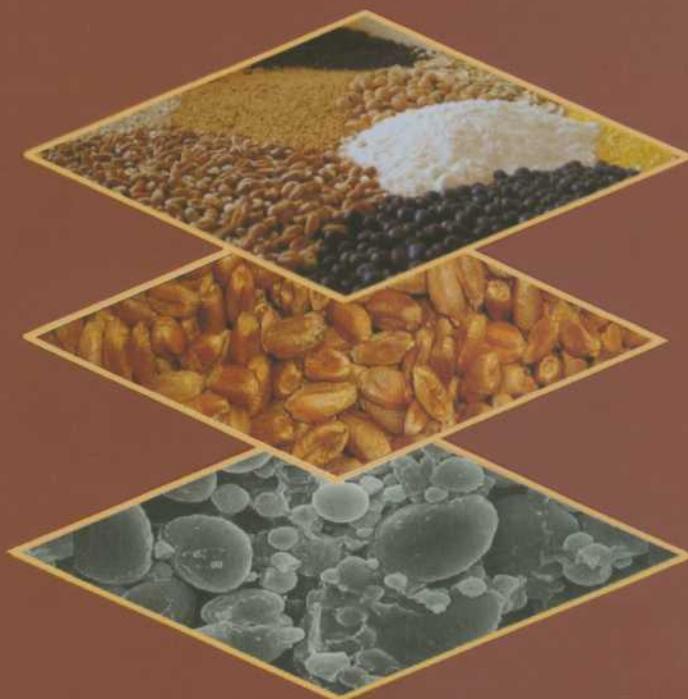




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International Workshop

Characterization of granular agro-materials and food powders

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INTRODUCTION

Storage, handling and processing of granular materials are employed in numerous industries and are of interest to various branches of science and technology such as physics, chemistry, mechanics, agriculture and engineering. Agriculture and the food industry are, next to chemical and pharmaceutical industries largest producers and users of granular materials. Two basic conditions have to be fulfilled by equipment for storage and processing of granular materials: predictable and safe operations and obtaining high quality of final products. Precise characterization of granular materials and better understanding of their interrelations with construction materials are needed to design efficient handling and processing systems.

Communications between producers, designers and users require common language and common procedures of material characterization. Despite unquestionable progress in development of measurement methods properties of granular materials measured in various laboratories can vary greatly. Comparison of results from different, cross-disciplinary laboratories require unification of terminology and standardization of measuring methods. Moreover some influencing factors that contribute to observed variability may still remain out of control.

The aim of the Workshop is to offer forum for exchange of experiences for researchers working with granular materials in such fields as: civil engineering, agricultural engineering and food industry. The scope of the meeting covers problems of characterization of physical properties of particulate agro-materials and food powders and their influence on technological processes. Invited lectures concern methods of computer modeling in granular mechanics, better understanding of silo behaviour, effects of physical properties of grain on silo structures and application of physical properties of cereal grain in processing.

We hope that the Workshop will contribute to better understanding and tightening links among specialists dealing with particulate solids in different fields of science and technology.

*Józef Horabik,
Marek Molenda*

APPLICATION OF PHYSICAL PROPERTIES OF RAW MATERIALS IN CEREAL PROCESSING

Janusz Laskowski, Dariusz Dziki

Department of Machine Operation in Food Industry, University of Agriculture
ul. Doświadczalna 44, 20-236 Lublin, Poland
e-mail: KEMPS@faunus.ar.lublin.pl

An analysis market of cereal has shown that about 5,8 million tones is used in cereal processing. Within the amount, 4,3 million tons of wheat, 1,2 millions tons of rye and about 250 thousands of other corns are milled. About 17 million tons of cereals is used for feed [Kisiel 2002].

These results show how huge mass of corn is processed. On the other hand, plenty of intermediate and final products are obtained. It is obvious that properties of cereals are closely connected with the obtained products. On the basis of the properties of the raw materials the end-use of cereals in processing can be evaluated. The demands for raw materials about accurate physical and technological parameters are systematically increased. The consumers make strict demands concerning the quality of cereal products. The physical, chemical and technological kernel properties are used for cereal quality evaluation. The results show close connections between these properties. It is concerned both with the connections between the raw material properties and the milling process, and dependencies between the intermediate products and the ones used for baking process.

The last years are characterized by the dynamic development of investigations concerning the physical and technological properties of foods, and rheological properties of dough obtained from different types of flour. The results of these works gave the basis to choose cereal quality properties, their division and diversification in trade. Similarly, on the basis of the rheological properties of dough, and technological properties, the criteria can be established for the end-use of flour for production of different kind of baking products.

An other wide group of physical and technological properties taken into account is the optimization of the cereal processing, both from the point of view of the quality of the products and the energy utilization. Many of works are also concerned with the physical and technological properties of the final products.

In conclusion we can say that the properties of cereals immediate and final products are connected in undisrupted technological chain, which requires much more accurate method of investigations of the physical and technological properties.

THERMAL FIELDS IN GRAIN DURING STORING – THEIR SOURCES AND EFFECTS ON SILO STRUCTURES

Andrzej Lapko

Institute of Civil Engineering, Białystok Technical University
ul. Wiejska 45e, 15-351 Białystok, Poland
e-mail: lapko@kmb.pb.bialystok.pl

A particulate organic solid stored in silo, like grain, should be considered as a man – made ecological system subjected to different kinds of actions and influences which may cause the deterioration or even spoiling effects in storing masse and sometimes also in silo bin structure. Among others the great importance play thermal and moisture fields appeared in the period of silo operation. Such thermal effects on silo structure and on stored agro – material are depending on numerous factors, first of all the type of material, size of the silo bin, its shape and structure and also climate and weather changes in period of storing process.

Basing on the relevant scientific publications and own research works have done at the Białystok Technical University an overview of thermal effects in reinforced concrete silo bins during storing of granular agro – materials will be specified and discussed in the paper focusing on the environmental influences. These effects were studied both experimentally and theoretically using numerical approaches.

The paper presents some results from experimental works conducted on grain silos in some Polish elevators concerning temperature distributions over the silo wall perimeter and also on the wall thickness registered in summer and winter periods. Also selected results of thermal strains appeared in silo bin walls during storing will be discussed and compared with the results of numerical estimation of such thermal effects in cylindrical reinforced concrete silo with grain. Taking into account the silo structure reliability requirements these studies showed the importance of analysis of thermal forces is silo structure design.

EFFECTS OF INHOMOGENEITY, ELASTICITY AND WEAR-IN ON LOADS IN A MODEL SILO

Marek Molenda, Józef Horabik

Institute of Agrophysics, Polish Academy of Sciences
ul. Doświadczalna 4, 20-290 Lublin, Poland
e-mail: mmolenda@demeter.ipan.lublin.pl

Standard procedures currently used to estimate the loads in silos assume constant grain properties throughout the silo and do not take into account such variations as the frictional interaction between grains or particle elasticity. This work addresses the variation in wall loads in thin, flat floor metal silos caused by the variation in the mechanical properties of the grain bedding and friction forces acting on the grain-wall interface. The majority of the work reported here was performed using a model silo 2.44 m in diameter and 7.3 m tall, having corrugated or smooth wall made of galvanized steel. The silos were constructed such that the wall and floor (flat bottom) were each supported independently on three load cells. This type of experimental configuration allowed for the determination of vertical wall and vertical floor loads as well as for the calculation of bending moments on the walls and floor of the silo.

Inhomogeneity of grain bedding

Certain loading and/or unloading conditions can create a non-uniform distribution of pressure within a grain silo during operation of a grain facility. Eccentric discharge of material from bins is most-often associated with non-symmetrical silo loads however other factors can contribute to the non-uniformity of loads. Considerable asymmetry of loads was observed even during notional axial filling of the model silo. Eccentric filling produced much larger non-uniformity of load distribution with the maximum bending moment reaching 70% of the bending moment observed during eccentric discharge after centric filling. Eccentric filling followed by eccentric discharge either magnified or reduced the non-uniformity of distribution of stress within the silo, depending on the mutual location of the filling and discharge gates.

Elasticity of grain

Experiments have shown that the elasticity of grain significantly influenced the wall and floor loads during discharge. During the final stages of discharge changes in the magnitude and direction of the resultant friction force against the bin wall

were observed which were attributed to the elastic strain recovery in the grain. The moisture content of grain was found to influence the magnitude of the maximum upward force because of changes in the grain compressibility.

The elasto-plastic hysteresis loading characteristics of grain was also found to affect the magnitude of the wall loads in a model silo. During experiments in which the model silo was partially loaded and unloaded the relationship between the ratio of vertical wall loads (VWL), both static and dynamic, to gross grain loads (GGL) were found significantly influenced by the number of fill-unload events. An envelope of experimental VWL/GGL ratio values as a function of the height to-diameter ratio (H/D) of the stored grain during cycles of fill, rest and at the beginning of unloading was approximated by a relationship derived using Janssen equation.

Grain stored in silos can become subject to rewetting if exposed to an adverse storage environment. When the grain absorbs moisture, kernels swell, and the grain bulk tends to expand. This expansion creates an increased loading on the bin structure. Experiments were performed in which the swelling pressure of wetted grain was measured in a model silo. An analytical model was developed which allows for the determination of the pressure increase in uniform bedding under established experimental conditions.

