

# Milling—A Further Parameter Affecting the Rapid Visco Analyser (RVA) Profile

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## ABSTRACT

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Rapid Visco Analyser (RVA) profiles were recorded for raw maize grits and two extruded nonexpanded pellets based on wheat and maize. Large differences were found between the profiles obtained when an impeller mill was used to prepare the samples compared with a disk mill. The differences were related to differences in particle properties of the ground products (particle-size distribution, particle shape, and protein content). Generally, milling the samples with the impeller mill resulted in greater starch conversion than with a disk mill. For raw maize grits, this was shown by X-ray diffraction, differential scanning calorimetry (DSC),

and alkaline viscosity measurements. Several other laboratory mills were tested and all produced particulates with a sieve range of 125–212  $\mu\text{m}$  that had substantially differing RVA profiles. Cooling the sample during milling did not nullify the milling effects. All the laboratory mills produced <20% of the particulates of the size range required for the RVA analysis. The mill used for sample preparation can exert a significant effect on the RVA for both raw and processed cereal samples, even if measurements are made on a defined sieve fraction.

The Rapid Visco Analyser (RVA) was originally developed by Newport Scientific Pty Ltd (NSW, Australia) to rapidly estimate sprout damage in wheat (Ross et al 1987). The RVA had advantages over the viscoamylograph because it required only a small amount of sample (4 g), was rugged and simple to operate, and gave results for this application in 3 min (Walker et al 1988). Subsequently, the RVA has been used extensively to study starch pasting characteristics for both raw materials (Walker et al 1988; Welsh et al 1991; Bason et al 1993; Bahnassey and Breene 1994; Cooreman et al 1995; Zeng et al 1997; Collado and Corke 1999; Jacobs et al 1999) and starch-based extruded or otherwise processed food (Ruy et al 1993; Whalen 1995; Whalen et al 1997; Guha et al 1998; Whalen 1999a, b).

RVA measurements were particularly valuable in characterizing an extrusion process. Based on the idea that pasting parameters generated from the RVA provide a relative measure of starch gelatinization, disintegration, swelling, and gelling ability (Ruy et al 1993), Whalen et al (1997) related the degree of starch conversion as introduced by extrusion to the RVA profile and found the RVA very sensitive and descriptive of processing effects caused by water content and thermal and mechanical input. With increasing specific mechanical energy input, changes in profile such as a shift from a gelatinization peak to a cold swelling peak occurs after the progressive loss of granular integrity, a lowering of the trough, and the final viscosity of native and extruded wheat flour (Fig. 1). The RVA profile depends strongly on the particle size used, particularly for an extruded profile where the cold swelling peak will depend on the rate of particle hydration. In the literature, different types of mills have been employed. The aim of the current investigation was to determine if the RVA profile is dependent on the mill type for a given sieve size fraction. This article presents the reasons milling for size reduction of the sample is a major consideration when studying starch-to-water interactions by RVA.

## MATERIALS AND METHODS

Three samples that represent many food products were used: raw maize grits (RM) considered a friable product, an extruded half product from maize (EM), and a half product from wheat (EW) (Fig. 2). Unlike RM, the extruded half products are hard and difficult

to grind and therefore the choice of mill is critical. Supplier and composition of raw materials are given in Table I.

Each product was milled with two mills: a disk mill (Tecator 1090 Cemotec mill [for RM and EW] and Perten FN mill 3303 [for EM]) and an impeller mill (Tecator 1093 Cyclotec mill [for RM, EM, and EW]). The same amount of product was introduced into each mill and was always milled in a single pass. The powder was sieved for 10 min with a sieve shaker (Analysette SRL5300, Fritsch, Idar-Oberstein, Germany) using sieves of 125, 212, and 250  $\mu\text{m}$  (equivalent to U.S. mesh sizes #120, 70, and 60, respectively). Samples from the sieve fraction 125–212  $\mu\text{m}$  (U.S. mesh #120–70) were used for RVA, microscopy, X-ray, differential scanning calorimetry (DSC), alkaline viscosity, and particle-size analysis.

Moisture content was determined by oven drying at 110°C for 15 hr and calculated as an average value of triplicates (% dry basis).

RVA measurements were performed using 4 g of sample (14% moisture, db), i.e., 3.509 g of dry sample and sufficient distilled water to achieve a total sample weight of 29 g. The measured moisture content of each sample after milling was used to calculate the weight of the powder to be used for the RVA analysis to ensure a constant solid-to-liquid ratio of 13.77%. Sample and water were weighed separately to  $\pm 0.001$  g and mixed. Measurement started within <20 sec to detect all cold swelling behavior. The viscosity was recorded (RVA Series 4) along with the accompanying software Thermocline for Windows Version 2.0. The profile was hold at 25°C for 6 min, ramp to 95°C over 5 min, hold at 95°C for 6.5 min, cool back to 25°C over 6 min, and hold at 25°C for 9 min. Each analysis took 32.5 min and was done in duplicate.

