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Breaking tester for examining strength of consolidated starch**

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A b s t r a c t. A new method based on the measurement of force required to break by bending a vertical column of consolidated powder was elaborated, and its results were compared with the ones obtained from the Jenike shear test. A new apparatus was built based on a vertical cylindrical chamber divided into two cylinders connected with a horizontal hinge. The apparatus was tested with samples of potato, maize and wheat starches with moisture content of 6, 12 and 17% and with the addition of a lubricant. Results of testing revealed significant differences in measured force required to rotate the upper part of the cylinder away from the lower one. The average force varied from 0.138 N for maize starch to 0.143 N for potato starch, while, for various moisture contents, the measured force varied from 0.135 N for 6% to 0.143 N for 17% mc. The results were compared with the results of a direct shear test.

K e y w o r d s: starch, powders, flowability, breaking, strength

INTRODUCTION

Currently, a number of methods and testers are available to determine the strength and flow properties of bulk solids. There are many studies on the flowability of powders (Juliano *et al.,* 2006; Teunou *et al.,* 1999). Selection of the right method for a 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 the procedure proposed by Jenike (1961) and Opaliński *et al.* (2012). The tester is used to determine flowability (Schwedes, 2003). As reported by industrial practitioners, many different shear testers have been developed for different applications (Carson and Wilms, 2006).

According to some specialists, the Jenike flowability method is considered very complex and time consuming (Bell *et al.,* 1994; Opaliński *et al.,* 2012). The characteristics obtained by the tests performed could be interpreted by limited number of specialists and are understandable to very few practitioners. There are still some improvements proposed in the Jenike shear tester, as proposed by the consulting group founded by Jenike and Johanson (2005) where the consolidation bench is added to the standard tester.

A ring shear apparatus is also available, that, apart from the typical applications of the Jenike tester, offers better measurement accuracy. As a result, more accurate measurements are possible, especially at low normal stresses (ASTM D6773-08; Schulze, 1996; Schwedes, 2003).

As it was mentioned by Wiącek *et al.* (2011), a quality control flowability test is an important task in the production and processing of granular materials because it allows for undisturbed flow of material and maintaining constant composition of processed powder. There are many powder testers available for the determination of simplified parameters which could be related to flowability and process parameters which change along the production line. The quality parameters are also desirable when portions of materials are introduced in mixing, especially in food and pharmaceutical industries. The quality of products may determine the commercial value of products. A comprehensive description of the requirements of quality testers is presented by Ploof and Carson (1994). Those authors stated that a tester should be easy and quick to operate with minimum skill and training from the operator side. The results

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should be precise, repeatable and obtained in a short time. The construction of such testers should be simple and compact, which allows for placement where needed.

There are existing quality testers introduced by many authors. Peschl (1989a, 1989b, 1989c) proposed a tester, a method of determination of powder parameters and a method of quality control of powders for industrial application. A new tester of Jenike and Johanson was presented and validated by Ploof and Carson (1994). Researchers of the Norwegian institution POSTEC proposed a type of indirect shear apparatus, described by Maltby and Enstad (1993).

The chemical, food and pharmaceutical industries need characteristics of fine powders. Carr Indices are commonly used and were standardised by the American Society for Testing Materials (ASTM D6393, 1999). Carr parameters with indexes describing flowability are determined using Powder Characteristics Tester PT-S, Hosokawa (Hosokawa Micron B.V., 2010).

Among all powders used in industry, starch is probably one of the most common. This material is one of the major components in our diet and plays an important role in the formulation of food products. Starches from various botanical sources have received extensive attention in relation to their structural and physicochemical properties. Starch contributes greatly to the textural properties of many food and has many industrial applications as a thickener, colloidal stabiliser, gelling agent, water retention agent and adhesive (Oniszczek et al., 2013; Singh *et al.,* 2003).

