Abstract. Processes of compaction of granular materials were described using selected models. The analysis of their accuracy on the example of wheat was the basis for the discussion on their applicability to the processing of plant-origin materials. Parameters of the model equations for wheat, compressed at 10-18% moisture content were calculated, and the relations between these parameters and wheat moisture were determined. It was found that the analyzed models described the pressure compaction of granular plant material with different accuracy, and were highly dependent on moisture. The study also indicated that the model of Ferrero et al. fits the experimental results well. The parameters of this model reflected very well the physical phenomena which occur during compression.

Keywords: pressure compaction models, wheat meal, moisture

Introduction

Processes such as extrusion or granulation require knowledge about the agglomeration of plant raw materials (Obidziński, 2012). Much of the research to determine the compaction properties of these types of plant materials have been conducted in a closed compartment (Laskowski et al., 2005). Experiments reveal that the pressure compaction of biological materials in closed compartments can be the basis for describing the phenomena associated with the granulation process and makes it possible to fully characterize both the process and the product. At the same time, determining the dependence between material density and load used for compaction (pressure - density relationship), along with the material behaviour after taking it out of the matrix, is also important. Materials of plant origin show a large variability in their physical features with the change in moisture content (Barnwal et al., 2012; Wiącek and Molenda, 2011). This is a cause for considerable difficulties when modelling the compaction process. Numerous references in the literature relate to modelling of metals, ceramic or pharmaceutical powders, or chemical materials (Chevanan et al., 2010; Feng et al., 2007; Haware et al., 2009; Panelli and Filho, 2001; Souriou et al., 2009). Equations have often been presented which set the dependencies between the load used (pressure) and the physical properties of powder, pressure vs. specific volume, and internal stress vs. material deformation. Uniaxial confined compression has been applied to describe the elastic properties of food powders (Molenda and Stasiak, 2002; Molenda et al., 2006). The process was described using both elastic and plastic models, i.e. Drucker-Prager, Di Maggio-Sandler, and Cam-Clay. They are discussed in numerous works (Chen et al., 2001; Cocks, 2001; Michrafy et al., 2002; Rolland et al., 2012; Sinka et al., 2003; Wu et al., 2005).

Previous publications (Laskowski et al., 2005; Laskowski and Skonecki, 2001) have presented results concerning the influence of moisture content and material temperature on the compaction parameters and susceptibility of cereal and legume grains to the agglomeration process. The study presented herein deals with the analysis of the compaction process. While an earlier work by Skonecki (2004) reported on the details of compaction analysis, this paper aims at evaluating the applicability of models (equations) applied for describing the compaction course for processing plant materials.

Materials and Methods

Wheat meal was used in this study. The mean particle size of the samples ground at 10+/-0.2% moisture content was 1.02 mm.

Research on the compaction processes was performed at moisture contents ranging from 10 to 18% (every 2±0.2%). The compaction tests were performed using a ZD40 hydraulic...
press (VEB WPM Leipzig, Germany). The press was equipped with a pressing assembly with a closed matrix (the diameter of the trial compartment was 25 mm). Values of the compression force and piston displacement were monitored by computer recording. A detailed description of the press and pressing assembly has been presented in previous publications (Laskowski et al., 2005; Skonecki, 2004). The following conditions were maintained during the compaction testing: sample weight 0.02 kg, sample temperature 293 K, piston speed 0.3 mm s\(^{-1}\), maximum pressure 200 MPa.

The compaction curves (relating the force to piston displacement) (Laskowski et al., 2005; Skonecki, 2004) were registered during every measurement. In the study the values of forces and displacements for five wheat grain moisture levels were registered, which made it possible to calculate the parameters applied in compaction equations, eg specific pressure (being the quotient of the force and cross-section area of the compaction compartment), in relation to the features of processed material (density, specific volume etc).

The results of experiments done were used to determine the parameters associated with each compaction model. All computations were made using Statistica (StatSoft, Inc. 2003). The non-linear estimation was applied using a quasi-Newtonian method.

Models used in the present paper are quoted and described below.

