0	6.1	-0.44	-0.93**	0.23
Final gelatinization					
temperature (°C)	62.0-72.0	67.4	0.49	1.00	-0.17
Gel consistency (mm)	36—96	50	-0.56	-0.17	1.00
Gel viscosity (cps)	547—1299	837	-0.66*	-0.82**	0.18
Degree of gelatinization (%	) 1832	24	-0.55	-0.63*	0.24
Water absorption index (g/)	g) $1.4 - 2.0$	1.7	0.39	-0.29	-0.55
Water solubility index (%)	1.3-7.7	2.4	-0.99**	-0.44	0.57
Cooked 6% paste					
viscosity (cps)	1500-4890	3570	-0.44	-0.11	-0.01
Starch lipids (%)	0.28-1.16	0.90	0.91**	0.36	-0.53
Non-starch lipids (%)	0.60—1.15	0.86	-0.76*	-0.77**	0.61

# TABLE 1 Range of, and Mean Properties of, Ten Milled Rice Flours<sup>a</sup> Used for Extrusion Cooking and their Correlation with Starch Properties

<sup>a</sup> IR29, IR43 (two samples), IR24, IR29708-41-2, IR48, IR45, IR36, IR42 and IR32. <sup>b</sup> Significant r = 0.632 at the 5% level (\*) and 0.765 at the 1% level (\*\*).

hardness with 15% moisture batters than with 20% and 25% moisture batters (Table 2). Despite the greater expansion ratio, the extrudates of 20% moisture batters were harder than 25% moisture extrudates. Moisture content of extrudate of 15% moisture batters was close to 10% for both rices. Gelatinization was essentially complete for extrudates of both rices. At 20% and 15% moisture, extrudates of IR43 had lower water absorption index (WAI) and higher WSI than extrudates of IR45 rice. Amylose content was not decreased by extrusion in both rices. Maximum absorbance of the starch-iodine complex was at  $600 \pm 20$  nm.

Reducing sugar content and total sugars extracted with hot 80% ethanol were 0.02-0.04% and 0.14-0.21%, respectively, for IR43 extrudates and 0.02-0.05% and 0.13-0.21% for IR45 extrudates. Cold water-soluble reducing sugars were 0.04-0.06% for IR43 extrudates and 0.04-0.07% for IR45 extrudates. The results confirmed the reported lack of increase in free sugars during extrusion cooking (Mercier & Feillet,

Mean extrudate property <sup>a</sup>	IR43 (16% amylose)			IR45 (25% amylose)		
	25 % <sup>b</sup>	20 %*	15%	25 %*	20 % <sup>b</sup>	15%
Expansion ratio	1.8	2.2	3.4	1.5	2.1	3.1
Instron hardness (kg)	8.3	11.0	7.1	14.4	28.7	<b>8</b> ·7
Moisture content ( $\frac{9}{6}$ wet basis)	11.2	11.4	9·4	9.5	9.8	10.0
Degree of gelatinization (%)	99	95	101	104	101	100
Water absorption index (g/g)	6.2	4.3	<b>4</b> ·2	6.8	7.1	6.6
Water solubility index (%)	27	33	40	24	27	25
Amylose content (% dry basis)	17.4	17.2	16.9	26.5	26.7	<b>26</b> ·7
Total water-soluble carbohydrates						
(% glucose)	39	47	56	10	15	30

TABLE 2Effect of Batter Moisture Content on Mean Properties of Extrudates of IR43 and IR45Rice Flours at 120, 135 and 150°C

<sup>a</sup> Mean of extrudate at 120, 135 and 150 °C.

<sup>b</sup> Batter moisture content.

1975). By contrast, mean total water-soluble carbohydrates for extrudates increased with decreasing moisture content of batter but were higher for IR43 than for IR45 (Table 2).

# Light and electron microscopic examination

Scanning electron micrographs of the fractured surface of extrudates at 150 °C showed large cells or cavities except for the smaller cavities in the IR43 extruded at 25 % moisture. The extrudate had a smooth surface with starch granules and protein bodies not distinguishable (Fig. 1). Only IR32 showed many air cells within the extrudate layer. Light microscopy showed that the protein was in the form of masses, mostly uniformly distributed throughout the gelatinized starch matrix (Fig. 2). The number of protein masses tended to follow the protein content of the rice sample. Sections of waxy IR29 were really artifacts because much of the starch leached out during processing. Transmission electron microscopy revealed that the integrity of crystalline protein bodies was destroyed. By contrast, the spherical protein bodies were present in the protein masses but were distorted in some of the samples, but mostly intact and retaining much of their original appearance (Fig. 3). Endosperm cell walls were



Fig. 1. Scanning electron micrograph of cross-section surface of IR45 extrudate at 150 °C and 15% moisture.

found in the protein masses. Cytoplasmic remnant structures (endoplasmic reticulum, ribosomes and membranes) were also destroyed.

