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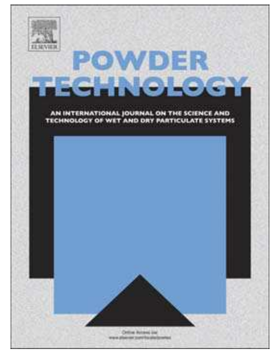

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Rheology of moist food powders as affected by moisture content

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Abstract

Dynamic testing to determine rheological characteristics of moist food powders (semolina, coarse wheat flour, potato starch) was carried out using a powder rheometer of a new construction. The unique feature of the rheometer is that scale of shearing was confined to the thickness of shearing band of powder bed only. It was found that flow pattern of moistened samples was noticeably and diversely affected by both moisture content (varying in the range of 0-15% w/w) and shear rate. The observed changes showed statistical significance $p<0.01$ in all trials carried out. What is noteworthy about the conducted research is that at some shear rate values, the shear stress of the bed reached the maximum for specific moisture content levels, irrespective of particle size of the bed. Such behavior may provide an indication of complex interference of different powder shearing mechanisms in the presence of moisture. For beds consisted of larger particles, shear stress values decreased considerably with increasing moisture content. To explain this, modeling of the shearing process with Discrete Element Method (DEM) was performed. The results obtained supported the idea that friction coefficients of particulate material were significantly

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reduced at higher moisture content of the powder bed in the whole range of shear rates applied.

Introduction

Powders or granular materials when flowing can be in different rheological state depending on the way the particles interact with each other. In rapid flow, the particles interact predominantly by short collisions and their energy is dissipated mainly due to their inelasticity. It is often assumed that particles in rapid flow are so energized that they show behavior similar to that of molecules in a gas or freely flowing fluid. Theoretical considerations concerning such flow regime are therefore usually based on the kinetic theory of gas and a general literature review on existing models is given by Tardos (1997). During slow or moderate flows, the particles are in contact for extended periods of time and the predominant mechanism responsible for energy dissipation is particles frictional sliding. The most important feature of the powder in the slow flow regime is that the bed behavior does not follow with a similarity to that of a solid or a fluid (Klausner et al., 2000). Owing to the complexity of such flows they received little attention and theoretical approaches to the flow mechanisms are diverse (Langroudi et al., 2010). More experimental methods and results are therefore needed in order to validate theoretical achievements and some known analytical solutions making them more applicable to particulate powder flow.

Methods of experimental investigation of slowly flowing granular materials originated from basis conceptions of soil mechanics. They were extended and modified by Jenike (1964) to apply mostly for the purpose of silo design. However, the Jenike shear tester may be an inappropriate method for testing granular materials of higher moisture contents (Opaliński et al., 2012). Alternatively for such materials, a dynamic shear cell (a powder rheometer) can be used (Klausner et al., 2000; Tardos, 1997). The shear process is realized in annular cell and the shear and normal stresses over a range of shear rate are measured. Powder rheometer is a valuable tool for measuring stresses in loosely compacted powder beds for which Jenike (1964) tester is not suitable. Although other methods of characterizing powder flow behavior at low stresses and under dynamic conditions have been reported (Hassanpour and Ghadiri, 2007; Wang et al., 2008, Pasha et al., 2013 and 2015), but for moist powders and continuous shearing rheometer has better control over the test conditions. However, similar to the techniques suggested by above authors, the continuous shearing provided by rheometer can be carried out at low normal stresses applicable to some industrial operations conditions, e.g. fluidization, mixing or pneumatic transport, as well as possibility to test powders in dynamic conditions. This provides better differentiation in flowability of powders processed in dissimilar packing states (Freeman, 2007). An example of this may be limestone powders modified with different hydrophobizing agents: stearic acid vapor and silicone solution (Vogt and Opaliński, 2009). Yield loci obtained for this powders (under high normal load) were almost the same as unmodified raw limestone powder. However, rheological characteristics of modified materials (obtained under low normal load) varied significantly between all the examined samples (Opaliński, Vogt, to be published).

As moisture content in a powder bed increases, the interactions between particles are strengthened usually by forming liquid bridges (Zou & Brusewitz, 2002; Harnby et al., 1996). The cohesion properties of food powders are also dependent on their chemical and biochemical properties. Landillon et al. (2008) found that starch and soluble pentosans may increase viscosity of liquid bridges, thereby increasing their strength. Soluble components of grains or seeds may lead to plasticizing of powder resulting in an increase in contact area and surface stickiness (Rennie et al., 1999). Fitzpatrick et al. (2007) showed that the food powders with greater amount of amorphous lactose were more sensitive to absorbing moisture giving rise to lumping and caking problems. The complex behavior of moist food powders was also found by Ganesan et al. (2008a, b) for DDGS (Distillers Dried Grains with Solubles the main bi-product of ethanol production from non-fermentable maize residues). They found that flow function values for DDGS decreased with increasing moisture content (10 to 20%), but increased for 25 and 30% moisture content. Thus, above a certain moisture content, a lubricating effect of moisture is possible and an improvement in flow can be observed. This contradictory effect of moisture content on powder flowability is particularly significant for powders flowing under small values of normal load where flow conditions are not suppressed by overwhelming action of contact forces as in the case of powder sheared in Jenike shear tester.

A key factor affecting flow of powders (particularly food powders) is surface friction defined as the frictional resistance to bulk flow that includes both particle-particle and particle-wall interactions. Some early data (Savage 1967) concerning flow of cohesionless bulk solids in a vertical converging channel showed that the particle-wall friction was more influential for the rate of flow than the angle of internal friction. Surface friction influences both wall and bulk solids properties as well as and handling conditions (Prescott et al., 1999; Bradley et al., 2000). Humidity of the surrounding air or moisture content of bulk solid significantly influences the surface properties of solid particles and as a result also the surface friction but the way the moisture interacts with particle material is complex. This is firstly due to the capability of food materials to absorb water to some extent and secondly due to some biological changes that may occur inside the particle material in presence of water. Despite general awareness of the friction phenomena for powder flow (Ganesan et al. 2008b) there is so far no quantitative data allowing prediction of the friction coefficient as a function of particles moisture content. This would provide a constructive approach to reflect and explain in quantitative terms the complex rheology of food powders and their flowability under various process conditions.

The aim of this work was (i) to show experimentally how moisture content affects the rheology of food powders, and (ii) to obtain computational values of friction coefficients of moistened food powders that provide agreement between measured and calculated results. The rheology of food powders was investigated by a new type of powder rheometer enabling a precise control and calculation of the velocity gradient in shear band of the examined powder bed. To obtain friction coefficients DEM modelling method was used and calculation were performed with PFC2D software (Itasca Consulting Group Inc.). Friction coefficients for both translational and rolling movement of contacting particles, as well as damping constants were considered and their changes with humidity and shear rate of the powder bed are given.

1. Materials and methods

2.1. Physical properties

The materials used were commercially available food powders: semolina, coarse wheat flour, common wheat-flour, potato starch and milk powder. Particle size distribution (PSD) for the powders were obtained using a Malvern Mastersizer 2000E laser diffraction analyzer. The powders were dispersed in isopropyl alcohol to allow for PSD measurement. The measured values of particle size are summarized in Table 1 and an example of the PSD for coarse wheat flour, potato starch and semolina are given in Fig. 1. More details on physical properties of the materials and the methods used to obtain them were given in earlier paper of the authors (Opaliński et al., 2012).

Hydration of materials was accomplished by direct addition of water to samples, plus mixing until a homogeneous consistency was obtained. In the case of highly dispersed food powders, e.g. potato starch or common wheat-flour, the moisturizing method entailed passing a stream of air of controlled humidity through the materials being mixed in the *V*-type drum mixer. This was a protracted process. To obtain higher moisture contents of the materials under investigation, it was necessary to keep mixing for many hours, as for example, it was found for the required final moisture content of common wheat-flour.

Moisture content usually applied in operations involving food powders varies in a rather broad range from 1-5% for milk powder, 10-15% for commercially available flours to 40-60% for pasta and bread processing. For this reason water content in current experiments was gradually increased until marked changes in rheological characteristics developed and this typically was observed for moisture contents not larger than 15-20%.

Moisture content was determined by weighing about a 5-7 g sample before and after drying at 70^{0}C. All examined samples were dried in laboratory vacuum heating chamber SPT-200 (ZUT COLECTOR, Poland). The drying and weighing procedure was repeated several times until constant mass of the sample was obtained.

2.2. Rheological measurements

The objective of this experimental investigation was to present and analyze the rheological characteristics of food powders as affected by moisture content. The rheological state that has been recognized as the most important from cognitive and practical point of view, was frictional flow realized at slow shear rates. In order to remain within the frictional flow regime it was necessary to carry out the experiments at shear rates as low as possible. The lowest stable rheometer speed was equal to about 5 rpm, i.e. approximately with blade tip speed of 2.5 cms^{-1} and this value was a starting point for each experiment. The experiments were however carried out also at much higher shear rates (in "fast flow" regime) to investigate the possible effect of moisture content on powder rheology.