Morphological, rheological, thermal and textural properties are of major interest in food technology, but, for storage, handling and transport, mechanical phenomena such as friction or flowability are important as well. Numerous methods are in use for the characterisation of the mechanical properties of powders. However, still a need exists for the development of a method for determination of powder flowability that would allow rapid evaluation directly and by unskilled operator. In this paper, a new method is elaborated and validated for the determination of breaking force of a cylindrical consolidated probe of three starches: potato, wheat and maize. The breaking force of the cylindrical consolidated sample of starch powder was measured and compared with the results obtained with a standard direct shear tester 60 mm in diameter. The examinations were performed for starches of different moisture contents and with the addition of magnesium stearate which is added to improve the flow of powder.

MATERIALS AND METHODS

The tests were performed on samples of potato starch produced by Melvit in Ostrołęka, Poland, and maize and wheat starches produced by Cargill in Warsaw, Poland. The characteristics of the starches were measured at the levels of moisture content occurring in practical technological processes. Materials with moisture content of 6, 12 and 17% were tested. Moistening of the powder was realized by humidifying in a closed cubic space for 48 h. Moisture content (wet basis) was measured gravimetrically by weighing a 10 g sample before and after drying at 105ºC for 24 h. Drier materials were obtained by 24 h drying in laboratory drier with forced circulation at stable temperature of 30ºC. Starches with 6% by weight addition of a lubricant and mixing in standard laboratory mixer for 2 h were also tested. Magnesium stearate (MS) is widely used in tablet formulation because of its lubrication action and ability to form hydrophobic thin film on the surface of the carrier granules. Use of magnesium stearate allows the reduction of power consumption and ejection forces in tablet production (Wang *et al.,* 2010).

Direct shear testing was performed in a shear tester equipped with a chamber 60 mm in diameter. The tests were performed following Eurocode 1 (2006) procedure for consolidation reference stresses σ_r of 4, 6 and 10 kPa and speed of shearing V of 2 mm min^{-1}. Yield loci were determined based on values of maximum shear stresses at two levels of consolidating stress σ_r and ½ σ_r. With the yield locus determined, Mohr circles were drawn that gave values of unconfined yield strength σ_c and major consolidating stress σ_1. The relationship $\sigma_c(\sigma_1)$, characterises the ability of a powder to flow and is termed the flow function of the material, *FF* (Jenike, 1961; Molenda *et al.,* 2006). Mechanical properties of potato starch given in this article are cited from our earlier work (Stasiak *et al.,* 2014).

The apparatus for determination of breaking force (F) is shown in Fig. 1. The rigid base of the device is supported by four adjustable screw feet. Its controls and indicators are placed on the upper surface of the housing: switch of the direction of the linear drive, LED direction indicators and speed control knob. The load cell of 1 N (100 g) capacity is fixed to the moving part of the linear drive and is connected with the measuring chamber using an inelastic (at the load applied) synthetic string and a hook. The measuring chamber has a shape of a vertical cylinder with an internal diameter of 15 mm, horizontally divided into two parts connected with a horizontal hinge which is connected by means of string with the upper part of the chamber. The mobile, upper part has a height of 50 mm, while the lower, motionless part is 28 mm high. Measuring cylinder is fastened to the Teflon base with quick release chuck that allows appropriate positioning of the measuring cylinder. During filling and consolidation both parts of the chamber are coupled with a scissor clamp. A cylindrical scoop of 1.08 cm^3 volume is used to fill the measuring chamber. To consolidate the sample, a 141 g cylindrical rod is used. Piston of the rod is equipped with a millimetre scale used during consolidation to raise the rod at a constant height of 10 mm for 3 times. The procedure of forming the sample and the idea of breaking with force measurement in the apparatus is presented in Fig. 2. The force transducer is