Wear-in effects in smooth wall silo

It was determined that the first fill-unload cycle in a new silo is the most critical for tall slender smooth-walled silos operating in the mass flow regime. In subsequent fill and unload cycles a reduction in the friction coefficient between the grain and the silo wall was observed. The values of $k\mu$ were found to decrease about three fold during 23 loading cycles. Vibrations that resulted in the oscillations of the wall and floor loads resulting from the stick-slip friction were observed during the first three load cycles. The dynamic-to-static wall load ratio increased in a factor of 1.15 during the first ten loading cycles.

DISCRETE ELEMENT MODELLING OF GRANULAR SYSTEMS

Jin Y. Ooi

Institute for Infrastructure and Environment, University of Edinburgh
The King's Buildings, Edinburgh EH9 3JN, United Kingdom
e-mail: j.ooi@ed.ac.uk

Over the last few decades, there has been an increasing research effort in developing computational models for the behaviour of granular solids. The two main categories are the continuum finite element (FE) method and the discrete element (DE) method. The FE continuum models can often give satisfactory answers to questions about bulk global behaviour, but they have difficulty representing discrete rupture surfaces and flow boundaries, because discontinuities in continua are still not easily modelled.

The DE method is a relatively new technique for simulating moving granular particles (Cundall, 1971). It is based on the use of an explicit numerical scheme in which the interactions between a finite number of particles is monitored contact by contact and the motion of the particles is modelled particle by particle. Newton's equations of motion for each particle effectively replace the equilibrium equations used in continuum mechanics, and the model of inter-particle contacts replaces the constitutive model. The essential feature of this approach is that each particle is modelled separately, so the integrated behavior of the mass is accurately represented, without the need for control tests to establish constitutive models of the bulk behavior.

This lecture will first presents an overview of the DE method. The strengths and the weaknesses of the method will be discussed. The method requires single particle properties as input parameters, so the methodologies for measuring these properties for use in DE simulations will also be discussed.

To date, the majority of the DE studies have been confined to either 2D or spherical particles or both. However real systems containing granular solids are almost always dealing with non-spherical particles in interaction with machinery surfaces. The second part of the lecture will focus on the recent development of a DE computational model for irregularly shaped 3D particles interacting with static/moving surfaces of complex geometry. This DE solver allows a much more realistic modelling of particles with increasing complexity. The recent successful coupling with FLUENT Computational Fluid Dynamics code now provides a platform for integrating particle, fluid and machine dynamics. Some examples applications will be described.

NUMERICAL MODELING OF SHEAR LOCALIZATIONS DURING GRANULAR FLOW IN SILOS

Jacek Tejchman

Civil Engineering Department, Gdańsk University of Technology
ul. Narutowicza 11/12, 80-952 Gdańsk-Wrzeszcz, Poland
e-mail: tejchmk@pg.gda.pl

During confined granular flow in silos, localizations of deformation occur in the form of shear zones along walls and inside of the flowing material which contribute to the oscillation of silo pressures and non-symmetry of flow.

The paper deals with a numerical modeling of shear localizations during silo flow with a finite element method based on an extended hypoplastic constitutive law. Hypoplastic constitutive laws are an alternative to elasto-plastic formulations for continuum modelling of granular materials. In contrast to elasto-plastic models, the decomposition of deformation components into elastic and plastic parts, yield surface, plastic potential, flow rule and hardening rule are not needed. They describe the behaviour of so-called simple grain skeletons assuming that the macroscopic state can be sufficiently described by mean values of stress and void ratio. They can reproduce essential features of granular bodies in dependence on the void ratio, pressure level and deformation direction. They are characterised by simplicity and a very wide range of applications. The material constants can be found by means of standard element tests and simple index tests.

Classical FE-analyses of shear zones are not able to describe properly both the thickness of localization zones and distance between them since they suffer from spurious mesh sensitivity (its size and alignment). The rate boundary value problem becomes mathematically ill-posed. Thus, classical constitutive models require an extension in the form of a characteristic length to regularize the boundary value problem. It can be achieved with polar, non-local and gradient terms.

The polar terms were introduced in a hypoplastic law with the aid of a polar (Cosserat) continuum. A Cosserat continuum takes into account two linked levels of deformation: micro-deformation at the particle level and macro-deformation at the structural level. Each material point has for the case of plane strain three degrees of freedom: two translational degrees of freedom and one independent rotational degree of freedom. The gradients of the rotation are connected to curvatures which are associated with couple stresses. It leads to a non-symmetry of the stress tensor and a presence of a characteristic length in the form of a mean grain size.

A non-local approach is based on spatial averaging of tensor or scalar state variables in a certain neighborhood of a given point (i.e. material response at a point depends both on the state of its neighborhood and the state of the point itself). To obtain a full regularization effect according to both the mesh size and mesh inclination, it is sufficient to treat non-locally only one internal constitutive variable whereas the others can retain their local definitions. The FE-calculations were carried with a non-local modulus of the deformation rate (stresses, strains and other variables remained local). The error density function (normal Gaussian distribution function) was chosen as a weighting function.

The gradient approach is based on the introduction of a characteristic length by incorporating higher order gradients of strain or state variables into the constitutive law. In the calculations, the second gradient of the modulus of the deformation rate was used. To evaluate the gradient terms, instead of additional shape functions, a standard central difference scheme was employed.

The FE-analysis was performed with an enhanced hypoplastic constitutive laws for dry granulate during quasi-static plane strain flow with controlled outlet velocity in a silo with convergent walls. The calculations were carried with different wall roughness, initial void ratio and silo size. The initial void ratio was stochastically distributed in the solid. The numerical results were compared with model tests.

CHOSEN MORE RECENT APPROACHES TO PLS MODELLING IN APPLICATION TO SPECTRAL DATA

Grażyna Balcerowska¹, Ryszard Siuda¹, Henryk Czarnik-Matusiewicz²

¹Institute of Mathematics and Physics, University of Technology and Agriculture
ul. Kaliskiego 7, 85-796 Bydgoszcz, Poland
e-mail: gbalcer@mail.atr.bydgoszcz.pl

²Department of Clinical Pharmacology, Faculty of Pharmacy, Medical University
ul. Bujwida 44, 50-345 Wrocław, Poland

A well-known problem in chemometrics is to estimate a linear relationship between two sets of variables, e.g. the set of spectra, \mathbf{X} , and the concentrations of some of sample constituents, \mathbf{Y} . Among the classical regression methods Partial Least Squares (PLS) is one of the most commonly used tool. Because the model parameters are estimated based on the empirical variance-covariance matrix of the data, the approach is very sensitive to outlying measurements. The presence of outliers is one of complications in application of PLS and it can be overcome by use of a robust calibration approach. Recently an effective algorithm of the robust PLS based on a robust covariance matrix has been developed [1-3].

Another complication which could negatively affect the interpretation of the PLS model refers to the systematic variation present in \mathbf{X} that is unrelated with the variation in \mathbf{Y} . This situation typically occurs when \mathbf{X} variables represent the absorbance or reflectance of light intensities measured at hundreds of wavelengths, and the measurements are influenced by various sources of different types of variation having nothing in common with the information of interest. Orthogonal Signal Correction (OSC) is a recently proposed preprocessing method that seems to be promising in this context [4-7]. This approach determines and removes from spectral data \mathbf{X} the part of information which is \mathbf{Y} -orthogonal (i.e. not correlated with \mathbf{Y}).

The purpose of the present paper is to illustrate how both mentioned above techniques work in application to NIR spectra of rapeseed meal with reference to the results obtained by standard use of PLS method.

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STANDARDIZATION CONCERNING GRANULAR MATERIALS IN POLAND 2004

Andrzej Bieganowski, Marek Molenda, Józef Horabik

Institute of Agrophysics, Polish Academy of Sciences
ul. Doświadczalna 4, 20-290 Lublin, Poland
e-mail: a.bieganowski@demeter.ipan.lublin.pl

During last 50 years substantial increase in amount of granular materials on the market took place. This was a result of quickly growing competition and scale of industrial operations caused by globalization. Material in granular form is very convenient for storage, transport, handling, mixing, dosing and other operations. Communications between producers and between designers require common language and common procedures of determination of material parameters that enforces standardization. Numerous national and international committees have been recently working around the world on standards concerning granular materials. This work is a review of Polish Standards done to reveal areas of current interest of technology in granular materials. Standards were selected from the list of Polish Committee for Standardization using keywords: *granu, sypek, zboz* (first letters of polish terms for granular, loose and grain). Documents concerning metallurgy and machines were rejected. Out of 182 standards that remained the majority of 97 addressed questions of food and agro materials, in the prevailing number of cases characterization of grain, seeds and food powders. Standards concerning chemical industry were present in a number of 48, while 21 standards addressed questions of protection of environment. The fourth group formed standards issued for civil engineering, mainly concerning characterization of aggregates. Testing techniques are interest of large group of analyzed standards. Among them the largest of number of 41 is the group regarding grain size distribution. Density and moisture content are the next properties in need. The remaining standards concern rather large group of different properties important for various branches of technology.

The review of standards show that in the fields of science and technology much more methods of material characterization are available, and most probably, soon will be adopted by industrial practice as standards.

THERMOPHYSICAL PARAMETERS OF CHOSEN GRANARY SAMPLES

Monika Božíková

Faculty of Agricultural Engineering, Department of Physics, University of Agriculture
Tr. A. Hlinku 2, 949 76, Nitra, Slovakia
e-mail: Monika.Bozikova@uniag.sk

This article deals with thermophysical properties of granary mass. It is necessary to know thermophysical performance of granary mass for protection of quality of technological process by processing to final products. Granary mass consist of grains complex of specific kind. It is non – uniform material in microscopic and macroscopic structure. There are enacted biophysical and physiological processes. Heat transfer can not be isolated by solid transfer and heat – moisture transfer. It means that specification of granary mass and granary fragments is difficult to determine. We researched thermal properties of granary mass.