Faborode and O’Callaghan (1986) achieved the dependence between load \( P \) and density \( \rho \) of the material in the form:

\[
P = \frac{q \rho_o}{b} \left[ \exp \left( \frac{\rho}{\rho_o} - 1 \right) - 1 \right],
\]

where: \( \rho_o \) – initial density, \( a, b \) – experimental constants, where \( q = k_o \) – compression bulk modulus, \( a \) – expresses material incompressibility, \( b \) – is the material porosity index.

Ferrero et al. (1991) described the relation for compaction of fibrous plant materials in the form:

\[
\rho = \rho_o + (A + BP)(1 - \exp(-CP)),
\]

The parameters \( A, B, C \) presented in the equation have the following physical meaning: 
\( A \) (g cm\(^{-3}\)) – maximum hypothetical increase in density (in relation to the initial one) during unloading (decompression of agglomerate). In fact, studies revealed that real density of agglomerate is slightly lower than \( \rho_o + A \);
\( B \) (g cm\(^{-3}\) MPa\(^{-1}\)) – apparent elasticity of material. Term \( B \rho_o \) is related to material compressibility;
\( C \) (MPa\(^{-1}\)) – expresses mainly the proportion of density increase during the compaction phase; it decreases along with an increase in pressure. The \( 4C \) product illustrates the slope of curve for unit load \( P = 0 \) (\( 4C \rho_o \) – initial compressibility).

Kawakita and Ludde (1971) presented the dependence between change of relative material volume and pressure; the equation is of the following form:

\[
\frac{V_o - V}{V_o} = abP \quad \text{or} \quad \frac{P}{c} = \frac{1}{ab} + \frac{P}{c},
\]

where:

\[
\frac{V_o - V}{V_o} = c \quad \text{change in volume of material, } a, b – \text{constants with their physical meaning, } a \quad \text{– refers to the initial porosity, } b \quad \text{– represents deformation of particular particles, } h^{-1} \quad \text{defines the qualitative associations with the limit of plasticity.}
\]

Heckel (1961), having focused on metal powders, presented the dependence that is applicable for high pressures (400 MPa). The Eq. (4) illustrates proportional dependence of the logarithm of reciprocal porosity of a material on pressure:

\[
\ln \frac{1}{\varepsilon} = kP + A \quad \text{or} \quad \ln \frac{1}{1 - D} = kP + A,
\]

where: \( D \) – compression coefficient; \( (1 - D) = \epsilon \) – powder porosity; \( k, A \) – experimental constants. The constant \( A \) defines the compaction degree reached at low pressure by rearrangement and displacement of particles (first compaction phase), while the constant \( k \) – ability to change the density by means of plastic deformation, \( k^{-1} \) – reciprocal of \( k \) equals approximately to 3\( \rho_o \) (\( \sigma_o \) – limit of plasticity).

Denny (2002), when comparing Heckel model with Kawakita-and-Ludde, claims that both of them have the same form at low pressures.

Lordi et al. (1997) developed a compaction equation on the basis of pressure \( P \) vs. specific volume \( V \), determined for various materials (sodium chloride, polyethylene, starch, etc.) at different loads, up to 400 MPa. Thus, the compaction equation takes the following form:

\[
PV = k(1 - \exp(bP)) + V_d P,
\]

where: \( k, b \) – parameters dependent on loading conditions (displacements, compression speed) and characteristics of material, \( V_d \) – dynamic limiting specific volume.

The authors distinguish three compaction phases within the framework of this model:

– At pressures \( P \leq 10 \text{ MPa} \), rearrangement of particles and their interactions dominate, calculated threshold – load \( P_d = V/b \),

– At increased pressure to \( P_d \) – mainly compaction. Load \( P_d \) defines the minimum load necessary to form agglomerate with sufficient durability,

– At pressures higher than \( P_d \), decrease of specific volume \( V - V_d \) is proportional to \( 1/P \).