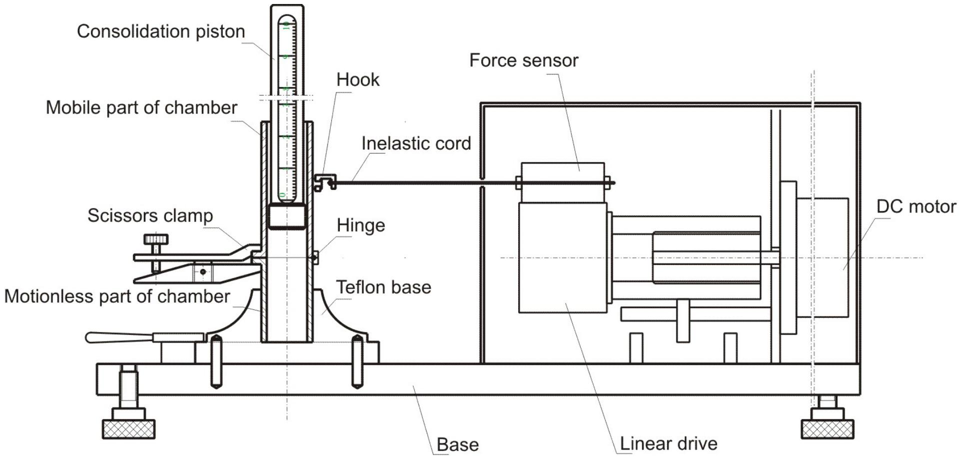

Fig. 1. Breaking force tester and measuring chamber.

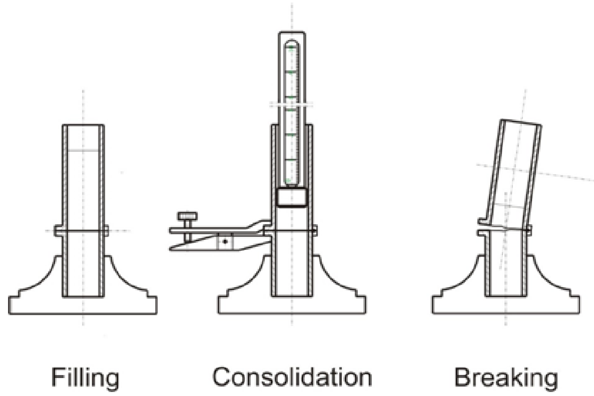

Fig. 2. Procedure of probe forming and measurement.

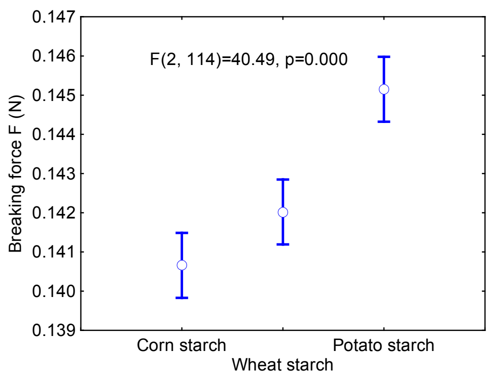

Fig. 3. Mean values of breaking force and 95% confidential intervals of three types of starch. Mean values calculated for whole range of moisture contents. Vertical bars denote 0.95 confidence intervals; F, p – analysis of variance parameters.

connected to data acquisition unit and computer. Force is recorded as a function of real time during the experiment. In the case of consolidation performed in our experiments under load conditions applied, the compaction of the sample reflects only compaction with translation and rotation movements of particles and elastic plastic-contact deformation with pore filling by fine particles. The same kind of translations and rotation movements was observed by Stasiak *et al.* (2010) in the beginning of the compaction process of tablets.

Each measurement of the force was replicated 10 times. Statistical ANOVA tests were used to analyse the results.

RESULTS AND DISCUSSION

A first series of measurements was performed to check the quality of the measurement chamber. Three chambers were tested in the apparatus to determine the influence of production quality and friction of the hinge. Differences between forces obtained by using the three chambers, were examined for three materials. In the following analyses only results of breaking force obtained in chamber 1 will be presented, because this one had the lowest frictional force of the hinge.