Thermophysical parameters were measured by instrument Isomet, it is made by company Applied Precision. It is used for quick and exact measurement of thermophysical parameters of various materials. Measurements were performed with spike probe with range $0.015\text{-}0.2\text{ W m}^{-1}\text{ K}^{-1}$. Spike probe was inserted into the analysed material. Probe is generating a heat. Time process of temperature which is related with thermophysical parameters of samples, that were analysed.

In the first series of measurements we measured thermal conductivity, thermal diffusivity of wheat, food wheat, malt barley and colza. The moisture content of samples was the same during the measurements. All measurements were realised at the room temperature.

Because the moisture content and the bulk density are very important parameters which determine thermophysical parameters of biological materials, we made in the second series of measurements relations of thermal conductivity and thermal diffusivity for chosen samples of wheat and food wheat to the moisture content in range 2-18%. In the third series of measurements we made relations of thermal conductivity λ to bulk density ρ_s and thermal diffusivity a to bulk density ρ_s . Samples had identical moisture content 6.5%. For different size of fragments – wheat grains and fragments of food wheat were measured relations $\lambda = f(\rho_s)$ and $a = f(\rho_s)$ for fragments of grains with dimension 0.5-2 mm and for fragments of grains with dimension 0.063-0.5) mm. Measured results are corresponding with results at the literature.

FLOWABILITY OF THE WHEAT FLOUR AS AFFECTED BY MOISTURE CONTENT, STORAGE TIME AND CONSOLIDATION

Ewa Domian, Karolina Poszytek

Department of Food Engineering and Process Management, Warsaw Agricultural University
ul. Nowoursynowska 159c, 02-787 Warszawa, Poland
e-mail: domian@alpha.sggw.waw.pl

The effect of moisture content, storage time and consolidation time on the flowability for two kinds of wheat flour: type 500 (poznanska) and type 2000 (whole meal) was evaluated. The flours differed each other with particle size distribution and chemical composition. Powder flowability was measured using uniaxial shear tester according to Jenike procedure at four levels of normal consolidating stress at the range 4.9-17.5 kPa. Flours were placed in a humidity chamber to obtain following samples: wheat flour poznanska at 0.33, 0.65 and 0.8 water activity, wheat flour whole meal at 0.65 and 0.8 water activity. Instantaneous shear tests were performed with controlled relative humidity (RH 33, 65 or 80%) at temperature 20°C on each flour, from which the instantaneous flow-function were obtained. Time o consolidation tests on the flour poznanska at 0.33 and 0.8 water activity were carried out using a Jenike shear cell and consolidating bench for the following two consolidation times: 1 and 7 days. Temporal flow functions of the flour poznanska were determined to quantify the combined effects of moisture content, time and compression stress. A number of physical properties, including particle size distribution, bulk density and water sorption isotherms were used in interpreting and comparing the flowability measurements for each flour.

ANALYSIS OF SEED DENSITY DISTRIBUTION

Marek Domoradzki, Wojciech Korpala, Wojciech Weiner

Faculty of Technology and Chemical Engineering, University of Technology and Agriculture
ul. Seminaryjna 3, 85-326 Bydgoszcz, Poland
e-mail: keram@atr.bydgoszcz.pl

Harvested plant seeds constitute a collection of particles of varied size and density. For many species higher density seeds terminate better than rest of the lot. During imbibition the volume of seeds increases and their density changes. As the densitometric classification reportedly gives fractionation that correlates better with seed germination it was of interest to study dependence of density distribution on the imbibition time.

In the course of seed imbibition seed density distribution undergo narrowing. This phenomenon could be rationalized by initial differences in water absorption velocity.

Content of seeds of very high density decreases with imbibition time and after 8 hours a normal distribution around the average of 1.10 g ml^{-1} .

As small density seeds as well as large density seeds are always present within harvested seed lot the presented method of gravimetric classification of imbibed seed in the range from 1.06 to 1.13 g ml^{-1} can be useful for separation of poorly germinating seeds.

DEPENDENCE OF GERMINATION PARAMETERS ON SEED SIZE

Marek Domoradzki, Wojciech Korpala, Wojciech Weiner

Faculty of Technology and Chemical Engineering, University of Technology and Agriculture
ul. Seminaryjna 3, 85-326 Bydgoszcz, Poland
e-mail: keram@atr.bydgoszcz.pl

After being harvested vegetable seeds undergo cleaning by sifting. Vibrating sieves with air flow are most often used to remove seeds that are too light or empty.

Both laboratory and field-testing of size-fractionated seeds have shown that that the seed germination and the seed vigor are often dependent of the size of the seeds. Larger and heavier seeds yield most often larger plants as well as better and more even crop establishment and increased further productivity.

Generally the extremely large seeds are full of cracks and contaminated by fungi and other pathogens while the smallest seeds are most often immature and germinate poorly.

Classification of seeds with respect to their size (diameter) is performed usually in various sieve sifters. In this investigation a continuous vibrational sifter constructed in the University of Technology and Agriculture in Bydgoszcz, Poland.

The sifter was equipped with sheet metal sieves with circular holes of diameter series: from 0.8 mm to 3.6 mm for every 0.2 mm and also from 2 mm do 6 mm for every 0.5 mm. The sifter was fed from a feeder yielding 5-10 kg hr⁻¹.

Classification of various vegetable seed lots was performed resulting in diversified fraction specific SG (seed germination). Such a classification facilitates: separation of well germinating from poorly germinating seeds, separation from the well germinating fractions those of extremely high SG (quality extra fraction) and rejection of poorly performing seeds and thus achievement of higher SD for the remaining seeds.

INFLUENCE OF WHEAT DEBRANNING RATIO AND MOISTURE CONTENT ON KERNEL MECHANICAL PROPERTIES

Dariusz Dziki, Janusz Laskowski

Department of Machine Operation in Food Industry, University of Agriculture
ul. Doświadczalna 44, 20-236 Lublin, Poland
e-mail: uldar@faunus.ar.lublin.pl

The aim of the present work was to determine the influence of kernel debranning ratio and moisture content on wheat mechanical properties. The Polish winter wheat cultivar (*T. aestivum*) Turnia, about different debranning ratio, was used for investigation. Samples were tempered to 12, 14 and 16% moisture. Individual kernels were compressed. The loading force was acting along kernel's thickness. The mechanical properties of wheat were carried out on testing machine Zwick Z020/TN2S. On the basis of the obtained curves the immediate resistance threshold and the collapse threshold was evaluated, and corresponding values of forces and deformations were determined. The values of work and specific work were also determined.

The results showed that the debranning caused decrease the deformation of kernel up to the immediate resistance threshold (average about 29%). However this deformation increased as the moisture of kernel increased. The debranning had the highest influence on force up to the immediate resistance threshold when the moisture of kernel was 12% (decrease of force form 123 to 75 N). The values of this force obtained for samples about 14 and 16% of moisture content were not statistically significantly different ($\alpha = 0.05$). The water content had a little influence on force up to the kernel collapse threshold. As the debranning ratio of kernel increased the force decreased (form 1500 to 550 N). It was also shown that the values of work up to the immediate resistance threshold obtained for debranning kernel were about 83% less form the values obtained for undebranning wheat. As the moisture of kernel increased the values of this work also increased (about 100%). However the moisture content has no significant influence on work up to the collapse threshold.

The results showed that the debranning of kernel had an influence on wheat mechanical properties. The results depended also on the moisture of kernel.

DESIGN MODEL OF CORN GRANULATION AND GRINDING PROCESS

Józef B. Flizikowski

Faculty of Mechanical Engineering, University of Technology and Agriculture
ul. Kaliskiego 7, 85-796 Bydgoszcz, Poland
e-mail: fliz@mail.atr.bydgoszcz.pl

The semi- or fully intelligent development machinery: grinders and granulators construction, especially corn of grain grinders is not only because of the standardization by the European Commission today a hot topic. One of the consequences is the increasing inefficiency of not automated in the next years. The paper deals with a robotized, a integron as the model for semi-intelligent development design of grain particles grinders. Integron, i.e. the mathematical model of runs and instantaneous, average values as well as standard expedient, deviations of energetic and plane shapening of sections, forces and energy of disintegration in creation with the application of genetic algorithms (AI), innovative solutions of new generation multisided seeds grinders has been presented in the study.

If we treat the grinder as an existence, individual features of which (genes) have been described in details with the application of intervals of admissible values, we shall be able to build a population composed of individuals (shedders) generated by way of "random selection". The constructional form of a working complex has been reduced to a multi-shield, multi-opening system and the following from the admissible Φ set are subject to selection: constructional features of the assembly, shields; numbers of shields, openings from the assigned interval of variability. The population shall be subject to evolution on the strength of selection based on construction *integrons*: intersection, force and energy.

In order to obtain a constructional solution, genetic operations such as crossing, mutation and selection are performed on each at random selected pair of individuals. Drawings presents the diagram of operation of a genetic algorithm applied in the strategy of construction innovation, construction development of grain particles grinders.

ELECTRIC PROPERTIES OF GRANULAR AND POWDERY MATERIALS UTILIZATION

Zuzana Hlaváčová

Slovak University of Agriculture
Tr. A. Hlinku 2, 949 76 Nitra, Slovak Republic
e-mail: Zuzana.Hlavacova@uniag.sk

Determination of electrical properties is utilized in a wide range of disciplines and industries. A brief compendium of granular and powdery agricultural and food materials electrical properties exploitation is presented. Electrical properties of granular and powdery materials are influenced by various factors. The most important of these factors are the moisture content and its asymmetrical distribution in materials, temperature, density, volume density or bulk density. The relationship between the resistivity, conductivity, impedance, relative permittivity and various influencing factors are inscribed.

