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Récents Progrès en Génie des Procédés, Numéro 101 - 2011
ISSN 1775-335X - ISBN 2-910239-75-6, Ed. SFGP, Paris, France

Changes on wood powder morphology and flowability due to thermal pretreatment

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Abstract

Torrefaction is a thermal pretreatment of lignocellulosic biomass before gasification. This mild form of pyrolysis, carried out at temperatures between 200 and 300 °C, changes the physical and chemical properties of the material. In particular, it improves the feedstocks homogenisation, enhances the aeration ability and makes the grinding easier.

Our project deals with the study of the effects of the combined torrefaction and grinding processes on (*i*) the major particle behaviour: grindability, surface state, particle size, shape distribution (*ii*) and the major powder behaviour: bulk density, compressibility, aerability.

In this article, the studied parameter is the torrefaction temperature. The characterisation of the particles and powders is performed using the following techniques: optical microscopy, Flodex® methodology and powder rheometry.

The rheological behaviours of spruce torrefied at different temperatures then ground in the same apparatus (a knife mill with a sieving grid of 500 µm) are compared. It is shown that spruce torrefied at 240 °C (S240) has similar rheological properties compared to natural spruce (NS) after being ground, while the one torrefied at 300 °C exhibits a different rheological behaviour. The results are correlated to reproduce the coupled effect of the torrefaction and grinding conditions on the powder rheological behaviour.

Key-words: lignocellulosic biomass, torrefaction, powder, rheological properties.

1. Introduction

Wood biomass can be transformed into gaseous or liquid fuels by thermal processes such as BTL (Boerrigter et al., 2005). The last step of this process could consist in the Fischer-Tropsch reaction which produces synthetic alcanes from the syngas, a mixture of carbon monoxide and hydrogen. For this catalyst-activated reaction to work properly, the syngas needs to have a high purity level. Gas cleaning methods may be used to treat the syngas prior its injection in the conversion reactor. Alternatively, the efforts can focus on directly producing a relatively clean syngas (CO, CH_4, H_2), particularly with low content of tar (acids, aldehydes, naphtalenes, phenols, …, (Elliott et al., 1988; Milne et al., 1998). In order to reduce the tar products, an operation consists in a thermochemical conversion at high temperature (> 1000°C) and high pressure ensuring a biomass residence time below a few seconds (Rajvanshi and Goswami, 1986). It can be achieved with gas-particle technologies like fast circulating fluidised beds or

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entrained flow gasifiers (Kunii and Levenspiel, 1991). Wood powder is one of the most suitable forms for feeding this kind of reactors.

To produce wood powder, a possible process consists in drying and torrefying wood chips before grinding them at the desired size. Torrefaction is a mild form of pyrolysis carried out at temperatures between 200 and 300°C. The severity of this thermal pretreatment is appropriately measured by the anhydrous weight loss (AWL) of the wood. As shown by (Repellin et al., 2010a), the AWL increases with temperature for a given wood specie. The AWL also depends on the species: to reach the same AWL, spruce needs to be heated 20°C more than beech, which can be correlated to their chemical composition in terms of hemicelluloses content (Prins, 2005).

Interestingly, the torrefaction increases the wood brittleness, thus significantly influencing the mechanical energy required for grinding. As measured by (Repellin et al., 2010b), for an AWL below approximately 8% (which corresponds to torrefaction temperature below 240°C for beech and 260 °C for spruce), the grinding energy is dramatically reduced. Over 8%, the effect of the torrefaction is less pronounced. For an AWL around 28% (which corresponds to torrefaction temperature about 280°C for beech and 300°C for spruce), the grinding energy was cut by 93%. These results are consistent with other studies showing that torrefaction improves feedstocks homogenisation and makes the grinding easier (Svoboda et al., 2009) due to the changes on its physical and chemical properties (Arias et al., 2008).

Our project consists in an experimental study of the coupled effects of torrefaction and grinding on the particles morphology and powder behaviour. All the experimental data will be correlated in order to predict the optimum of torrefaction and grinding conditions for transport and aeration purposes.

2. Experimental procedure

The raw material is composed of natural spruce chips of approximately 5 x 20 x 30 mm. It was torrefied in a hot batch rotating pilot kiln at two different temperature conditions, i.e. 240°C and 300°C during 5 minutes (see Repellin et al. 2010a for details). The final anhydrous weight loss of each batch of biomass was measured. The torrefied biomass was then ground and sieved with a knife mill equipped with a grid of 500 µm using the same experimental protocol.