Particle-size analysis was performed with low angle laser light scattering (LALLS) using a Malvern Mastersizer S (Malvern Instruments Ltd., Worcester, UK) fitted with a small volume dispersion unit. The scattered light data from  $\approx 2,000$ –5,000 snapshots of 10  $\mu\text{sec}$  each were recorded within one measurement and transformed to a distribution of particle-size information according to the Mie theory by the accompanying software Mastersizer Sv2.19. A poly-disperse mode of analysis and a refractive index of 1.533 for starch with an adsorption of 0.001 were chosen. A sample of  $\approx 1$  g was dispersed in 20 mL of isopropanol and then sonicated for 5 min to disperse agglomerates. The sample dispersion was added to the circulating liquid until an obscuration of 15–20% was recorded, and the measurement was started after 30–45 sec of agitation. Two sample dispersions were prepared per sample, two runs were performed, and each reading was duplicated, resulting in eight measurements in total. Particle-size data were given as % (v/v) particles between two sizes, and parameters were calculated as median ( $D(n, 0.5)$ ), 10th and 90th percentiles ( $D(n, 0.1)$  and  $D(n, 0.9)$ ). To obtain information about particle shape, the samples were examined

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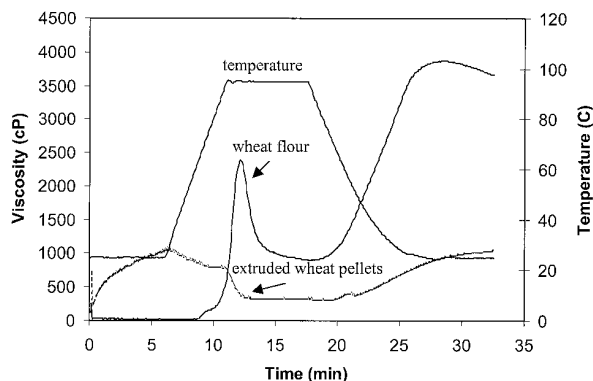
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visually using a microscope. Small amounts of sample were suspended in isopropanol and observed with an optical microscope (Leitz Diaplan, Wild Leitz GmbH, Wetzlar, Germany). Particle size and shape were focused with overall magnifications of 40×. Polarized light was used to distinguish between intact and damaged or gelatinized starch.

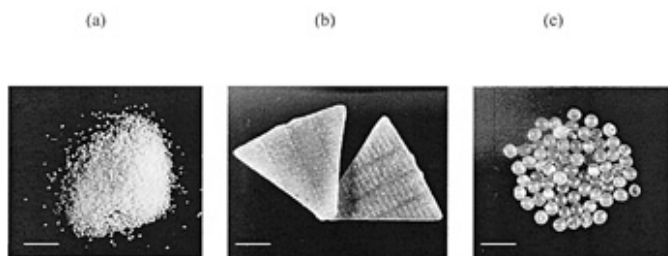
Protein was determined according to the Dumas method using a nitrogen analyzer (FP 2000 Leco Instrument UK Ltd., Stockport, Cheshire, UK). The sample (1 ± 0.5 g) was combusted at 1,150°C in a sealed furnace. Measurements were validated by analyzing a calibration sample (EDTA) and a standard material of known protein content before the actual sample, as well as the calibration sample after. The protein content was expressed as g of N/100 g of sample.

Crystallinity was measured by wide angle X-ray scattering using an X-ray diffractometer (Siemens D5005, AXS GmbH, Karlsruhe, Germany). The X-ray generator equipped with a copper tube operating at 40 kV and 50 mA produced radiation of ≈1.54 Å wavelength. Data were recorded over an angular range of 4–38° (2θ) with an angular interval of 0.02°. The degree of crystallinity (%) was calculated from the ratio of the sum of the individual peaks (crystalline peaks) to the total area of the diffractogram at 4–32° (2θ) using a computer program (Diffrac Plus, Bruker AXS, Karlsruhe, Germany).

Gelatinization enthalpy was recorded on a differential scanning calorimeter (DSC-7, Perkin-Elmer Ltd, Beaconsfield, UK). Ground maize or extrudate (10 mg) and 30 mg of distilled water was weighed into large-volume stainless steel pans to give a starch-to-water ratio of 1:3 and allowed to equilibrate in the sealed pans over night at ambient temperature. For analysis, the DSC cells were heated from 20 to 200°C at a scanning rate of 10°C/min, then cooled from 200 to 20°C at a cooling rate of 180°C/min, held for 5 min at 20°C to equilibrate, and then heated again from 20 to 200°C at a scanning rate of 10°C/min as control. The peak onset, peak, and completion transition temperatures were determined, and the transition enthalpy was calculated from the peak area as J/g of dry matter using software developed by Perkin Elmer. Measurements were performed in duplicate.



**Fig. 1.** Typical RVA profile of wheat flour and extruded wheat pellets showing the shift from the gelatinization peak to a cold swelling peak and a lowering of trough and final viscosity values caused by processing.