The electrical conductivity have utilization at the salinity of soils and irrigation water determination. Biological material properties are determined from their leachates too. The conductivity measurement are applied for determination of various characteristics of agricultural materials and food, for example for determination of the frost sensitiveness, of chilling and freezing tolerance, of moisture content, of seeds germination, of mechanical stress, of pasteurization, of other properties of grains, seeds, sugar, soil, fruit and vegetable, infected food, ... The utilization of dielectric properties are also described; for example in agricultural materials and food quality sensing (moisture content, potential insect control in seeds, radio frequency heating, ...). The classification of permittivity measurement techniques is mentioned.

MEASUREMENTS OF ELECTRICAL PROPERTIES FOR GRANULAR AGRO-MATERIALS*

Marek Horyński

Faculty of Electrical Engineering, Technical University of Lublin
ul. Nadbystrzycka 38A, 20-618 Lublin, Poland
e-mail: mhor@elektron.pol.lublin.pl

As far as organic dust can be removed by electrical methods, it is essential to recognize dust electrical properties. It is also interesting if the methods based on measurements of electrical properties can be applied to verify milled fraction ratio for dielectrics of plant origin. The paper presents a laboratory stand and measuring methodology for milled fractions obtained from grain. The frequency characteristics of real component and imaginary component of electric permittivity for tested dust fractions obtained from wheat grain milling have been described on the basis of the tests that have been carried out within the research. The suitable fractions and size class of the dust have been prepared by means of a small disc mill. Moreover, the other measurements have been carried out for wheat flour fractions made during industrial processing in a mill. On the basis of these measurements the research has been carried out on correlations between the influence of frequency on dust electric permittivity components for both origins from the laboratory mill and from the industrial milling. The results of measurements and computations have been statistically analysed.

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COMPARISON OF MLR AND PLS MODELS IN COMPOSITIONAL ANALYSIS OF RAPESEED MEAL FROM NIR SPECTRA

Jarosław Jankowski¹, Henryk Czarnik-Matusiewicz², Ryszard Siuda¹

¹Institute of Mathematics and Physics, University of Technology and Agriculture
ul. Kaliskiego 7, 85-796 Bydgoszcz, Poland
e-mail: zebee@poczta.onet.pl

²Department of Clinical Pharmacology, Faculty of Pharmacy, Medical University
ul. Bujwida 44, 50-345 Wrocław, Poland

Near infrared reflectance spectroscopy (NIRS) followed by multivariate calibration is an analytical procedure of practical usability in compositional analysis of some constituents of rapeseed (see, e.g., [1-5]). Multivariate calibration methods like MLR (Multiple Linear Regression), PCR (Principal Components Regression) and PLS (Projection on Latent Structure or, alternatively, Partial Least Squares) are typically used to determine chemical composition from NIR spectra [6], of them PLS seems to be a standard tool [7]. One of the advantageous features of PLS is that it uses the so called latent variables combined from the whole set of independent (predictor) variables in such a way as to maximise correlation with a given dependent (response) variable. In spectroscopy, however, the measurements provide hundreds or thousands of channels (variables), and often considerably part of the variables carries no, or almost no, important information except noise. For this reason MLR, although not directed to explore connections between structures of predictor and response sets, can provide results better than PLS. Therefore it is worth checking which of the methods works better in a given application. The present contribution aims to compare the results of both methods when applied to calibration of basic constituents (oil, dry matter, protein, ash, fibre) of rapeseed meal from NIR reflectance spectra. The set of spectra consists of 69 spectra recorded between 1100 to 2500 nm. Selection of wavelength needed to apply MLR has been made with two procedures. Both PLS and MLR calibrations were performed with PLS_Toolbox 3 package.

In modelling with MLR a key question is proper choice of selected predictor variables. In general, performance parameters (like R^2 , SEC and SECV) of the models show that better results can be obtained for all constituents from MLR (eg.), although for ash both methods provide not satisfactory results (R^2 close to 0.6).

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DETERMINATION OF THE INFLUENCE OF TILLAGE ON THE SOIL STRUCTURE BY FRACTAL ANALYSIS

Lubomír Kubík¹, Ladislav Nozdrovický²

¹Department of Physics, Slovak University of Agriculture
Tr. A. Hlinku 2, 949 76 Nitra, Slovak Republic
e-mail: Lubomir.Kubik@uniag.sk

²Department of Machinery and Production Systems, Slovak University of Agriculture
Tr. A. Hlinku 2, 949 76 Nitra, Slovak Republic

The contribution deals with an evaluation of soil structure by unconventional method of fractal analysis. The soil is represented as the pore medium with statistically self-similar structure which represent fractal structure. The fractal dimension is presented as decisive representative of the structural properties of soil. The fractal dimension is non-integer number which express a degradation of soil structure which was caused by pores. This number include complexly the modification of structural properties of soil induced by arbitrary influence. The fractal dimension of clayey-loamy soil was related to the activity of technogen factors which represented the influence of exactly defined tillage technologies (conventional and reduced) in the depths of 0-3 cm and 6-9 cm. Furthermore, the impact of fertilisation (NPK) on the surface structure of soil was examined. Intensity and statistic significance of individual factors were determined by analysis of variance. The effects of technologies on the modification of surface soil structure represented by fractal dimension and the influence of the tillage depth and fertilisation treatments were confirmed.

INVESTIGATION OF FABA BEAN MECHANICAL PROPERTIES FOR GRINDING BEHAVIOR PREDICTION

Grzegorz Łysiak, Janusz Laskowski

Department of Machine Operation in Food Industry, University of Agriculture
ul. Doświadczalna 44, 20-236 Lublin, Poland
e-mail: Lysiak@faunus.ar.lublin.pl

Paper presents results of studies concerned to the relationships between material mechanical properties and its grinding process parameters. Polish variety of faba bean *Bobik Nadwiślański* was used in the studies. Prior to experiments seeds were tempered to different moisture contents, in a range of 8-18% (w.b.). Universal testing machine Zwick Z020 was used to material properties determination. On the basis of load-deformation curves, characteristic values of loads, seed deformations, and energy inputs during crushing were determined. Grinding experiments were done with a help of small laboratory hammer mill, Polymix Micro Hammermill. Specific energy utilization and particle size distribution were determined.

Influence of moisture on the character of load-deformations curves and specific energy requirements during grinding were evaluated. For low material moisture levels, sharp fracture as a result of load (internal stresses) was observed. More relevant contribution of strains, plastic in nature, for moist seeds resulted in their distinct fracture mechanism. The increase of seeds moisture caused their larger deformability relative to the loads applied. Grinding process of moist bean seeds required more energy to be utilized. Relevant relations of energy requirement according to change in seeds moisture and their mechanical properties were observed.

DYNAMIC EFFECTS DURING EMPTYING OF LARGE SILOS

Maciej Niedostatkiewicz

Department of Fundamentals of Buildings and Material Engineering, Faculty of Civil Engineering
Gdańsk University of Technology
ul. Narutowicza 11/12, 80-952 Gdańsk, Poland
e-mail:mniedost@pg.gda.pl

Dynamic effects in bulk solids are an inherent characteristic of each silo discharge. They occur in form of pulsations and shocks. In dry granular materials (non-cohesive) only pulsations are created. In case of cohesive bulk solids, pulsations and shocks can be observed at the same time. The intention of this paper is to present measurements of dynamic effects in large silos caused by 2 different reasons: resonance between the bulk solid and silo system and resonance between technological devices and silo system.

Measurements in large silos were performed with silos containing polymer granulate, sugar and soya bean. In the case of a silo containing polymer granulate ($d = 1.80$ m, $h = 4.40$ m), dynamic effects were due to resonance between the bulk solid and silo system. In a silo, where sugar was stored ($d = 1.90$ m, $h = 4.80$ m), dynamic effects were induced by a mechanical exciter to promote flow of sugar due to cohesion. In the case of a silo containing soya bean ($d = 12.5$ m, $h = 24.5$ m), dynamic effects were caused by a selector and feeder.

During tests in large silos, vertical and horizontal accelerations on the silo wall were measured. Moreover, the effect of the outlet velocity on accelerations was investigated.

APPLICATION OF TOMOGRAPHY TO GRANULAR SILO FLOW

Maciej Niedostatkiwicz, Jacek Tejchman, Zbigniew Chaniecki

Department of Fundamentals of Buildings and Material Engineering, Faculty of Civil Engineering
Gdańsk University of Technology
ul. Narutowicza 11/12, 80-952 Gdańsk, Poland
e-mail:mniedost@pg.gda.pl

The intention of this paper is to present results of density changes by tomography during silo flow with very strong dynamic effects. The method is suitable for cylindrical silos with a diameter lower than 0.35 m. The tomographic images were obtained using the Electrical Capacitance Tomography (ECT) system capable to gather 100 images of density changes per second. The 12-electrode sensors with a factor of the length to the diameter equal to 1 were used. The sensors were located outside of the cylindrical silo model made of perspex (height 2000 mm, diameter 200 mm, wall thickness 4 mm) at two different levels. The silo model was supported by a steel frame. The experiments were carried out in co-operation with the Technical University of Łódź, Faculty of Electronics and Electrical Engineering, Computer Engineering Department and Warsaw University of Technology, Faculty of Electronics and Information, Nuclear Technology and Medical Electronics Division.