The ground torrefied material is analysed twofold. The mechanical properties are determined by several techniques inspired from Freeman studies (Freeman and Cooke, 2006; Freeman et al., 2009). The flowability, compressibility and aeration behaviours are characterised with the FT4 Rheometer® and the Flodex®. The FT4 Rheometer® methodology consists in the determination of several parameters describing the powder properties: *(i)* its conditioned bulk density (CBD), *(ii)* its compressibility resulted from the application of a normal stress on the powder, and *(iii)* its flowability. In the case of the compressibility test, a vented piston applied a normal stress on 10 mL of powder filling a 2.5 cm diameter and 3.0 cm length cylindrical vessel. As for the other tests, the basic flow energy (BFE) required by a rotating blade to move across a given volume of powder (25 mL for the flowability test and 35 mL for the aeration test) filling a 2.5 cm diameter cylindrical vessel (with 6.1 cm length vessel for the flowability test and 8.0 cm length vessel for the aeration test) is determined as a function of the helix vertical translation velocity (rotation speed of 2 rpm) or as a function of the air velocity injected in the particles bed. In this second case, the powder reach a state, similar to the minimum fluidisation state, at the characteristic air velocity (CAV) – when the flow energy, named aerated energy (AE) at the CAV has stabilised.

Since the flowability of a powder is strongly correlated to the size and shape of the particles, a special emphasis is brought to the morphological characterisation of the latter. For this purpose, an experimental set-up has been designed to homogeneously distribute a given amount of powder on a flat surface and analyse them by optical microscopy. More precisely, as depicted on Figure 1, an airtight vessel of 15 cm diameter is arranged on a horizontal plate and vacuum is made by a pump down to 267 mbars. Typically 20 mg of powder is then poured at atmospheric pressure in an intermediate chamber placed above the vessel. Finally, the gate between the admission chamber and the vessel is open. The powder is entrained by the air penetrating the empty vessel and homogeneously dispersed by turbulences. With this method, there is almost no overlapping and each grain can be analysed individually.

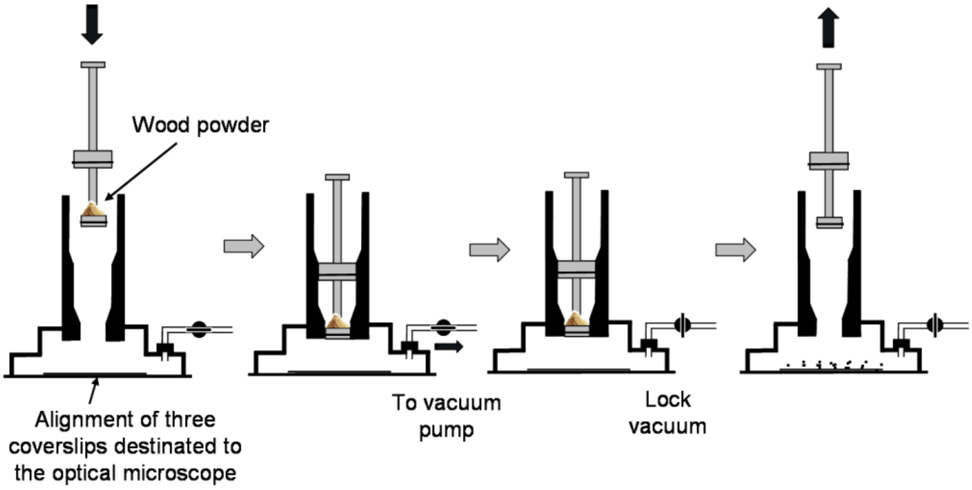

Figure 1: Scheme of the tool designed for particles distribution.

A portion of 30 x 8 mm of the dispersion plate is analysed by optical microscopy. A complex image analysis procedure was finally developed to obtain a statistically representative characterisation of the morphology (particle size, shape distribution, surface state) as (Cao et al., 2010) recently recommended. The reproducibility is ensured by the large number of particles (between 20000 and 90000 particles for each of the three measurements).

3. Results and discussion

3.1 Rheological study

Results obtained with the FT4 Rheometer® and the Flodex® method show (Figure 2, Table 1, Table 2) that, when torrefied at 240°C, spruce powder (S240) has the same behaviour as in natural state (NS). For the S300 powder (torrefaction at 300°C), the flowability is much better (see BFE values in Table 1). Thus, the biomass flowability increases with the torrefaction temperature. The comparison with the behaviour of a powder of mono-dispersed rigid spherical glass particles (GB 560 µm) shows the influence of the size, shape and surface state of the particles on their flowability (Table 2).