The annular shear cell-type powder rheometer used in the present work was similar to that described by Klausner et al., (2000). A simplified sketch of the rheometer is shown in Fig. 2. The powder sample was sheared in the annular space between the upper shear plate and the bottom rotating cell. The space has external and internal diameters of 102 and 88 mm respectively and a depth of 9 mm. The upper shear plate remained fixed by a moment arm and the bottom cell was driven by a Lenze GKR geared motor with a rotational speed in the range of 5-300 rpm controlled by Lenze vector frequency inverter. Both upper and bottom parts of the rheometer were made of hardened and polished steel. To avoid escaping of the powder from the annular space during experiments, a flat ring seal was used which was fixed to the protrusion of upper plate of the rheometer. The seal was made of polyethylene sheet and covered with sand paper of grit size suitable for the examined powder particle size.

A Hottinger Baldwin Messtechnik (HBM) C9B and U9B force transducers, both with a range of 200 N and an accuracy class of 0.5% were used to measure the tangential force resulting from powder shearing and normal force resulting from powder compression and dilation respectively. The current position of the upper plate was adjusted with the upper plate positioner and measured with HBM WA/10MM-T displacement transducer with measuring distance range of 0-10 mm and linearity deviation of 0.1%. For every individual measurement, the position was established according to required compaction of the sheared powder sample. The measured force data were amplified with HBM SPIDER8 amplifier and collected with HBM Catman®Easy software.

The unique feature of this rheometer design is the possibility to settle precisely the height of the powder layer under examination and to ensure that the height corresponds to the shear band thickness, i.e. about 15 – 20 particle diameters (Klausner at al., 2000, Horabik 2001). Thus the experimental shear rate values could be correctly determined, making the obtained results reliable for comparison with theoretical data.

The powder sample of mass, m, required to achieve the desired powder bed height was evenly placed in the annular rheometer space and the shear plate was established in a position to obtain an appropriate powder compaction. The lower plate speed was than adjusted to a definite values of 5 rpm and it was allowed to rotate until the normal and shear stresses reached a constant values. The data on loads F_n and F_t , bed height, δ, and rotational speed, n, were recorded. After the measurements were accomplished, the shear plate was moved up until it was no longer in contact with the powder in the rheometer annular space. The plate was then rotated again with the same speed and the loads, f_n and f_t, resulting from friction of polyethylene seal against the plate wall were recorded.

The treatment of the data collected depended on whether the load cell was in tension or compression and it was the same as that given in Klausner et al. (2000):

a) normal stress for load cell in compression:

$$\sigma_n = \frac{W+F_n-f_n}{\pi(R_o^2-R_i^2)} \tag{1}$$

and in tension:

$$\sigma_n = \frac{W-F_n-f_n}{\pi(R_o^2-R_i^2)} \tag{2}$$

where W = 15,83 N is the weight of the upper shear plate, R_o= 51 mm is the outer annular radius, and R_i = 44 mm is the inner annular radius. F_n and f_n are the measured normal loads at working and tare conditions respectively.

b) mean shear stress:

$$\tau = \frac{3(F_t - f_t)L}{2\pi\left(R_o^3 - R_i^3\right)} \tag{3}$$

where F_t and f_t are the measured tangential loads at working and tare conditions respectively, L = 175 mm is the moment arm length.

c) shear rate:

$$\gamma = \frac{u}{\delta} = \frac{\pi\, d\, n}{\delta} \tag{4}$$

where δ is powder bed height in the annular gap, d = 95 mm is the mean value of annular gap diameter and n is the rotational speed.

In order to ensure the proper conditions of powder shearing, i.e. an ordered movement of the bed particles through the whole thickness of the shear band, the surfaces of the upper shear plate and the bottom of rotating cell were covered with sandpaper of an appropriate grits corresponding to the particle size of the material being sheared in the rheometer.

To avoid any substantial changes taking place in powder physical and chemical properties as a result of shearing, the following precautions were taken: i) the time spent at each shearing rate was limited to about 1 minute, i.e. the value needed to establish steady-state conditions only; ii) the normal loads applied to the shear layer were restrained to the lowest possible values – they were close to zero before the experiments started, and eventually increased to give shear stress values of approximately several kPa (at steady-state conditions) due to powder dilation. The small loads and limited time of experiments provided proper shearing conditions with no heat generation and no essential powder particles degradation.

The experimental procedure applied to obtain food powder rheological characteristics included measuring the shear and normal stresses resulting from powder resistance to shearing and the shear layer thickness, as well as moving and controlling the rheometer plates stability and their leveling during experiments. The measurements were generally difficult to accomplish and the main problem was to maintain the rheometer rotating plates in stable and precisely parallel position. Moreover the upper plate of the rheometer had to be maintained at a given and constant distance (equal to the thickness of shear layer) from the lower one exactly to about $\pm$ 0.1 mm. The reported data are the arithmetic average results of at least three measurements for each powder sample. Statistical evaluation of the obtained data was given in form of error bars, added to all experimental points in Figures 3-6.

3. Results and discussion

3.1. Effect of moisture content on powder rheology

In Fig. 3 the variation of shear stress with shear rate for the bed of semolina of different moisture contents is shown. All the results shown in this Figure and the succeeding ones were obtained at constant volume of the powder bed, i.e. constant bed height in the rheometer cell. For smaller water contents ($s = 0$, $s = 5\%$), the powder shear stress increases with increasing shear rate (with statistical significance, $p < 0.01$) in the whole range of shear rates. This is a typical feature of a powder being in the frictional regime in which the prevailing interaction between particles is considered to be surface friction (Klausner et al., 2000). For larger moisture contents ($s = 10$, $s = 15\%$), the powder behavior is different. The characteristic feature is that the shear stress reaches a maximum at a certain value of shear rate, and then begin to decline. The descending part of the curves at higher shear rates indicates that the powder is now in transitional region between a slow frictional regime and a rapid flow one.

The observed variation of the shear stress with moisture content clearly indicates that moisture favorably affects the rheology of powder bed. The shear stress values are considerably smaller at larger water contents, particularly in the range of higher shear rates. A possible explanation is that due to water absorption, the properties and contact conditions of solid bed particles are changing. Especially, this concerns the surface layer of particles where presence of water can result in substantial changes of some particle material properties, possibly reduction of surface friction coefficients that ease the flow.

In Fig. 4 the effect of moisture and shear rate on normal stress values for semolina is shown. The normal stress-strain rate relationship is relatively flat for each moisture contents in the whole range of shear rates covering the slow and transitional flow regimes. Similar results for the same flow regimes were obtained by Klausner et. al, (2000) for silica and polymer dry powders. As suggested by the authors, the very little increase in normal stress with increasing shear rate corresponds to the characteristics of frictional and also transitional flow, where the stress contribution from interparticle collisions is small. From the results obtained in the present work it follows that in these flow regimes the presence of liquid in the powder bed significantly affects the shear stress variation with shear rate but not normal one which is similar in dry and moist powder bed.

The ratio of shear-to-normal stress is shown in Fig. 5. It follows approximately the same behavior as for shear stress since the normal stress does not change noticeably in the range of shear rates applied. Similar results were obtained for other food powders consisting of more coarse particles. An example is the shear stress-shear rate relationship for coarse wheat flour shown in Fig. 6.

Using the same experimental procedure to study rheological behavior of fine food powders it was shown that moisture can affect powder flowability in a different manner. A typical example is rheology of potato starch shown in Fig. 7. The shear stresses for this fine powder increased with moisture increase, i.e. an opposite trend was obtained as compared with materials of greater particle sizes like semolina (Fig. 3). Similar flow patterns were obtained for milk powder and common wheat flour but the results were somewhat scattered. A possible explanation of this opposite trend is that fine powders, in general are more cohesive, i.e. their resistance to shear is larger. The bed of such a powder has usually small porosity, and the presence of water may cause the bed to be more compacted and as a result even more resistant to shear. As splitting such a dense bed structure into smaller flowing particle assembles requires more energy, the shear stress increases with humidity. Another reason is that some soluble components of food powders may cause plasticizing of powder resulting in greater contact area and surface stickiness (Rennie et al., 1999). This may also increase the agglomerates strength and as a result the shear stress needed to break the agglomerates apart becomes larger.

3.1. Theoretical description of frictional flow of bulk powders

Theoretical description of a fluid motion is usually based on mass and momentum conservation laws. To obtain fluid density and velocity fields it is necessary to complete these equations with an appropriate constitutive equation, i.e. the relation between two physical properties of a material, that is the response of that material to external forces. Using the constitutive equation with mass and momentum balance to obtain equations of motion is a common procedure applied in fluid mechanics.

An attempt to use this fluid mechanics approach for description of slow frictional flow of powders was given by Tardos (1997). He assumed the powder assembly as a compressible continuum for which conservation laws of mass and momentum can respectively be applied as

$$\frac{\partial \varrho_b}{\partial t} + \nabla \cdot (\varrho_b u) = 0 \tag{5}$$

$$\varrho_b \frac{D\boldsymbol{u}}{Dt} = \varrho_b g + \nabla \cdot T \tag{6}$$

where ρ_b is the powder bulk density, *u* is the velocity vector, *g* is gravitational vector, and *T* is the stress tensor.