Figure 3 presents the results of breaking force for three starches. The highest value of mean breaking force of 0.145 N was observed in the case of potato starch. This material had distinctly different mechanical behaviour as compared to other starches. A similar effect was noted in earlier investigations in direct shear testing (Molenda *et al.,* 2006 and Stasiak *et al.,* 2013). The lowest value of force (below 0.141 N) was obtained for maize starch. In the case of wheat starch, mean value of breaking force was of approximately 0.142 N, while for potato starch the force was distinctly higher and equalled 0.145 N.

The highest value of breaking force measured for potato starch is a result of the morphology of the granules. As mentioned by Molenda *et al.* (2006), the large granules and clearly bimodal composition resulted in higher surface of contact in consolidated powder. The smaller granules fill spaces between the larger ones and this is a probable reason of different behaviour of potato starch. In the case of wheat starch, granules are lens-shaped and very flat. There are also small granules in wheat starch which can be classified as spherical. In the case of maize starch, granules are medium size, from 10 to 25 μm, and of a rather irregular shape. This resulted in smaller contact surfaces after consolidation and it could be the reason of smaller breaking force. The granules of potato starch are clearly non-spherical, while those of maize starch are nearly spherical. The shape factor WK for potato starch was found three times higher than the one for maize starch and two times higher than WK of wheat starch granules (Stasiak *et al.,* 2013). Strengthening of the consolidated samples was observed for maize and wheat starches with an increase in moisture content (Fig. 4). In the case for potato starch, the strengthening was negligible.

In the case of wheat starch, the mean value of breaking force increased from 0.139 to 0.149 N with an increase in moisture content from 6 to 17%, and this was the largest change for all tested materials. In the case of maize starch, the average breaking force increased from 0.140 N for 6 and 12% mc up to 0.143 N for 17% mc. In the case of maize starch, breaking force was higher only for 17% moisture content. No influence of moisture content on the breaking force was observed for potato starch. Granules of potato starch were found to differ significantly in morphology as well as in mechanical properties (Molenda *et al.*, 2006; Stasiak *et al.*, 2013). The results obtained with the new apparatus were found in good agreement with values of angle of repose measured with Hosokawa Powder Tester, as well as with flow function FFC determined using Schulze ring shear tester by Stasiak *et al.* (2013). Those authors stated that in all starches the angle of repose increased with an increase in moisture content. The FFC, determined with the Schulze tester, decreased with an increase in moisture content, indicating a decrease in flowability. For potato starch, the angle of repose increased from 38 to 58º and FFC determined in Schulze ring shear tester decreased from 12.4 to 8.6, showing transition from free flowing to easy flowing. For wheat starch, the angle of repose increased from 42 to 56°, FFC decreased from 5.95 to 2.41, which means that the powder became cohesive. For maize starch, the angle of repose increased from 39 to 53º, while FFC changed from characteristic for easy flowing materials to cohesive materials (from 4.36 to 2.65) (Stasiak *et al.,* 2013).