**Fig. 2.** Raw maize grits (a), extruded maize pellets (b), and wheat pellets (c). Bar = 1 cm.

Alkaline viscosity was measured following the method of Paterson et al (1986). Dried samples (0.2 g) were mixed with ethyl alcohol (0.5 mL) before 5M KOH (4 mL) was added. The samples were stirred for 18–24 hr, resulting in a clear solution. The solution was diluted with distilled water to 40 mL (0.5% starch). Aliquots (30 mL) of the resulting solution were measured using a controlled stress rheometer (model CS 10, Bohlin Instruments Ltd., Cirencester, UK). Double gap cylindrical measuring geometry (DG 40/50) was chosen as most suitable for measuring low viscosity fluids. The measurements were performed over the increasing shear rate range of 1–100/sec at a constant temperature of 25°C. Measurements were performed in triplicate. Data were compared at a constant shear rate of 10/sec.

## RESULTS AND DISCUSSION

Figure 3 shows the RVA profiles of the three samples from different types of mills. All the profiles were obtained in duplicate and the variation in viscosity is typically <7% at any point along the curve. The three profiles indicate that the samples are different one from another, both in terms of origin and milling. Figure 3a shows the typical profile expected for a native sample with no cold swelling peak but a marked gelatinization peak starting when the temperature within the RVA was >66°C. Figure 3b shows the RVA profile for the maize samples produced by a thermo-mechanical extrusion process. The viscosities recorded were ≈5- to 10-fold lower than those for the native sample. The profiles show a cold swelling peak and some additional increase in

**TABLE I**  
Composition of Raw Materials (% db)<sup>a</sup>

	Raw Maize Grits	Wheat Flour	Corn Flour
Moisture	12 – 14	17	9 – 12
Starch (s)/Carbohydrates (c)	75 – 85 (s)	82 (c)	86 (c)
Fat	0.9 – 1.0	2	3.5 – 5.5
Protein	8 – 10	16	8 – 10
Ashes	0.2 – 0.5	0.5	0.6

<sup>a</sup> As given by suppliers: Maize Céréales Technologies, France; Peyer, France; and Azteca Milling L.P., Texas, respectively.

**TABLE II**  
Nitrogen Content of Raw Maize Grits

Sieve Fraction	Disk Mill			Impeller Mill		
	N <sup>a</sup>	SF <sup>b</sup>	N/F <sup>c</sup>	N <sup>a</sup>	SF <sup>b</sup>	N/F <sup>c</sup>
<125 μm	1.10	5.38	0.059	1.16	45.05	0.523
125–212 μm	1.17	9.61	0.112	1.25	31.12	0.389
>212 μm	1.25	85.02	1.063	1.35	23.83	0.322
Nonsieved	1.23	100	1.23 <sup>d</sup> / 1.234 <sup>e</sup>	1.24	100	1.24 <sup>d</sup> / 1.233 <sup>e</sup>

<sup>a</sup> Nitrogen content (g of N/100 g of sample).

<sup>b</sup> Sieve fraction (g/100 g).

<sup>c</sup> Nitrogen per fraction (g of N).

<sup>d</sup> Measured value.

<sup>e</sup> Calculated value.

**TABLE III**  
Starch Damage on Molecular Structure Caused by Milling Raw Maize with a Disk or Impeller Mill

Mill	SF <sup>a</sup> (μm)	M <sup>b</sup> (%)	X-ray <sup>c</sup> (%)	AV <sup>d</sup> (mPas)	DSC <sup>e</sup> (J/g)
None	All	13.47	14.33	1.562	10.83
Impeller	All	11.04	14.27	1.508	8.20
Disk	125–212	11.86	9.78	1.579	10.05
Impeller	125–212	9.60	9.03	1.508	4.25

<sup>a</sup> Sieve fraction.

<sup>b</sup> Moisture, triplicate measurement, standard deviation <±0.21.

<sup>c</sup> X-ray (% relative crystallinity), single measurement.

<sup>d</sup> Alkaline viscosity, triplicate measurement, standard deviation ±0.025.

<sup>e</sup> Differential scanning calorimetry, duplicate measurement.