In order to describe the powder velocity and density fields with these equations, constitutive equations relating the stress tensor to the rate of deformation is required and it was given by Schaefer (Tardos et al., 1998)

$$T_{ij} = p\left[I_{ij} + \sqrt{2}sin\varphi \frac{D_{ij}}{|D_{ij}|}\right] \tag{7}$$

where $p = (T_{xx} + T_{yy} + T_{zz})/3$ is the average normal stress, φ is the angle of internal friction and $I_{i,j}$ is the unit tensor.

The rate of deformation tensor ($D_{i,j}$) and the magnitude of the rate of the deformation $|D_{i,j}|$ are given as:

$$D_{ij} = -\frac{1}{2}\left(\frac{\partial u_i}{\partial x_j} + \frac{\partial u_j}{\partial x_i}\right) \tag{8}$$

$$|D_{ij}| = \left[\sum_{ij} D_{ij}^2\right]^{1/2} \tag{9}$$

The applicability of the constitutive equation given by Eq. (7) with reference to cohesive powders flow was examined using a powder rheometer by Klausner *et al*. (2000).

Applying the Eq. (7) for the simple Couette flow (which closely corresponds to that in the powder rheometer), it follows that (Tardos et al., 1998)

$$\sigma = p \text{ and } \tau = -psin\varphi \tag{10}$$

where p is the pressure and φ is the internal angle of friction of the powder bed.

Clearly this is not correct since the shear-to-normal stress ratios obtained experimentally are shear rate dependent. This was shown both in experiments by Klausner et al. (2000) as well as in those performed in this work – Figs 5-7. Klausner et al. (2000) suggest some possible reasons for the disagreement between the model given by Eq. (7) and the experiment. In their opinion the reasons are: (i) the interparticle friction is not necessarily constant with shear rate and (ii) in addition to friction, there exists a mechanism associated with cohesion, which provides a resistance to shearing.

It appears that the results obtained in our experiments and concerning the effect of moisture content on rheology of powder bed fully confirm the Klausner's suggestions. The mechanism associated with cohesion, which provides a resistance to shearing seems to be based on the moisture of the bed, which causes changes of interparticle friction coefficients with increasing rate of shearing. To confirm this idea, modeling of powder rheology was performed using DEM method which allowed to obtain theoretical values of friction coefficients of moistened food powders that provide agreement between measured and calculated results.

3.2. Predicting the rheology of moist powders with DEM method

In addition to fluid mechanics approach for description of flow of powders, another approach to examine powder flowability is to use of DEM method. It has proved to be a successful way in simulating behavior of a powder bed since the first work on this subject published by Cundall and Strack (1979). Over the last twenty years the DEM has become an important tool for understanding particle movement within a powder bed and an accepted means of addressing problems in granular and discontinuous media providing information for the design and optimization of operation with bulk solids (Weerasekara et. al. 2013, Radjaï and Dubois, 2011; Matuttis and Chen, 2014). Regardless of the advantages, the important shortcoming of the DEM method is the limited number of particles that can be built-into the model based on available computational power. For this reason calculations were performed for beds composed of larger and rather regular shape particles i.e. semolina (d_p=0.2-0.3 mm).

The DEM methodology is well established and described in many books and review articles (Radjaï and Dubois, 2011; Matuttis and Chen, 2014). It allows monitoring of movement of particles in the investigated system. Using force law calculations the translational and rotational velocities as well as the positions of particles can be determined on the basis of acting forces for all contacts. A force arising in each contact point i is a sum of normal (F_{iN}) and tangential (F_{iS}) force components:

$$F_i = F_{iN} + F_{iS} \quad (11)$$

Here authors applied well-known nonlinear model by Hertz-Mindlin described in (Cundall, 1988) with described following modification.

The normal force (F_{iN}) in i-th contact point is nonlinear function of normal overlap Δn:

$$F_{iN} = \frac{2\hat{G}\sqrt{2\hat{r}}}{3(1-\hat{\nu})}\sqrt{\Delta n} \quad (12)$$

where $\hat{r}$ is averaged particle radius for both (A and B) particles in contact:

$$\hat{r} = \frac{2r^{[A]}r^{[B]}}{r^{[A]}+r^{[B]}} \quad (13)$$

$\hat{G}$ is averaged Kirchhoff modulus of both particles A and B:

$$\hat{G} = \frac{1}{2}(G^{[A]}+G^{[B]}) \quad (14)$$

and $\hat{\nu}$ is averaged Poisson's ratio of both particles A and B:

$$\hat{\nu} = \frac{1}{2}(\nu^{[A]}+\nu^{[B]}) \tag{15}$$

The tangential component (F_{iS}) of the contact depends on its value in previous timestep ($F_{iS(t-\Delta t)}$) and it is limited by Coulomb fractional limit (i.e. transitional friction coefficient μ and normal force F_{iN} ratio). According to Hertz-Mindlin model in case the tangential force exceeds the limit, a mutual slip occurs instead of shear and the tangential force F_S is set to $\mu|F_{iN}|$, where μ is transitional friction coefficient. Authors propose a modification that seem to reasonable within compacted beds and is based on assumption that the tangential force is controlled not by transitional (μ) but by rolling friction coefficient μ_{rol} :

$$F_{iS} = \begin{cases} F_{iS(t-\Delta t)} + \frac{2\sqrt[3]{3\hat{r}\hat{G}^2(1-\hat{\nu})|F_{iN}|}}{2-\hat{\nu}} V_{rel}\Delta t \ \text{ if } F_{iS} \leq \mu|F_{iN}| \\ \mu_{rol}|F_{iN}| \ \text{ if } F_{iS} > \mu|F_{iN}| \end{cases} \tag{16}$$

where Δt is calculation timestep and V_{rel} is relative velocity of particles in contact (A and B):

$$V_{rel} = V^{[B]} - V^{[A]} - \omega^{[A]}r^{[A]} - \omega^{[B]}r^{[B]} \tag{17}$$

with V and ω being particles linear and rotational velocities respectively.

The resultant force acting on each particle is calculated as a sum of contact forces in n number of contact points taking into account the dissipative proportion described by the damping constant (C):

$$F = \sum_i^{i=n} F_i - C\sum_i^{i=n} F_i \tag{18}$$

The 64 bit version of the commercial software package (PFC2D, version 4.00-123 by Itasca Consulting Group, USA) was used for all simulations in this study. Annular particle factory was interpreted in 2D-spaced software as a 1/10 section of the real annulus with periodic space approach applied. This approach is schematically presented in Fig 8. The shear test consisted of the generation of particles with properties given in Table 1 and Table 2. The particles randomly positioned within rheometer working space (in the shape of annular groove) were settled under gravitational force to form approximately 20-diameter layer. Next, 1.192 N (This value resulted from the weight of the upper plate of the rheometer, as applied to the powder layer at the beginning of experiment) normal force was applied to upper wall and simultaneously the vertical motion of the bottom wall was started with fixed velocity (v_x). In repeated tests the bottom wall velocity (v_x) was adjusted to obtain shear rates γ in the range of (5-400) s^{-1}. The upper wall mass value was controlled during the tests by servomechanism and established on the level of 121.5 g, i.e. value corresponding to the initial load of 1.192N. Once the normal stress σ gained the expected level (8 kPa ± 1%), the horizontal load (τ) exerted on the bottom wall was registered versus time. Each of the tests was carried out until steady (τ) value was achieved (the test was stopped when ten following values were congenial with allowable 1% relative error).

The described procedure was repeated for different values of transitional (μ) and rolling (μ_{rol}) friction coefficients changing in the ranges of (0.01-0.9) and (0.01-0.5) respectively and for damping constants (C) varying from 0.5 to 0.9. Finally, the τ values obtained in simulations were compared with corresponding experimental results to find the effects of transitional (μ) and rolling (μ_{rol}) friction coefficients values as well as the damping constant (C) values on rheological behavior of examined samples of semolina. The parameters resulting in the best fit of DEM simulations with experiments for different moisture levels and shear rates are discussed below.

In the case of dry semolina particles (water content s = 0%) the experimental values of shear to normal load ratio (τ/σ) grows proportionally (from 0.014 to 0.587) with the applied shear rate γ in the range 6-380 s^{-1} – Fig. 9. The experimental data are found to be best fitted (with determination coefficient R^2 = 0.976) with simulation results performed with fixed damping constant (C) of 0.7, transitional friction coefficient (μ) of 0.9 and rolling friction coefficient

(μ_{rol}) of 0.5. Invariability of the three parameters for the entire shear rate range (the fact that the simulation results could be fitted with fixed values for all shear rates) could indicate that surface properties of semolina particles are preserved in the studied range of shear rate (γ) and confirms the presumption that the powder flow was in the frictional regime.

