Physical Properties of Biofuel Pellets Vacuum Treated with Vegetable Oil

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ABSTRACT

Wood pellet production as solid biofuels is increasing rapidly in Europe. One of the challenges from this industry is to manufacture firm pellets with the highest energy content as possible and with a long shelf life. Addition of oil is useful for the aforementioned last two reasons, but it weakens the pellets when present in the compaction process. This work examines the addition of oil after the compaction process through a vacuum infusion process. The results show that oil can be added to the pellets while keeping their strength if pressure is well controlled in the infusion process.

INTRODUCTION

Gobal production of wood pellets is growing significantly, in 2012 was estimated a total of 22.4 million tonnes which constitute an increase of more than 55% compared to the productions in 2010. The European Union is the world market pellet leader and consumes around 67% of the worldwide production[1, 2].

Used or waste vegetable oils by the food industry have high potential to be incorporated into solid biofuels because of their high energy content. Oils also provide a hydrophobic shell that increases the shelf life of pellets[2, 3]. Waste vegetable oils are available in large quantities and they do not require pre-treatments[3, 4]. Other advantages of waste vegetable oils are that they have low quantities of heavy metal and low contents of water. Oil also have beneficial properties to the process, it acts as a lubricant in the pelleting process, reduces die friction and therefore decreases power consumption.

The negative side of oils for pelleting process, is that addition of oil reduces significantly the physical strength of the pellets and reduces the compressibility of sawdust[3]. The previous study we conducted[3], showed that addition of oil as low as 2.2% reduces significantly the strength of pellets when tested under diametric compression.

The present work aims at increasing the energy content of biofuel pellets through the addition of oil, at the same time affecting as little as possible the strength of pellets. Specifically, this work studies how the physical properties of pellets are affected when incorporating oil in a post pelleting operation. The idea behind infusing oil after the compaction process is to avoid interfering with the formation of bonds.

Biofuel pellets are dense compacts with usual densities of around 1200 kg/m^3. The voids inside the pellets are small and it is hard for the oil to penetrate deep into the pellet because of the oil viscosity, surface tension and small porous size.

The pelleting process is a continuous process. If a process step requires a batch, it is sought to be as quick as possible to avoid large installations holding the materials. This research will examine the convenience of infusing oil through vacuum, in a similar way as in the fish feed industry with extruded pellets. The fish feed industry commonly refers to this as vacuum coating.

Today it is possible to find a number of producers of batch vacuum coaters that are compatible with continuous pellet production in the market, therefore, it is of interest to analyse the convenience of using this processing technique.

MATERIALS AND METHODS

Vacuum treatment with vegetable oil

Wood pellets were procured from the largest producer of wood pellets in Sweden, Laxå pellets AB. Present production is 95 000 tonnes/year. The diameter of the pellets is 8 mm and are made from fresh spruce chips mixed with dry pine chips that, according to the producer[5], gives "dense, firm and solid" pellets.

Table 1. Main properties of the pellets.

Parameter	Value*	Unit
Ash content	< 0.5	%
Water content	~ 8	%
Energy content	>4.8	kWh/kg

*Values provided by Laxå Pellets AB

The oil selected for the infusion process is rapeseed oil since it is commonly used in food frying process and it is collected from restaurants and food industry once is used.

The experimental arrangement to infuse oil in the pellets through vacuum is shown in Fig. 1. The system is able to recreate the physical environment surrounding the pellets on industrial vacuum coaters. The difference with the industrial oil infusion process, is that on our experimental arrangement, we are able to infuse oil into individual pellets placed into cylindrical holes. The idea is to measure the oil absorption for each pellet. The cylindrical holes have a diameter of 10 mm. The experimental procedure is as follows: (1) pellets are introduced into the PTFE container (Fig. 1). The PTFE container is thereafter inserted into a glass chamber

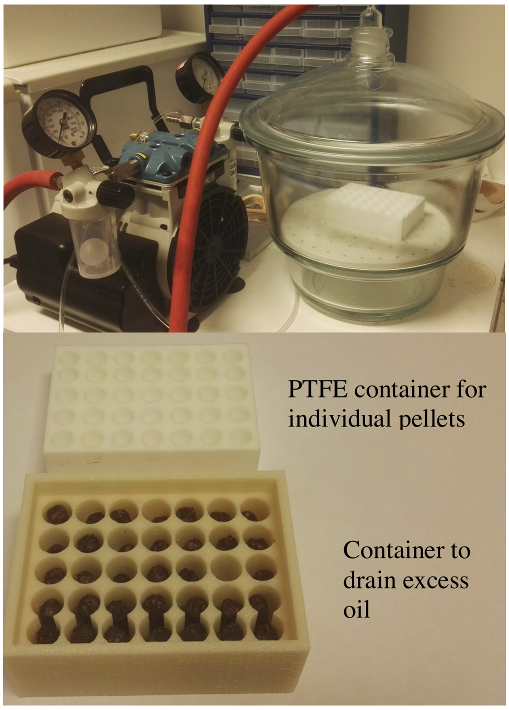

Figure 1. Arrangement to infuse oil through vacuum. The top picture shows a glass vacuum chamber having a PTFE container inside. The chamber is connected to a vacuum pump. At the bottom, a detailed view of the PTFE container and a container to drain the excess oil.