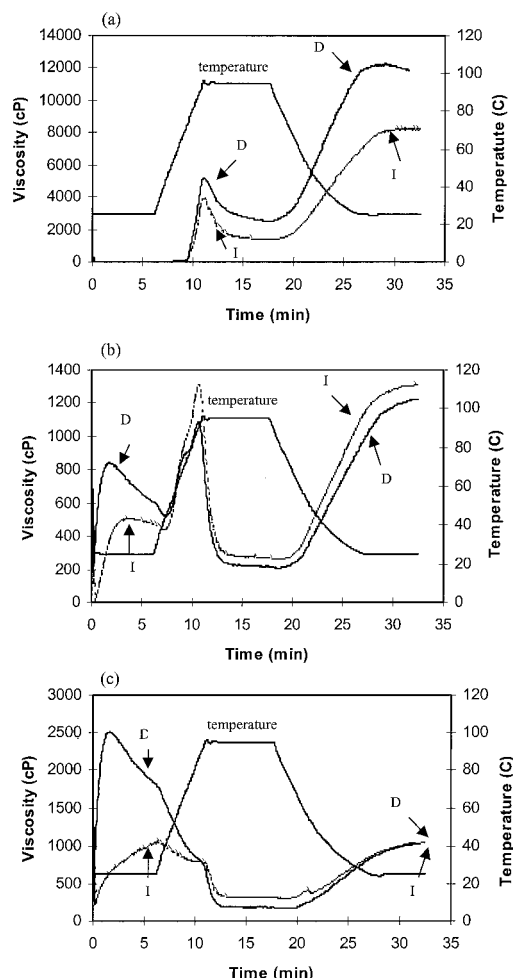
viscosity as the temperature increases to the gelatinization range. Final viscosities are  $\approx 1,000$  cP compared with 10,000 cP for the native sample. For the extruded wheat sample, the highest viscosities were observed at low temperatures, presumably due to cold water swelling of the particulates. The final viscosities for the samples were approximately the same as those for the extruded maize samples. These results would confirm the findings of others that the RVA is a sensitive method for distinguishing between samples processed differently.

Despite using the same sieve size fraction for both mills (125–212  $\mu\text{m}$ ) there are large differences between the RVA profiles (Fig. 3) for the samples prepared from the two different mills. The samples indicating the greatest difference due to milling are the wheat ball pellets, where the cold viscosity peak from the disk-milled sample is more than twice that from the impeller mill (Fig. 3c).

Clearly, the differences due to milling are significant and ought to be understood in some detail to ensure that the secondary changes caused by milling are not interpreted as starch conversion due to the initial processing.

Both mills cause some moisture loss and this is typically less for the disk mill than for the impeller mill. Table III indicates that milling and sieving raw maize samples can cause a 37% loss of moisture. Large moisture losses were also observed when processed samples of both maize and wheat were milled (data not shown). Again, the higher moisture loss was associated with the impeller mill.

The actual moisture contents of the milled samples were taken into account for the samples weights used for the RVA analysis. Hence, the particle sizes of samples used for the comparison of the mills are of the same small and narrow range (125–212  $\mu\text{m}$ )



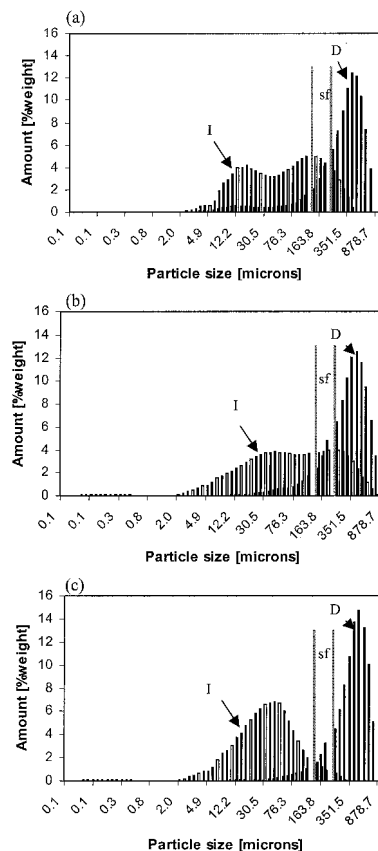
**Fig. 3.** Effect of milling on RVA profiles of raw maize grits (a), extruded maize pellets (b), and extruded wheat pellets (c) milled with a disk mill (D) and impeller mill (I). RVA profiles using sieve fraction of 125–212  $\mu\text{m}$ .

and samples have the same solids content, yet the mills cause major variations in the profile. Variations in RVA profiles may be due to several factors: particle-size distribution was not the same within the range; particle shapes were different; chemical composition of the samples for the two mills was different; or the mills changed the particle components, particularly the starch.

### Particle-Size Distribution and Shape

The particle sizes produced from the impeller and disk mills for raw maize grits in the 125–212  $\mu\text{m}$  sieve fraction were checked using the Malvern Mastersizer. The samples were mixed with isopropanol and sonicated before analysis. It is possible that the particles might swell in the alcohol and this would influence the size. Aggregated groups of particles could become disassociated in the liquid environment compared with the dry conditions used in sieving. However, physical breakup of the aggregates could be expected in the RVA where there is liquid present and the sample is stirred.

