# Compression rheology and physical quality of wood pellets pre-handled with four different conditions

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## ABSTRACT

The effects of drying temperature and storage time on the compressibility and strength of Scots pine pellets were analysed in this article. Compressibility was not affected, whereas the highest pellet strength was obtained from the wood with longest storing and highest drying temperature.

### INTRODUCTION

Traditionally, rest products from saw mills have been used as raw material for pellets. Increased demands have resulted in less available material from this resource. New raw materials are therefore needed to increase the wood pellet production. New raw materials most probably have different rheological properties like compressibility (i.e. compacting stress \_ density relationships<sup>1</sup>), compactibility (i.e. the ability of a material to form strong compacts) and resistance to flow, which influence the pelleting performance and product quality<sup>2, 3</sup>.

It is a general statement<sup>4-7</sup> that the storing time of the raw material plays a major role on the durability and strength of

the pellets. Storage of the raw material is necessary due to logistics, transport, etc. However, storage also increases the power demands of pelleting and reduces throughputs due to the loss of extractives that provides a lubrication effect in the die<sup>5</sup> (also called matrix).

For a feasible pellet production, the raw material has to be dried to a moisture content between 7 to 13% (w.b.), which only is achievable through artificial drying. One of the most common drying methods of today is drum drying using flu gas with temperatures around 450 °C. With such temperatures, part of the extractives will vaporize. Extractive content will be reduced even when drying the raw material below 100 °C.

Less extractives content, due to drying and storage of the raw material, will most likely influence the compressibility and compactibility of the raw materials, giving other pellet properties as a result.

Compressibility analysis can be performed through compression tests which have been used widely in pharmaceutics, ceramics, metallurgy, civil engineering, as

well as in the food powder field, because it is a simple and convenient technique to measure powder compressibility and flowability<sup>8</sup>. Compressibility indicates the stress level that is required to compact a material to a given density. This information is useful to select or design process equipments. Compressibility data are also important to describe the flowability of loose materials. Compressibility provides information for storage, transport and handling<sup>1, 8, 9</sup>. A stored powder having high compressibility can be highly compacted at the bottom, reducing its flowability. This can cause problems at the discharge of silos, bins and all transport ducts where the material is moved by gravity. Consequently, compressibility information is important in process design.

Compactibility is also important to determine the amount of stresses (i.e. consolidating stresses) required to form strong compacts or pellets. Compactibility provides information about durability of the pellet which is important for storage, transport and handling of pellets.

The main goal of this research is to characterize the changes in compressibility and compactibility of ground Scots pine materials treated by two different storage times and by two drying methods. Another goal is to analyze the capability of a new die pelleting rig to perform such studies. Scots pine was chosen because it is economically attractive for large scale production<sup>10</sup>.

# MATERIALS AND METHODS Design features

Wood pellets were produced using a new customized die pelleting rig (see Fig. 1) as a fixture in a Lloyd LR 5K Plus texture analyzer. The die pelleting rig was connected to the texture analyzer using the same assembly that was described and used by Salas-Bringas et al.<sup>11</sup> and Rukke et al.<sup>12</sup>. The die pelleting rig (see Fig. 1) consists of a barrel made of brass having a compressing channel along the centre. The maximum

load that can be produced in the texture analyzer is 5 kN. The compressing channel has a diameter of 9.5 mm and a 9.4 mm diameter rod is used to press the samples. The system can produce compacting stresses up to 72 MPa.

The barrel is connected to a closed or blank die made in brass. The barrel and die are covered with a jacket heater of 550 Watts. The temperature is measured by a thermocouple that is used by a PID controller to keep a steady temperature. The rod that is shown in Fig. 1 is made of iron to impose an adequate stiffness to avoid buckling at the maximum pressures.





Preparation of the wood samples

The material used in the experiments was chipped from unbarked stemwood from a pine stand located in Bygland in Setesdal in Southern Norway (EUREF N 6 533 862, E 82 480 ) at 220 m altitude. The stand was classified as F14 in the Norwegian site class system<sup>13</sup>.

Parts of the chips were stored in a 6 meter high pile for 11 months (from May 2008 to April 2009). Samples from this pile is referred to as "Stored". The "fresh" material, stored for three months, was felled from a nearby and identical stand to the first.

The high temperature drying, referred as "HT", was performed in an industrial drum dryer using flue gas with an inlet temperature of 450 °C.

