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RVA Pasting Properties of Australian Wheat Starches

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Viscosity properties of wheat starch and flour measured by the Rapid Visco Analyser (RVA) were characterised by RVA running conditions and varietal difference. RVA variables including starting temperature of measurement, heating rate and final cooling temperature were found to be the most important factors determining RVA viscosity properties. Starting temperature mostly affected peak time, peak viscosity, breakdown and setback viscosity for starch, and peak time and breakdown for flour. Heating rate influenced peak time, peak viscosity, holding

strength and final viscosity for starch, and peak time, peak viscosity, breakdown and holding strength for flour. Setback viscosity for starch and flour was determined by final cooling temperature. Discrimination of wheat varieties was greatest with the following RVA running profile: starting temperature of 50 °C, heating rate of 20 °C/min and final cooling temperature for both starch and flour of 43 °C. This profile also showed clear classification of wheat varieties tested and accordingly good prediction for softness of cooked white salted noodles.

1 Introduction

Starch viscosity properties determine the textural characteristics and storage quality of many cereal food types. These pasting properties can be measured using various instruments such as the Brabender Amylograph, the Brookfield Viscometer and most recently the Rapid Visco Analyser (RVA). Many RVA procedures have been developed for a range of cereal food applications [1–6]. Different procedures vary in sample concentration, starting temperature, heating and cooling rates, holding times and temperature. Recently a general method analysing the pasting quality of wholemeal flour, flour or starch from various plant sources has been established as a standard by the International Cereal Research Associations [7]. This standard method was adequately designed to suit the particular purpose. For example, the quality of rice flour was well tested by RVA for the prediction of cooked rice quality. The optimum concentration of rice flour in the assay and the operating condition of RVA were suggested [2]. Potato starch was prepared at a range of concentrations and examined on various running profiles differing in temperature and time [5]. The RVA result was then com-

pared with the Amylograph result showing high correspondence between the two methods. These methods have either been developed to mimic the conditions the starch is subjected to during food processing, or to discriminate starch quality for a broad range of applications. For example, a confectionary manufacturer may require a starch that pours effectively into moulds when hot, and then sets to form a very firm texture when cooled. Test conditions to measure starch for this application can be clearly defined based on those used for production. Alternatively, wheat breeders may be seeking to screen large numbers of samples for a range of end uses including noodles, bread and non-food applications. In this instance, a single temperature/time profile must be selected to screen the wheat genotypes for a wide range of end uses. Accordingly it would be necessary to examine all RVA running variables causing the change of pasting properties and to determine appropriate RVA running conditions for the prediction of the quality of cereal products such as pan bread or noodles.

Pasting properties of starch also depend on genetic variation of starch source. 59 New Zealand wheat cultivars were tested using Minipaster [8, 9]. The results revealed that the cultivar was the most important factor in determining variation in wheat starch pasting properties. In Australia, these findings were confirmed with 65 wheat cultivars. These properties were also reported to be important parameters for the quality of noodles [3, 6] and Chinese steamed bread [10].

The objective of the present work was to investigate the effects of a) RVA running conditions on the measurement of wheat flour and starch properties and b) varietal differences on RVA running conditions. Samples were selected to represent differences between wheat genotypes due to grain hardness, and the presence or absence of the null 4A allele for granule bound starch synthase (GBSS), which has been shown to influence starch properties [11].

2 Materials and Methods

2.1 Wheat samples

Five Australian wheat varieties (Cadoux, Rosella, Hartog, Egret and Sunco) were selected for this study. Cadoux (control), Rosella and Egret are soft wheats and Sunco and Hartog are hard wheats. Cadoux, Rosella and Hartog are null 4A genotypes, whilst Egret and Sunco are normal. The wheats were milled into 60 % extraction flour using a Bühler test mill. Raw starch was then separated from flour by handwashing with deionised water using the method previously reported [12]. Starch was exhaustively defatted with 85 % aqueous methanol.