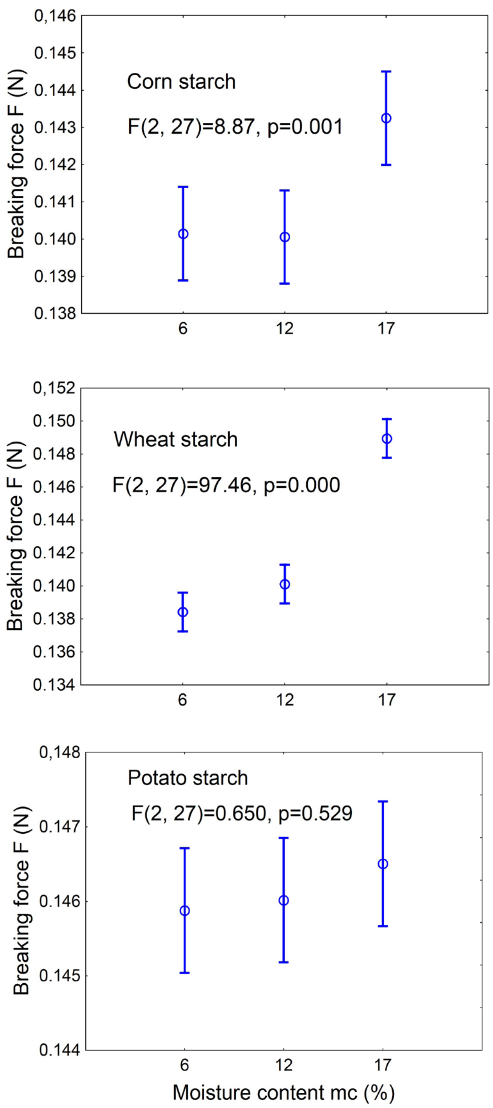

Fig. 4. Mean values of breaking force for three starches and three levels of moisture content. Vertical bars denote 0.95 confidence intervals; F, p – analysis of variance parameters.

In the following series of tests the influence of magnesium stearate on breaking force of consolidated samples of starches was examined (Fig. 5). A significant decrease in breaking force was observed in all tested materials. In the case of maize starch, a significant decrease in breaking force, from 0.140 to 0.138 N, was observed. For potato starch, the breaking force decreased from 0.146 N to approximately 0.142 N, and for wheat starch it decreased from 0.140 to 0.138 N. The potato starch showed a markedly different behaviour in the case of addition of the lubricant as well. The probable reason of such behaviour is surface reaction of magnesium stearate powder with bigger starch particles and covering of particles by the lubricant. A decrease in the breaking force in maize starch resulted from much smaller granules surrounded perfectly by granules of the lubricant.

A linear regression was performed to compare the breaking force with material parameters obtained using the standard Jenike shear test from earlier experiments by Stasiak *et al.* (2013). Table 1 presents the parameters of linear equation fitted with values of parameters obtained in Jenike shear tester and the breaking force values measured for three starches. In the case of potato starch, an increase in moisture content resulted in an increase in breaking force (F), cohesion (C) and flowability index i. The angle of internal friction φ of potato starch was the only parameter decreasing with increasing moisture content. The breaking force, angle of internal friction and flow index decreased, while cohesion increased in potato starch with an addition of the lubricant.

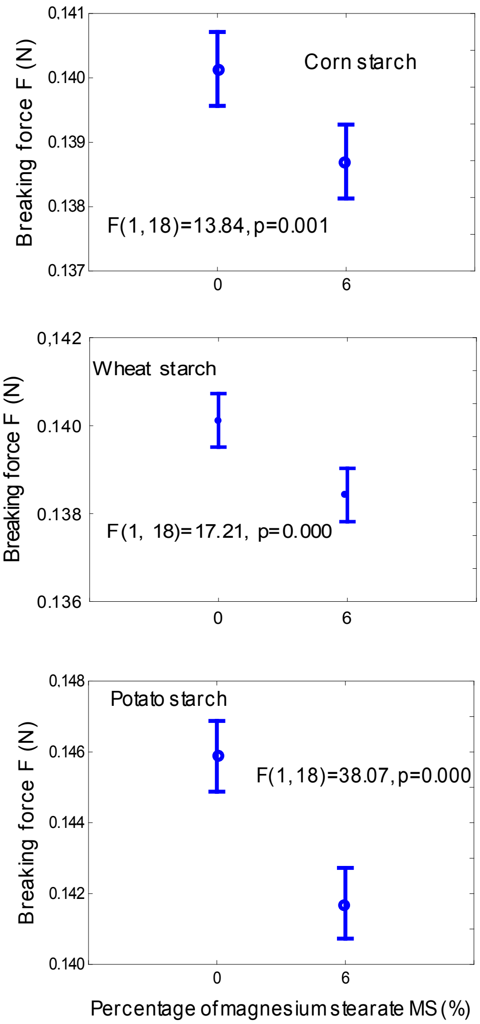

Fig. 5. Mean values of breaking force for materials of 6% moisture content with and without addition lubricant. Mean values calculated for whole range of moisture contents. Vertical bars denote 0.95 confidence intervals; F, p – analysis of variance parameters.