In the case of moist semolina sample (s= 5%) the behavior of examined powder bed was different and changes of τ/σ ratio versus shear rate (γ) are shown in Fig 9. The observed horizontal loads (τ) and consequently τ/σ ratio values became lower (between 0.08 and 0.444) as compared to dry powder results in the same range of shear rate (γ). Additionally, the increase of τ/σ ratio values is not so evident for shear rates higher than 180 s^{-1}. Slightly decreased, as compared to dry sample of semolina, values of τ/σ ratio were satisfactorily fitted (R^2 = 0.977) with computer simulation results executed with damping constant (C) of 0.725, transitional friction coefficient (μ) of 0.8 and rolling friction coefficient (μ_{rol}) of 0.5 for shear rates γ in the range (6-180) s^{-1}. The rolling friction coefficient (μ_{rol}) value had to be gradually reduced from 0.5 to 0.25 for shear rates (γ) above 180 s^{-1}. Evidently, as it is seen from the given data, the moisture content substantially affects the flow behavior of the tested semolina sample. Slightly increased value of damping constant (C) reflects the effect of higher energy dissipation caused by wetted particles' surface. The experimentally observed changes of flow pattern, as seen in the range of shear rates (γ) higher than 180 s^{-1}, can be assigned to the diminution of rolling friction coefficient (μ_{rol}).

Increasing water content to 10% brought about substantial turn in τ/σ ratio versus shear rate (γ) - Fig 9. Roughly proportional growth of τ/σ ratio (from 0.001 to 0.393) was registered only for shear rates (γ) values below 190 s^{-1}. Higher rotational speeds of rheometer resulted in considerable drop of measured values of τ and τ/σ ratios (from 0.393 for γ = 190 s^{-1} down to 0.062 for γ = 375.4 s^{-1}). Computer simulation results proved to be consistent with experimental data in the first range of shear rates γ when values of the damping constant (C) of 0.75, transitional friction coefficient (μ) *of*0.75 and rolling friction coefficient (μ_{rol}) of 0.5 were assumed as values of rheological model parameters. Substantial drop of τ/σ ratios above shear rate (γ) of 190 s^{-1} implied that transitional and rolling friction coefficients were gradually reduced from 0.75 to 0.1 and from 0.5 to 0.05, respectively, if acceptable level of agreement between experimental and model results is to be obtained (R^2 = 0.883).

Further growth of water content (s= 15%) resulted in lowering measured τ/σ ratio to the values below 0.1 within almost the whole range of applied shear rate (γ) values (6-361 s^{-1}) – Fig. 9. The exception is the region close to the shear rate (γ) value of 126.4 s^{-1} where slightly higher level of τ/σ_N (0.113) was noticed. This observation proves that substantial changes of powder rheological behavior occur at this level of moisture content. In order to reproduce the observed experimental results, DEM simulations were executed with damping constant (C) value increased to 0.775. Transitional and rolling friction coefficient values were proved to be constant (μ = 0.25 and μ_{rol} = 0.5, respectively) up to shear rates not exceeding 126.4 s^{-1}. A small decrement of τ/σ ratio for higher shear rates was associated with simultaneous reduction of both transitional and rolling friction coefficients from 0.25 to 0.16 and from 0.5 to 0.05 respectively, as used in DEM computations (determination coefficient value, R^2 = 0.778).

The combined effects of shear rate (γ) and moisture content (*s*) on both friction coefficients (μ and μ_{rol}) are shown in 3D plots (Figs. 10 A and B) that reveal the aforementioned rapid decrement area in the range of high shear rates (γ) and moisture contents (*s*).

5. Conclusions

In this work it is found that the moisture content considerably affects the shear stress-strain rate relationship of food powders and hence their rheology. As moisture content increases, water can act either as plasticizing agent for food-powder particles of hygroscopic nature like semolina and coarse wheat-flour or as lubricating factor for non-hygroscopic powder particles where it is also possible for water layer to exist on particles surface and ease the flow.

For moist food-powders with water content up to 15% it does not seem to be reliable hypothesis that moisture can exists on powder particles in the form of water layers. Instead, a realistic assumption is that water is absorbed into the core of the hygroscopic grains and it is also the case for the surface layer of particles. For this reason presence of water can result in substantial changes of some surface particle material properties, possibly reduction of surface friction coefficients and damping factor as it was suggested by the computer simulations for semolina. The calculations clearly show that friction coefficients, both transitional (μ)and rolling (μ_{rol}) were most affected by the moisture; their values declined from 0.9 to 0.1 and from 0.5 to 0.05 respectively in the range of moisture content between 0 and 15%. To a lesser degree this refers to another surface property of powder particles - damping constant (C)which values raised from 0.7 to 0.775 in the given range of water content.

The values of the above mentioned material parameters (μ , μ_{rol} and C) that had to be adjusted during the DEM simulations to fit the experimental data can be treated as a quantitative measure of material's rheological properties. Sharp decrease of friction coefficients for moist material could be an indication of material surface properties modification followed by powder rheology changes, under assumption that the applied DEM contact model is correct. The obtained results are a premise to continue the experimental verification of DEM contact models in the range of high shear rates as well as for moist powder conditions and elaborate for more generalized DEM contact model.

Using rheometer reliable information on the rheological characteristics and powder flowability can be obtained including a possibility to identify differences between very similar powders, like coarse and common wheat flour The opposite trends of the shear stress-strain rate relationships with increase of moisture content as found in the present research for these materials confirmed that rheology and flowability of food powders is a complex function of many physical and even biochemical food properties. Therefore further research is required to quantify the specific mechanistic relationships that can explain the changes observed during these experiments as well as extending the range of powders to confirm the applicability of the models applied to describe the flow.

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Fig. 1. Particle size distribution for coarse wheat flour, potato starch and semolina

Fig. 2. A simplified sketch of the annular cell-type rheometer. 1- shear plate positioner, 2-shear plate, 3-tangential load transducer, 4-displacement transducer, 5-rotating cell, 6-driving geared motor, 7-annular space, 8- normal load transducer

Fig. 3. The effect of moisture content *s* and shear rate γ on shear stress τ for semolina; particle size (0.1-0.3 mm)

Fig. 4. The effect of moisture content *s* and shear rate γ on normal stress σ for semolina; particle size (0.1-0.3 mm)

Fig. 5. The effect of moisture content *s* and shear rate γ on shear-to-normal stress ratio τ/σ for semolina; particle size (0.1-0.3mm)

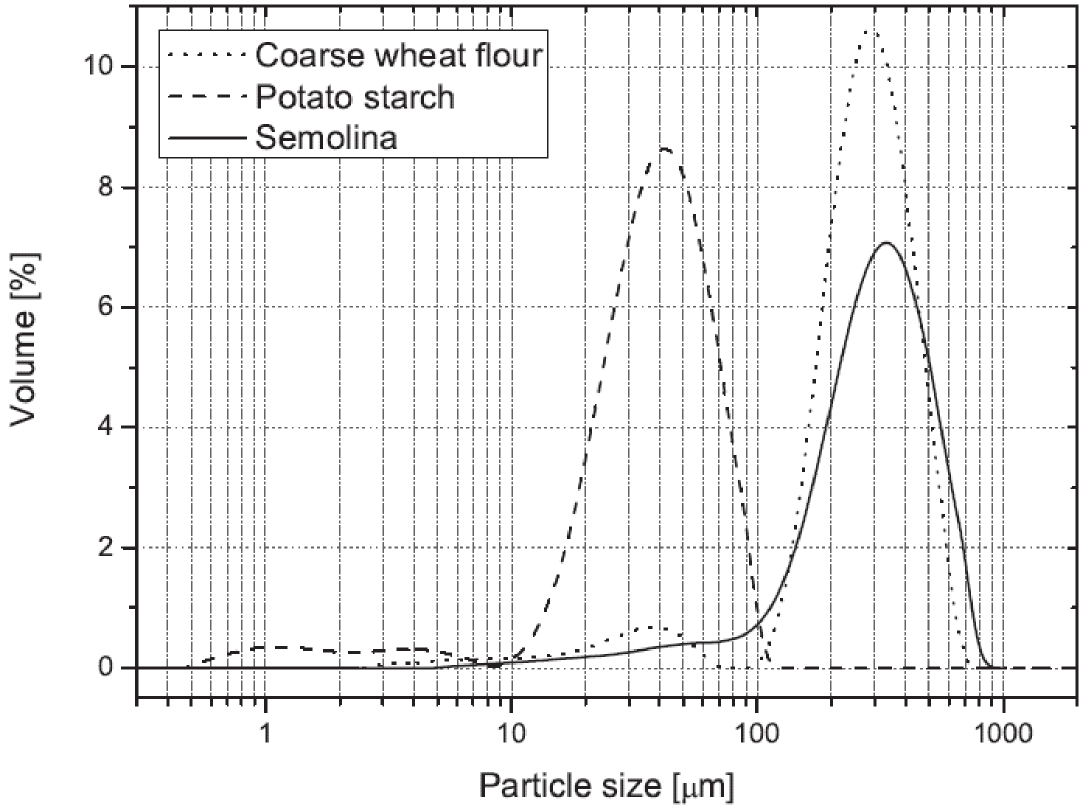
Fig. 6. The effect of moisture content *s* and shear rate γ on shear-to-normal stress ratio τ/σ for coarse wheat flour; particle size (0.1-0.25mm)

