Influences of Wet Torrefaction on Pelletability and Pellet Properties of Norwegian Forest Residues

Quang-Vu Bach¹, Nevena Mišljenović², Khanh-Quang Tran¹, Carlos Salas-Bringas² and Øyvind Skreiberg³

¹Department of Energy and Process Engineering, Norwegian University of Science and Technology, NO-7491 Trondheim, Norway
²Department of Mathematical Sciences and Technology, Norwegian University of Life Sciences, P.O. Box 5003, N-1432 Ås, Norway
³Department of Thermal Energy, SINTEF Energy Research, NO-7465 Trondheim, Norway

ABSTRACT
The compressibility of Norway spruce and birch tree branches torrefied in subcritical water conditions and the mechanical strength of the obtained pellets were experimentally studied in comparison with the raw materials. The pelletization was performed on a single pellet press. The pellet strength was investigated via diametric compression tests, employing a 60 mm diameter probe connected to a Lloyd LR 5K texture analyzer. The results showed that wet torrefaction improved the compressibility and strength of the tested material. In addition, compressing pressure affected both the pellet density and strength, while pelleting temperature influenced the pellet strength only.

INTRODUCTION
Wet torrefaction (WT), which may be defined as pretreatment of biomass in hot compressed water at temperatures within 180-260 °C¹, ², is a promising method for production of high quality solid fuels (hydrochars) from low cost wet biomass resources such as forest residues, agricultural waste, aquatic energy crops, and sewage sludge. The concept of WT is very similar to “hydrothermal carbonization” (HTC)³ and sometimes is discussed under the general term “hydrothermal conversion”⁴ or “hydrothermal treatment”⁵. The main improvements in fuel properties of hydrochars produced from WT of biomass include the change from hydrophilic to hydrophobic nature, increased heating values, and improved grindability. However, the bulk and volumetric energy densities of biomass are reduced by WT⁶-⁸. In addition, hydrochar becomes more flaky and dusty, compared to the raw biomass⁶-⁸. These drawbacks may cause problems for the storage, logistics, and further utilizations (combustion, gasification, and pyrolysis) of hydrochars⁶-⁸. Therefore, an additional step of pelletization is usually required to overcome the drawbacks.

Pelletization is a mechanical process that convert bulky solid biomass fuels into pellets with uniform shapes and reduced dust formation. More importantly, the bulk and volumetric energy densities of solid biomass fuels are both significantly improved via pelletization⁸,¹⁰. The pellet form of biomass fuels is suitable for many industrial and residential applications⁸,¹¹,¹².

In the open literature, there are few reports dealing with pelletization of biomass pretreated in subcritical water conditions⁶-⁸.
It was reported that pellets produced from hydrochars were denser, more durable and mechanically stronger than pellets produced from the corresponding raw biomass \(^6\text{-}^8\). Nevertheless, pelletizing hydrochars is more challenging than the raw biomass because the friability and hydrophobicity of hydrochars reduce significantly the bonding capacity between hydrochar particles \(^6\text{-}^8\). However, many factors such as pelletizing temperature, compacting pressure, type of feedstock, processor type may affect the compressibility and the physical properties of pellets\(^8\). The effects of these factors have not been fully understood and therefore more research in this area is needed.

This present study aimed to investigate the effects of WT on the pelletability and physical properties of Norwegian forest residues (FRs). Norway spruce and birch tree branches were used as feedstocks and torrefied in subcritical water conditions at different temperatures.

**Table 1. WT conditions and fuel properties of the raw forest residues and their hydrochars.**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Solid yield(^a)</th>
<th>MC(^b)</th>
<th>Ash(^a)</th>
<th>VM(^c)</th>
<th>FC(^a)</th>
<th>HHV(^c)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spruce</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Raw</td>
<td>–</td>
<td>10.30</td>
<td>0.23</td>
<td>86.50</td>
<td>13.27</td>
<td>20.42</td>
</tr>
<tr>
<td>Torrefied for 30 min in water at 70 bar and different temperatures</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>175°C</td>
<td>88.27</td>
<td>6.67</td>
<td>0.11</td>
<td>85.72</td>
<td>14.17</td>
<td>20.81</td>
</tr>
<tr>
<td>200°C</td>
<td>78.45</td>
<td>4.90</td>
<td>0.12</td>
<td>83.92</td>
<td>15.95</td>
<td>21.33</td>
</tr>
<tr>
<td>225°C</td>
<td>69.74</td>
<td>4.26</td>
<td>0.14</td>
<td>74.74</td>
<td>25.12</td>
<td>22.97</td>
</tr>
<tr>
<td>Birch</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Raw</td>
<td>–</td>
<td>9.74</td>
<td>0.28</td>
<td>89.46</td>
<td>10.26</td>
<td>19.94</td>
</tr>
<tr>
<td>Torrefied for 30 min in water at 70 bar and different temperatures</td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>175°C</td>
<td>79.53</td>
<td>6.10</td>
<td>0.09</td>
<td>88.57</td>
<td>11.34</td>
<td>20.21</td>
</tr>
<tr>
<td>200°C</td>
<td>64.64</td>
<td>5.05</td>
<td>0.09</td>
<td>85.15</td>
<td>14.76</td>
<td>20.78</td>
</tr>
<tr>
<td>225°C</td>
<td>58.01</td>
<td>4.69</td>
<td>0.13</td>
<td>73.78</td>
<td>26.09</td>
<td>22.93</td>
</tr>
</tbody>
</table>