Figure 4 shows the corresponding particle-size distribution patterns determined with LALLS for samples from two mills. The average particle size produced by the impeller mill is much smaller than that produced by the disk mill. The 125–212  $\mu\text{m}$  size range is indicated in Fig. 4 at the lower end of the disk-milled sample and on the upper end of the impeller-milled sample. As a consequence, the  $D(n, 0.5)$  values were markedly different for the two sieved samples. The disk-milled sample was much greater ( $D(n, 0.5) \approx 298 \mu\text{m}$ ) than the impeller-milled sample ( $D(n, 0.5) \approx 170 \mu\text{m}$ ). It is also a very small fraction,  $\approx 10.6, 12.4, 8.1\%$  for the impeller mill, and  $\approx 19, 15.3, 5.9\%$  for the disk mill for the raw maize grits, extruded maize, and extruded wheat, respectively, of the whole sample. It is obviously poor practice to use such a small proportion of the sample for analysis. The particles that represent the small size end of the distribution are probably those that are



**Fig. 4.** Particle-size distribution of raw maize grits (a), extruded maize pellets (b), and wheat pellets (c) milled with impeller mill (I) or disk mill (D). Dotted lines indicate sieve fraction (sf) of 125–212  $\mu\text{m}$  used for RVA.

the easiest to reduce in size or are the fragments of the largest particles. While at the other end of the distribution, the particles that are most difficult to reduce in size will be represented. The two aliquots used for the analysis, therefore, could have very different physical characteristics. Moreover, the particles may differ from one from another in chemical composition.

A range of sieved impeller-milled samples were investigated to determine whether there was a simple relationship between particle size and pasting curves. Figure 5 shows that particle size does influence the viscosity values when pasting milled samples of raw maize grits, extruded maize pellets, and wheat pellets. The relative size of the peaks, the final shape of the peak, and the shape of the curve depended on the size fraction of material. For all the samples, the greater the particle size, the higher the viscosity at the end of the RVA profile. For extruded materials, the large particle sizes (>250  $\mu\text{m}$ ) produced the highest peaks in the early part of the pasting profile. In contrast, for the raw maize grits, the greatest final viscosity came from the large particle size aliquot. For this native sample, the initial gelatinization peak is delayed, occurs lower, and shows little breakdown compared with the profiles from smaller size particles.

It is, therefore, apparent that prediction of the RVA profile could not be achieved just from knowledge of particle size, knowledge of the sample is also required. The differences between the profiles using the two mills (Fig. 3) would indicate that there does seem to be a greater difference in the profiles of samples that would be considered the most difficult to mill. The wheat ball pellets are particularly difficult to reduce to a small particle size.

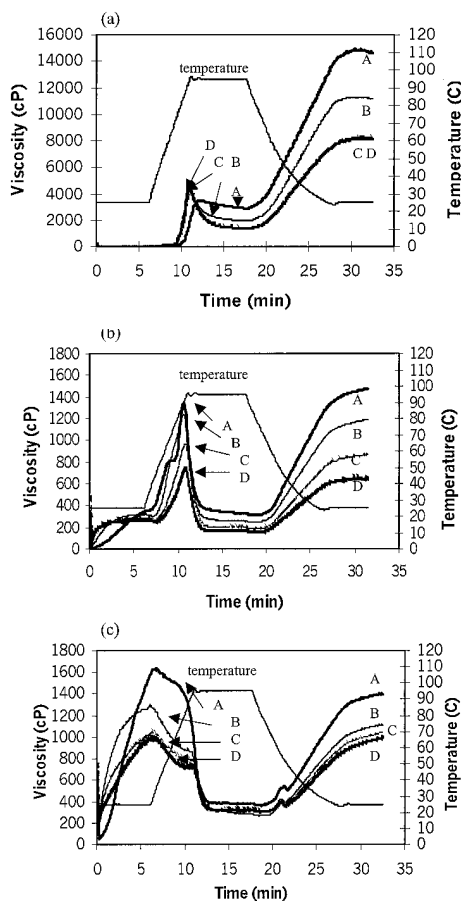
The size reduction methods of the disk mill and the impeller mill are somewhat different. Within the disk mill, particles are broken along lines of weakness by impact and shearing forces; the

resultant particles are typically not very small and with poor uniformity of particle size. Particles milled with the impeller mill are forced against an abrasive ring by the high-speed rotating impeller; therefore pieces of the material are worn away from the bulk material. Another factor is the shape of the particles. Water uptake and swelling behavior of a particle is influenced by the particle shape and, hence, will influence the RVA curves.

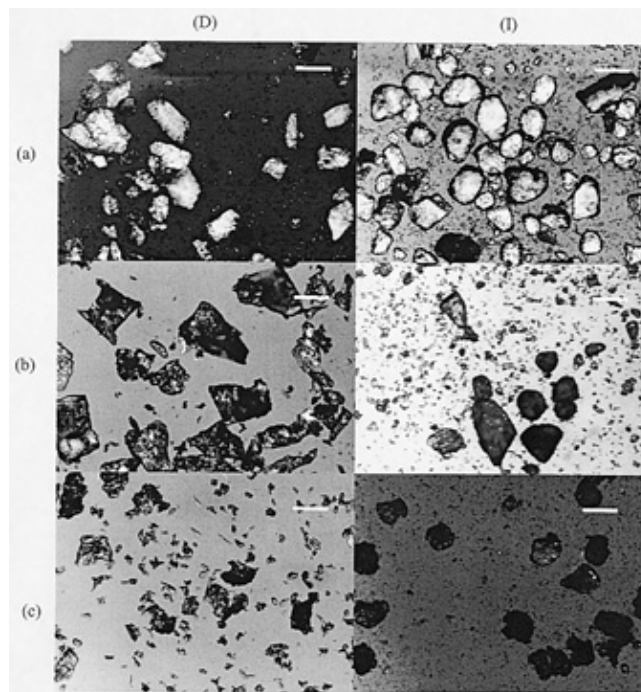
Figure 6 shows polarized light micrographs of particles of raw maize grits, and maize and wheat extrudates milled with the disk mill or impeller mill. A polygonal form with sharp edges characterized the shape of particles milled with the disk mill. This may indicate a straight breakage through the particle once the force was applied. The same sample milled with the impeller mill formed larger particles that were of a rather round shape accompanied by small particulates. Clearly the use of the mill will give rise to different sized and shaped particulates and these will influence the RVA pasting profile.