Fig. 7. The effect of moisture content *s* and shear rate γ on shear-to-normal stress ratio τ/σ for r potato starch; particle size (36-75 µm)

Fig. 8. Scheme of 2D representation of 3D annular particle generation region for DEM modelling with periodic space approach

Fig 9. DEM simulation results compared with experimental results for the effect of moisture content and shear rate on shear-to-normal stress ratio for semolina

Fig 10. Effects of shear rate and moisture content on the fitted (A) transitional (μ)and (B) rolling (μ_{rol}) friction coefficients of semolina particles applied in DEM simulations of annular rheometer



Coarse wheat flour
Potato starch
Semolina
Volume [%]
0
2
4
6
8
10
1
10
100
1000
Particle size [μm]

Figure 1

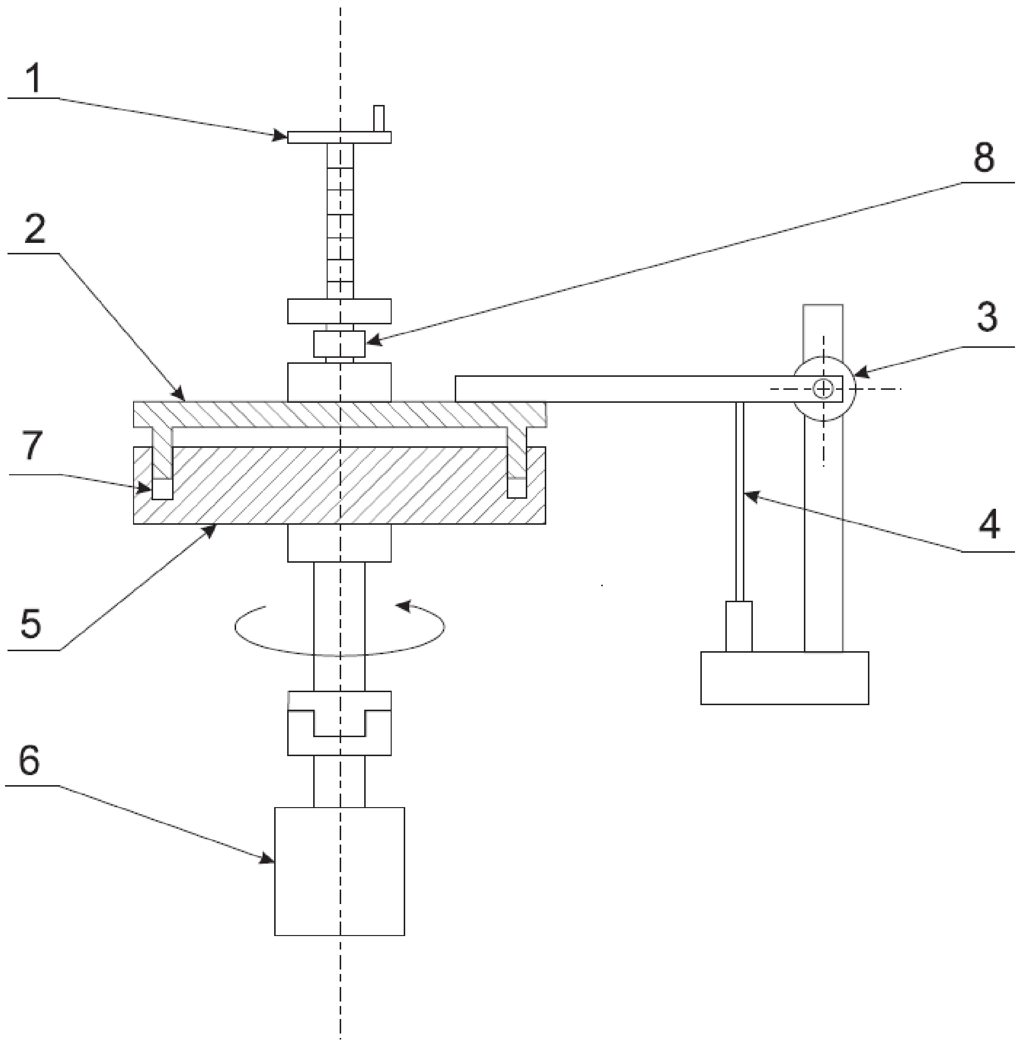


Figure 2

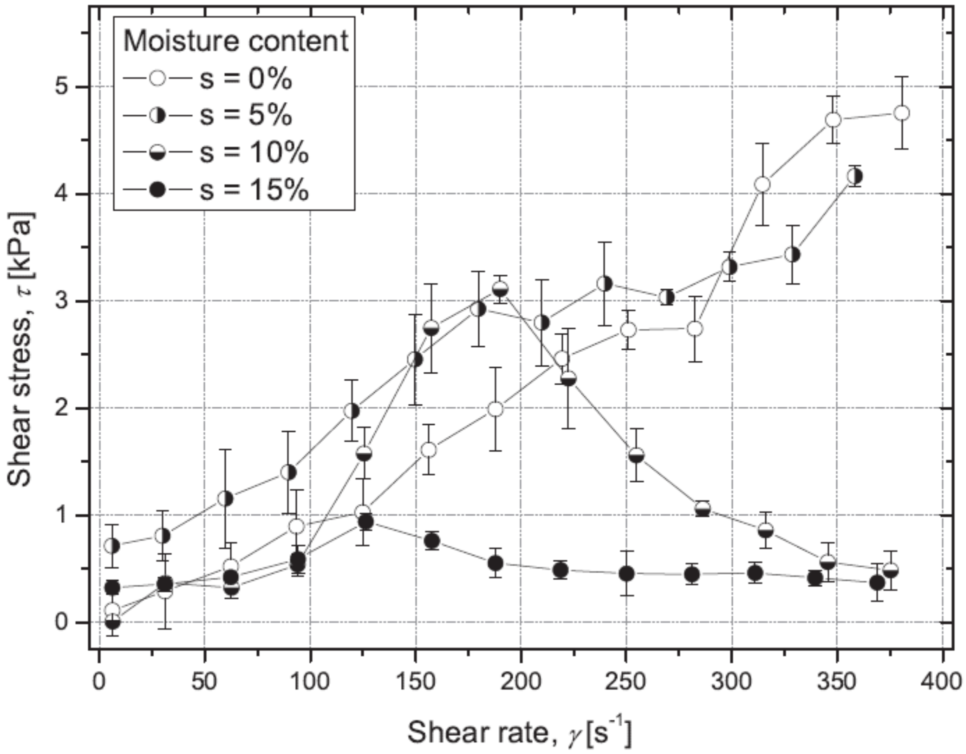
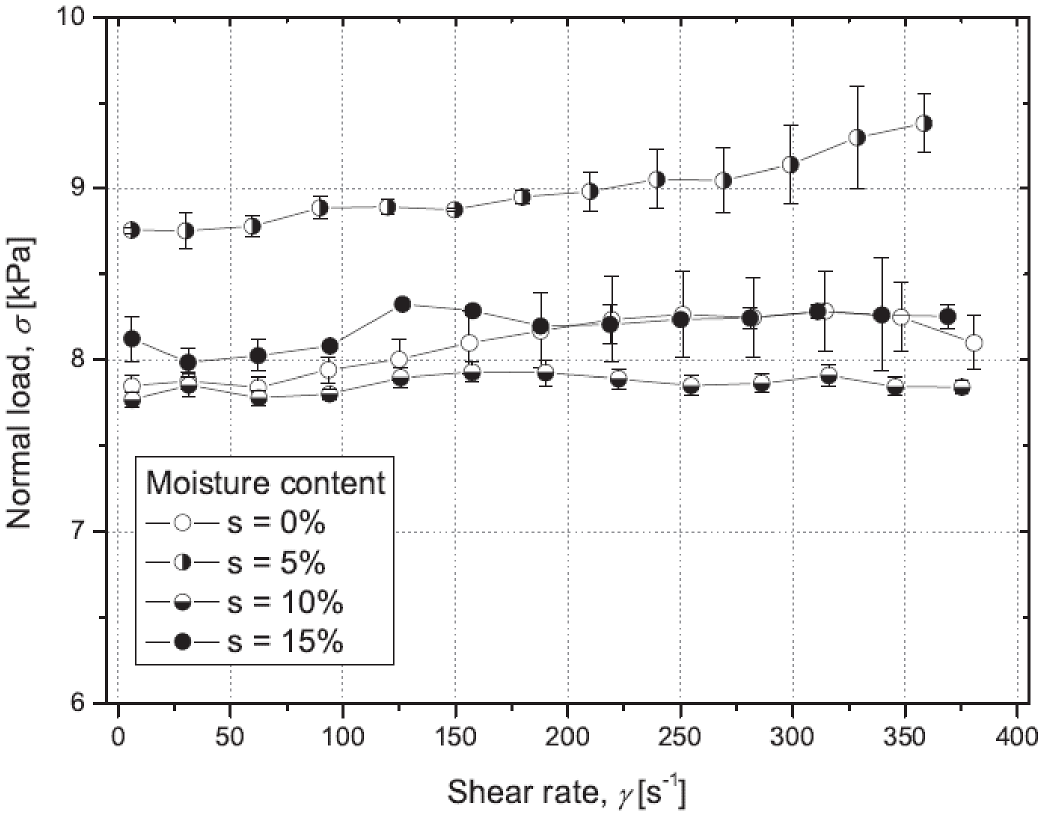
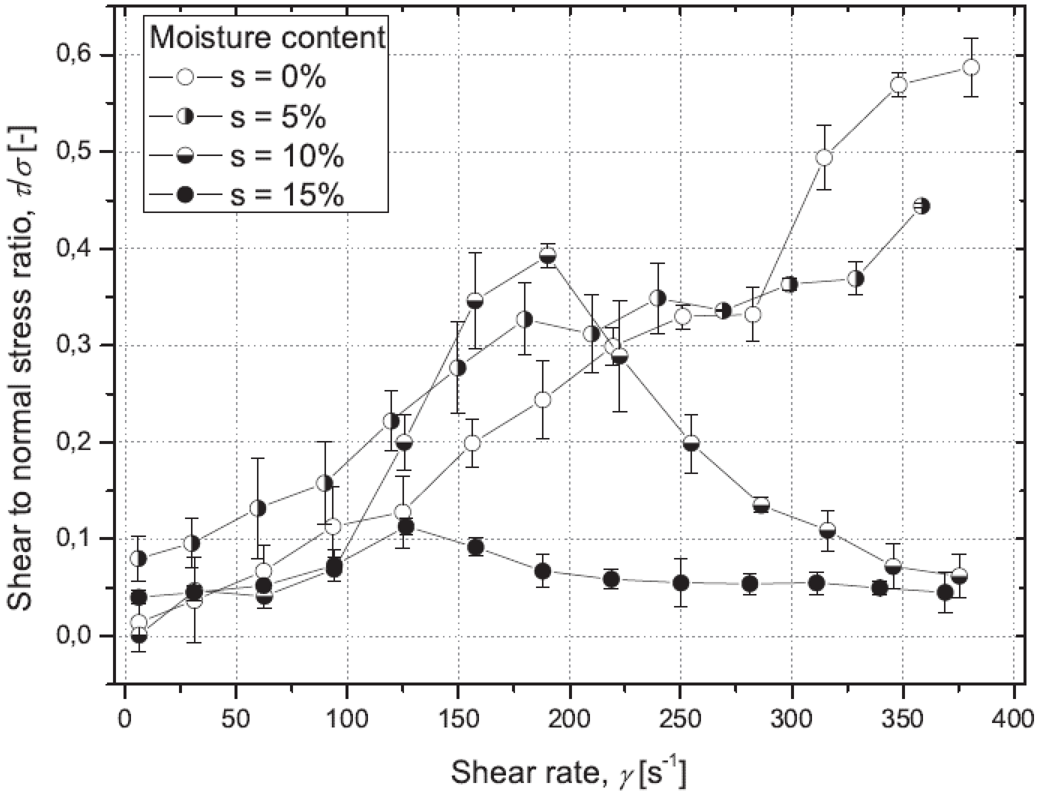