2.2 Analytical methods

Protein and lipid contents of starch and flour samples were measured by AACC Methods 46-12 and 30-10 respectively [13]. Starch damage and falling number of flour samples were measured by AACC Methods 76-30A and 56-81B, respectively [13]. Starch granule size distribution was obtained using the Malvern Particle Sizer as previously reported [3]. Differential Scanning Calorimetry (DSC) experiments were performed using a Seiko DSC-120 (Kawasaki Kanagawa, Japan). An empty pan was used as reference. Approximately 15 mg of starch were accurately weighed in a pan and distilled water was added to obtain a ratio of 1:2 (w/w). The sample was heated from 10° C to 140° C at a rate

Tab. 1. Chemical composition of samples.

of 10 °C/min. Onset temperature (T_0) , peak temperature (T_n) and completion temperature (T_c) and the enthalpy of gelatinisation (∆*H*) were determined from the endothermic curve. Softness of cooked white salted noodles was measured by Instron as previously described [6].

2.3 Experimental design for RVA

The wheat variety Rosella was selected to screen the RVA variables using the experimental design program "Discovery" (International Qualtech. Ltd, Minneapolis, USA). A fractional factorial design was applied firstly for peak time and peak viscosity with four independent RVA variables [starting temperature of measurement (ST), holding time at starting temperature (TST), heating rate (HR) and holding time at 95 °C (T95)]. The five variables for breakdown and holding strength were starting temperature of measurements holding time at starting temperature, heating rate, holding time at 95 °C and cooling rate (CR). Finally seven variables were tested for their effect on setback and final viscosity [starting temperature, holding time at starting temperature, heating rate, holding time at 95 °C, cooling rate, final cooling temperature (FCT) and holding time at final cooling temperature (TCT)]. The levels of the variables were determined on the basis of expected gelatinisation characteristics of wheat starch and RVA heating and cooling capacity as follows: ST, 40 °C to 70 °C; TST, 0 min to 3 min; HR, 1 °C/min to 20 °C/min; T95, 1 min to 5 min; CR, 1 °C/min to 15 °C/min; FCT, 30° C to 50° C; TCT, 1 min to 5 min. 2 g starch or 3 g flour were suspended in 25 mL of deionised water for all viscosity tests. Combinations of conditions were then selected from the screening test which were shown to most strongly influence starch and flour properties. Details of the conditions are presented in the Results and Discussion section.

3 Results and Discussion

3.1 Sample description

Selection of wheat sample was based on white salted noodle eating quality. The soft wheat variety Cadoux was developed in Australia to meet the quality requirements for Japanese style white salted noodles and used as control in

¹ The size which has 50 % particles smaller than itself. ⁴ Mean diameter derived from the volume distribution

³ The size which has 10 % particles smaller than itself.

² The size which has 90% particles smaller than itself. ⁵ Measure of the ratio of the total volume of particles to the total surface area.

Tab. 2. DSC results¹.

¹ *T*o, onset temperature; *T*p, peak temperature; *T*c, completion temperature of gelatinisation; ∆*H*, enthalpy of gelatinisation.

this study. Among five varieties Cadoux, Rosella and Hartog have a different genetic characteristic of starch granule bound starch synthase (null 4A) from the other two varieties (normal 4A), which was reported to be an important factor to determine the eating quality of white salted noodles [14]. Tab. 1 shows chemical data of five flour samples and corresponding starches. Protein content of the soft wheat flours of Cadoux, Rosella and Egret flours ranged from 8.4 % to 9.3 %, whereas the hard wheat varieties, Sunco and Hartog, showed a protein content of 12.2 and 11.1 %, respectively. No significant difference in lipid content was observed between the samples. Hard wheat flour samples (Hartog and Sunco) showed higher level of starch damage compared to soft wheat flour. There was no apparent difference in falling number between the flour samples.

Starch samples showed low protein and lipid contents with negligible difference between the varieties (Tab. 1). Starch granule size distribution was similar for the four varieties except for the Cadoux sample having the lowest percentage of B granules ($< 10 \mu m$). The average size of Cadoux starch granules was bigger than that of the other starch samples. This means that Cadoux contained more A granules than the other varieties tested in this study. No obvious trends in protein content, lipid content and starch granule size distribution were observed among the five samples. Overall granule size distribution did not appear to have a significant effect on RVA viscosity properties in this study.