Similar tendencies in the change of values of parameters with an increase in moisture content from 6 to 17% were observed for wheat and maize starches, wherein breaking force F, flow index and cohesion increased. The decrease in angle of internal friction with an increase in moisture content of the materials was observed, which corroborated the results presented earlier by Stasiak *et al.* (2013) for materials examined by using the standard Jenike and Schulze tester. The strongest increase in the value of breaking force was observed in the case of wheat starch (parameter *a* of linear fit equal to 0.0009), while the lowest increase was observed for potato starch ($a = 6.23\ 10^{-5}$).

The mean values of breaking force decreased with an addition of the lubricant for all tested starches. The highest decrease in the breaking force was observed in the case of potato starch (a = -0.0007) and the lowest decrease in the breaking force occurred for maize starch (a = -0.0002). As mentioned above, that result is governed by the diameter of a single particle which is the lowest for maize starch. A majority of the parameters obtained in the Jenike shear tester also decreased. The expectation was cohesion for potato starch, which was an effect of linear fit. In the case of addition of lubricant, the breaking force showed the same tendency as the parameters determined during the shear test.

A linear regression analyses between breaking force and flow index and cohesion was performed to check the sensitivity of new method for possible determination of flowability. The results are presented in Fig. 6. The analysis was performed in 10 replications, for breaking forces obtained for materials with moisture content ranging from 6 to 17% and for original materials with moisture content of 6% and with lubricant. In the case of moisture content, a positive correlation between flow index and cohesion, and breaking force was obtained for all materials. A moderate correlation with flow index i was found in the case of maize starch (r = 0.596), which increased in the case of wheat starch (r = 0.872). A weak coefficient of correlation (r = 0.187) was obtained for potato starch.

A positive correlation of breaking force and cohesion was obtained in the case of three starches for three levels of moisture content. A moderate (r = 0.594), strong (r = 0.853) and weak (r = 0.076) correlation was obtained for maize, wheat and potato starch, respectively. In the case of addition of magnesium stearate, a positive correlation of breaking force with flow index for three starches was found. The coefficients of correlation r = 0.659, r = 0.699 and r = 0.823 were obtained for maize, wheat and potato starch, respectively. Correlation between force (F) and cohesion (C) was strong and positive for maize starch and wheat starch, while for potato starch it was negative (r = 0.659, r = 0.699 and r = -0.824).

The standard direct shear method (Jenike cell) is considered very reliable, but, as has been pointed out by Bell *et al.* (1994), it is time-consuming and not easy in interpretation. The specialists are searching for new methods for characterization of strength of powders that would be easier and might be applied for specific applications. Simultaneously, flowability gains a wider interest as a quality control factor and new methods of its determination are proposed. At the current stage of the development of the powder technology, an increase in the number of new methods is observed rather than a tendency to concentrate and standardise. A relatively large number of methods and parameters are in use, and some disadvantages and limitation are discussed. In the presented project, a method of characterization of consolidated powder by using a new apparatus was presented. The force required to break the cylindrical sample was determined using a test chamber divided into two parts connected with a horizontal hinge. The force was recorded as a function of time during experiments. The new method of determination of breaking force was proven to be efficient, quick and repeatable. Our tester may be compared

