

## MICROSTRUCTURE AND MECHANICAL PARAMETERS OF FIVE TYPES OF STARCH

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Key words: mechanical properties, flowability, shear test, uniaxial compression, starch, food powders

Research was conducted to recognize interrelations between morphology and mechanical properties of starch. Microscopic examination, determination of particle size distribution, as well as direct shear and uniaxial compression testing were performed on five types of starch extracted out of: potato, wheat, corn, tapioca and amaranth. Regarding geometrical features (morphology) of granules, three distinct groups of materials were distinguished. Classification of materials based on the results of mechanical testing was found in a close agreement with classification based on morphology. Potato and wheat starches that had relatively large granules ( $d(0.5)$  of 41.5 and 20.2  $\mu\text{m}$ ) and bimodal particle size distribution showed stress-strain curves with fluctuations, particularly high in the case of potato starch. Tapioca and corn starches had smaller granules of similar sizes ( $d(0.5)$  of 15.6 and 13.8  $\mu\text{m}$ , respectively). The uniaxial compression stress-strain curves of the two materials were also very similar, as well as their angles of internal friction. Granules of amaranth starch with  $d(0.5)$  of 3.0  $\mu\text{m}$  were several times smaller than those of corn and tapioca starches (parameter  $d(0.5)$  is the size in microns at which 50% of the sample is smaller and 50% is larger). As a result, amaranth starch was characterised by relatively weak compressibility and flowability, the lowest of all the materials tested.

### INTRODUCTION

Starch, the main storage component in multiple plants, is not only an important energy source for developed seedling, but also a valuable material for food and non-food industries due to its unique structure-forming properties. Cereals, pseudo-cereals, legumes, roots and tubers are the raw materials for starch extraction. The most important source of starch is corn. In the United States (producer of more than 60% of the total world starch), it is almost the sole industrial material for starch extraction. In the whole world the share of starch from corn is about 83%, followed by wheat (7%), potato (6%) and tapioca (4%). In Europe an increase in the use of wheat as raw material for starch production has been observed recently. The reason of this is that its cultivation is favoured by the climatic conditions, it is available at a relatively low price, and finally, exploitation of by-products of starch extraction (e.g. gluten) is possible [Baere, 1999]. In the EU 15, where the total starch production is approximately  $8.0 \times 10^6$  tons per year, starch from wheat is produced in the amount of  $2.8 \times 10^6$  tons. Nearly 53% of starch total production is used in the food sector (sweets – 18%, soft drinks – 11%, other food – 24%). Of the non-food sector (total share of 46%), 28% is used for production of paper, cardboard and corrugated board, and 13% is used for fermentation [Bergthaler, 2004].

Technological processes of starch production usually differ depending on the raw product, but in general comprise such basic unit operations as: cleaning of material, steeping (in the case of corn), rasping (potato), addition of substrates (as e.g. sulphur dioxide, sulphuric acid or sodium bisulphite

solution), separation of anatomical parts (germ, seed coat), washing of ground material (extraction), dewatering, drying, refining, sifting and packaging. Refining (using centrifuges or hydrocyclones) is a very important phase of the technological process that is based on differences in specific density of starch (1.55  $\text{g}/\text{cm}^3$ ), cell walls (1.05), water (1.00) and mineral impurities ( $>2$ ). Starch dried in moderately hot air (potato to 19% of moisture content, cereal starches to 13% m.c.) is pneumatically transported to a silo for additional screening and bagging [TM5-2www ISI; TM18-2www ISI].

Diversity of raw materials used for starch extraction leads to obtaining a broad spectrum of starches, varying in particle size distribution, morphology and physicochemical properties. According to the size of individual granules, starches may be grouped into four classes following Lindeboom *et al.* [2004]: large – above 25  $\mu\text{m}$ , medium – from 10 to 25  $\mu\text{m}$ , small – from 5 to 10  $\mu\text{m}$ , and very small – below 5  $\mu\text{m}$ . In some cases, bimodal size distribution (predominantly small and large granules) is observed. Granules of potato starch belong to the first class, most of cereal starches show bimodal size, oat, buckwheat, rice millet represent the class of small starch. Starches of very small granules are obtained, among others, from amaranth, taro, quinoa, or cow cockle. Considering their morphology, starch granules may be classified as oval, oblong, spherical, polygonal, irregular or lens-shaped. The size distribution of granules in a specimen may be the main factor responsible for properties of starch in bulk (aggregation, clustering) that influence its behaviour during transportation and storage, which in turn may affect the quality of the product.

Parameters of internal friction and flow properties of powders influence handling and processing operations such as flow from silos and hoppers, transportation, mixing, compaction or packaging [Knowlton, 1994]. With increasing scale of industrial operations, there is a growing demand for information about food powder parameters to design reliable processes and efficient equipment. The flow characteristics of food powders have recently gained special importance as measures of quality of final product on-line, as well as during later handling and on-shelf storage [Molenda & Stasiak, 2002].