\(^a\) wt%, dry basis; \(^b\) Moisture content, wt%, wet basis; \(^c\) MJ/kg, dry and ash free basis.

**MATERIALS AND METHODS**

**Materials**

Fresh branches with diameter of 2-2.5 cm of Norway spruce and birch trees were collected from a local forest in Trondheim, Norway, to simulate Norwegian FRs. The WT procedure and fuel characterization methods employed for this present work are adopted from our earlier study\(^1\). The feedstocks were torrefied in a Parr 4650 autoclave reactor at three different temperatures (175, 200, 225°C), for a constant holding time of 30 min and at a constant pressures of 70 bar. The WT conditions and some fuel properties of the tested materials are presented in Table 1.

**Pelletization**

The pelletization was carried out using a single pellet press\(^10\) presented in Figure 1, which allows precise control and adjustment of compressing pressure and pelleting temperature. The unit consists of a steel cylinder (8 mm inner diameter) and a tungsten carbide pressing rod. The press is heated by a jacket heater (450W) of which the temperature was controlled by a PID. The compressing force is applied to the rod using an Instron 100 kN texture...
analyzer. Two pelletizing temperatures (120, 180°C) and five compacting pressures (20, 40, 80, 160, 240 MPa) were tested. More details of the press and pelleting procedure can be found in our previous study.10

Figure 1. Single pellet press unit: picture of the equipment (left) and section view A-A (right).

Characterization of pellets

The density of pellets was calculated by dividing the weight by the volume of the pellets. The length and diameter of the pellets were measured by means of a digital caliper (from Biltema Sweden).

The compressing tests were carried out at 48 h after the pellets were produced. A 60 mm diameter probe connected to a Lloyd LR 5K texture analyzer (Lloyd Instruments, England) was employed for this test. The compression speed was set to 1 mm/min, and the maximum normal force at breakage was recorded automatically. The pellet strength was expressed as the maximum force per length of the pellet (N/mm).

RESULTS

Effects of wet torrefaction on the grindability and particle size distribution

A pulverization step was required prior to pelletization. A quantitative evaluation of the specific grinding energy (SGE) was carried out for all samples being used for pelleting. Results from the evaluation are presented in Fig. 2, which indicates that WT improved the grindability of the biomass. When torrefaction temperature increases, the SGE decreases. The reduction in SGE was up to 13.3 times for spruce and 27.5 times for birch torrefied at 225°C for 30 min, compared to the raw materials.

The particle size distribution (PSD) of the ground sample was determined by a Mastersizer 3000 laser diffraction particle size analyser, and the results for this test are presented in Fig. 3. The distribution curves show that WT resulted in lower fractions of the coarser particles and larger fractions of finer particles. This effect of WT for birch was more pronounced than that for spruce. Moreover, the curve for spruce torrefied at 225°C exhibits two peaks, while only one peak is observed for the other samples.

Figure 2. Specific grinding energy of raw and wet-torrefied forest residues.

Effects of wet torrefaction on the compressibility

The compressibility of a biomass fuel can be evaluated via examining the density of the pellet produced from the fuel powder at various pelletizing pressures.
Figure 3. Particle size distribution of ground (A) spruce and (B) birch.

The results from such an evaluation for the raw FRs and hydrochars produced in different WT conditions are shown in Fig. 4, in which the density values at zero pelletizing pressure indicate the bulk density of the ground materials. As expected, the pellet density in all cases increased with increasing pelletizing pressure. The effect of pelletizing temperature was not pronounced, but the effect of biomass type was clear. In the identical condition, birch pellets were denser than spruce pellets and the effect of torrefaction temperature was more pronounced for birch than spruce. More importantly, WT improved the density of pellets. In other words, WT increases the compressibility of the tested materials. In the case of spruce, pellets made from the material torrefied at 175 and 200°C had higher density than it raw material. However, the hydrochar produced at 225°C was the least compressible at low compacting pressures. From the pressure of 80 MPa, the compressibility of this hydrochar sharply increased and became higher than that of the raw spruce. This increasing trend continued and got close to the compressibility of the spruce hydrochars, produced at the other temperatures, at the highest compressing pressure (240 MPa). Similar trends were observed for birch but the improvements in the compressibility by WT were more pronounced than those for spruce. However, unlike spruce, the birch torrefied at 225°C was better compressible than the raw birch at any pelletizing pressure. The highest density of 159 kg/m³ was obtained from the spruce torrefied at 175°C and pelletized at 20 MPa, 180°C, whereas it was 213 kg/m³ for the birch torrefied at 175°C, pelletized at 40 MPa, 180°C.