### Chemical Composition of Sieve Fractions

The low recoveries achieved for the common sieve fraction of 125–212  $\mu\text{m}$  may indicate different portions of the sample within the RVA, thus the protein of raw maize grits sieved fractions was determined. The nitrogen content of raw maize grits (given as g of N/100 g of sample) increased with increasing particle size (Table II). A direct comparison of the 125–212  $\mu\text{m}$  sieve fractions showed a higher nitrogen content for impeller-milled samples. Using  $N \times 6.5$ , to ascertain the protein levels in the fractions, the impeller-milled 125–212  $\mu\text{m}$  sieved fraction samples would have 7.81% protein while the equivalent disk-milled sample would have 7.31%. This represents a difference of 0.02 g of protein in the 4-g sample used for the RVA measurement. The additional 0.02 g of protein would mean that there would be less starch and this variation in the starch level could be detectable in the RVA profile and would result in lower viscosity readings (Almeida-Dominguez et al 1997). The sample with the higher protein levels is the sample producing the lower viscosity profiles (Fig. 3). However, the differences observed for the raw maize grits prepared by the two



**Fig. 5.** Effect of particle size on RVA profiles of raw maize grits (a), extruded maize pellets (b), and extruded wheat pellets (c) milled with impeller mill. RVA profiles of sieve fractions >250  $\mu\text{m}$  (A), 212–250  $\mu\text{m}$  (B), 125–212  $\mu\text{m}$  (C) and <125  $\mu\text{m}$  (D).

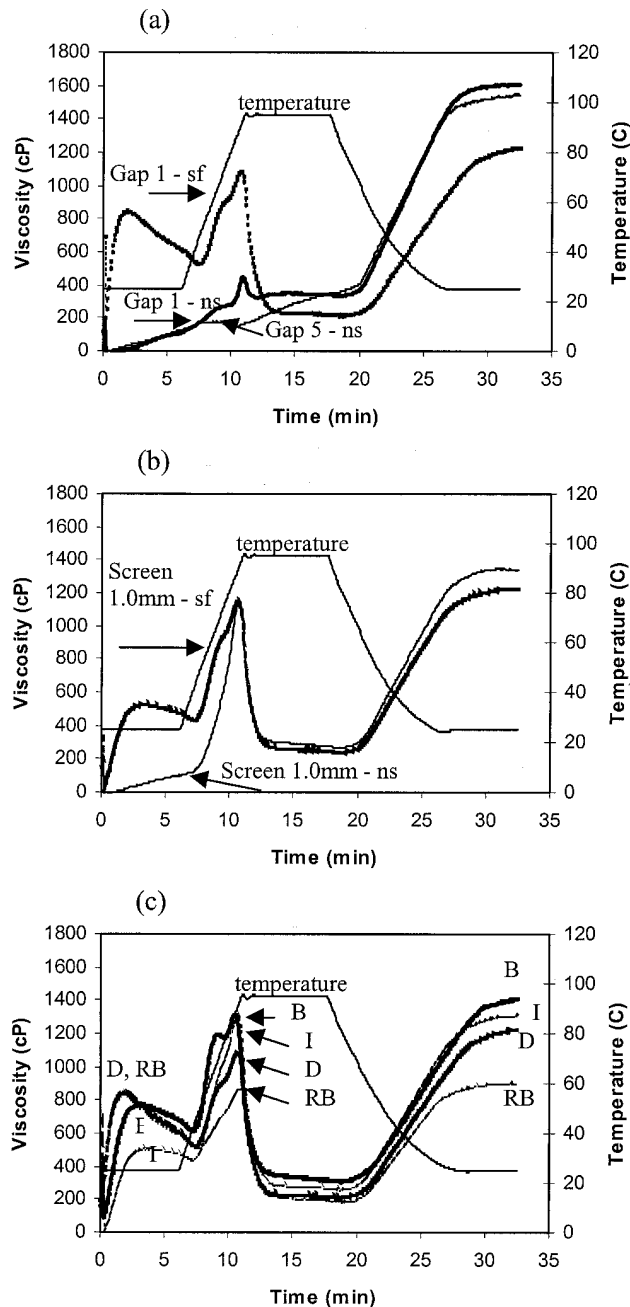


**Fig. 6.** Polarized light micrographs of raw maize grits (a), extruded maize pellets (b), and wheat pellets (c), milled with the disk mill (D) or impeller mill (I). Raw maize grits and extruded wheat pellets from sieve fraction 125–212  $\mu\text{m}$ ; extruded maize pellets from nonsieved sample. Samples are suspended in isopropanol. Magnification 40 $\times$ . Bar = 0.1 mm.