Advantages of Lordi et al. model comprise of:

– Excellent compliance with experimental data during compaction phase for \( P > P_o \) (data fitting for \( P \leq 10 \text{ MPa} \) is not appropriate),
– Calculated parameters of the dependence (5) of $PV$ on $P$ are:

$k$ – unit compaction work (J g$^{-1}$); 
$P_o=1/b$ – initial compaction load (MPa); 
$V_d$ – dynamic limiting specific volume of material (cm$^3$ g$^{-1}$).

RESULTS AND DISCUSSION

The calculated parameters of selected models (equations) for variations in moisture content of wheat are presented in Tables 1-5. Some exemplary dependencies resulting from measured values (observed values) and those from fitted function (model) are presented for the selected wheat moisture $w=10\%$ in Figs 1-5. The dependence of these parameters on grain moisture was described by means of linear regression and the results are included in Table 6.

The parameters $a$ and $b$ of the Faborede and O’Callaghan model, for various wheat moisture contents, are listed in Tables 1 and 6. Utilizing this model (Eq. (1)), determination coefficients over the range of moisture contents tested varied from 0.998 to 0.984 (Table 1), which indicates that the model fits well to the experimental data. The determination coefficient decreases slightly for higher moisture content samples $w=10\%$ the model fits the experimental data better at lower moisture levels than at higher moisture levels. Figure 1 demonstrates the small differences between values calculated from the equation and the experiments.

The parameters ($A$, $B$, $C$) used in the model proposed by Ferrero et al. (Eq. (2)) for wheat at various moisture contents are presented in Table 2. The determination coefficients over the range of moisture contents tested varied from 0.947 to 0.929. The lowest value was achieved for wheat at moisture content of $14\%$, while the highest occurred at $18\%$ moisture. Experimental and predicted values described by this model are presented in Fig. 2 for wheat moisture of $w=10\%$.

The relations between parameters $A$, $B$, and $C$ as a function of wheat moisture content are described using linear regression equations (Table 6). The coefficient $A$ increases with a rise in moisture content. The measurements of the agglomerate density (after taking out of the matrix) revealed that this density increased with moisture increase up to 16%. The true density of the agglomerate varied from 1.18 to 1.23 g cm$^{-3}$. The value of the calculated sum for constant $A$ and initial density $\rho_o$ amounted to 1.26-1.34 g cm$^{-3}$ $w=10\%$ higher than that of the agglomerate, which is consistent with the findings of Ferrero et al. (1991).

An increase in wheat moisture content causes a decrease in parameter $B$ which defines material elasticity. This reveals that as the moisture content increases the wheat becomes more plastic. Wheat at a moisture content of $18\%$, was characterized by low elasticity. Studies by Laskowski et al. (2005), and by Laskowski and Skonecki (1999), on the influence of moisture on compaction parameters and the susceptibility to compaction are consistent with the results achieved from the compaction model.

<table>
<thead>
<tr>
<th>Moisture content (w, %)</th>
<th>$a$ (MPa cm$^3$ g$^{-1}$)</th>
<th>$b$</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>5.529</td>
<td>4.923</td>
<td>0.998</td>
</tr>
<tr>
<td>12</td>
<td>2.141</td>
<td>5.739</td>
<td>0.996</td>
</tr>
<tr>
<td>14</td>
<td>1.965</td>
<td>5.823</td>
<td>0.992</td>
</tr>
<tr>
<td>16</td>
<td>0.517</td>
<td>7.228</td>
<td>0.986</td>
</tr>
<tr>
<td>18</td>
<td>0.309</td>
<td>7.978</td>
<td>0.984</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Moisture content (w, %)</th>
<th>$A$ (g cm$^{-3}$)</th>
<th>$B$ (g cm$^{-3}$ MPa$^{-1}$)</th>
<th>$C$ (MPa$^{-1}$)</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>0.408</td>
<td>0.0029</td>
<td>0.628</td>
<td>0.940</td>
</tr>
<tr>
<td>12</td>
<td>0.443</td>
<td>0.0027</td>
<td>0.719</td>
<td>0.937</td>
</tr>
<tr>
<td>14</td>
<td>0.435</td>
<td>0.0024</td>
<td>0.729</td>
<td>0.929</td>
</tr>
<tr>
<td>16</td>
<td>0.487</td>
<td>0.0021</td>
<td>0.751</td>
<td>0.941</td>
</tr>
<tr>
<td>18</td>
<td>0.515</td>
<td>0.0020</td>
<td>0.833</td>
<td>0.947</td>
</tr>
</tbody>
</table>
The parameter \( C \) (Tables 2 and 6) in this model increased with an increase in moisture content. This reveals that material at higher moisture levels demonstrates a higher increase in density at the first compaction phase. And similarly, the product \( AC \) (slope of the curve for unit pressure \( P = 0 \)) varies with an increase in moisture content.