Mechanical strength of pellets

Results from the mechanical strength tests of the pellets produced from raw FRs and their hydrochars are presented in Figure 5. The figure shows that the pellet strength was significantly improved by WT. Moreover, both torrefaction temperature and pelletizing temperature affected the mechanical strength of the pellets. A general trend observed from the figure is that the pellet strength increases with the pelletizing temperature. The smallest increases in the strength of the hydrochar pellets compared to the pellets of the raw materials were 1.3 and 0.7 times for spruce and birch, respectively. On the other hand, the largest increases were 3.4 and 2.7 times for spruce and birch, respectively. In addition, when WT temperature was increased from 175 to 200°C, the spruce pellet strength increased but that for birch decreased. At low compacting pressures, the strength of the pellet made from the FRs torrefied at 225°C was not as good as that of the pellets made from the FRs torrefied at 175
and 200°C, but better than that of the pellets of the raw materials. Thereafter, the strength of pellets made from the materials torrefied at 225°C increased rapidly and became the strongest at the highest compressing pressure (240 MPa).

Figure 4. Density of pellets made at different pelletizing pressures and temperatures.

Since the density and mechanical strength of the pellets were both increased with pelletizing pressure, the correlation between these two properties was evaluated. Results from this evaluation are presented in Figure 6, in which fitting curves also are included. The figure shows an exponential relationship between the two properties. The pellets with higher density also had higher mechanical strength. At the same density value, hydrochar pellets were mechanically stronger than the raw material pellets. The effect of WT temperature was more pronounced for spruce than birch. Below the density of 1000 kg/m³, large increases in density results in only small increases in the strength. However, this relationship was reversed when the density was higher than 1000 kg/m³.
DISCUSSION
WT not only enhanced the fuel properties but also reduced the SGE of the FRs. Compared to the raw FRs, wet-torrefied FRs had larger fractions of fine particles, and the particle size distribution peaks shifted to a smaller particle size range. Except for the spruce torrefied at 225°C, most of the pellets produced from the wet-torrefied FRs had higher density than the raw material pellets. These indicate the WT improved the compressibility of the FRs. However, in order to obtain hydrochar pellets with higher density than pellets of the raw materials, torrefaction temperatures higher than 225°C and pelletizing pressures above 80 MPa should be applied for spruce. Moreover, all of the pellets produced from wet-torrefied FRs exhibited higher mechanical strength than the pellets produced from the raw FRs, at the same pelletizing pressure. These results are in good agreement with the other studies.6-8
Increasing the pelletizing temperature from 120 to 180°C had a little effect on the pellet density but improves the pellet strength. This is addressed to the behaviours of lignin below and above its glass transition temperature ($T_g$), which is around 135-165°C\(^8\). At a temperature higher than the $T_g$, lignin softens and enhances the inter-particles binding, which improves the mechanical strength of pellets\(^{13}\). Therefore, the pellets made at 180°C were stronger than pellets produced at 120°C, at the same compacting pressure.

Pelletization at higher compacting pressure produced pellets with higher density and strength. It is also shown that a small increase in density resulted in a large increase in strength if the density of pellet was higher than 1000 kg/m\(^3\), which can be achieved by applying a compacting pressure above 80 MPa. Although more energy is required, it is recommended a pressure higher than 80 MPa for the production of pellets due to the benefit form the exponential relationship between the pellet strength and density.

**CONCLUSION**

WT improved the fuel properties and reduced specific grinding energy of the FRs. The average particle size of ground hydrochar was smaller than that for raw FR and gradually decreased with increasing WT temperature. The pellets of wet-torrefied FR were better compressible and stronger than the pellets of raw FR. Increases in density for the hydrochar pellets compared to the pellets of raw materials was up to 159 kg/m\(^3\) for spruce and 213 kg/m\(^3\) for birch. Improvements in the strength of torrefied pellet compared to raw pellet were up to 3.4 and 2.7 times for spruce and birch, respectively. The effect of pelleting temperature on pellet density
was unpronounced but the effect on pellet strength was significant due to different behaviours of lignin below and above its glass transition temperature. Increasing compacting pressure increased the mass density and strength of the pellets.

ACKNOWLEDGMENTS
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REFERENCES