The Differential Scanning Calorimetry (DSC) results indicated that Sunco was characterised by a low gelatinisation temperature, whilst the soft wheat Rosella showed the highest onset temperature of gelatinisation with the lowest value of (T_c-T_o) (Tab. 2). Otherwise no significant differences in DSC properties were observed between the sample groups in terms of hardness and genetic property. As defatted starches showed almost the same gelatinisation properties on DSC as raw starches, raw starch samples were used for RVA investigations in this study.

3.2 Selection of RVA variables

The effects of the RVA variables on starch and flour pasting viscosity parameters using the screening test were calculated using a fractional factorial design (Tabs. 3 and 4). In the case of starch samples HR and ST significantly affected RVA peak time. Starch peak viscosity was not greatly influenced by RVA variables tested in this screening test suggesting that the variance of starch peak viscosity may be attributed to varietal difference or the influence of other sample properties. ST, T95 and CR showed significant effects on breakdown in that order, whereas holding strength was highly affected by T95. Setback, which is known to be a critical RVA parameter for white salted noodle quality [6], was strongly affected by FCT followed by the other variables, ST, CR and HR.

Flour pasting properties showed somewhat similar effects to starch properties by RVA variables (Tab. 4). ST and HR significantly affected peak time as with the starch samples, while peak viscosity and breakdown viscosity of flour were not significantly affected by RVA variables. This finding may explain the previous observation in which no significant relationship was found between the eating quality of white salted noodles and starch and flour peak viscosity [6]. Holding strength of flour paste was influenced by different RVA variables than that of starch paste. HR appeared to be the major factor to determine holding strength. Furthermore CR significantly affected flour holding strength whereas that of

¹ Figures based on effect as % of mean of RVA parameter.

¹ Figures based on effect as % of mean of RVA parameter.

starch was not influenced. The most important RVA variable for flour setback viscosity was FCT, which was also important to starch setback viscosity.

From the observations shown in Tabs. 3 and 4, combinations of the RVA variables most affecting viscosity were selected to test all starch and flour samples. Since T95 and TCT contributed minor effects on RVA viscosity parameters, T95 was set to 3 min and TCT to 2 min. TST did not show any effect on RVA parameters and set to 0 min. CR did not show a significant effect and was dependent to some extent on the water temperature supplied, therefore 15° C/min – which can be consistently achieved using the tap water temperature $(17^{\circ}C - 23^{\circ}C)$ – was chosen for the cooling rate. Other variables and their conditions tested were as follows: ST, 50 °C (below normal wheat starch gelatinisation temperature) and 70 °C (above wheat starch gelatinisation temperature); HR; 10 °C/min, 15 °C/min and 20 °C/min; FCT, 43 °C and 49.5 °C. In total, 12 RVA running profiles were tested in this study.

3.3 Pasting properties of starch and flour on various RVA running profiles

Results of RVA pasting viscosity properties on 12 RVA running profiles tested in this study are shown in Tabs. 5 and 6 for starch samples and Tabs. 7 and 8 for flour samples.

3.3.1 Effects of RVA running profiles on starch pasting properties

RVA pasting properties of the starches tested showed considerable variation due to RVA running profiles and variety. **Tab. 5.** RVA pasting viscosity properties of starch samples on 12 RVA temperature-time profiles.

¹ min. ² RVU (Rapid Visco Analyser Unit).

Tab. 6. RVA pasting viscosity properties of starch samples on 12 RVA temperature-time profiles.

Starting temp. $[^{\circ}C]$ Heating rate $[^{\circ}C/\text{min}]$ Cooling temp. $[°C]$		50			70		
		15 10		20	10	15	20
		43/49.5	43/49.5	43/49.5	43/49.5	43/49.5	43/49.5
	Cadoux	91/58	94/67	90/67	77/68	73/68	75/63
	Rosella	82/67	83/69	83/68	74/65	75/63	76/63
Setback ¹	Hartog	77/45	84/51	80/50	77/53	63/53	76/50
	Egret	48/25	62/33	59/36	80/57	79/51	70/57
	Sunco	61/36	69/43	68/43	70/48	69/48	68/45
Final $vis1$	Cadoux	159/111	171/122	174/131	146/135	149/134	160/131
	Rosella	137/121	146/126	157/133	137/130	139/129	152/132
	Hartog	132/79	145/91	152/98	147/103	145/105	155/102
	Egret	75/ 47	100/61	100/72	128/102	127/91	122/102
	Sunco	98/64	115/ -80	121/83	121/89	117/92	128/90

1 RVU (Rapid Visco Analyser Unit).

Tab. 7. RVA pasting viscosity properties of flour samples on 12 RVA temperature-time profiles.