Flow in silos can take the form of one of two patterns: mass or funnel flow [Jenike, 1964]. During mass flow, all the powder is in motion and moving downward towards the discharge opening, while in the case of funnel flow powder discharges through a flow channel formed within the grain bulk and no sliding along the wall takes place. A serious industrial problem that may occur during technological operations on food powders is cessation of flow. This is usually a result of an arch forming across the discharge opening, which has strength sufficient to be self-supporting. Jenike [1964] proposed the theory of flow of granular material and methods of determination of material parameters, including the shear cell technique, for the determination of powder flow properties. Based on results of two-dimensional stress analysis, this author introduced the method of estimation of the minimum hopper opening dimension for mass flow from conical and wedge shaped hoppers. The design requires the determination of the following material characteristics: the flow function  $FF$ , the effective angle of internal friction  $\delta$  and the angle of wall friction  $\varphi_w$ . The flow function is a plot of unconfined yield strength  $\sigma_c$  of the powder against major consolidating stress  $\sigma_1$ , and represents the strength of the consolidated powder that must be surpassed to initiate flow of the powder. Regarding the values of flow function, powders may be characterised as free flowing, easy flowing, cohesive and strong cohesive. Based on linearized flow function, the flow index  $i$  is defined as the slope of the flow function.

A typical application of the flow function as a material characteristic in industry is quality assessment of powders [Bell *et al.*, 1994]. It has been reported by a number of researchers that powder morphology strongly influences flow properties of powders. Fitzpatrick *et al.* [2003] determined flow properties of 13 food powders of various particle sizes, moisture contents, bulk densities and particle densities using an annular shear cell. Based on the results obtained, the authors categorized materi-

als in groups from easy flowing to very cohesive. These authors concluded that particle size distribution and moisture content markedly influenced flowability, but no strong enough relationship was found to relate the flowability of the food powders based solely on these physical properties. It was also stated that surface forces between particles influence flowability to a considerable extent. Teunou *et al.* [1999] reported results of flowability determination in an annular shear tester for 4 food powders and discussed possible relationships between flowability and physical properties and relative humidity of surrounding atmosphere. The authors presented an evaluation of the effect of storage time and consolidation on the flowability of the food powders. All food powders tested demonstrated a time-consolidation effect such that their flowability was reduced with an increase in consolidation time.

Currently a number of methods and testers are available to determine the strength and flow properties of bulk solids. Choosing the right method for the specific application requires knowledge and some experience in handling bulk materials, as outlined by Schwedes [2002]. Most frequently, the flow properties of bulk solids have been determined by performing a shear test following slightly modified procedure proposed by Jenike [1964]. The testing consists of two stages: consolidation under normal reference pressure and measurement of shear force. Repeatability of the test results may be attained only if the consolidation was identical [Schwedes, 2002].

Starch is one of the major components in the diet and plays an important role in the formulation of food products, with respect to both food functionality and nutritional quality. Up to date a lot of information is available on chemical nature and physicochemical properties of different starches, but data on their physical properties are scarce. Investigations should be conducted to gain more information on properties of starches of different botanical origin that can be interesting for both engineers and technologists.

The objectives of the reported project were: a) determination of mechanical characteristics and parameters of five kinds of starch, b) characterization of morphology (form and structure) and particle size distribution of examined materials, and c) an attempt to find possible relationships between morphology and mechanical behavior of the examined materials.

## MATERIALS AND METHODS

The materials examined were starches of potato, wheat,

TABLE 1. Proximate composition of starches from different biological sources and their selected physicochemical properties (according to: Juszcak *et al.* [2003a,b], Walkowski *et al.* [1997], Lingeboom *et al.* [2004]).

Starch source	Amylose [Walkowski <i>et al.</i> , 1997]	Protein [Walkowski <i>et al.</i> , 1997]	Fat [Walkowski <i>et al.</i> , 1997]	Granule diameter [Lindeboom <i>et al.</i> , 2004; Walkowski <i>et al.</i> , 1997]	Specific [Juszcak <i>et al.</i> , 2003a,b]		Mesopore [Juszcak <i>et al.</i> , 2003a,b]	
	%	%	%		Density (g/cm <sup>3</sup> )	Surface (m <sup>2</sup> /g)	Volume (cm <sup>3</sup> /g)	Diameter (nm)
Potato	22.5	0.06	0.05	5–100	1.52	0.24	0.35	5.72
Wheat	27.1	0.40	0.80	1–45	1.51	0.53	0.76	5.70
Corn	28.0	0.60	0.44	2–30	1.50	0.69	1.10	6.42
Tapioca	17.3	0.10	0.10	4–35	1.51	*	*	*
Amaranth	4.0	0.53	0.27	0.75–2.0	*	*	*	*

\* – data not available

corn and tapioca available on the local market and purchased by Starch and Potato Research Laboratory of Potato Industry, Luboń, Poland, and amaranth starch extracted by the method described in details by Walkowski *et al.* [1997]. The former starches were chosen due to their share in the world market, while amaranth starch was tested because of increasing interest of the food and non-food industry in small granule starches. Selected physicochemical properties of starches used for this study, reported earlier in literature, are shown in Table 1.