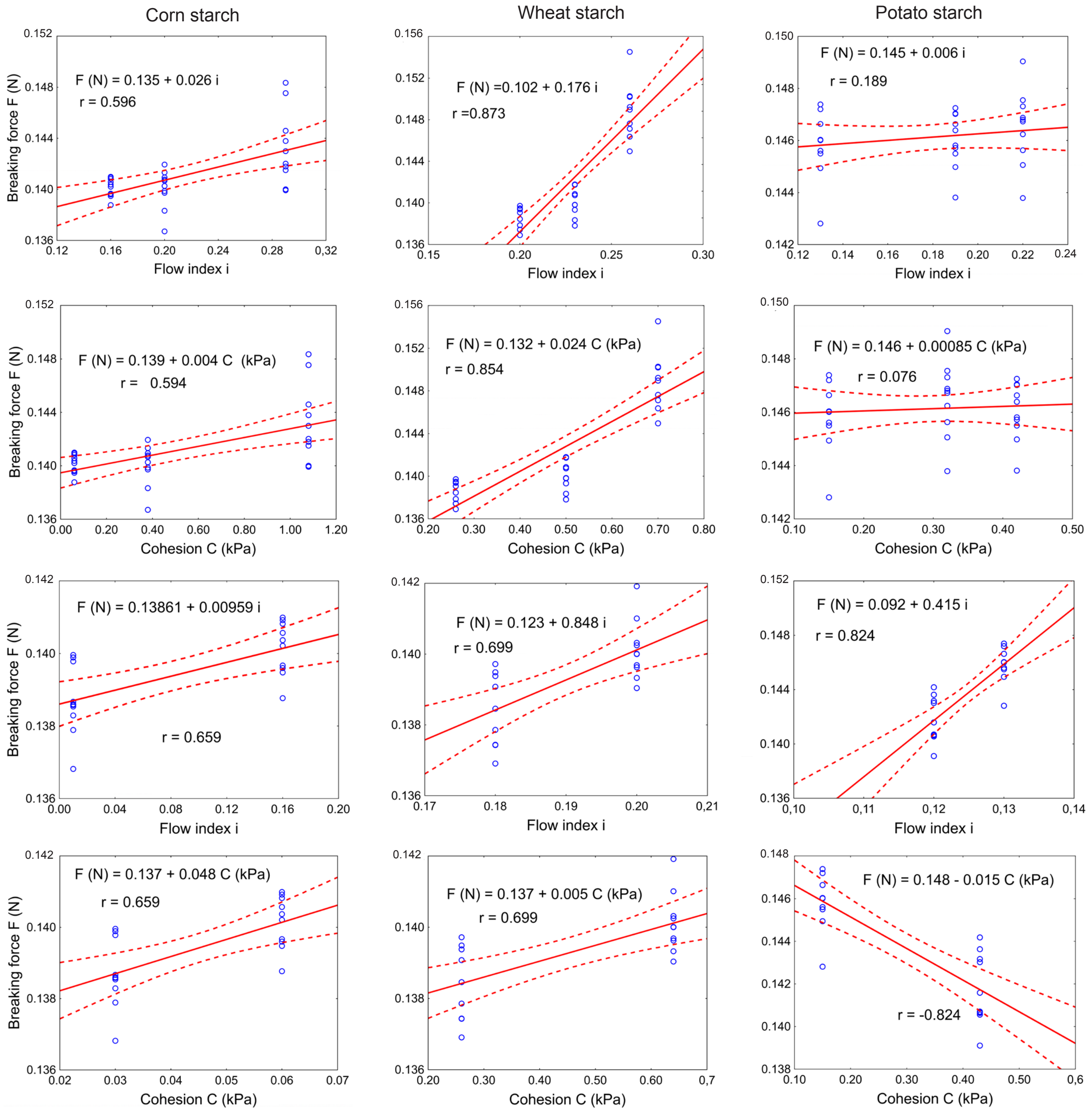

Fig. 6. Analyses of linear regression between flow index i and cohesion C (kPa) and breaking force F (N) for three starches calculated as averages for various moisture content and with lubricant. Dotted lines denotes 95% confidence intervals.