Starting temp. $[°C]$ Heating rate [°C/min]		50			70		
		10	15	20	10	15	20
	Cadoux	5.35	4.28	3.88	3.37	2.82	2.95
	Rosella	5.42	4.22	3.88	3.35	2.95	2.97
Peak time ¹	Hartog	5.42	4.35	3.95	3.35	3.15	3.08
	Egret	5.22	4.08	3.75	3.28	3.02	2.90
	Sunco	5.08	3.95	3.70	3.08	2.68	2.78
Peak vis^2	Cadoux	152	158	170	167	163	167
	Rosella	168	175	178	185	180	182
	Hartog	170	186	185	188	191	181
	Egret	113	142	147	153	151	167
	Sunco	117	145	146	154	151	145
Breakdown ²	Cadoux	75	82	84	99	94	89
	Rosella	88	96	94	108	102	100
	Hartog	75	83	80	102	95	89
	Egret	33	50	52	69	63	71
	Sunco	52	66	69	81	76	67
Holding $str2$	Cadoux	77	76	86	68	69	78
	Rosella	80	79	84	77	78	82
	Hartog	95	103	105	86	96	92
	Egret	80	92	95	84	88	96
	Sunco	65	79	77	73	75	78

¹ min. ² RVU (Rapid Visco Analyser Unit).

RVA variables, which were selected from preliminary experiments, showed significant effects on most RVA viscosity parameters.

ST had significant effects on peak time, breakdown and setback viscosity for all starch samples. At low ST longer peak time and lower breakdown viscosity were clearly observed for all samples. However ST had a different effect on setback viscosity, showing a decrease for Cadoux, Rosella and Hartog (all null 4A genotypes) and an increase for Egret (minor increase for Sunco). ST affected the final viscosity of Egret, while no significant effect was observed for the other varieties for this parameter.

RVA peak time of all starch samples decreased when HR increased. Difference in peak time between the HRs of 10 °C/min and 15 °C/min was larger than that between the HRs of 15 °C/min and 20 °C/min indicating that there was no

significant difference in peak time at high heating rates. No apparent trends between the heating rates for the setback viscosity value were observed in this study. In general, peak viscosity increased with the increase in HR, for ST of 50 °C, for all varieties, whereas at an ST of 70 °C no obvious difference in peak viscosity was observed for the different heating rates. Obvious increases in holding strength and final viscosity at a ST of 50 °C were observed with the increase of HR in all varieties. Overall HR had major effects on most RVA parameters at a ST of 50 °C, whereas at an ST of 70 °C no significant effect of HR was observed on RVA parameters.

FCT clearly affected RVA setback and final viscosity for all starches. Low setback viscosity and final viscosity were observed at a high FCT of 49.5 °C. Differences in setback and final viscosity between two FCTs varied for varieties.

Tab. 8. RVA pasting viscosity properties of flour samples on 12 RVA temperature-time profiles.

Starting temp. $[°C]$ Heating rate $\lceil \degree C / \text{min} \rceil$		50			70		
		10	15 43/49.5	20 43/49.5	10 43/49.5	15 43/49.5	20 43/49.5
	Cooling temp. $[°C]$						
	Cadoux	89/72	87/66	90/68	81/59	76/ 59	84/ 57
	Rosella	93/73	93/71	94/72	87/67	90/66	95/67
Setback ¹	Hartog	108/86	105/82	106/84	95/74	100/75	99/78
	Egret	111/57	116/90	117/90	109/85	106/81	115/81
	Sunco	81/65	82/ -66	85/68	76/64	82/67	83/67
Final $vis1$	Cadoux	166/151	163/146	176/149	149/128	145/128	162/149
	Rosella	173/148	172/148	178/157	164/142	168/143	177/145
	Hartog	203/181	208/179	211/190	181/166	196/169	191/178
	Egret	191/138	208/179	212/185	193/175	194/174	211/173
	Sunco	146/135	161/142	162/151	149/140	157/149	161/147

¹ RVU (Rapid Visco Analyser Unit).