During experiments, both dry and cohesive sand (mixture of sand with water and mixture of sand with glicerine) were used. In the case of dry sand, the initial density varied. The influence of the outlet diameter and wall roughness on the results was also examined. In addition, vertical, horizontal and circumferential accelerations on the silo wall were registred.

CHARACTERISTIC FEATURES OF SILO WALL PRESSURE DISTRIBUTION FOR SILO DESIGN

Jin Y. Ooi, J. Michael Rotter

Institute for Infrastructure and Environment, University of Edinburgh
The King's Buildings, Edinburgh EH9 3JN, United Kingdom
e-mail: j.ooi@ed.ac.uk

Over the last few decades many silo failures have been reported in all developed countries. Many of these failures have been attributed to a significant mismatch between the design silo wall pressure loads and those arising in service. To address this problem, many experimental studies have been conducted over the last few decades to measure and study the pressures that develop on silo walls during filling, storing and discharging of solids.

Unfortunately, many of these experiments have been conducted with an inadequate appreciation of what is required, especially in terms of the pattern of pressure developing on the silo walls. As an example: it is often assumed that the highest pressure is the most damaging to the silo structure and it is seldom recognised that a pressure which varies between 20 kPa and 40 kPa around the circumference can be much more dangerous to the structure than a uniform pressure of 60 kPa. Thus the lowest pressure at a certain instant may be an important observation, but such a value is almost never reported from experimental studies.

This paper presents an analysis of a series of very carefully conducted experiments on a 46 m high concrete silo in Sweden. The focus of the work is to identify the key features of the pressure variation around the circumference at different levels on the silo walls, using statistical analysis of the full set of pressure observations to identify characteristic values that could be used for structural design assessments. The changes in pressure from filling to discharge are similarly described. The statistical analysis gives a rigorous basis for the interpretation of silo pressure experimental data and leads to a reliable quantification of unsymmetrical loads for silo design.

THE PRESSURE RATIO OF RAPE SEEDS

Robert Rusinek, Mateusz Stasiak

Institute of Agrophysics, Polish Academy of Sciences
ul.Doświadczalna 4, 20-290 Lublin, Poland
e-mail: rrusinek@demeter.ipan.lublin.pl

The horizontal to vertical pressure ratio k is one of the three most important parameters required for the calculation of stresses that granular materials exert on the wall and floor of a silo, introduced by Janssen [1895]. The author assumed that the vertical pressure was uniform in a horizontal section of the silo and that ratio k was constant everywhere within the material. The pressure ratio depends on type of grain, moisture content, bulk density and bedding structure of grain formed during the filling process. The silo design standards recommends to determine experimentally parameters of stored materials for each specific silo design.

The objective of reported project was to determine the pressure ratio of rape seeds for four levels of moisture content. This project utilized model silo and uniaxial compression tester for determination the horizontal to vertical pressure ratio. The model silo 0.6 m high and 0.6 m in diameter was constructed and instrumented to measure mean pressure ratio k , mean tangent stress on the wall σ_t , as well as distribution of vertical pressure σ_z along the radius of the silo. Second method was uniaxial compression test, this is the most popular method of experimental determination of the lateral to vertical pressure ratio. An experimental set for the uniaxial compression was built according to the general guideline of the Eurocode 1 standard. The experimental set allowed for determination of the mean lateral pressure σ_x , mean vertical pressure on the bottom σ_z , and the mean vertical pressure on the top plate σ_{z0} . Experiments were performed for four levels of moisture content (6, 9, 12 and 15%) for rape seeds variety Lirajet and Licosmos. Each variant of the experiment was performed in three replications.

The friction force and the cohesion between seeds increase with increasing moisture content. As a result the pressure ratio decreases, both of tests for rape seeds variety Lirajet and Licosmos for large (0.17 m^3) and little (0.0035 m^3) capacity. The values of pressure ratio obtained in model silo were higher from uniaxial compression test, only at 6% of moisture content were similar. Experimental values of the pressure ratio from compression tests were similar to theoretical values obtained for yielding at the center of a silo in the active case.

**APPLICATION OF OPTICAL TECHNIQUE DIPV
ON THE INVESTIGATION OF GRANULAR MATERIAL BEHAVIOUR
IN BINNS/HOPPERS**

Irena Sielamowicz¹, Tomasz A. Kowalewski²

¹Civil and Environmental Engineering Department, Chair of Structural Mechanics
Białystok Technical University, ul. Wiejska 45 E, 15-351 Białystok, Poland
e-mail: sieliren@pb.bialystok.pl

²Institute of Fundamental Technological Research, Polish Academy of Sciences
ul. Świętokrzyska 21, Warszawa, Poland
e-mail: tkowale@ippt.gov.pl

The objective of this paper is to describe the inherently transient process of granular material flows quantitatively in a plane model of silo built of Plexiglas using the optical technique DIPV (Digital Particle Image Velocimetry). The transparent walls of the model allow for flow visualisation and PIV (Particle Image Velocimetry) measurements of velocity fields and local stresses in the material. Two granular materials were used in the experiments – amarantus seed and flax-seed. These materials develop negligible static electricity when flowing and sliding over Plexiglas. The experimental setup consists of high resolution camera (SensiCam) permitting to acquire about 200 pairs of 1280x1024 pixels images with frequency of 3.75 Hz. Evaluation of grains displacement (and velocity) is performed for each pair of images, taken at the interval 0.266 s. The high speed camera is used to obtain temporal characteristics of the flows. Vector fields, stream lines, velocity distributions and the traces of flowing particles in consecutive stages of the flow for two selected materials is presented. A sequence of digital images of moving materials in the model is analysed with PIV. The zones, where the material is in motion and the stagnant zones are characterised. These zones are not constant but slightly changing their location with respect to time. The change of local velocity with time is demonstrated with this method providing an insight into the dynamic behaviour of the granular material. The fluctuations of material motion lead to changes of the wall stresses. In the initial phase of the flow the whole material lowers vertically and the vector lines in the bottom part of the model are directed towards the outlet. While flowing of the grains of oblong shape some irregular vector lines are observed. Another stage of the flow – material still lowers and the boundaries of the flow are vertical but only the boundaries near the outlet are curved. Some irregular vector lines appeared in the pictures detect the phenomenon of static electricity of the material and correspond to the shape of the grains. The results on the profiles of vertical velocity components across the cavity obtained at height $h = 15$ cm at time steps 3.75 s, 30 s, 52.5 s after the beginning of the experiments are presented in the paper. Velocity distributions of the flowing material at the consecutive time steps and on the certain sections in the model at time steps 3.75 s, 15 s, 30 s, 40 s, 52.5 s and 60 s are shown as well.

ANALYSIS OF STRESS EVOLUTION IN CONVERGING HOPPERS*Irena Sielamowicz¹, Zenon Mróz²*

¹Civil and Environmental Engineering Department, Chair of Structural Mechanics
Białystok Technical University
ul. Wiejska 45 E, 15-351 Białystok, Poland
e-mail: sieliren@pb.bialystok.pl

²Institute of Fundamental Technological Research, Polish Academy of Sciences
ul. Świętokrzyska 21, Warszawa, Poland
e-mail: zmroz@ippt.gov.pl

The simplified elasto-plastic analysis of deformation and transient stress states and wall pressure evolution in converging hoppers during filling and discharge of a granular material is presented. The method for solving the set of the differential equations governing the transient flow of an incompressible, cohesionless granular material is discussed. The equilibrium conditions and stress-strain relations are satisfied for slice elements. It is assumed the material to be in elastic or elasto-plastic states within the hopper satisfying the Coulomb yield condition and the non-associated flow rule. The analysis is performed using a simple model of the material – elasto-perfectly plastic with no dilatancy or compaction effects. The analysis indicates that the transient evolution is characterised by a moving interface between passive plastic state and elastic state from the bottom surface upward toward the upper free surface. Such transient switch between passive and elastic zones generates the excessive wall pressure with its maximum at the interface. The paper presents a detailed analysis of pressure evolution of a granular material on the hopper wall during the discharge process when the initial active state of pressure is transformed into the passive state. The growth of wall pressure associated with this process is demonstrated. The analytical treatment presented in this paper are compared to the respective finite element solution.

APPLICATION OF DIELECTRIC METHODS FOR DETERMINATION OF MOISTURE IN GRANULAR MATERIALS

Wojciech Skierucha, Marek Malicki, Andrzej Wilczek

Institute of Agrophysics, Polish Academy of Sciences
ul. Doświadczalna 4, Lublin 20-290, Poland
e-mail: skieruch@demeter.ipan.lublin.pl

There is a continuous need to develop measurement methods and sensors of moisture in porous and granular media, especially for soil and food products measurements. The currently applied methods are too laborious, time consuming, produce significant errors or need expensive instrumentation. Also the scientists need new tools for the verification of models of physical processes taking place in porous materials. The indirect measurement of moisture using dielectrical properties of porous bodies seems to be the right direction for researchers. The dielectric permittivity of water is much higher than the other porous bodies, like soil, constituents because of the unique dipole nature of water molecules. Therefore having measured the porous body dielectric permittivity it is possible to determine indirectly the amount of water in the analyzed medium. The time domain reflectometry for soil moisture determination based on the above principle has become very popular. The experience and hardware solutions developed for soil as the analyzed medium can be applied to other porous media, also granular materials. The aim of the study is to present the comparison of two methods of the determination of the complex permittivity of soil and other porous or granular agricultural materials: a three-rod TDR probe and an open-ended coaxial probe.