Moisture content
s = 0%
s = 5%
s = 10%
s = 15%
Shear stress, τ [kPa]
Shear rate, γ [s⁻¹]
0
1
2
3
4
5
0
50
100
150
200
250
300
350
400

Figure 3



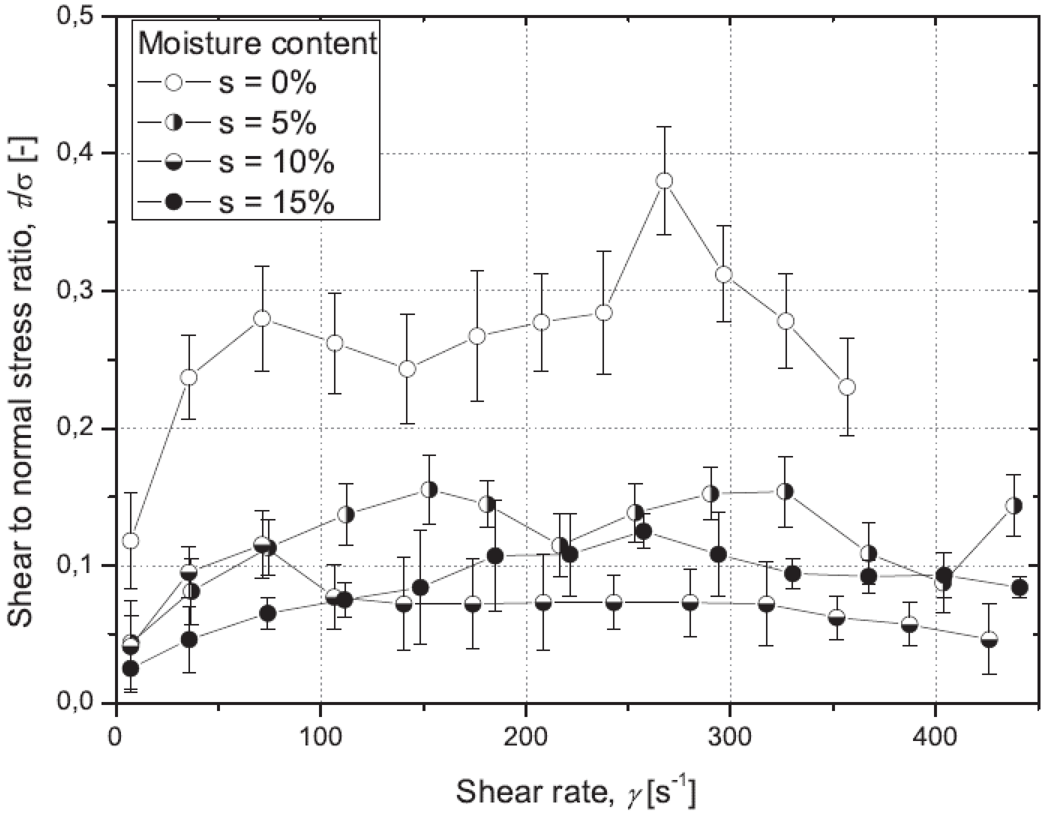
10
9
8
7
6
Normal load, σ [kPa]
0
50
100
150
200
250
300
350
400
Shear rate, γ [s-1]
Moisture content
s = 0%
s = 5%
s = 10%
s = 15%

Figure 4



Moisture content
s = 0%
s = 5%
s = 10%
s = 15%
Shear to normal stress ratio, τ/σ [-]
Shear rate, γ [s-1]
0,0
0,1
0,2
0,3
0,4
0,5
0,6
0
50
100
150
200
250
300
350
400

Figure 5



Moisture content
s = 0%
s = 5%
s = 10%
s = 15%
0,0
0,1
0,2
0,3
0,4
0,5
0
100
200
300
400
Shear to normal stress ratio, τ/σ [-]
Shear rate, γ [s⁻¹]