For the low temperature drying, referred as "LT", it was used a container with perforated bottom and air at 75 °C as drying medium.

The materials were dried to a moisture content between 7 to 13 % (w.b.) which is common when pelleting wood. However, a narrower range in moisture contents was sought, to avoid any moisture content effect on the rheological properties. As a consequence, the samples were further kept in a climatic chamber (Termarks, Type KBP6395 F, Bergen, Norway) during 48 h using a relative humidity of 50 % and 20 °C to even out the moisture content. The final range in moisture content from the climatic chamber was reduced to a range of 9 - 10%(w.b.).

# Production of wood pellets

The ground and dried samples were added in the cylindrical compressing channel until the channel was fully covered. Wood pellets were produced using 20, 30, 40, 50, 60 and 70 MPa of normal stress. Three samples were produced for each normal stress.

There are disagreements in the indications of the production pressures in pellet presses. Some authors describe that the die pressures do not need to be higher than 50 MPa to produce pellets from Spruce sawdust<sup>14</sup>. Others give a general statement that die pressures are not higher than 70

MPa<sup>15</sup>, while some estimate even higher pressures, around 300 MPa<sup>5</sup>. However, noticing the disagreements, it is important to keep in mind that the pressure drop through the die hole depends on the mass flow rate (throughputs), die diameter, die length, entry angle and the rheological properties of the materials related to resistance to flow, which also depend on particle size, moisture content, temperature, processing history, etc.

The pressing speed was set to 2 mm/s with a short retention time (1 s). Retention time under high pressure is very short in pellet presses due to the fast and intermittent pressure given by the rotating rollers inside the die ring in the nip area.

After the retention time period, the pressure was released and the blank die removed. The same compressing rod and texture analyzer were used to further move the pellet inside the channel until its gentle discharge from the barrel.

The source of error in this type of compression tests is that the friction between the material and the side walls of the channel reduces the stress from top to bottom<sup>1</sup>. This might result in a wood pellet having a density gradient, with its lowest density close to the blank die. However, the pellets that were produced, having a diameter of 9.5 mm, were relatively short (18 mm). Consequently, density gradients within the single pellets were neglected. The average density was calculated using the pellet weight and its volume, based on their well defined cylindrical shape.

The temperature was set to 110 °C for all tests. At this temperature the amorphous thermoplastic material lignin<sup>16</sup>, acts as a binder since it is over its glass transition temperature<sup>16, 17</sup> and bellow its melting point<sup>18</sup>. Temperature in the range of glass transition is important to make durable particle-particle bonding in pellets<sup>16</sup>.

The experiments were repeated three times and the results are presented in a compacting stress (MPa) versus density (kg m<sup>-3</sup>) plot.

Measurement of pellet strength

The physical quality of the wood pellets were analyzed by measuring the maximum peak force registered during a diametrical compression, as similarly used by Rhén et al<sup>14</sup>. The first preliminary tests showed a plastic and ductile nature of the wood pellets. It was not possible to measure the tensile stresses based on the same criteria that have been used for brittle pellets<sup>19, 20</sup>. Instead maximum yield load producing a ductile failure (ref. Fig. 4) divided by the pellet length (kN mm<sup>-1</sup>), which gives information about the shear strength per unit length, was used. This method has also been used by other authors characterizing wood pellets<sup>14</sup>. The results showing the normal force (kN), are plotted in Fig. 4. Maximum yield loads (kN mm<sup>-1</sup>) are plotted in Fig. 5. The test speed was set to 1 mm/min and the test was ended when the probe reached 2.2 mm below the diameter of the pellet (strain ~ 0.23).



Figure 2. Representation of the diametrical compression of a wood pellet using a specially designed conical probe of 60 mm diameter. *F* is the normal force (kN).

# RESULTS AND DISCUSSIONS <u>Production of wood pellets</u>

The compressibility of all wood samples showed a clear ductile type of compression, similar to ductile powders<sup>21</sup> (see Fig. 3) following a power law trend. A power law curve fit resulted in high correlations ( $\mathbb{R}^2 > 0.94$ ), n = 18, where *n* is the number of samples, three samples for each of the six compacting stresses 20, 30, 40, 50, 60 and 70 MPa.

Following the power law curve, a large increase in density is expected at low compacting stresses followed by a smaller increase at higher stresses. Consequently, much larger changes in compacting stresses are required to increase the density of wood pellets at high stresses.