# Varietal differences in extrudate properties from 15% moisture batters

For the ten rices extruded as 15% moisture batters at 120, 135 and 150 °C, maximum expansion ratio occurred at various temperatures. Only at 135 °C was expansion ratio positively correlated significantly with amylose and negatively with gel consistency (Table 3). Instron hardness values were lowest at 150 °C and highest at 120 °C. At 150 °C, IR29 (waxy) seemingly had the hardest extrudate, but the sample was not crispy, unlike the non-waxy extrudates. Among the non-waxy rices, IR42 had the hardest extrudate at 135 and 150 °C and IR48 and IR29798-41-2 had the softest extrudates at 135 °C, and IR43 (19% amylose) was softest among 150 °C extrudates. Only hardness values at 135°C and 150 °C were significantly correlated ( $r = 0.83^{**}$ ). Yanase *et al.* (1982) reported greater volume expansion and more brittle extrudate from waxy rice than from non-waxy rice at 11–15% moisture, 140–150°C and 123–147 bars.

Mean amylose content was not affected by extrusion cooking (Tables 1







**Fig. 3.** Transmission electron micrograph of section of IR45 15% moisture—150°C extrudate showing protein mass with distorted protein bodies in gelatinized starch matrix.

and 3). Maximum absorbance of the starch -iodine complex of non-waxy extrudates was at  $600 \pm 20$  nm, which was lower than that of the raw flours at  $610 \pm 10$  nm. Reducing sugars of extrudate ranged from 0.14% to 0.28% glucose and total sugars 0.12-0.30% in 80\% ethanol extract. Soluble sugars in water were 1.5-2.7% glucose of extrudate, of which 0.1-0.4% glucose were reducing sugars. The results suggest amylose degradation, if any, did not reduce amylose chain length to below the DP 45 glucose units required for a blue-colored complex on iodine binding (Bailey & Whelan, 1961).

Gel consistency was reduced to 100 mm in all ten extrudates (Table 3). All extrudates had some insoluble white suspended particles in the gel. The insoluble white particles of IR42 extrudate were collected by centrifugation, washed with water and tested. This showed a positive Lowry reaction for protein but only a slightly blue color in the iodine test for starch, suggesting that the insoluble particles were mainly protein. Extrusion cooking also drastically decreased gel viscosity which correlated significantly positively with amylose content and negatively with gel consistency of raw flour (Table 3). IR29 (waxy) rice had the highest raw gel viscosity (1299 cps) and the smallest extrudate gel viscosity

#### TABLE 3

Range of, and Mean Properties of, Extrudates of 15% Moisture Batters of Ten Milled Rice Flours and Their Correlation with Raw Rice Amylose Content and Gel Consistency

Property of extrudate	Extrusion temperature - (°C)	Values		Correlation coefficient"		
		Range	Mean	with		
				Amylose	Gel consistency	
Expansion ratio	120	2.2-3.8	3.1	0.12	-0.03	
Expansion ratio	135	2.9-3.7	3.3	0.66*	-0·79**	
Expansion ratio	150	2.5-3.9	3.2	0.23	-0.01	
Instron hardness (kg)	120	$8 \cdot 2 - 12 \cdot 3$	9.4	0.07	0.05	
Instron hardness (kg)	135	6.9 - 8.8	7.6	0.05	0.01	
Instron hardness (kg)	150	4.4 - 7.0	5.7	-0.25	0.24	
Mean amylose						
content (%)	120-150	1.5 - 30.8	20.9	0.99**	-0.53	
Mean gel						
consistency (mm)	120-150	100	100	0	0	
Mean gel						
viscosity (cps)	120-150	13-33	20	0.64*	-0.73*	
Mean degree of						
gelatinization (%)	120-150	88-104	97	0.05	-0.30	
Cold 12% paste						
viscosity (cps)	120	45-2840	1072	0.56	-0·74*	
Cold 12% paste						
viscosity (cps)	135	55-2510	952	0.55	-0.76*	
Cold 12% paste						
viscosity (cps)	150	50-1860	817	0.47	-0.71*	
Water absorption						
index $(g/g)$	120	0.8 - 7.5	5.0	0.84**	-0.80**	
Water absorption						
index $(g/g)$	135	0.7 - 7.7	5.2	0.84**	-0.78**	
Water absorption						
index $(g/g)$	150	0.77.8	5.4	0.77**	-0.80**	
Water solubility						
index (%)	120	18-86	36	-0.94**	0.72*	
Water solubility						
index (%)	135	20 - 86	39	-0.90**	0.77**	
Water solubility						
index ( %)	150	17-87	32	-0.90**	0.77**	

"Significant r = 0.632 at the 5 % level (\*) and 0.765 at the 1 % level (\*\*).

(13 cps). Among the nine non-waxy rices, IR42 had the highest gel viscosity of raw flour (1130 cps) and extrudate (33 cps).