Figure 6

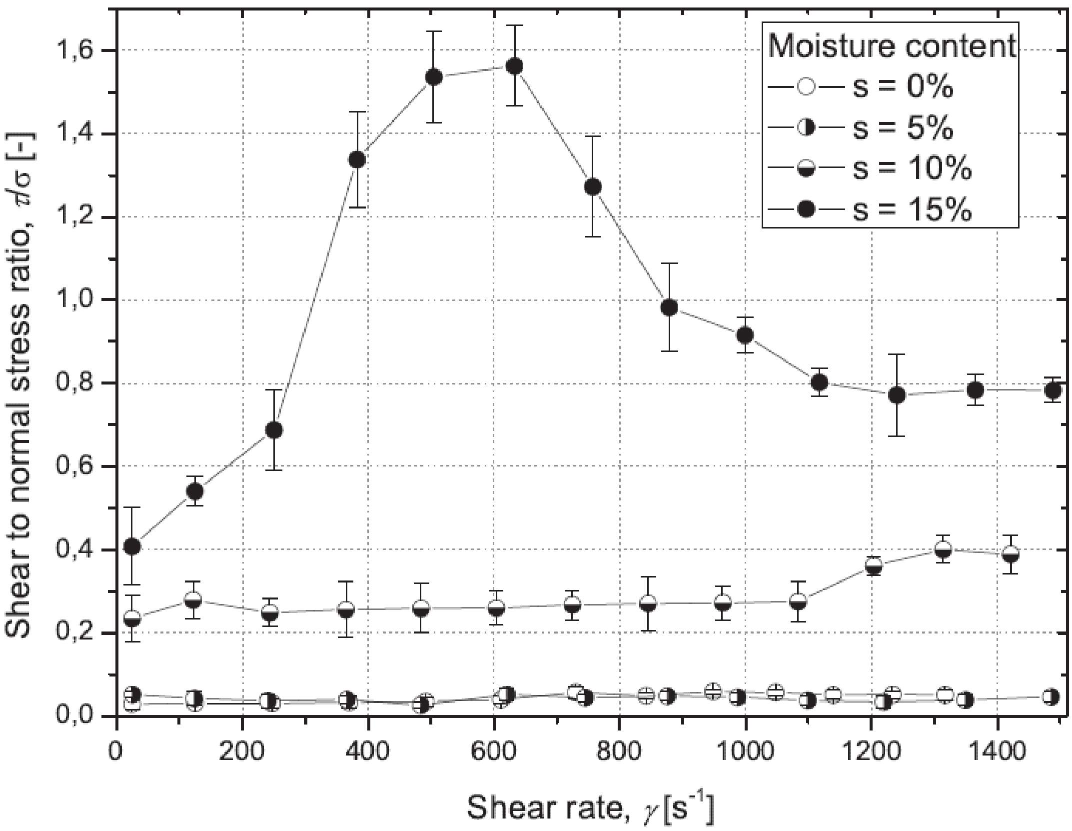

Moisture content
s = 0%
s = 5%
s = 10%
s = 15%
Shear to normal stress ratio, τ/σ [-]
1,6
1,4
1,2
1,0
0,8
0,6
0,4
0,2
0,0
0
200
400
600
800
1000
1200
1400
Shear rate, γ [s⁻¹]

Figure 7

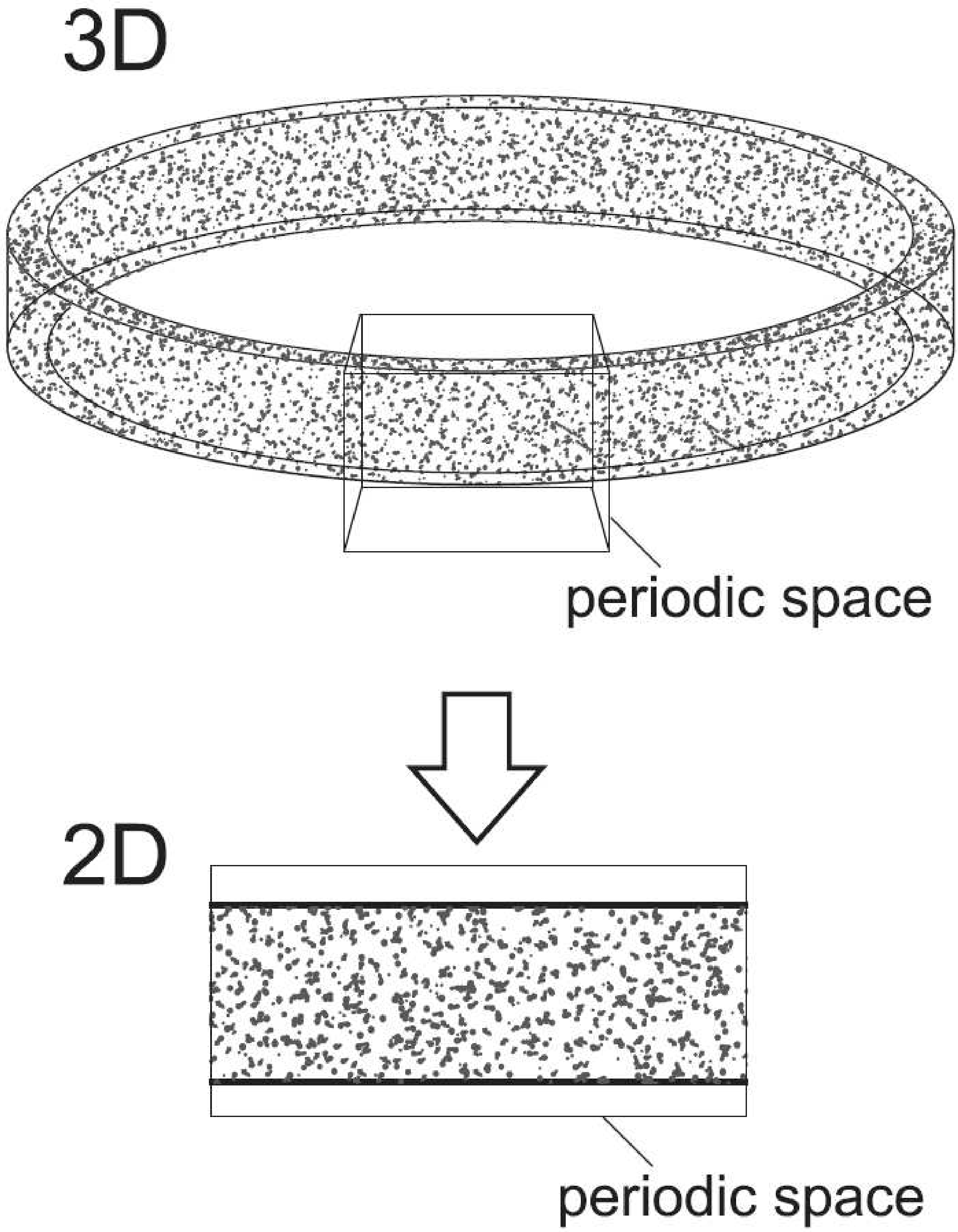
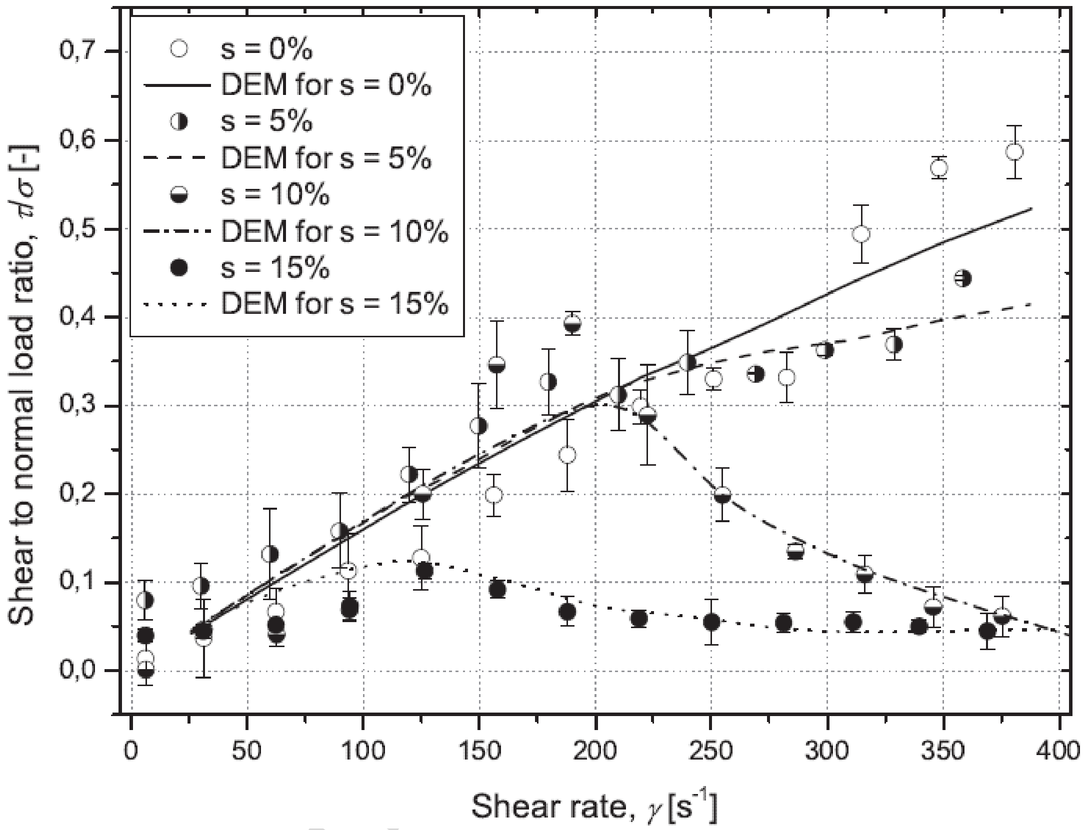


Figure 8



s = 0%
DEM for s = 0%
s = 5%
DEM for s = 5%
s = 10%
DEM for s = 10%
s = 15%
DEM for s = 15%
Shear to normal load ratio, τ/σ [-]
Shear rate, γ [s-1]
0,7
0,6
0,5
0,4
0,3
0,2
0,1
0,0
0
50
100
150
200
250
300
350
400