No significant differences (p>0.05) in compressibility was found between the regression curves for the four different raw materials (see Fig. 3).



Figure 3. Compressibility data for pine under four pre-handling conditions. No significant differences were found between the four groups (p>0.05).

#### Measurement of pellet strength

A preliminary view of some of the results given by the texture analysis indicates the relationship between the ductile nature of wood pellets and Maximum Yield Load (Fig. 4).

Fig. 4 shows a long plastic region for the pellets having the lowest densities. This occurred to many of the pellets that were produced with low compacting stresses (20 MPa) and the least expected quantities of extractives (HT Stored and LT Stored). All pellets made from "HT Stored" raw material and one pellet from "LT Stored" having the lowest density (approximately 870 kg m<sup>-3</sup>),

did not present any clear abrupt failure of its structure, but a continuous shear or frictional deformation in a long yield region, denoting a plastic nature. For these cases it was not possible to estimate a maximum yield load. This is why two of the linear regressions made in Fig. 5 were made with smaller number of samples. It is also possible to observe from Fig. 4 that the plastic pellet having 870 kg m<sup>-3</sup> presents the smallest slope between 200-600 kN of normal force which indicates the smallest elastic modulus and thus the least stiffness.



Figure 4. Examples of stress analysis for the wood pellets (HT Stored). The curves show the ductile nature of the wood pellets and how the ductility decreases as the density increases.

As the pellet density increases (>954 kg  $m^{-3}$ ) the yield starts to show a maximum value (Fig 4), ductile failure appears in "HT Stored" pellets, allowing the determination of a maximum yield load which is used in Fig. 5.

It can be observed that as the density increases the slopes between 200-600 kN also increases, indicating that pellet stiffness increases with higher densification. The positive correlation between Maximum yield load and pellet density illustrated in Fig. 5 was significant (p<0.05).

Another observation is that as the density increases, the yield peak becomes sharper. This could indicate the loss of ductility as pellet density increases. Possibly at a much higher density, the wood pellets could become brittle in nature.

Fig. 5. indicates similar strength values (Max. Yield load  $\cdot$  length<sup>-1</sup>) for three of the samples (LT Fresh, HT Fresh and LT Stored) since these groups did not present any significant differences (p>0.05).

On the other hand the pellets based on stored and high temperature dried raw material (HT Stored), had significantly higher maximum yield load values (p<0.05) compared to the others. However, it was only possible to measure a maximum yield load to the pellets with densities above 950 kg m<sup>-3</sup> for this kind of material, indicating that the linear relationship shown in Fig. 5 only is valid in the density range above 950 kg m<sup>-3</sup>.



Figure 5. Strength of wood pellets produced with different densities. The word "Linear" indicates that a linear regression was applied to the data sets (p<0.05). *n* is the number of samples.

The significant higher maximum yield load values found for the "HT stored" material is in accordance with Nielsen et al.<sup>5</sup> saying that pellets produced from wood with lower extractive content have higher strength due to closer contact between the bonding sites of the lignocellulose particles. Still the basis for the found relationship is not clear. Samuelsson et al.<sup>4</sup> and Finell et al.<sup>7</sup> found that the reduction of fatty acids and resin acids after storage gave higher pellet durability. On the other hand, Arshadi et al<sup>6</sup> could not find that the amount of fatty acids and resin in sawdust affected the pelleting process and pellet quality. They indicated that since no useful model was found in their data, there are other properties causing the difference between fresh and stored sawdust. During storage, in addition to loss of extractives, some degradation of cellulose, hemicellulose and lignin take place, depending on active fungi species present. This most probably also will affect the the pelleting process and pellet properties.

Despite the small density changes present at higher compacting stresses ( $\sim$ 50-70 MPa), a significant (p<0.05) and a continuous increase in pellet strength occurred for all compacting stress levels (20-70 MPa).

# CONCLUSIONS

Ground Scots pine (*Pinus sylvestris*) presented a ductile compression following a power law curve. The pellets produced in the die pelleting rig were plastic and ductile materials.

Drying temperatures and storing conditions did not change the compressibility of the wood materials significantly (p>0.05).

Ground Scots pine pre-handled by the highest drying temperature and longest storage time, produced pellets with the highest strength values under diametrical compression test.

A continuous increase in pellet strength occurred when increasing the compacting stresses from 20 to 70 MPa.

The die pelleting rig proved to be a useful device to perform compressibility tests and to create wood pellets under defined compacting stresses and temperatures.

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