Mean degree of gelatinization was high for all extrudates and was not correlated with any starch property. Mean values were 96.9% at 120 °C, 98.0% at 135 °C and 96.4% at 150 °C. Surprisingly, the lowest degree of gelatinization of 88% was shown by the low GT IR24, whereas the other low-GT rices showed values of 97-101% (mean 100%). The extrudates of intermediate-GT rices had gelatinization degree values of 90-104% (mean 97%).

Cold 12% aqueous paste viscosity of extrudate was consistently lower than cooked 6% paste viscosity of raw flour (Tables 1 and 3). Cold paste viscosity of extrudate did not correlate significantly with amylose content but was significantly negatively correlated with raw flour gel consistency but not with gel viscosity (r = -0.04 to 0.05). Waxy rice (IR29) showed the largest drop in paste viscosity from extrusion cooking from 3280 cps to 45–55 cps. The high amylose rices, IR36 and IR42, with hard gel consistency had the least decrease in paste viscosity. The high amylose soft gel consistency IR32 had the lowest extrudate paste viscosity (300–470 cps) and the lowest raw paste viscosity (1500 cps).

WAI of extrudate was higher than that of raw flour (Table 3) except for the waxy sample IR29 which decreased from 1.4 to 0.7-0.8. IR36 and IR42 had the highest WAI. WAI of extrudate significantly correlated positively with amylose and negatively with gel consistency of raw flour (Table 3). WAI of raw flour was not significantly correlated with WAI of extrudate ( $r = 0.58 \sim 0.63$ ). WSI of extrudate was also higher than that of raw flour and was significantly negatively correlated with amylose and positively correlated with gel consistency. IR29 had the highest WSI of 86–87 %, and IR42 had the lowest WSI of 17–20 %, followed by IR36 with WSI of 21–25 %. WSI of raw flour significantly correlated positively with WSI of extrudate ( $r = 0.91^{**} \sim 0.95^{**}$ ) and negatively with WAI of extrudate ( $r = -0.79^{**} \sim 0.82^{**}$ ).

Among the extrudate properties, expansion ratio at 135 °C and gel viscosity correlated significantly with cold paste viscosity and water absorption and solubility indexes (Table 4). Expansion ratio and gel viscosity are also significantly correlated. Only expansion ratio was significantly correlated with non-starch lipids. Non-starch lipids also correlated significantly with extrudate WAI ( $r = -0.75^* \sim -0.82^{**}$ ) and WSI ( $r = 0.78^{**} \sim 0.83^{**}$ ). Non-starch lipids also correlated significantly with extrudate water with extrudate significantly with extrudate significantly with extrudate significantly with extrudate significantly with extrudate water with extrudate water with extrudate significantly with extrudate water water with extrudate water water water with extrudate water with extrudate water water water water water with extrudate water water water water water with extrudate water water with extrudate water wat

#### **TABLE 4**

Correlation Between Expansion	Ratio at 135°C and C	Gel Viscosity of Extruda	ite and Cold
Paste Viscosity, Wate	r Absorption and So	lubility Indexes at 135	°C

	Property of extrudate at $135^{\circ}C$	Correlation coefficient <sup>a</sup> with			
		Expansion ratio	Gel viscosity		
Gel visco	osity	0.84**	1.00		
Cold pas	te viscosity	0.89**	0.94**		
Water at	psorption index	0.92**	0.73*		
Water so	lubility index	-0.81**	-0.67*		
Non-star	ch lipids	-0.65**	-0.49		

"Significant r = 0.632 at the 5% level (\*) and 0.765 at the 1% level (\*\*).

but not at 120° and 135°C ( $r = -0.60 \sim -0.61$ ). Starch lipids also correlated significantly with extrudate WAI ( $r = 0.73^* \sim 0.75^*$ ) and WSI ( $r = -0.82^{**} \sim -0.91^{**}$ ). WAI or WSI of extrudates at 120°, 135° and 150°C of the ten rices are also significantly correlated ( $r = 0.96^{**} \sim 0.99^{**}$ ). WAI-WSI correlation coefficients were  $-0.90^{**}$ to  $-0.96^{**}$ .

#### CONCLUSIONS

Among ten rices differing in starch properties extrusion cooked through a twin screw extruder at  $15 \,\%$  moisture and 120, 135 and 150 °C, only expansion ratio at 135 °C significantly correlated with amylose content and gel consistency of raw flour. Amylose content and gel consistency of raw flour correlated significantly with WAI and WSI of extrudate at all temperatures and with gel viscosity of extrudate, but only gel consistency of raw flour correlated with cold paste viscosity of extrudate. Final gelatinization temperature of starch was not correlated with any extrudate property. Extrusion cooking drastically reduced viscosity in 0.2N KOH and in water without any decrease in amylose content and with little increase in reducing sugars. Spherical protein bodies were intact in the protein masses uniformly dispersed in the gelatinized starch matrix.

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