The parameters used in the model proposed by Kawakita-and-Ludde (Eq. (3)) are listed in Table 3. The coefficient \( a \), which is used to describe the initial porosity, is almost stable across the range of moisture contents used in this study (it varies from 0.434 to 0.461) (Table 6). The

### Table 3. Parameters of Eq. (3) for compaction of wheat with various moisture contents as well as determination coefficients \( R^2 \)

<table>
<thead>
<tr>
<th>Moisture content (w, %)</th>
<th>( a ) (MPa)</th>
<th>( b ) (MPa(^{-1}))</th>
<th>( R^2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>0.434</td>
<td>1.110</td>
<td>0.899</td>
</tr>
<tr>
<td>12</td>
<td>0.446</td>
<td>1.274</td>
<td>0.912</td>
</tr>
<tr>
<td>14</td>
<td>0.440</td>
<td>1.328</td>
<td>0.899</td>
</tr>
<tr>
<td>16</td>
<td>0.458</td>
<td>1.421</td>
<td>0.947</td>
</tr>
<tr>
<td>18</td>
<td>0.461</td>
<td>1.498</td>
<td>0.951</td>
</tr>
</tbody>
</table>

### Table 4. Parameters of Eq. (4) for compaction of wheat with various moisture contents as well as determination coefficients \( R^2 \)

<table>
<thead>
<tr>
<th>Moisture content (w, %)</th>
<th>( k ) (MPa(^{-1}))</th>
<th>( A )</th>
<th>( R^2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>0.022</td>
<td>0.912</td>
<td>0.945</td>
</tr>
<tr>
<td>12</td>
<td>0.030</td>
<td>0.921</td>
<td>0.949</td>
</tr>
<tr>
<td>14</td>
<td>0.031</td>
<td>0.925</td>
<td>0.956</td>
</tr>
<tr>
<td>16</td>
<td>0.036</td>
<td>0.931</td>
<td>0.947</td>
</tr>
<tr>
<td>18</td>
<td>0.039</td>
<td>0.959</td>
<td>0.912</td>
</tr>
</tbody>
</table>

### Table 5. Parameters of Eq. (5) for compaction of wheat with various moisture contents as well as determination coefficients \( R^2 \)

<table>
<thead>
<tr>
<th>Moisture content (w, %)</th>
<th>( k ) (J g(^{-1}))</th>
<th>( b ) (MPa(^{-1}))</th>
<th>( V_d ) (cm(^3) g(^{-1}))</th>
<th>( R^2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>7.714</td>
<td>0.033</td>
<td>0.594</td>
<td>0.998</td>
</tr>
<tr>
<td>12</td>
<td>5.564</td>
<td>0.054</td>
<td>0.605</td>
<td>0.998</td>
</tr>
<tr>
<td>14</td>
<td>4.914</td>
<td>0.066</td>
<td>0.610</td>
<td>0.998</td>
</tr>
<tr>
<td>16</td>
<td>3.375</td>
<td>0.099</td>
<td>0.620</td>
<td>0.998</td>
</tr>
<tr>
<td>18</td>
<td>3.010</td>
<td>0.115</td>
<td>0.630</td>
<td>0.998</td>
</tr>
</tbody>
</table>

Fig. 1. Fitting the models of: a – Faborode and O’Callaghan, b – Ferrero et al., c – Kawakita-and-Ludde, d – Heckel, e – Lordi et al. (Eq. (1)-(5), respectively) to experimental data from compaction of wheat grain at moisture content 10%. — Fitted model, ○ experimental data.
The values of the Heckel model parameters (Eq. (4)) \( k \) and \( A \) for different wheat moisture levels are presented in Table 4. The determination coefficients over the range of moisture contents tested varied from 0.912 to 0.956. Over the range from 10 to 16% moisture content the model fits the data well. However, for more moist (more plastic) materials, the fitting of values calculated from the model to the experimental data was worse. Experimental and predicted values using this model are shown in Fig. 4 for moisture content of \( w = 10\% \).