The measurements were performed using the TDR system working with the 20 ps rise time step pulse and as well as a vector network analyzer (VNA) operating in the frequency range from 20 kHz to 8 GHz. The sensors were calibrated on liquids of known dielectrical properties and were used to measure the complex permittivity of sand, silt and clay soils of different moisture. The real part of the complex permittivity calculated from S_{11} parameters measured by VNA for frequencies near 1 GHz is in agreement with the values measured by TDR method using three-rod probes. The values of the real and imaginary parts of the complex permittivity of the soil samples measured by the applied probes are also comparable. The comparison of the hardware differences between the systems for the measurement of the complex permittivity of porous materials working in time and frequency domains is also discussed.

**THE INFLUENCE OF THERMAL TREATMENT AND MOISTURE
CONTENT ON THE WALL FRICTION COEFFICIENT
OF *CAMELINA SATIVA* SEED**

Marcin Stadnik¹, Elżbieta Kusińska¹, Józef Horabik², Józef Łukaszuk²

¹Department of Engineering and Food Machinery, Agricultural University
ul. Doświadczalna 44, 20-236 Lublin, Poland
e-mail: marcin.stadnik@interia.pl
e-mail: kisinska@faunue.ar.lublin.pl

²Institute of Agrophysics, Polish Academy of Sciences
ul. Doświadczalna 4, 20-290 Lublin, Poland

The aim of this study was to determine the influence of thermal treatment and moisture content of *Camelina sativa* seeds on the wall friction coefficient of seeds against stainless steel and galvanized steel. The experiments were conducted on samples at the moisture content of 4, 8, 12, 16, 20% subjected to three different methods of thermal treatment.

The thermal treatment was conducted by means of a microwave oven, a infrared – radiator and a convection-type furnace. Moisture content of raw material before thermal treatments was 15%. The angle of wall friction was determined with the aid of Jenike shear tester, friction area was 0,0028 m². Measurements were conducted at 4 normal pressures of 30-100 kPa.

Obtained results pointed out that the moisture content and an application of thermal treatment had statistically significant effect on the friction coefficient. Character of changes of examined parameters depending on kind of applied construction material was the same. Lower values of the friction coefficient were measured against galvanized steel. The increase of moisture content was coupled with the increase of the friction coefficient. The highest values of the friction coefficient of *Camelina sativa* seeds against galvanized steel was 0,167 and against stainless steel was 0,266 were obtained at the moisture content of 20% and normal pressure of 100 kPa. The increase of normal pressure was coupled with the increase of examined parameter. Examination of samples subjected to thermal treatment pointed out that in all cases the highest values were observed at the normal pressure of 30 kPa. The increase of the normal pressure to 100 kPa caused the 30% decrease of the friction coefficient.

**THE INFLUENCE OF THERMAL TREATMENT AND MOISTURE
CONTENT ON THE ANGLE OF INTERNAL FRICTION
OF *CAMELINA SATIVA* SEED**

Marcin Stadnik¹, Elżbieta Kusińska¹, Józef Horabik², Józef Łukaszuk²

¹Department of Engineering and Food Machinery, Agricultural University
ul. Doświadczalna 44, 20-236 Lublin, Poland
e-mail: marcin.stadnik@interia.pl
e-mail: kisinska@faunue.ar.lublin.pl

²Institute of Agrophysics, Polish Academy of Sciences
ul. Doświadczalna 4, 20-290 Lublin, Poland

The aim of this study was to determine the influence of thermal treatment and moisture content of *Camelina sativa* seeds on the angle of internal friction and cohesion. The experiments were conducted on samples at the moisture content of 4, 8, 12, 16, 20% subjected to three different methods of thermal treatment.

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Obtained results pointed out that the angle of internal friction and cohesion depend on the moisture content. No significant effect of method of thermal treatment were found. At moisture content of 45 the angle of internal friction was 29,2⁰ and cohesion value was 0,78 kPa. The increase of moisture content to the level of 20% caused the decrease of the angle of internal friction by 13% and increase of cohesion to 5,70 kPa. The thermal treatment of seeds with the aid of microwave oven caused the increase, whereas an application of the convection-type furnace and the infrared-radiator caused slight decrease of the angle of internal friction. These changes weren't significantly different. In regard to cohesion, increase in comparison with the control sample was found for samples after thermal treatment in the convection-type furnace as well as in the infrared-radiator. Obtained differences also weren't significantly different.

DIRECT SHEAR TESTING OF FOOD POWDERS FLOWABILITY

Mateusz Stasiak, Robert Rusinek

Institute of Agrophysics, Polish Academy of Sciences
ul. Doświadczalna 4, 20-290 Lublin, Poland
e-mail: mstasiak@demeter.ipan.lublin.pl

Handling and processing of powders being produced in industry, especially food powders, need information about their mechanical properties. Design of equipment for food industry and effective processing require determination of material parameters to obtain high quality final product. The knowledge of physical properties of food powders is also important for quality assessment and because they intrinsically affect material behaviour during storage, handling and processing. Properties of the material serve as design parameters for engineers designing storage systems and processing plants.

The flowability of powders and their shear behaviour under pressure are important in handling and processing operations, such as storage in hoppers and silos, transportation, mixing, compression and packaging. Many testing methods and instruments exist to determine the strength and flow properties of bulk solids but simple shear tester is still most commonly used. Numerous papers were published that considered the influence of physical and mechanical properties of investigated material on their flow properties measured in shear tester but there is still lack of information about influence of speed of shearing and stiffness of apparatus on flowability.

The objective of reported project was to determine the flow parameters (flow function FF and flow index i) of two groups of food powders used in industry: cereal powders (pearl barley groats, coarse flour, manna, oat meal, semolina) and others (sugar, icing sugar, powdered milk, potato flour, salt). Materials were different in average linear sizes of particles d^* in a range from 0.03 mm for potato flour to 4.45 mm for oat meal. Experiments were performed in 60 mm in diameter direct shear tester (Jenike shear tester) for four values of consolidating stresses σ_z : 30, 60, 80 and 100 kPa.

Second part of investigations was determination of conditions producing stick-slip effect in food powders and examination of influence of speed and stiffness of apparatus on flowability. Experiments were performed in the same shear tester 60mm in diameter for four different food powders: corn flour, wheat starch, potato starch and fine potato starch. Tests were performed following Eurocode procedure in a low range of consolidation stress varying from $\sigma_r = 4$ kPa to $\sigma_r = 10$ kPa, for

two speeds of shearing $V = 2$ and $V = 4$ mm/min and for three levels of stiffness of washer supporting test box: G0 $E = 2 \cdot 10^5$ MPa, G1 $E = 6,46$ MPa and G2 $E = 4,78$ MPa.

FF represents the relationship of unconfined yield strength σ_c and the major consolidation stress σ_r . The flow index i of each powder, which is given by the slope of the FF , took values characteristic for free flowing and easy flowing materials for the range of applied consolidating stresses. The highest values and the widest range of variability (from 0.5 kPa to 35 kPa) of FF were obtained for pearl barley groats. For the other powders values of FF were more stable and did not exceed a range of values of flow function characteristic for easy flowing materials ($i < 0.1$). The highest values of FF of the other materials were obtained for icing sugar (from 19 kPa to 24 kPa) and the lowest for salt (from 3 kPa to 7 kPa). The widest variability of FF values for the other materials were obtained for powdered milk and potato flour.

Oscillations in second part of experiments were present for all of the examined materials, speeds and stiffness values with except of potato starch for 2 mm/min and stiffness G0 for all consolidation pressures. Amplitude of oscillations increased with an increase in consolidation stress. Changes in velocity from 2 mm/min to 4 mm/min resulted in an increase in amplitude of oscillations depending on materials and consolidation pressure. The highest values of amplitude of oscillations at approximately 2.6 kPa were obtained for corn flour for 4 mm/min, stiffness G2 for 10 kPa of consolidation stress.

Flow functions obtained for wheat starch, fine potato starch and potato starch for 2 mm/min speed of deformation and for three levels of stiffness of the washer took values characteristic for easy flowing materials in a range of applied consolidation stress. For corn flour and G1 in a range of applied consolidation stress as well as for $\sigma_r = 4$ kPa and $\sigma_r = 6$ kPa for G0 and G1 flow functions took values characteristic for cohesive materials. No changes in values of flow functions were observed with changing speed of deformation for wheat flour, fine potato starch and potato starch. For corn flour with an increase in speed flow functions took values characteristic for cohesive materials.

THERMOPHYSICAL PROPERTIES OF SELECTED FOOD MATERIALS

Vlasta Vozárová

Slovak Agricultural University, Tr. A Hlinku 2, SK-94976 Nitra, Slovak Republic
e-mail: Vlasta.Vozarova@uniag.sk

Properties of food depend on properties of raw materials – agricultural products and on processing technology. The temperature and the moisture content are the most important physical properties that influence physical and chemical processes running in this material. Important properties from the point of view of food quality are also mechanical, thermophysical, sorptive and other properties of materials.

This work deals with thermophysical properties of vegetable food materials – selected grains and seeds. Measurements of specific heat at the constant pressure by differential scanning calorimetry and thermal conductivity by hot-wire method are realised.

Description of measurement methods and measurement equipment are presented. Differential scanning calorimeter is used for measuring the temperature dependence of the specific heat. The probe modification of the standard hot wire method is utilised as the measuring technique and computer-controlled experimental apparatus, that allows the determination of the thermal conductivity of solid, powders and granular materials is used. The moisture content of the grains was determined by electronic (conduction) moisture meter and by thermogravimetric method.