**Description of morphology of powders.** Microscope investigations were performed using the JSM 5200 microscope at 10 keV. Specimens were placed on double-side adhesive tape mounted on aluminium specimen holders, coated with gold in a vacuum evaporator JEOL 400. Particle size distribution was analysed using Mastersizer 2000 produced by Malvern, with water as dispersant.

**Determination of mechanical characteristics.** Experiments were performed in a direct shear tester and in a uniaxial compression tester for four powders: corn starch, wheat starch, tapioca starch, potato starch and amaranth starch. Direct shear tests were conducted using an apparatus 60 mm in diameter. Tests were performed following the standard procedure for consolidation reference stresses  $\sigma_r$  of 10, 20, 30, 60 and 80 kPa and speed of shearing  $V$  of 2 mm/min. For determination of yield locus, values of maximum shear stresses at two levels of consolidating stress  $\sigma_r$  and  $1/2 \sigma_r$  (Figure 1) were used, following the recommendations of standards [Eurocode 1 2003; Polish Standard PN-B-03262-2002]. With yield locus determined, Mohr circles were drawn that gave values of unconfined yield strength  $\sigma_c$  and major consolidating stress  $\sigma_1$ . Relationship between these two parameters,  $\sigma_c(\sigma_1)$  is termed the flow function, *FF*, of the material (Figure 2). The flow function characterises the ability of a powder to flow.

Uniaxial compression tests were performed following Eurocode 1 [2003] in a 54 mm diameter apparatus (Figure 3) with a 47 mm high sample. During the test, the top cover of the apparatus was moving down with a constant speed of 1.5 mm/min until the vertical reference stress  $\sigma_z$  of 80 kPa was reached. After reaching the prescribed level of  $\sigma_z$ , movement of the top cover was stopped and unloading took place with the same speed of deformation until the stress level of 0 kPa was detected.

Both direct shear and uniaxial compression tests were repeated three times.

**RESULTS**

**Microstructure of starch granules**

The results of particle size analysis are presented in Table 2. The parameters are defined following Malvern – Operators Guide [1999]. The parameter  $d(0.5)$  is the size in microns at which 50% of the sample is smaller and 50% is larger,  $d(0.9)$  gives the size of particle below which 90% of the sample lies. The specific surface area is defined as the total area of the particles divided by the total weight, based on the assumption that particles are both spherical and non-porous. Surface weighted mean is  $\Sigma d^3/\Sigma d^2$ , and volume weighted mean is  $\Sigma d^4/\Sigma d^3$ . Span is the measurement of the width of the distribution. The narrower the distribution, the smaller the span

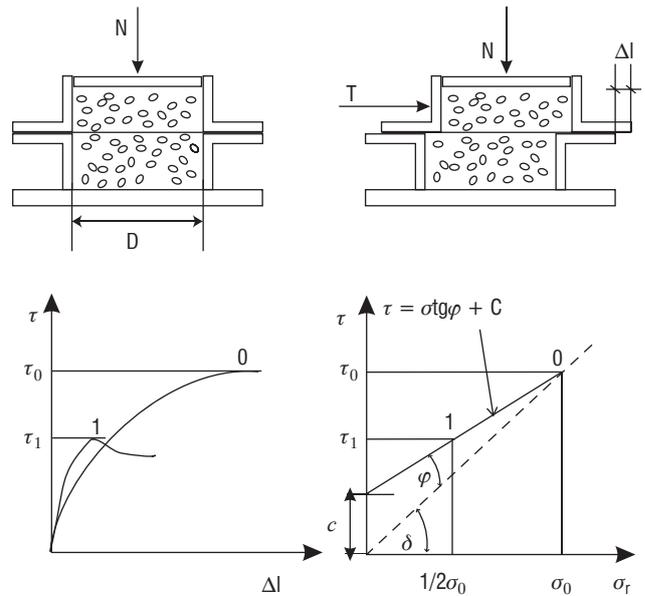


FIGURE 1. Direct shear apparatus and interpretation of experimental data following Eurocode 1:  $\tau$  – shear stress [kPa],  $\sigma_r$  – consolidation reference stress [kPa],  $\Delta l$  – relative displacement.

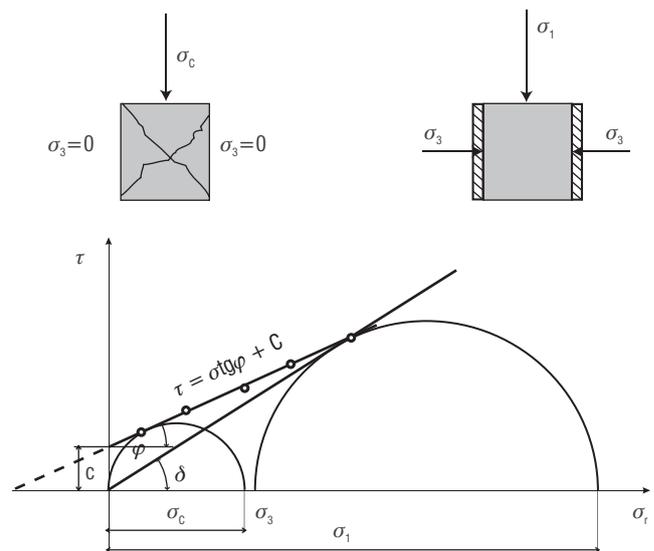


FIGURE 2. Determination of flow function  $FF = \sigma_c(\sigma_1)$  of powder.