which is connected to a vacuum pump. Oil is added until reaching the top surface of the PTFE container for each cavity holding a pellet. Once filled with oil, the glass cover is closed and the vacuum pump is activated reducing the pressure by 900 mbar. The pellets remain in the chamber under vacuum for 4 min. During this time, foaming at the surface was observed and it decreased substantially when reaching 4 min. This gave an indication that most of the air inside the pellet was extracted. Thereafter, the vacuum was gently released during a period of 2 and 4 min.

Oil uptake (%) is calculated as mass of absorbed oil relative to the initial mass of wood pellet.

Surface Roughness

The surface roughness of the pellets was determined in a needle profiler (Surftest SJ-210, Japan) that follows the ISO 4287:1997 standard. Based on 10 pellets, the average surface roughness (R_a) ± 95% CI and the mean peak-to-valley height (R_z) ± 95% CI are presented for the two treatments with 2 and 4 min of vacuum release time.

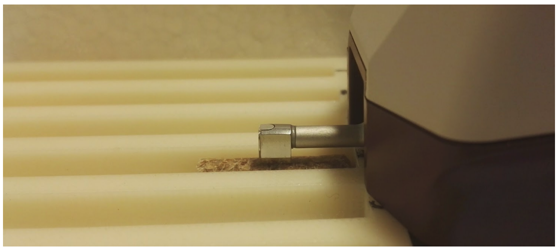

Figure 2. Needle profiler measuring the surface roughness of a pellet

As observed from Fig. 2, a single pellet is placed in a specially designed and 3D printed ABS holder.

Bending stress

To determine the strength of pellets under bending, a three-point bending test was conducted. In the same way as in our previous work[6], the three-point bending test was selected in lieu of the typically used diametric compression test because of the inability to precisely determine the length of the pellets, and thus to correctly interpret the results. The same type of tests were also used by other authors on pellets[1]. This test is able to replicate the type of loading during storage, transportation, and handling[6]. A detailed description of the bending test method can be obtained from Li et al.[7].

A single pellet was placed over a specially designed holder attached to a Lloyd LR 5K texture analyser (Fig. 3). The bending speed applied over the pellet was set to 1 mm/min until reaching the maximum peak bending load. The maximum bending stress (σ) for cylindrical pellets is calculated using Eq. 1., were F is the maximum bending load, L the support spam (8.5 mm) and d the pellet diameter.

$$\sigma = \frac{8FL}{\pi d^3} \qquad (1)$$

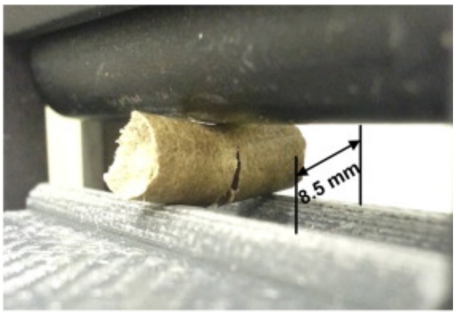

Figure 3. Three-point bending test. The picture shows a pellet fracture during testing. Image taken from Misljenovic et al.[6]

Contact angle

Surface hydration properties of the pellets and its changes over time were assessed by measuring optically the contact angle (CA) of a sessile drop placed on a diametrically positioned pellet. CA measurements were conducted at room temperature (23 °C) in an optical contact angle meter OCA 15EC (DataPhysics Instruments GmbH, Germany). A 3 µl drop of distilled water was disposed from the dosing pump of the equipment onto de cylindrical surface of the pellet. Videos of the drop over the pellets were recorded. The videos were thereafter analysed by SCA 20 software 15 EC (DataPhysics Instruments GmbH, Germany). The method used in pellets (Fig. 4) have been fully explained in our recent publications[3, 6, 8].