different mills are greater than can be explained by 0.02 g less of starch. The difference in starch would be equivalent to  $\approx 30$  cP at the peak viscosity. For the extruded material, the protein and starchy components would be expected to be well integrated and would not expect to segregate on milling and sieving. Therefore, the change in composition due to the different milled regimes cannot totally explain the differences in viscosity profile.

### Starch Damage

A proposed use of the RVA is to determine the amount of starch conversion or the degree of cook that has occurred during the processing of starchy materials. However, grinding treatments can induce physical conversion of the starch granules causing a change of properties of starch similar to gelatinization (Lelievre 1974).



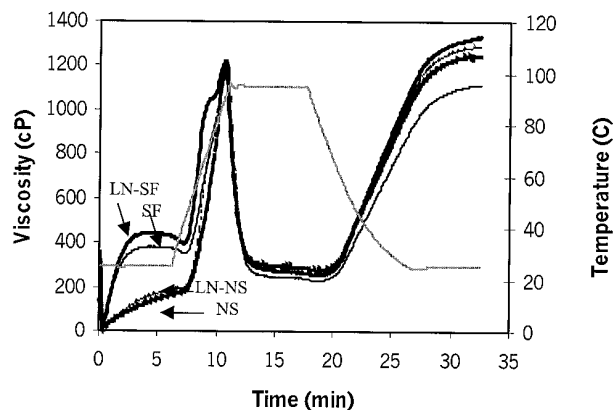
**Fig. 7.** Comparison of RVA profiles of sieve fraction (sf) 125–212  $\mu\text{m}$  and nonsieved (ns) extruded maize pellets emphasizing the importance of sieving (a,b). RVA profiles of extruded maize pellets (sieve fraction 125–212  $\mu\text{m}$ ) from disk mill (D), impeller mill (I), blade mill (B) and rotor beater mill (RB) (c).

The effects of mechanical damage summarized by Evers and Stevens (1985) include increased capacity to absorb water, increased susceptibility to amylolysis, and loss of organized structure (manifested as loss of X-ray pattern, birefringence, DSC gelatinization endotherm).

Samples from the two different mills were investigated for parameters indicative of starch conversion. Table III summarizes the results for raw maize grits, while the extruded samples showed no DSC endotherms and little X-ray order.

A notable difference is the drop of moisture content that occurred after the impeller milling. This was taken into account for all the assays, including the X-ray samples, which were allowed to equilibrate before testing.

The relative crystallinity values of raw maize grits were higher than those for the milled samples and the 125–212  $\mu\text{m}$  fraction showed less crystallinity than the total sample. Gelatinization en-



**Fig. 8.** Effect of cooling with liquid nitrogen (LN) during impeller milling on RVA profiles of maize extrudates, nonsieved (ns), and sieve fraction (sf) 125–212  $\mu\text{m}$ .

**TABLE IV**  
Mean Particle Size and Amount of Material Collected by Sieving 125–212  $\mu\text{m}$  of Extruded Maize Pellets Milled with Disk or Impeller Mill at Varying Mill Settings

Gap Setting <sup>a</sup>	Disk Mill		Impeller Mill		
	Median (D50) ( $\mu\text{m}$ )	Material <sup>b</sup> (%)	Screen Aperture Size (mm)	Median (D50) ( $\mu\text{m}$ )	Material <sup>b</sup> (%)
1	369	2.83	0.5	68	19.14
2	484	2.67	0.8	120	19.84
3	577	1.98	1.0	206	17.17
4	668	1.46	2.0	281	12.41
5	672	1.36	No screen	93	11.51

<sup>a</sup> Width of gap between two grinding disks is set by a simple crank adjustment typically using a scale of 1 graduation = 0.01 mm.

<sup>b</sup> Weight (%) of material at 125–212  $\mu\text{m}$ .

**TABLE V**  
Effect of Cooling with Liquid Nitrogen During Milling with Impeller Mill on RVA Profiles of Maize Extrudates

Sieve Fraction	M <sup>a</sup> (% db)	RVA Viscosity <sup>b</sup>		
		Peak (cP)	Final (cP)	X-ray <sup>c</sup>
125–212 $\mu\text{m}$				
Liquid nitrogen	14.06 $\pm$ 1.33	1,202 $\pm$ 22.14	1,332 $\pm$ 36.14	2.38
Nonliquid nitrogen	10.61 $\pm$ 2.83	1,152 $\pm$ 87.68	1,109 $\pm$ 12.02	1.90
Nonsieved				
Liquid nitrogen	13.77 $\pm$ 1.13	1,189 $\pm$ 29.60	1,297 $\pm$ 22.28	2.33
Nonliquid nitrogen	10.53 $\pm$ 2.72	1,221 $\pm$ 102.53	1,246 $\pm$ 184.55	2.21

<sup>a</sup> Moisture, triplicate measurement.