Dependency of thermophysical properties, namely specific heat and thermal conductivity on the temperature and on the moisture content are presented. Obtained results are discussed. It is shown that the temperature and the moisture content are some of essential factors which influence thermophysical properties of food materials and food quality in consequence.

**ARTIFICIAL NEURAL NETWORKS
IN COMPOSITIONAL ANALYSIS OF RAPESEED MEAL
FROM NIRS – ASSESSMENT OF APPLICABILITY**

Marek Wróbel¹, Henryk Czarnik-Matusiewicz², Ryszard Siuda¹

¹Institute of Mathematics and Physics, University of Technology and Agriculture
ul. Kaliskiego 7, 85-796 Bydgoszcz, Poland
e-mail: albertmw@wp.pl

²Department of Clinical Pharmacology, Faculty of Pharmacy, Medical University
ul. Bujwida 44, 50-345 Wrocław, Poland

Rapeseed (*Brassica napus*) is a crop of great importance as a constituent of both food and feed, hence methods capable to evaluate quality and/or composition of the crop in a rapid and cost-effective way are the subject of enduring interest. Near infrared reflectance spectroscopy (NIRS) followed by multivariate calibration is an analytical procedure of practical usability in applications to compositional analysis of rapeseed (see, e.g., [1–5]). Multivariate calibration methods, like MLR, PCR and PLS, are typically used to determine chemical composition from NIR spectra [6,7], while efficacy of artificial neural networks (ANN), more recently becoming a popular tool, has not been evaluated yet.

The present paper is aimed to assess applicability of artificial neural networks as a tool for determination of the content of main nutritional components of rapeseed meal (dry mass, protein, oil, ash, fiber) on the basis of NIRS measurements (69 spectra recorded in 1/R mode). Two most popular types of ANN (MLP and RBF) are tried in this work. Despite the set of spectra is not very numerous, it has been split up into training and testing subsets with an algorithm described in [8]. Inputs for ANNs have been selected separately for each chemical constituent with a procedure developed for the use in MLR [9]. All computations have been carried out with Matlab 6.5 package [10, 11].

Preliminary results prove that MLP gives a little better predictive ability than RBF for all five constituents. Comparison of the best results from MLP with the results from PLS shows the same or a little better predictive ability for MLP, except of ash.

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AUTHORS*

<i>Pedro Aguado</i>	Escuela Superior y Técnica de Ingenierías Agrarias University of León, Spain		7
<i>Francisco Ayuga</i>	Escuela Técnica Superior de Ingenieros Agrónomos Polytechnic University of Madrid Madrid, E.T.S.I. Agronomos, Ciudad Universitaria s/n 28040 Madrid, Spain	ayuga@cvr.etsia.upm.es	7
<i>Grażyna Balcerowska</i>	Institute of Mathematics and Physics University of Technology and Agriculture ul. Kaliskiego 7, 85-796 Bydgoszcz, Poland	gbalcer@mail.atr.bydgoszcz.pl	16
<i>Andrzej Bieganski</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland	a.bieganski@demeter.ipan.lublin.pl	17
<i>Monika Božiková</i>	Faculty of Agricultural Engineering Department of Physics, University of Agriculture Tr. A. Hlinku 2, 949 76, Nitra, Slovakia	Monika.Bozikova @uniag.sk	18
<i>Zbigniew Chaniecki</i>	Department of Fundamentals of Buildings and Material Engineering, Faculty of Civil Engineering Gdańsk University of Technology ul. Gabriela Narutowicza 11/12 80-952 Gdańsk, Poland		31
<i>Henryk Czarnik- Matusiewicz</i>	Department of Clinical Pharmacology Faculty of Pharmacy, Medical University ul. Bujwida 44, 50-345 Wrocław, Poland		16, 26, 42

* e-mail of corresponding author

<i>Ewa Domian</i>	Department of Food Engineering and Process Management, Warsaw Agricultural University ul. Nowoursynowska 159c 02-787 Warszawa, Poland	domian@alpha.sggw.waw.pl	19
<i>Marek Domoradzki</i>	Faculty of Technology and Chemical Engineering University of Technology and Agriculture ul. Seminaryjna 3, 85-326 Bydgoszcz, Poland	keram@atr.bydgoszcz.pl	20, 21
<i>Dariusz Dżiki</i>	Department of Machine Operation in Food Industry University of Agriculture ul. Doświadczalna 44, 20-5236 Lublin, Poland	uldar@faunus.ar.lublin.pl	9, 22
<i>Józef B. Flizikowski</i>	Faculty of Mechanical Engineering University of Technology and Agriculture ul. Kaliskiego 7, 85-796 Bydgoszcz, Poland	fliz@mail.atr.bydgoszcz.pl	23
<i>Eutiquio Gallego</i>	Escuela Técnica Superior de Ingenieros Agrónomos Polytechnic University of Madrid Madrid, E.T.S.I. Agrónomos, Ciudad Universitaria s/n 28040 Madrid, Spain		7
<i>Zuzana Hlaváčová</i>	Slovak University of Agriculture Tr. A. Hlinku 2, 949 76 Nitra, Slovak Republic	Zuzana.Hlavacova@uniag.sk	24
<i>Józef Horabik</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland		11, 17, 37, 38
<i>Marek Horyński</i>	Faculty of Electrical Engineering Technical University of Lublin ul. Nadbystrzycka 38A, 20-618 Lublin, Poland	mhor@elektron.pol.lublin.pl	25

Jarosław Jankowski	Institute of Mathematics and Physics University of Technology and Agriculture ul. Kaliskiego 7, 85-796 Bydgoszcz, Poland	zebee@poczta.onet.pl	26
Wojciech Korpala	Faculty of Technology and Chemical Engineering University of Technology and Agriculture ul. Seminaryjna 3, 85-326 Bydgoszcz, Poland		20, 21
Tomasz Kowalewski	Institute of Fundamental Technological Research Polish Academy of Sciences ul. Świętokrzyska 21, Warszawa, Poland	tkowale@ippt.gov.pl	34
Lubomír Kubík	Department of Physics Slovak University of Agriculture Tr. A. Hlinku 2, 949 76 Nitra, Slovak Republic	Lubomir.Kubik@uniag.sk	28
Elżbieta Kusińska	Department of Engineering and Food Machinery Agricultural University ul. Doświadczalna 44, 20-236 Lublin, Poland	kusinska@faunus.ar.lublin.pl	37, 38
Janusz Laskowski	Department of Machine Operation in Food Industry University of Agriculture ul. Doświadczalna 44, 20-236 Lublin, Poland	KEMPS@faunus.ar.lublin.pl	9, 22, 29
Józef Łukaszyk	Institute of Agrophysics, Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland		37, 38
Andrzej Łapko	Institute of Civil Engineering, Chair of Structural Concrete, Białystok Technical University ul. Wiejska 45e, 15-351 Białystok, Poland	lapko@kmb.pb.bialystok.pl	10
Grzegorz Łysiak	Department of Machine Operation in Food Industry University of Agriculture ul. Doświadczalna 44, 20-236 Lublin, Poland	Lysiak@faunus.ar.lublin.pl	29

<i>Marek Malicki</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland		36
<i>Marek Molenda</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland	mmolenda@demeter.ipan.lublin.pl	11, 17
<i>Zenon Mróz</i>	Institute of Fundamental Technological Research Polish Academy of Sciences ul. Świętokrzyska 21, Warszawa, Poland	zmroz@ippt.gov.pl	35
<i>Maciej Niedostatkiwicz</i>	Department of Fundamentals of Buildings and Material Engineering, Faculty of Civil Engineering Gdańsk University of Technology ul. Narutowicza 11/12 80-952 Gdańsk, Poland	mniedost@pg.gda.pl	30, 31
<i>Ladislav Nozdrovický</i>	Department of Machinery and Production Systems Slovak University of Agriculture Tr. A. Hlinku 2, 949 76 Nitra, Slovak Republic		28
<i>Jin Y. Ooi</i>	Institute for Infrastructure and Environment University of Edinburgh The King's Buildings, Edinburgh EH9 3JN United Kingdom	j.ooi@ed.ac.uk	13, 32
<i>Karolina Poszytek</i>	Department of Food Engineering and Process Management, Warsaw Agricultural University ul. Nowoursynowska 159c 02-787 Warszawa, Poland		19

<i>Álvaro Ramírez</i>	Escuela Técnica Superior de Ingenieros Agrónomos Polytechnic University of Madrid Madrid, E.T.S.I. Agronomos, Ciudad Universitaria s/n 28040 Madrid, Spain		7
<i>Michael Rotter</i>	Institute for Infrastructure and Environment University of Edinburgh, The King's Buildings Edinburgh EH9 3JN, United Kingdom		32
<i>Robert Rusinek</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland	rrusinek@demeter.ipan.lublin.pl	33, 39
<i>Irena Sielamowicz</i>	Civil and Environmental Engineering Department Chair of Structural Mechanics Białystok Technical University ul. Wiejska 45 E, 15-351 Białystok, Poland	sieliren@pb.bialystok.pl	34, 35
<i>Ryszard Siuda</i>	Institute of Mathematics and Physics University of Technology and Agriculture ul. Kaliskiego 7, 85-796 Bydgoszcz, Poland		16, 26, 42
<i>Wojciech Skierucha</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland	skieruch@demeter.ipan.lublin.pl	36
<i>Marcin Stadnik</i>	Department of Engineering and Food Machinery Agricultural University ul. Doświadczalna 44, 20-236 Lublin, Poland	marcinstadnik@interia.pl	37, 38
<i>Mateusz Stasiak</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland	mstasiak@demeter.ipan.lublin.pl	33, 39