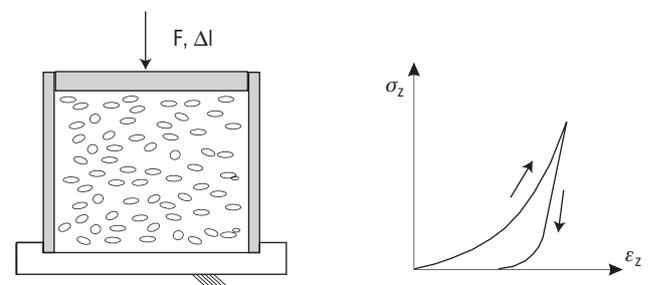


FIGURE 3. Uniaxial compression apparatus.

becomes.

Particle size analysis showed that potato starch had the largest granules with  $d(0.5)$  two times higher than the next in the order, wheat starch. Corn and tapioca starches had relatively close values of parameters of distribution, while granules

TABLE 2. Particle size analysis of starches.

Starch	Diameter d(0.5) ( $\mu\text{m}$ )	Diameter d(0.9) ( $\mu\text{m}$ )	Specific surface area ( $\text{m}^2/\text{g}$ )	Surface weighted mean ( $\mu\text{m}$ )	Volume weighted mean ( $\mu\text{m}$ )	Span $d(0.9)-d(0.1)/d(0.5)$
Potato	41.5	72.0	0.315	19.07	43.82	1.22
Wheat	20.2	32.49	0.564	10.64	20.63	1.10
Corn	13.8	21.49	0.809	7.419	14.00	1.00
Tapioca	15.6	26.14	0.801	7.491	16.10	1.19
Amaranth	2.97	6.59	2.75	2.185	3.48	1.86

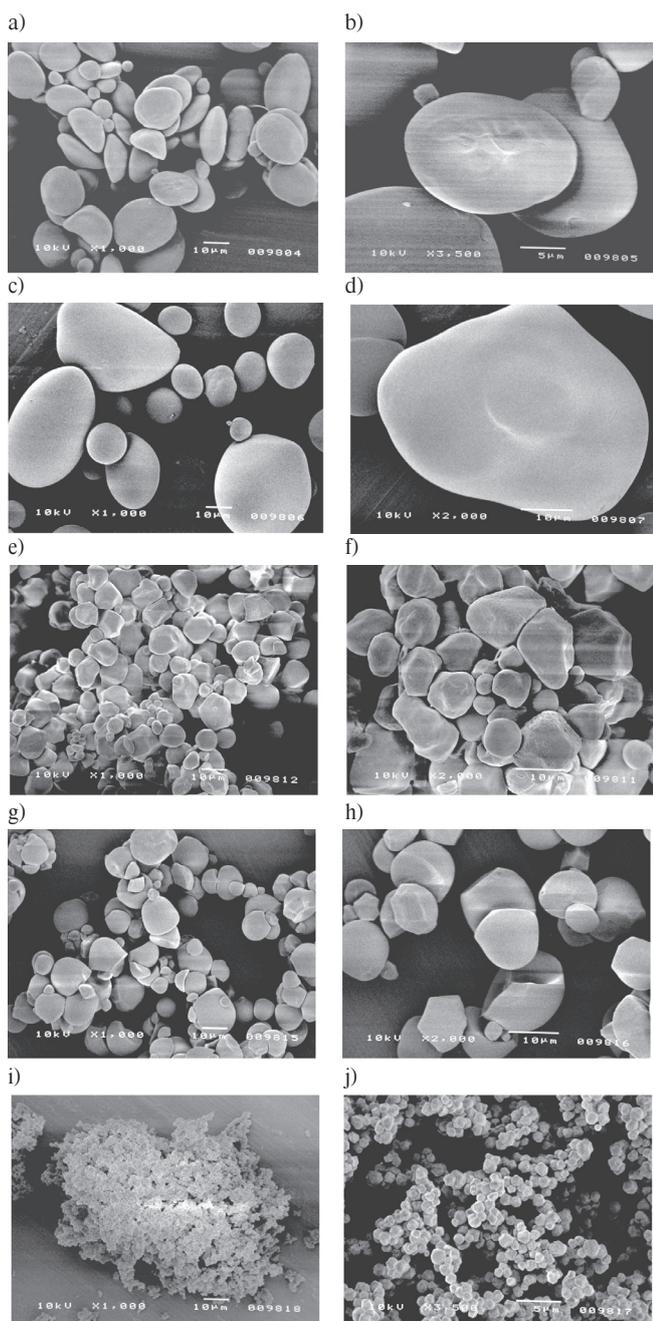


FIGURE 4. Microscope images of tested starches: a, b – potato; c, d – wheat, e, f – corn; g, h – tapioca; i, j – amaranth

of amaranth starch were approximately five times smaller.