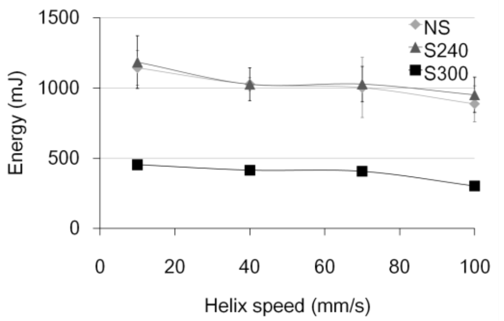

Figure 2: Mechanical energy measured with FT4 Rheometer® and required by a rotating blade to move across 25 mL of powder filling a 2.5 cm diameter and 6.1 cm length cylindrical vessel as a function of the helix vertical translation velocity (rotation speed of 2 rpm) for different wood powders.

Table 1: Conditioned bulk density (CBD) and basic flow energy (BFE) of wood powders NS, S240 and S300.

Sample	NS	S240	S300
CBD (g/mL)	0.13 ± 0.00	0.16 ± 0.01	0.21 ± 0.00
BFE (mJ)	1057 ± 20	1024 ± 27	296 ± 8

Table 2: Comparison of the flowability of several types of wood powder using Flodex®.

Sample	Spruce			Glass beads
	NS	S240	S300	GB 560 µm
Min. diameter[a] (mm)	34	34	26	4

[a]Minimum diameter required for the powder to freely fall through a hole.

Compressibility measurements were done with the FT4 Rheometer®. Results show a lower deformation for spruce torrefied at 300°C (ΔV/V = 22% @ 15 kPa) than for spruce torrefied at 240°C (ΔV/V = 32% @ 15 kPa) and for natural spruce (ΔV/V = 43% @ 15 kPa) (Figure 3). As for the aeration test, S300 characteristic air velocity (CAV) is 2 mm/s (Figure 4, Table 3). According to Freeman Technology W7015 instruction, this can be likened to a minimum fluidisation velocity when the flow energy is reduced to near zero (typically less than 10 mJ). Therefore, in the compressibility and aeration tests, it appears that a torrefaction at 300°C leads to powders with low cohesivity and better aeration characteristics than natural spruce or spruce torrefied at 240°C (Figure 3, Figure 4, Table 3). These results are consistent with those previously obtained with the Flodex® (Table 2).

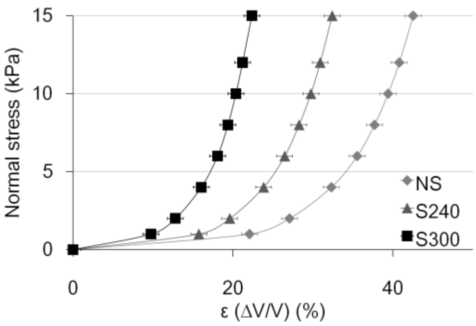

Figure 3: Stress-strain curves for different wood powders.

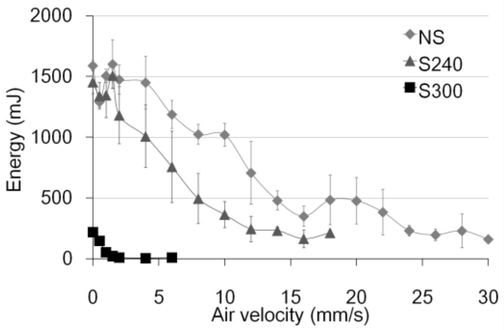

Figure 4: Mechanical energy measured with FT4 Rheometer® and required by a rotating blade to move across 35 mL of powder filling a 2.5 cm diameter and 8.0 cm length cylindrical vessel as a function of the air velocity for different wood powders.

Table 3: Characteristic air velocity (CAV) reached for wood powders NS, S240 and S300 and its corresponding aerated energy (AE).

Sample	NS	S240	S300
CAV (mm/s)	26	14	2
AE (mJ)	161 ± 19	213 ± 24	10 ± 1

3.2 Morphological study

After dispersion with the in-house developed system (Figure 1), all samples were observed by optical microscopy. The particle equivalent circular diameter (ECD) distribution (Figure 5) is wide with a mode at 560 µm for NS, at 890 µm for S240 and at 140 µm for S300. It seems that, in the case of S240, some of particles with larger size than 500 µm passed through the sieving grid, that is surely due to segregation of particles in the powder.

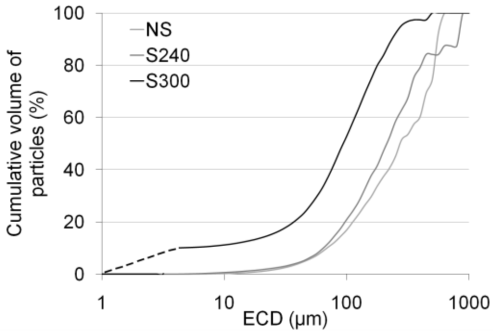

Figure 5: Particle diameter distribution of NS, S240 and S300 particles.