In an overview of five wheat starch samples under various RVA running profiles, discrimination of varieties in peak time at any ST was not large compared to the effect of HR. However, the other RVA parameters showed more distinct differences between varieties at a ST of 50 °C than at 70 °C. For example, varietal differences in setback and final viscosity at an ST of 50 °C were larger than those measured at 70 °C indicating better discrimination at 50 °C for this group of samples. HRs at STs of 50 °C showed no clear effect for the discrimination of varieties in all RVA parameters. Therefore the maximum HR, 20° C/min, can be applied to reduce RVA running time. However, it must be noticed that the order of varieties in most RVA parameters changed with a change of HR, particularly at high ST. Similarly a range of varietal responses to setback and final viscosity were observed between two FCTs. However, the ranking of varieties for setback viscosity at 50 °C changed when the ST was 70 °C, and this was not observed for the other RVA parameters. The range of varietal differences in setback viscosity at two different FCT with the ST of 70 °C was narrow. It was distinctively wider at the ST of 50 °C. This result indicates that the ST of 50 °C would be preferable for discrimination of the samples in setback viscosity. Varietal differences in final viscosity by combined analysis of ST and FCT showed that a wide range of differences in final viscosity was observed at a ST of 50 °C and a FCT of 43 °C. However, it would be necessary to increase the number of samples tested before any statistical analyses for a particular end product quality could be assessed. Depending on processing conditions, it may be necessary to change RVA running profiles to more closely represent the processing conditions These results also show that starch gelatinisation properties are affected not only by RVA running condition but also by starch sample characteristics. It was not possible to draw conclusions concerning the effects of grain hardness or the null 4A GBSS status of the cultivars as the effects of environment were not able to be addressed in this trial.

3.3.2 Effect of RVA profiles on flour gelatinisation properties

The same experiment was carried out with the corresponding flour samples. Flour samples showed somewhat different RVA results from starches on the same conditions indicating that starch and flour properties could be analysed and utilised independently.

Peak time was greatly affected by ST. At low ST a much longer peak time was clearly observed for all flour samples. Interestingly flour samples with normal 4A gene showed shorter peak time compared to the samples with null 4A. Peak viscosity at an ST of 50 °C increased with the increase of HR, whereas no change was observed at an ST of 70 °C. Higher breakdown viscosity was observed at an ST of 70 °C for all flour samples. As already indicated in preliminary experiments, no significant effects of ST were found for the other RVA parameters including holding strength, setback and final viscosity.

Peak time decreased when HR increased in all the varieties. At an ST of 50 °C difference in peak time between the HRs of 10° C/min and 15° C/min was much larger than that between the HRs of 15 °C/min and 20 °C/min. However difference in peak time between the HRs of 15 °C/min and 20 °C/min at an ST of 70 °C was negligible for all samples. There were no varietal differences in peak time in terms of grain hardness regardless of HR. Breakdown measured at an ST of 70 °C decreased when HR increased, in contrast to an ST of 50° C, where breakdown increased with increasing HR. Cadoux and Rosella showed the same holding strength at the HRs of 10° C/min and 15° C/min, whereas Hartog, Egret and Sunco showed a similar value of holding strength at the HRs of 15 °C/min and 20 °C/min.

Higher setback and final viscosity were always observed at an FCT of 43 °C than at an FCT of 49.5 °C. Varietal difference in setback viscosity was clearly observed at an FCT of 43 °C.