Microscopic images of structure of the investigated starch granules are presented in Figure 4. Regarding the shape, the

granules are typical to photographs presented elsewhere. Granules of potato starch belong to the class of large and are clearly bimodal. The clear differences in shape and diameter are visible in Figure 4a. The largest granules with diameter of nearly  $60\ \mu\text{m}$  are oval, round or irregular. The second group of granules, medium, has particle size from  $10$  to  $30\ \mu\text{m}$ , some of them being regularly spherical. Surface of these granules at higher magnification is rather smooth and shows some irregularities at the centre of the granule (Figure 4b). Such a large diversity in granule diameters was the means of industrial separation of potato starch due to different properties (viscosity, chemical reactivity) of the two fractions determining their usability [Szymońska & Krok, 2003].

The granules of wheat starch also represent bimodal type (Figures 4c and d). The majority of population could be classified as lens-shaped, being very flat. Granules of lower fraction are small (from  $5$  to  $10\ \mu\text{m}$ ) or very small (below  $5\ \mu\text{m}$ ) and have spherical shape. Similarly to potato starch, the separation of starch into two size classes has also been proposed [Bergthaler, 2004]. At higher magnification granules showed surface deformation and signs of mechanical damage (Figure 4d). It is worthy noticing that the orientation of lens-shaped granules in bulk (horizontal or vertical) could be a reason of the mechanical properties of starch.

The next two examined starches, corn and tapioca, represent the class of medium sized granules ( $10$ – $25\ \mu\text{m}$ ). Granules of both are irregular in shape – from spherical to polygonal with the higher share of the latter in corn starch (Figures 4e–h). At higher magnification (Figures 4f and h), numerous pits can be observed on the surface of corn granules whereas tapioca is characterised by naturally deformed or damaged ones.

The amaranth starch is unique considering the granule size. It belongs to the class of very small granule starches (Figures 4 i–j). Majority of granules have polygonal shape with a diameter of about  $1\ \mu\text{m}$ . They tend to agglomerate in round or irregular structures.

#### Direct shear testing

Relationships between shear stress  $\tau$  and relative displacement  $\Delta/D$  were obtained from direct shear tests. In the case of both potato and wheat starches, fluctuations of shear force were observed for all tested values of normal pressure, while the experimental curves of corn, tapioca and amaranth starches followed smooth paths. Typical stress *versus* relative displacement curves obtained at  $60\ \text{kPa}$  of normal stress  $\sigma$  for potato, wheat and amaranth starches are shown in Figure 5. The strongest stick-slip effects were observed in the case of potato starch where variation in shear stress had an amplitude of approximately  $12\ \text{kPa}$  or  $20\%$ . Fluctuations of shear

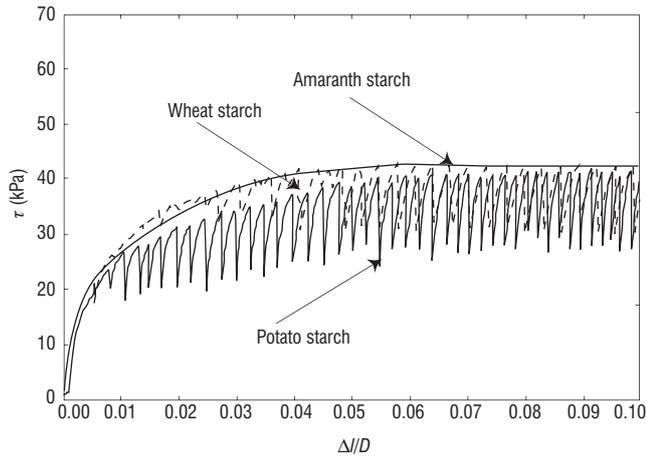


FIGURE 5. Shear stress  $\tau$  – relative displacement  $\Delta/D$  relationship of potato, wheat and amaranth starch for normal pressure  $\sigma$  of 60 kPa.

force in the case of wheat starch were less regular and had lower frequency. Slip-stick effects cause difficulties in interpretation of testing results. In the case of the reported project, frictional parameters for materials showing fluctuations were estimated using the maximum values of shear stress to obtain the largest (or the worst case) values. In terms of the theory of vibration, the measurement system may be treated as a linear system with one degree of freedom excited by displacement causing an increase in shear force. Its response depends on the ratio of elasticity and damping present in the system and on the velocity of deformation. As shown by Stasiak & Molenda [2004], it is possible to reduce the slip-stick effects by a change in the stiffness of measurement equipment.