The regression equations relating the parameters \( k \) and \( A \) to variations in wheat moisture are shown in Table 6. The parameter \( k \) increases with an increase in moisture content. This indicates that when moisture increases, the value of \( k^{-1} \) \((3\sigma_0)\) decreases causing the material susceptibility to compaction to be enhanced. The parameter \( k^{-1} \) varied from 45.7 to 25.8 MPa \((\) for the moisture range 10-18\%). Moisture also affected the compaction process by means of particle rearrangement (higher value of indicator \( A \), Table 4).

The parameters used in the Lordi et al. model (Eq. (5)), \( k \), \( b \), and \( V_d \), for a given moisture content are presented in Table 5. The determination coefficients described by Eq. (5) over the range of moisture contents studied reached the value of 0.998. This indicates that this is a very good fitting model to the experimental data. The exemplary dependence achieved from measurements (experimental data) and fitted function according to Eq. (5) for moisture level \( w = 10\% \) is shown in Fig. 5. It confirmed very good conformity of experimental results with those achieved from the model.

The relationships of the model parameters to wheat moisture content are shown in Table 6. The coefficient \( k \) decreased with an increase in moisture content. Hence, increased moisture causes a decrease in the specific compaction work \( (k) \), which is invoked by the lubricating effect of a liquid and the change in material mechanical properties (it becomes more plastic). Values of \( k \) varied from 7.7 to 3.01 J g\(^{-1}\). The experimental data revealed that specific compaction work varied from 15.2 to 9.5 J g\(^{-1}\), which was larger than the values of \( k \) established from the model.

The parameter \( b \) (Tables 5 and 6) increased \((\)ie initial pressure \( P_o = b^{-1} \) decreased\()\) along with wheat moisture. Its values varied from 0.033 to 0.115 MPa\(^{-1}\). Over the range of moisture contents tested, pressure \( P_o \) varied from 30 to 8.7 MPa, demonstrating that particle rearrangement as described by this model takes place at relatively low loads. These results indicate that higher compaction work occurred at values of higher pressure \( P_o \). The dependence between pressure \( P_o \) and work \( k \) is linear.

Similarly, the limiting specific volume \( V_d \) as described by the model increased with an increase in the moisture content of wheat (Tables 5 and 6), ie the limiting density \( P_d \) decreased. For these values of moisture (10-18\%), the limiting density of wheat varied from 1.68 to 1.59 g cm\(^{-3}\). This density is greater than the maximum density of the material observed in the compartment during compaction, which was 1.58-1.50 g cm\(^{-3}\). The pressure \( P_d \) (upper limit) determined from the model varied from 86 to 18 MPa. Those values are too low to achieve agglomerate with sufficient strength (the pressure determined from experimental data on the basis of compaction curve for moisture contents of 10-18\% ranged from 143 to 82 MPa).

Despite the very good fit of this model to the empirical data, the constants in this model, such as compaction work and limiting specific volume, differed significantly from experimental results and characterized the change in the material properties with respect to moisture in a biased way. Therefore, this model can be used to describe the compaction of granular biological materials only to a limited degree.

**CONCLUSIONS**

1. Evaluation of the influence of wheat moisture on the parameters of compaction equations indicates that the effect can be described by linear equations. The degree of fit of the models to the experimental data depends on the moisture content.
The compaction of granular plant-origin material according to Ferrero et al. model can be divided into three characteristic phases revealing changes in the structure of compressed material. The increase of moisture invokes higher rise in the material density within the first two phases (initial deformation and main compaction). A slight density increase at high pressure increase (linear dependence) was recorded in the third phase of compaction.

REFERENCES