with the well known Warren Spring tensile strength tester (Schwedes, 2003), wherein one half of a diametrically split cell is movable in horizontal direction. After the sample is consolidated, the movable part of the cell is pulled by wire in the horizontal direction without normal stress acting on the sample to enforce tensile failure. The force at failure divided by vertical cross-section of the sample yields tensile strength. The method proposed in our project may be also compared with the Schweiger and Zimmermann (1999) device. Those authors used a strength tester wherein a tensile force of separation for a thin layer of powder material was measured. The authors determined tensile force for maize starch and Lactose. They obtained the force of 400 mN for separation speed of 0.1 mm min^{-1} for the measurement a powder covered area of a watch glass of 0.343 cm^2, and the force of approximately 100 mN for speed of 3 mm min^{-1} and area of 0.397 cm^2.

The presented results showed that the new method for determination of breaking force allows the estimation of strength of potato, wheat and maize starches at three levels of moisture content and with the addition of a lubricant. The obtained results reflected different behaviour of potato starch compared to wheat and maize starches, which has been observed earlier in standard tests (Stasiak *et al.*, 2013, 2014).

Table 1. The parameters of linear equation fitted with values of parameters obtained in Jenike shear tester φ (°), i, C (kPa) and the breaking force F (N) values measured for three starches

Starch	Moisture content mc (%)	Magnesium stearate addition MS (%)
Potato starch	F (N): y = 0.145 + 0.00006 mc, r = 0.957	F (N): y = 0.146 - 0.0007 MS
	φ (°): y = 41.104 - 1.020 mc, r = -0.876	φ (°): y = 36.600 - 2.8 MS
	i: y = 0.084 + 0.008 mc, r = 0.991	i: y = 0.130 - 0.0017 MS
	C (kPa): y = 0.105 + 0.016 mc, r = 0.663	C (kPa): y = 0.150 + 0.047 MS
Corn starch	F (N): y = 0.138 + 0.0003 mc, r = 0.958	F (N): y = 0.140 - 0.0002 MS
	φ (°): y = 42.30 - 1.371 mc, r = -0.974	φ (°): y = 35 - 0.967 MS
	i: y = 0.081 + 0.012 mc, r = 0.964	i: y = 0.160 - 0.025 MS
	C (kPa): y = -0.560 + 0.091 mc, r = 0.965	C (kPa): y = 0.060 - 0.005 MS
Wheat starch	F (N): y = 0.132 + 0.0009 mc, r = 0.911	F (N): y = 0.138 - 0.0003 MS
	φ (°): y = 35.12 - 0.722 mc, r = -0.773	φ (°): y = 32.50 - 2.317 MS
	i: y = 0.167 + 0.005 mc, r = 0.997	i: y = 0.200 - 0.003 MS
	C (kPa): y = 0.020 + 0.040 mc, r = 0.999	C (kPa): y = 0.260 + 0.063 MS

CONCLUSIONS

The new device was found to be efficient in the estimation of strength of consolidated powder through the determination of the force required to break a vertical column of consolidated powder placed in a cylindrical test chamber. The following conclusions could be formulated:

1. The values of breaking force determined by using the device for potato, wheat and maize starches at three levels of moisture content were significantly different.

2. The highest value of breaking force was obtained for potato starch and the lowest one for maize starch.

3. An increase in moisture content resulted in an increase in breaking force, which is in agreement with the results obtained earlier by using a direct shear tester.

4. An addition of a lubricant resulted in a decrease in breaking force.

5. The tester proposed proved to be a promising tool for easy estimation of flowability of compacted powder.

6. The breaking force could be a convenient and quick index for the comparison of various batches of starch.

Conflict of interest: The Authors do not declare conflict of interest.

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