<i>Jacek Tejchman</i>	Civil Engineering Department Gdańsk University of Technology 80-952 Gdańsk-Wrzeszcz ul. Narutowicza 11/12, Poland	tejchmk@pg.gda.pl	14, 31
<i>Vlasta Vozárová</i>	Slovak Agricultural University Tr. A Hlinku 2, SK-94976 Nitra, Slovak Republic	Vlasta.Vozarova@uniag.sk	41
<i>Wojciech Weiner</i>	Faculty of Technology and Chemical Engineering University of Technology and Agriculture ul. Seminaryjna 3, 85-326 Bydgoszcz, Poland		20, 21
<i>Andrzej Wilczek</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland		36
<i>Marek Wróbel</i>	Institute of Mathematics and Physics University of Technology and Agriculture ul. Kaliskiego 7, 85-796 Bydgoszcz, Poland	albertmw@wp.pl	42

AUTHORS*

<i>Pedro Aguado</i>	Escuela Superior y Técnica de Ingenierías Agrarias University of León, Spain		7
<i>Francisco Ayuga</i>	Escuela Técnica Superior de Ingenieros Agrónomos Polytechnic University of Madrid Madrid, E.T.S.I. Agronomos, Ciudad Universitaria s/n 28040 Madrid, Spain	ayuga@cvr.etsia.upm.es	7
<i>Grażyna Balcerowska</i>	Institute of Mathematics and Physics University of Technology and Agriculture ul. Kaliskiego 7, 85-796 Bydgoszcz, Poland	gbalcer@mail.atr.bydgoszcz.pl	16
<i>Andrzej Bieganski</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland	a.bieganski@demeter.ipan.lublin.pl	17
<i>Monika Božiková</i>	Faculty of Agricultural Engineering Department of Physics, University of Agriculture Tr. A. Hlinku 2, 949 76, Nitra, Slovakia	Monika.Bozikova @uniag.sk	18
<i>Zbigniew Chaniecki</i>	Department of Fundamentals of Buildings and Material Engineering, Faculty of Civil Engineering Gdańsk University of Technology ul. Gabriela Narutowicza 11/12 80-952 Gdańsk, Poland		31
<i>Henryk Czarnik- Matusiewicz</i>	Department of Clinical Pharmacology Faculty of Pharmacy, Medical University ul. Bujwida 44, 50-345 Wrocław, Poland		16, 26, 42

* e-mail of corresponding author

<i>Ewa Domian</i>	Department of Food Engineering and Process Management, Warsaw Agricultural University ul. Nowoursynowska 159c 02-787 Warszawa, Poland	domian@alpha.sggw.waw.pl	19
<i>Marek Domoradzki</i>	Faculty of Technology and Chemical Engineering University of Technology and Agriculture ul. Seminaryjna 3, 85-326 Bydgoszcz, Poland	keram@atr.bydgoszcz.pl	20, 21
<i>Dariusz Dżiki</i>	Department of Machine Operation in Food Industry University of Agriculture ul. Doświadczalna 44, 20-5236 Lublin, Poland	uldar@faunus.ar.lublin.pl	9, 22
<i>Józef B. Flizikowski</i>	Faculty of Mechanical Engineering University of Technology and Agriculture ul. Kaliskiego 7, 85-796 Bydgoszcz, Poland	fliz@mail.atr.bydgoszcz.pl	23
<i>Eutiquio Gallego</i>	Escuela Técnica Superior de Ingenieros Agrónomos Polytechnic University of Madrid Madrid, E.T.S.I. Agrónomos, Ciudad Universitaria s/n 28040 Madrid, Spain		7
<i>Zuzana Hlaváčová</i>	Slovak University of Agriculture Tr. A. Hlinku 2, 949 76 Nitra, Slovak Republic	Zuzana.Hlavacova@uniag.sk	24
<i>Józef Horabik</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland		11, 17, 37, 38
<i>Marek Horyński</i>	Faculty of Electrical Engineering Technical University of Lublin ul. Nadbystrzycka 38A, 20-618 Lublin, Poland	mhor@elektron.pol.lublin.pl	25

Jarosław Jankowski	Institute of Mathematics and Physics University of Technology and Agriculture ul. Kaliskiego 7, 85-796 Bydgoszcz, Poland	zebee@poczta.onet.pl	26
Wojciech Korpala	Faculty of Technology and Chemical Engineering University of Technology and Agriculture ul. Seminaryjna 3, 85-326 Bydgoszcz, Poland		20, 21
Tomasz Kowalewski	Institute of Fundamental Technological Research Polish Academy of Sciences ul. Świętokrzyska 21, Warszawa, Poland	tkowale@ippt.gov.pl	34
Lubomír Kubík	Department of Physics Slovak University of Agriculture Tr. A. Hlinku 2, 949 76 Nitra, Slovak Republic	Lubomir.Kubik@uniag.sk	28
Elżbieta Kusińska	Department of Engineering and Food Machinery Agricultural University ul. Doświadczalna 44, 20-236 Lublin, Poland	kusinska@faunus.ar.lublin.pl	37, 38
Janusz Laskowski	Department of Machine Operation in Food Industry University of Agriculture ul. Doświadczalna 44, 20-236 Lublin, Poland	KEMPS@faunus.ar.lublin.pl	9, 22, 29
Józef Łukaszyk	Institute of Agrophysics, Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland		37, 38
Andrzej Łapko	Institute of Civil Engineering, Chair of Structural Concrete, Białystok Technical University ul. Wiejska 45e, 15-351 Białystok, Poland	lapko@kmb.pb.bialystok.pl	10
Grzegorz Łysiak	Department of Machine Operation in Food Industry University of Agriculture ul. Doświadczalna 44, 20-236 Lublin, Poland	Lysiak@faunus.ar.lublin.pl	29

<i>Marek Malicki</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland		36
<i>Marek Molenda</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland	mmolenda@demeter.ipan.lublin.pl	11, 17
<i>Zenon Mróz</i>	Institute of Fundamental Technological Research Polish Academy of Sciences ul. Świętokrzyska 21, Warszawa, Poland	zmroz@ippt.gov.pl	35
<i>Maciej Niedostatkiwicz</i>	Department of Fundamentals of Buildings and Material Engineering, Faculty of Civil Engineering Gdańsk University of Technology ul. Narutowicza 11/12 80-952 Gdańsk, Poland	mniedost@pg.gda.pl	30, 31
<i>Ladislav Nozdrovický</i>	Department of Machinery and Production Systems Slovak University of Agriculture Tr. A. Hlinku 2, 949 76 Nitra, Slovak Republic		28
<i>Jin Y. Ooi</i>	Institute for Infrastructure and Environment University of Edinburgh The King's Buildings, Edinburgh EH9 3JN United Kingdom	j.ooi@ed.ac.uk	13, 32
<i>Karolina Poszytek</i>	Department of Food Engineering and Process Management, Warsaw Agricultural University ul. Nowoursynowska 159c 02-787 Warszawa, Poland		19

<i>Álvaro Ramírez</i>	Escuela Técnica Superior de Ingenieros Agrónomos Polytechnic University of Madrid Madrid, E.T.S.I. Agronomos, Ciudad Universitaria s/n 28040 Madrid, Spain		7
<i>Michael Rotter</i>	Institute for Infrastructure and Environment University of Edinburgh, The King's Buildings Edinburgh EH9 3JN, United Kingdom		32
<i>Robert Rusinek</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland	rrusinek@demeter.ipan.lublin.pl	33, 39
<i>Irena Sielamowicz</i>	Civil and Environmental Engineering Department Chair of Structural Mechanics Białystok Technical University ul. Wiejska 45 E, 15-351 Białystok, Poland	sieliren@pb.bialystok.pl	34, 35
<i>Ryszard Siuda</i>	Institute of Mathematics and Physics University of Technology and Agriculture ul. Kaliskiego 7, 85-796 Bydgoszcz, Poland		16, 26, 42
<i>Wojciech Skierucha</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland	skieruch@demeter.ipan.lublin.pl	36
<i>Marcin Stadnik</i>	Department of Engineering and Food Machinery Agricultural University ul. Doświadczalna 44, 20-236 Lublin, Poland	marcinstadnik@interia.pl	37, 38
<i>Mateusz Stasiak</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland	mstasiak@demeter.ipan.lublin.pl	33, 39

<i>Jacek Tejchman</i>	Civil Engineering Department Gdańsk University of Technology 80-952 Gdańsk-Wrzeszcz ul. Narutowicza 11/12, Poland	tejchmk@pg.gda.pl	14, 31
<i>Vlasta Vozárová</i>	Slovak Agricultural University Tr. A Hlinku 2, SK-94976 Nitra, Slovak Republic	Vlasta.Vozarova@uniag.sk	41
<i>Wojciech Weiner</i>	Faculty of Technology and Chemical Engineering University of Technology and Agriculture ul. Seminaryjna 3, 85-326 Bydgoszcz, Poland		20, 21
<i>Andrzej Wilczek</i>	Institute of Agrophysics Polish Academy of Sciences ul. Doświadczalna 4, 20-290 Lublin, Poland		36
<i>Marek Wróbel</i>	Institute of Mathematics and Physics University of Technology and Agriculture ul. Kaliskiego 7, 85-796 Bydgoszcz, Poland	albertmw@wp.pl	42