Based on the experimental curves, the angle of internal friction,  $\varphi$ , effective angle of internal friction,  $\delta$ , and flow functions,  $FF$ , of materials were determined. Relationships between the angle of internal friction and consolidation stress  $\sigma_r$  are presented in Figure 6. The obtained values of angle of internal friction  $\varphi$  were stable in the range of the consolidation stress applied. The highest value of angle of internal friction – equal to  $38.9^\circ$  – was obtained for potato starch at 20 kPa of consolidation stress, while the lowest was the value of  $22.9^\circ$  obtained for tapioca starch at 10 kPa of consolidation stress. Relationships between angle of internal friction

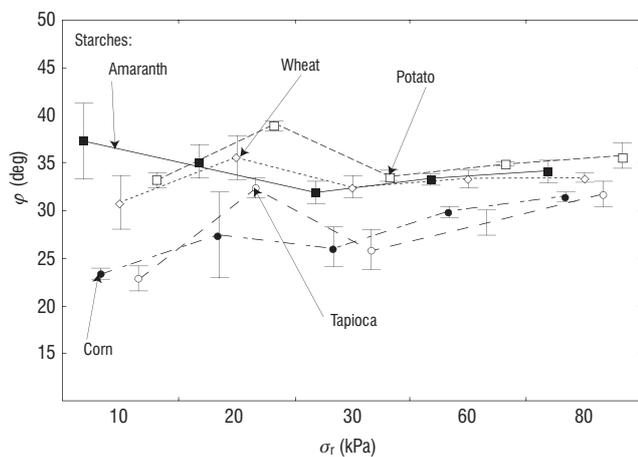


FIGURE 6. Relationships between angle of internal friction  $\varphi$  and consolidation reference stress  $\sigma_r$  for the tested starches.

tion and consolidation stress for tapioca and corn starches were located approximately 30% lower than the experimental curves for other experimental materials. Such a behavior of those two starches (tapioca and corn) is likely a result of their particle size distribution, size of the single particles and their shape and roughness. Standard deviations of the angle of internal friction were found higher for lower values of consolidation stresses as an effect of non-replicable structure of the materials under low range of consolidation stress.

Relationships between effective angle of internal friction  $\delta$  and consolidation reference stress  $\sigma_r$  are shown in Figure 7. Effective angle of internal friction was higher than angle of internal friction due to slight cohesion observed in the examined materials. The highest value of  $\delta$  of  $43^\circ$  was found for amaranth starch at 10 kPa of consolidation stress, while the lowest effective angle of internal friction of  $29^\circ$  was that of

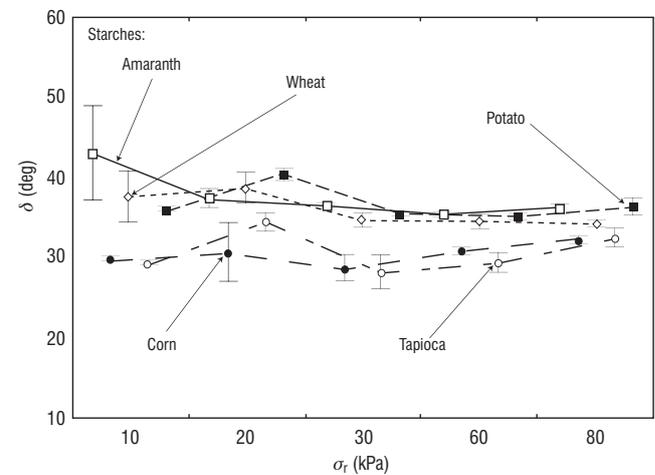


FIGURE 7. Relationships between effective angle of internal friction  $\delta$  and consolidation reference stress  $\sigma_r$  for the tested starches.

corn starch at 30 kPa of consolidation stress.

Flow functions obtained for tested starches are shown in Figure 8. The highest values and the widest range of variability (from 4 kPa to 16 kPa) of  $FF$  were obtained for amaranth starch. For the other starches, values of  $\sigma_c$  increased slower with an increase in  $\sigma_r$ . A probable reason for lower flowabil-

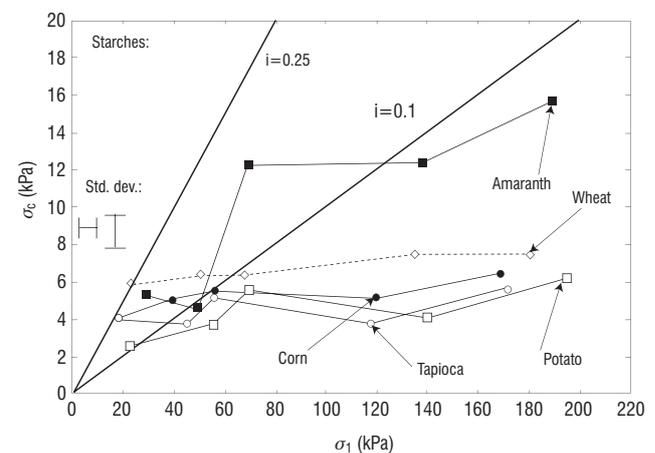


FIGURE 8. Flow functions  $FF = \sigma_c(\sigma_r)$  of tested starches.

ity of amaranth starch were distinctly lower dimensions of its granules and relatively high specific surface area of  $2.75 \text{ m}^2/\text{g}$  *i.e.* more than threefold higher than  $0.809 \text{ m}^2/\text{g}$  (Table 2), the highest of all the remaining materials found for corn starch. The higher particle surface area per unit mass provided larger surface area for cohesive forces to act, which resulted in higher cohesion and lower flowability. Flow indices *i* (*i.e.* the slopes of the *FF*) of the starches tested took values typical of free flowing and easy flowing materials.