As a result, strong varietal differences in peak time, breakdown and holding strength were obtained with flour samples when ST was 50 °C, whereas negligible varietal differences in setback and final viscosity were observed between two STs. The orders of varieties in most viscosity parameters were changed when they were compared with starch results. Differences between varieties and RVA running conditions are shown in Tabs. 7 and 8. Although ST affected peak time of flour in the same manner as that of starch, varietal difference and order were different to the results of starches in peak time. Even though the values of setback and final viscosity were marginally higher at an FCT of 43 °C than 49.5 °C, the order of varieties appeared to be the same. This finding provides the possibility of using the higher FCT for flour samples.

3.4 RVA running profiles and their significance in the relationship with the softness of noodles

The softness of white salted noodles was measured by Instron and the result is shown in Tab. 9. As there were strong relationships between peak viscosity and breakdown, between holding strength and setback and between setback and final viscosity [6], breakdown viscosity and setback viscosity were selected to examine the relationship with the softness of cooked noodles. Breakdown viscosity measured by the 12 RVA running profiles did not show any relationship with the softness of cooked noodles. However the setback viscosity showed interesting results. RVA profile 50° C (ST)- 10° C/ min (HR)-49.5 °C (FCT) showed a different pattern of setback viscosity compared with the other profiles tested in this study. All soft wheat varieties showed a consistent increase of firmness – which is undesirable for white salted noodles – with the increase of setback viscosity. A similar trend was observed with hard wheats. When two groups of wheats were combined, however, no consistent trend was observed. It implies the importance of grain hardness, so grouping the wheat samples on hardness may be necessary to improve the relationships between physicochemical data and noodle eating quality. The RVA profile 56° C (ST)-0 min (TST)-20 °C/min (HR)-3 min (T95)-15 °C/min (CR)-49.5 °C (FCT)-2 min (TCT) appeared to be the most suitable profile for the cooked noodle softness among the profiles tested in this study. Cadoux, Hartog and Rosella containing similar genetic characteristic showed higher viscosity figures than the other varieties (Egret and Sunco). This emphasises the significance of genetic property of variety compared to RVA running condition adjustment.

Tab. 9. Softness (force/time) of white salted noodles measured by Instron.

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Effects of Phosphorylating Salts and Temperature on the Preparation of Rice Starch Phosphates by Extrusion

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Rice starch phosphates were prepared by the extrusion of rice starch with phosphate salts including sodium tripolyphosphate (STP), sodium trimetaphosphate (SMP), and a 50:50 mixture of STP and SMP (STP-SMP). The incorporation of phosphorus in the starch increased with increased barrel temperature ranging from 120 to 180 °C. At 180 °C, phosphorus contents for products treated with STP, SMP, and STP-SMP were 0.376, 0.194, and 0.360 %, respectively. The effects of phosphorylation on functional and rheological properties of the starch varied, depending on the extent of molecular degradation and the forma-

tion of intermolecular cross-linking caused by the heat and shear of extrusion. Generally, extrusion stabilized the pasting properties of the extrudates, whereas phosphorylation enhanced the pasting consistency of the starch products. Good correlation was observed between the extrusion specific mechanical energy (SME) and the product of solubility and intrinsic viscosity, indicating a close relationship between the energy input in the extrusion process and the performance behavior of the extruded phosphorylation products.

1 Introduction

Starch has been used as a food ingredient in a wide variety of products. However, native starch needs modification to develop desirable functional properties, such as solubility, texture, adhesion, dispersion, and heat tolerance [1]. Starch can be conveniently phosphorylated through esterification of the hydroxyl groups with phosphorylating agents such as sodium tripolyphosphate (STP), sodium trimetaphosphate (SMP), and phosphorus oxychloride. Starch phosphate esters were reported to give clear pastes of high viscosity and consistency, with good freeze-thaw stability and resistance to

retrogradation [2, 3]. The conventional process requires an excess amount of reagents. The removal of the unused reagent afterward could be expensive and a cause of concern for possible pollution to the environment. Efforts have been made to overcome these shortcomings by using extrusion technologies to improve the efficiency of the process as well as the food-use quality of the products. *Kim* et al. [4] were able to increase the phosphorus incorporation in starch by reducing its moisture content during the extrusion. *Salay* and *Ciacco* [5] prepared corn starch phosphates by extrusion through a series of unit operations including pH adjustment of starch slurry, filtering, drying, powdering, and moisture