<sup>b</sup> Triplicate measurement.

<sup>c</sup> X-ray (% relative crystallinity), single measurement.

thalpies of impeller-milled raw maize grits measured by DSC were much lower than the unmilled or the disk-milled samples. The impeller-milled sample also showed a lower alkaline viscosity compared with the disk-milled sample. This suggests that the impeller mill caused some polysaccharide degradation.

### Choice of Mill

The previous results indicate that the grinding mechanism of a mill determines the properties of the particles. The particle size, size distribution, shape, surface, starch damage, and starch concentration are all influenced by the milling procedure adopted.

The question that must be addressed is: "What is the right mill to use for grinding samples, particularly processed samples before RVA measurements?" The disk mill grinds the particles between two rotating disks, resulting in less starch damage than the impeller mill. The viscosity profiles for the maximum and minimum gap settings for the disk mill are shown in Fig. 7a. The profiles are different one from another in that gap 1 shows a specific peak viscosity that is not obvious with the gap 5 setting. Also shown in Fig. 7a is the equivalent profile as for gap 1 but on a sieved sample. The profile is markedly different, it has a high cold swelling peak, a peak maximum, and low final viscosity compared with the nonsieved fractions. The major differences between the sieved and nonsieved fraction is also shown for the impeller mill (Fig. 7b). Our results concur with the general recommendations that samples should be sieved for reproducible RVA viscosity profiles. However, the problem remains as how to produce enough of the sample at the correct size to represent the whole of the sample. The portion of sample produced through the disk mill that had a sieve size of 125–212  $\mu\text{m}$  represented a minor fraction of the total sample and was at the smallest particle tail end of the distribution. Varying the disk gap determines the particle-size distribution (Table IV) and the majority of the our data corresponded to the smallest gap setting ( $D(n,0.5)$  369  $\mu\text{m}$ ). Hence, to achieve a substantial portion of the sample at the appropriate size, repeated grinding would seem to be unavoidable, although this would cause further starch damage.

The impeller mill grinds particles by smashing them against and shearing them along an abrasive grinding ring until they pass a screen of determined aperture size. Hence, the screen aperture size determines the time a particle is milled in the grinding chamber before it is small enough to exit. Typically, a screen of 0.5-mm aperture size was used for the comparison, resulting in particle-size distributions of which the sieve fraction of 125–212  $\mu\text{m}$  was at the upper end. Milling maize extrudates with a screen of 0.8 or 1.0 mm provided particle-size distributions most representative of the sieve fraction (Table IV). Yet the amount of product at the correct sieve size was <20%. Newport Scientific recommends using a screen of 1.0 mm aperture size for milling extrudates. However, our results show that the impeller mill caused significant starch damage in addition to the starch conversion caused by extrusion or other process.

During the milling, heat was generated and temperatures of  $\leq 50^\circ\text{C}$  were observed while grinding pellets with the impeller mill. Various cooling techniques were applied including cooling the sample while grinding with liquid nitrogen. None of the cooling regimes was successful in preventing the changes in the RVA profiles, suggesting that the thermal starch damage does not play a major role in causing the starch conversion. Figure 8 shows the effect of cooling with liquid nitrogen during milling on RVA profiles of maize extrudates. It indicates that the RVA profiles for the samples cooled with liquid nitrogen are very similar to those without the coolant. There was no significant difference between the X-ray crystallinity when nitrogen was used to cool the samples, but the presence did reduce the moisture loss from the sample (Table V).

Clearly both the disk mill and the impeller mill produced samples that gave RVA profiles that were very reproducible using a

strict protocol. Using the same protocol RVA profiles would reflect changes in the processing conditions used to prepare the cereal material. However, data produced using different mills cannot be compared directly to give information on the starch quality. Neither the disk mill nor the impeller mill gave high percentages of particles at the required size of 125–212  $\mu\text{m}$  and both inflicted additional damage to the starch.

Other mills were investigated to see whether any fulfilled the requirements. A knife mill and a cross beater mill were used to grind maize extrudates and some of the results are shown in Fig. 7c. The viscosity profiles were different one from another and different from those using the other two mills.

## CONCLUSIONS

Grinding is more than a step in sample preparation of extrudates prior to RVA measurements. It should rather be considered as further parameter affecting the RVA profile.

Properties of the ground flour extrudates, resulting from the milling process, influenced the pasting profile as recorded by the RVA. The effect of milling can be even more obvious than the degree of starch conversion resulting from extrusion processing. Hence, once a mill is set up for the job, the same mill and milling conditions should be used for all experiments to be compared.

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