#### Compressibility determined in uniaxial compression

Uniaxial compression tests revealed distinct differences between values of relative displacement reached at maximum consolidation stress (Figure 9). The minimum value of relative displacement equal to 0.14 was found for amaranth starch, whereas the maximum relative displacement of 0.21 was that for wheat starch. Differences between maximum relative displacement result likely from the shape and dimensions of individual particles of powders. For wheat starch, in which the highest values of relative displacement were obtained, single grains are lens shaped (Figure 4). During deformation in a uniaxial compression test, granules change their position. This explains maximal relative displacement

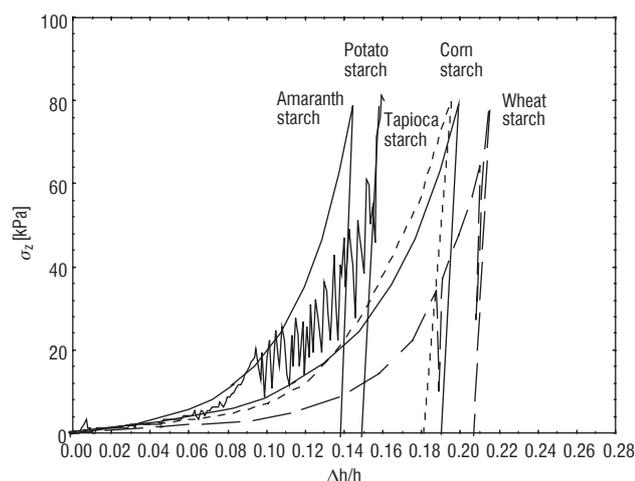


FIGURE 9. Stress strain relationships in uniaxial compression of examined starches.

obtained in the case of this material.

Regarding the mechanical properties, the starches examined may be classified into three groups: (1) corn and tapioca characterised by  $\varphi$  and  $\delta$  significantly lower than that of the other materials, (2) amaranth with the flow index distinctly higher than that of the other materials, and (3) potato and wheat. SEM images of the starch granules give certain ideas for the interpretation of results of mechanical tests. Comparison of SEM images shows that groups formed based on mechanical properties differ tangibly also in the size and shape of the granules. Potato and wheat starches have larger granules and are bimodal, which may result in unusual behaviour during compression (see Figure 4a). Two ramps up and down are seen at  $\sigma_z$  versus relative displacement curve for wheat starch, while a series of fairly regular fluctuations were observed on the experimental curve of potato starch. In

the case of potato starch, the first phase of compaction up to approximately  $0.9 \Delta h/h$  was also distinctly different, showing material hardening with slight fluctuations that reflected filling voids between large granules by small ones.

Apart from surface irregularities mentioned above (Figure 4b), much smaller structures are revealed by AFM. According to Juszczak *et al.* [2003a,b], these surface structures are  $0.7 \mu\text{m}$  in height. Szymońska & Krok [2003] observed also oblong sub-particles on the starch granule surface with a ridge and valley structure. Semi-spherical structures reported by Krok *et al.* [2000] are of  $100\text{--}130 \text{ nm}$  in diameter. Baldwin *et al.* [1998] classified surface elements as protrusions of  $50\text{--}300 \text{ nm}$  above flatter surface characterised by the presence of  $10\text{--}50 \text{ nm}$  particles. The two latter surface structures are believed to be the ends of starch chain polymers corresponding to blocklets (groups of amylopectine side-chains clusters). All these types of surface elements might influence the internal friction coefficient and thus the behaviour of bulk of starch during compression. Compared to potato, wheat starch revealed a different mechanical behaviour. Only two distinct ramps up and down appeared at the compression curve. The AFM pictures show much more smoother surface of granules with  $10\text{--}50 \text{ nm}$  structures that may result in smooth rearrangement of granules during compaction.

Stress-strain curves of corn and tapioca starches follow nearly identical paths, but distinctly different than those noted for the other three tested materials. A reason for this similar compressive behaviour is probably similar particle size distribution with grains from  $2$  to  $30 \mu\text{m}$  in the case of corn starch and from  $4$  to  $35 \mu\text{m}$  in the case of tapioca starch. The two materials have also close values of angle of internal friction and effective angle of internal friction that are significantly lower than  $\varphi$  and  $\delta$  of the other starches. Unlike the rest of the tested starches, tapioca and corn starches are characterised by an increasing angle of friction with an increase in compaction pressure  $\sigma_c$ . This is likely a result of polygonal shape of the granules that introduces plane to plane interlocking. They fit very well to each other in a “puzzle”-like mode. Interlocking results in an increase of shape component of shearing resistance that grows with an increase in normal pressure faster than linearly.