Figure 9

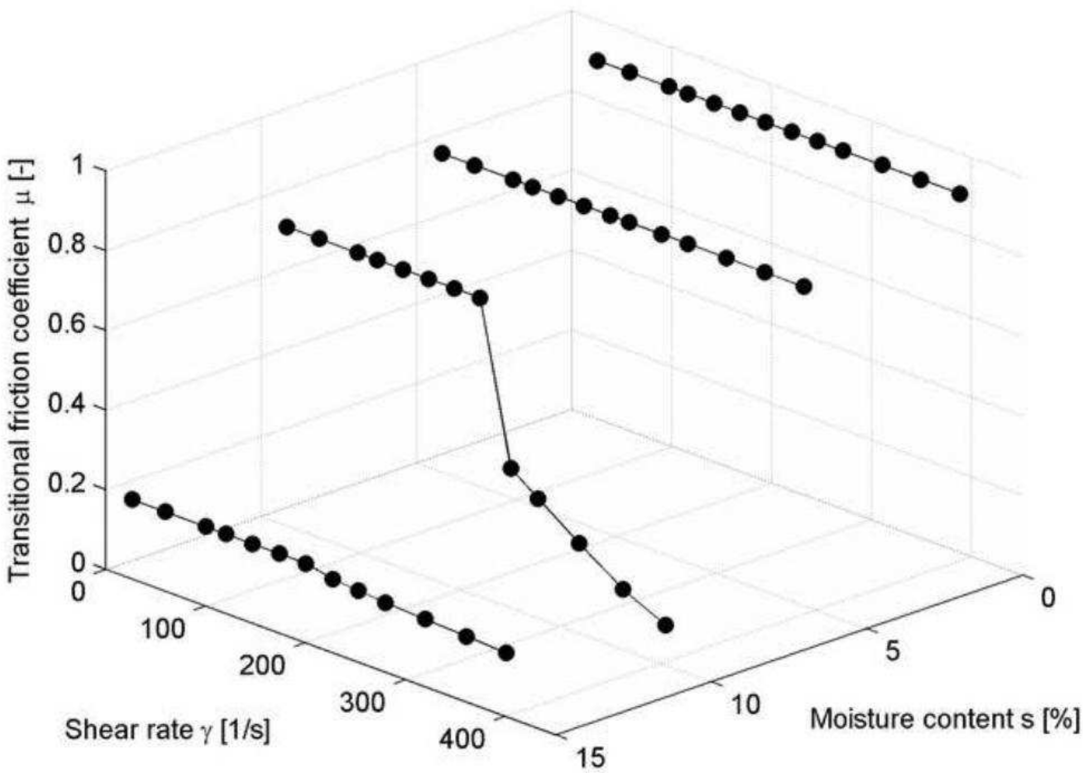
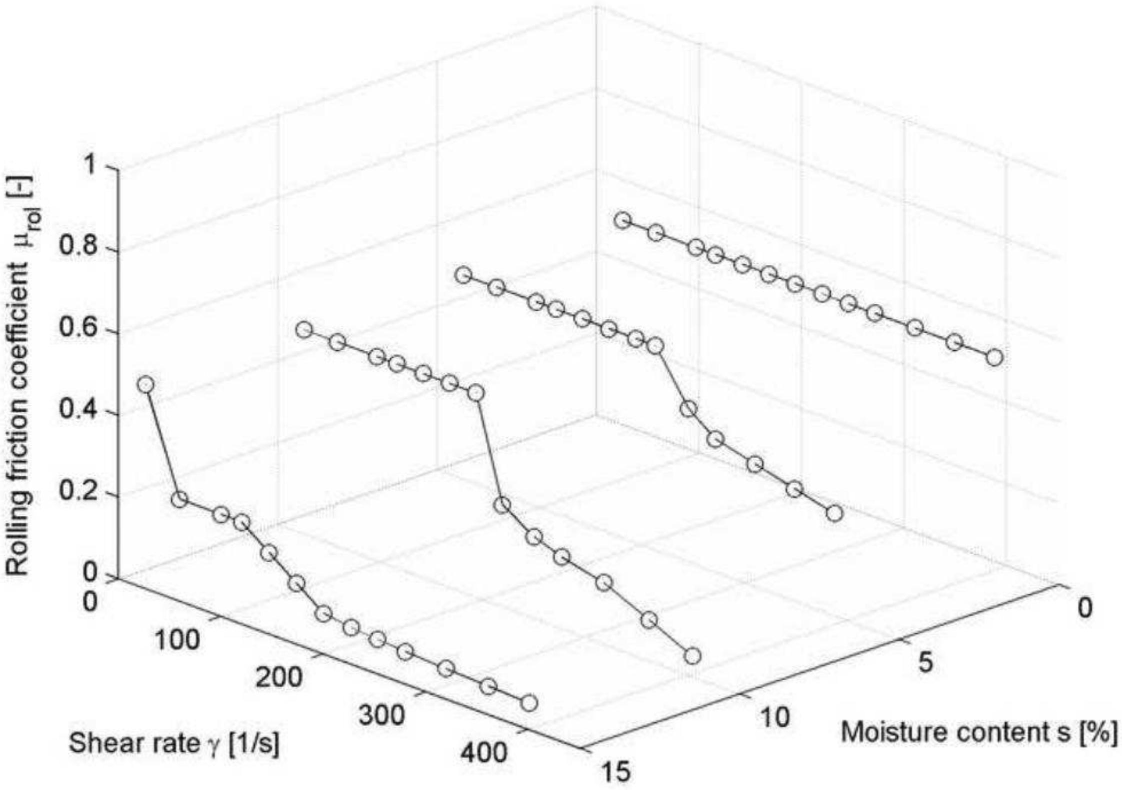

Transitional friction coefficient μ [-]
1
0.8
0.6
0.4
0.2
0
0
100
200
300
400
Shear rate γ [1/s]
0
5
10
15
Moisture content s [%]

Figure 10A



Rolling friction coefficient μ_{rol} [-]
1
0.8
0.6
0.4
0.2
0
0
100
200
300
400
Shear rate γ [1/s]
0
5
10
15
Moisture content s [%]

Figure 10B

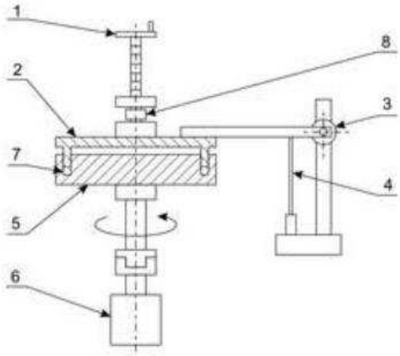
Table 1. Physical properties of studied powders

Material	d_{50}* [μm]	d_{10}* [μm]	d_{90}* [μm]	$d_n = \frac{d_{90}-d_{10}}{d_{50}}$ [-]
Semolina	288	118	517	1.39
Coarse wheat flour	140	106	189	0.60
Common wheat flour	62.5	28	97	1.10
Potato starch	24	13	37	1.01
Milk powder	111	44	239	1.75

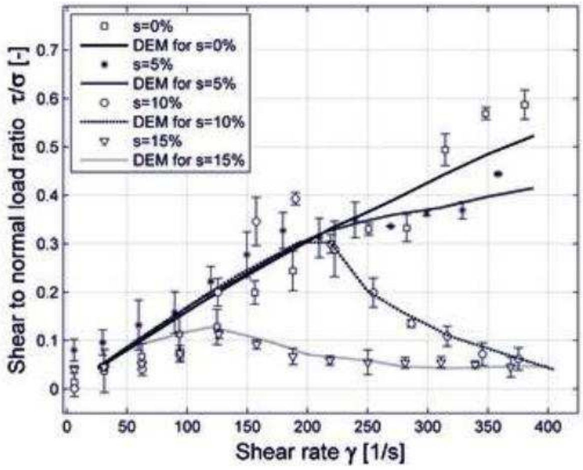
*) d^{50}, d^{10}, d^{90} are values of the particle diameter at 50%, 10% or 90% in the cumulative size distribution, respectively.

Table 2. Data used for DEM simulation of semolina particles

Quantity	Symbol	Value
Number of particles, -	N	4 000
Particle diameter, m	ds	1.6-2×10^{-4} (normal distribution)
Density, kg/m^3	ρ	1550
Kirchhoff modulus, Pa	G	5.09×10^9
Poisson's ratio, -	ν	0.2
Transitional friction coefficient, -	μ	0.01 - 0.9
Rolling friction coefficient, -	μ_{rol}	0.01 - 0.5
Damping constant, -	C	0.7 - 0.775
Normal load, kPa	σ	8
Rotating cell velocity, m/s	v_x	0.017 5- 1.5



A simplified sketch of the annular cell-type rheometer:
1- shear plate positioner, 2-shear plate, 3-tangential load transducer,
4-displacement transducer, 5-rotating cell, 6-driving geared motor,
7-annular space, 8- normal load transducer



Graphical abstract

Highlights

- Rheology of food powder samples were tested using rheometer of a new construction
- Changes of shear rate and moisture content show effects on shear to normal stress ratio
- DEM modeling supported the idea that friction coefficients decrease with moisture