A typically used morphological parameter to characterize particles of a given powder, the Sauter mean diameter (d_{32} or D[3, 2]), can also be determined. It is defined as the diameter of a sphere that has the same volume/surface area ratio as a particle of interest:

$$d_{32} = \Sigma d^3 / \Sigma d^2 \quad (1)$$

This diameter reduces when the torrefaction temperature increases: 170, 132 and 68 µm are respectively obtained for NS, S240 and S300. It can be explained by the decreasing in wood ductility and the increasing in wood fragility which induces smaller particles when ground.

Optical microscopy allowed us to also obtain several form factors like circularity, elongation and roughness (Figure 6, Figure 7, Figure 8). Wood particles are not spherical even for high temperature torrefaction (S300) (mode between 0.35 and 0.4 in Figure 6) and their particularity deals with their elongated form. Figure 7 shows this elongation (length/height ratio) with a mode between 1.5 and 2. Based on the proposal of (Bouwman et al., 2004), the roughness of the particles is calculated by:

$$\text{Roughness} = 1 - (\text{Convex Perimeter} / \text{Perimeter}) \quad (2)$$

The roughness of our powders ranges between 0.02 and 0.4 (Figure 8).

These results characterizing particles morphology show no significant difference of the shape factors between the three observed powders. The torrefaction seems to have a low effect on the morphology of particles obtained after grinding.

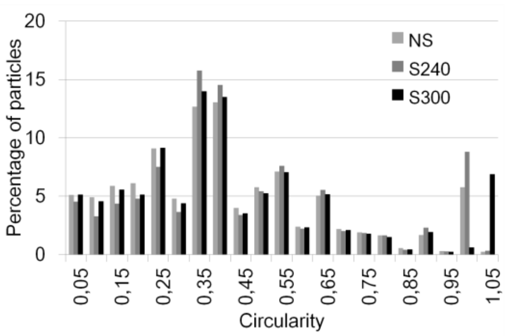

Figure 6: Circularity distribution of NS, S240 and S300 particles.

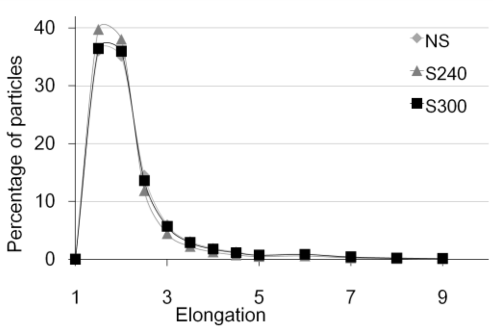

Figure 7: Elongation distribution of NS, S240 and S300 particles.

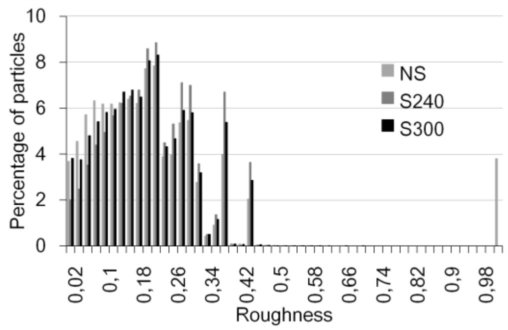

Figure 8: Roughness distribution of NS, S240 and S300 particles.

4. Conclusion

Contrary to the uniaxial compression test which shows a difference between NS and S240 powders, BFE determination, Flodex® method and aeration test show that they approximately have the same behaviour. However, an important observation made in these last three tests deals with a flowability significantly higher for S300 than for the two other powders.
Besides, the morphological analysis allows us to conclude that, for given grinding conditions (grid size of 500 µm), the severity of the torrefaction:
- does influence significantly particle size distribution. This influence is indirect, made through grinding.
- does not influence the particle morphology: circularity, elongation and roughness. As for the correlation between the micro-level (particles size and morphology) and the macro-level (flowability as measured by the three first tests), we observe that the Sauter mean diameter is the only morphological parameter that may influence the powder flowability.

The torrefaction severity seems to influence particle size but having a better knowledge of its direct effect on the rheological powder properties is our next perspective. Experimentally it will consist in testing a new powder production protocol based on grinding natural wood followed by its torrefaction. Although energetically defavourable, this kind of experiment would be helpful to understand how the size and morphology but also the surface state (fluffs), chemical composition and electrostatic properties may be affected by the thermal treatment. The last three parameters are believed to play an important role in the flowability. These studies are currently under progress.

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