In the case of amaranth starch, the decisive factor for mechanical behaviour was the distinctly smaller size of granules, in a range from  $0.75$  to  $2.0 \mu\text{m}$  (Table 1). This resulted in a shortest of all displacement to reach  $\sigma_z$  of  $80 \text{ kPa}$  in uniaxial compression (Figure 8). Another effect of smaller size of granules is strengthening of the action of cohesive forces that resulted in the highest location of *FF* and thus the weakest flowability of amaranth starch as compared to the other examined starches.

## CONCLUSIONS

Results of the reported study confirmed the relevance of morphology to the mechanical properties of starches of potato, wheat, corn, tapioca and amaranth.

Potato starch, that had the largest granules (with  $d(0.5)$  *i.e.* size of 50% of granules smaller than  $41.5 \mu\text{m}$ ), revealed strong fluctuations of stress-strain curve (slip-stick effect) recorded during uniaxial compression. Another factor that might contribute to stronger slip-stick action in potato starch

was relatively wide particle size distribution – 93% of granules were distributed among 8 fractions from 10 to 90  $\mu\text{m}$ . Only slight fluctuations were observed in the case of wheat starch that had  $d(0.5)$  of 20.2  $\mu\text{m}$  and 97% of granules contained in three fractions from 10 to 30  $\mu\text{m}$ ; no fluctuations were present at the curves for three other tested materials. Compressibility of wheat starch was the highest of all the materials, with strain of approximately 0.21 for 80 kPa of normal pressure  $\sigma_z$ , while the strain of the sample of amaranth starch at the same  $\sigma_z$  was approximately equal to 0.15. Tapioca and corn starches that had similar particle size distributions with  $d(0.5)$  of 15.6 and 13.8  $\mu\text{m}$  showed very close stress-strain curves and compressibility. The above results point out to the important role of particle size distribution for the compressibility of starch.

Regarding the frictional properties, the starches tested may be classified into two groups, where corn and tapioca starches had angle of internal friction  $\varphi$  and effective angle of internal friction  $\delta$  significantly lower than the values of  $\varphi$  and  $\delta$  found for the remaining three starches tested. The tendency of angle of internal friction  $\varphi$  to increase with an increase in normal stress was also distinct for corn and tapioca starches, for the three remaining materials no clear tendency was observed. The two mentioned effects are probably a result of relatively narrow particle size distributions of these materials, with above 95% of granules belonging to three fractions below 30  $\mu\text{m}$ , that reduced frictional interactions between granules. In the case of amaranth starch, 98% of granules were smaller than 10  $\mu\text{m}$ , which caused high partition of cohesive component in internal friction.

In terms of flowability, the amaranth starch may be classified as an easy flowing material, while the other tested starches have been found free flowing. Thus the decrease in granules size resulted in a decrease of flowability of starch, which in practice may introduce undesirable behaviour of the material during processing. Basic effects constituting flowability remain unclear and the phenomenon requires further investigation with special focus on the sources of cohesion between granules.

#### ACKNOWLEDGEMENTS

The paper was prepared in the frame of activity of the Centre of Excellence AGROPHYSICS – Contract No.: QLAM-2001-00428, sponsored by EU within the 5FP.

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Received July 2005. Revision received November 2005 and accepted January 2006.

## MIKROSTRUKTURA I PARAMETRY MECHANICZNE PIĘCIU TYPÓW SKROBI

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Celem badań była identyfikacja związków pomiędzy cechami morfologicznymi a właściwościami mechanicznymi skrobi. Wykonano analizę mikroskopową, analizę rozkładu wielkości cząstek oraz testy jednoosiowego ściskania i bezpośredniego ścinania dla pięciu rodzajów skrobi: ziemniaczanej, pszennej, kukurydzianej, tapiokowej i amarantusowej. Pod względem budowy morfologicznej wydzielono trzy grupy materiałów. Wykazano, że zachowanie materiałów w testach mechanicznych było związane z ich budową morfologiczną. Skrobie ziemniaczana i pszenna, złożone z większych cząstek (o  $d(0.5)$  równym 41.5 i 20.2  $\mu\text{m}$ ) oraz mające dwudzielny rozkład wielkości, wykazywały oscylacje przebiegów zależności naprężenie – odkształcenie, większe w przypadku skrobi ziemniaczanej. Skrobia tapiokowa i kukurydziana były złożone z mniejszych granul (o  $d(0.5)$  wynoszącym, odpowiednio 15.6 i 13.8  $\mu\text{m}$ ). Wartości kąta tarcia wewnętrznego oraz wyniki eksperymentalne testów jednoosiowego ściskania były zbliżone. Cząstki skrobi amarantusowej o  $d(0.5)$  3.0  $\mu\text{m}$  były kilka razy mniejsze od cząstek skrobi kukurydzianej i tapiokowej. Badania mechaniczne wykazały najmniejszą ściśliwość i sypkość skrobi amarantusowej.