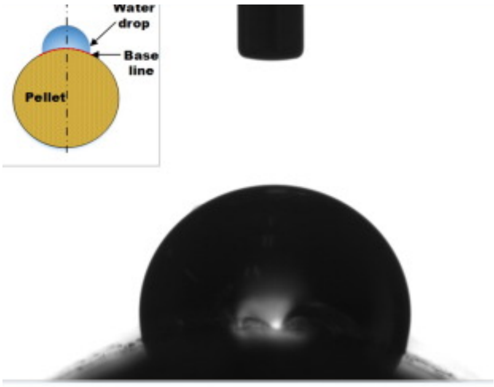

Figure 4. Example of a drop profile extraction and the measurements of contact angle on a cylindrical surface of a pellet. Image taken from Misljenovic et al.[6]

RESULTS AND DISCUSSIONS

Oil uptake

The oil uptake for the vacuum treated pellets with 2 min of pressure release was 6.6 ± 0.4 and 6.7 ± 0.3 % for pellets vacuum treated with a pressure release of 4 min. No statistical differences ($p > 0.05$) were found between the two treatments. In a previous work with single pellet press[3], when adding oil before pelleting, we were only able to achieve 5.8% oil uptake, above that value, the pellets were unable to hold their shape.

Surface Roughness

The average surface roughness of coated pellets, R_a, and the mean peak-to-valley height (R_z) increased ($p < 0.05$) after applying rapid oil infusions (2 min release pressure). However, when increasing the release time of vacuum to 4 min, i.e. decreasing dP/dt during oil infusion process, the surface roughness of pellets was not altered ($p > 0.05$). These results indicate the importance of subjecting the pellets to slow rates of pressure change to lower oil flow rates through the porous structure.

The forces influencing changes of surface roughness when quickly releasing vacuum, i.e. disruptions of bonds, are possibly the shear stresses and the strains occurring at the porous walls caused by the flow of oil. None of the pressure changes in the absence of oil, as seen in Fig. 5 and Fig. 6, influenced the roughness of the pellets ($p > 0.05$). Consequently, only rapid pressure changes during oil infusions produced changes in surface roughness.

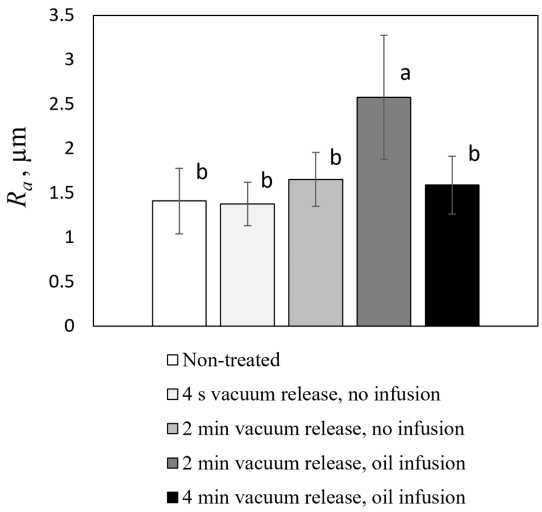

Figure 5. Average surface roughness, R_a, for non treated and vacuum treated pellets. Pressure changes were applied with and without oil.

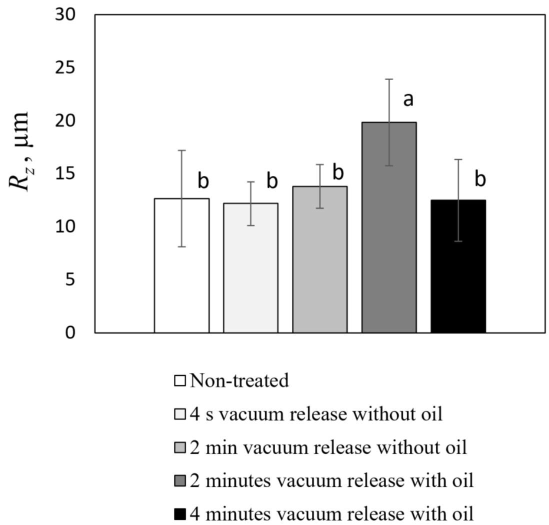

Figure 6. Mean peak-to-valley height, R_z, for non treated and vacuum treated pellets. Pressure changes were applied with and without oil.

Bending stress

As can be observed from Table 2, the maximum bending stress that pellets tolerated were between 7.6 ±1.04 and 9.1 ±1.16 MPa. The effects of vacuum coating over the maximum bending stress were not large enough to be statistically different with a CI of 95%. Averages of bending stresses were calculated from the samples presented on Fig. 7, Fig. 8 and Fig. 9.

Table 2. Maximum bending stresses ± CI with 95% confidence for the different treatments

Treatment	Bending Stress (MPa)
Non-Treated	9.1[a] ±1.16
2 min vacuum release with oil	7.6[a] ±1.03
4 min vacuum release with oil	7.6[a] ±1.04

A similar letter indicate no significant differences ($p > 0.05$)

When comparing the changes in strength from the pellets treated with oil from this work (post pelleting, i.e. post bond formation) to the results we obtained previously from pellets pelleted with up to 5.8% oil[3], it is clearly concluded that addition of oil is not recommended during pelleting process. Oil also acts as a lubricant and it reduces the compaction pressure in die ring pellet presses.

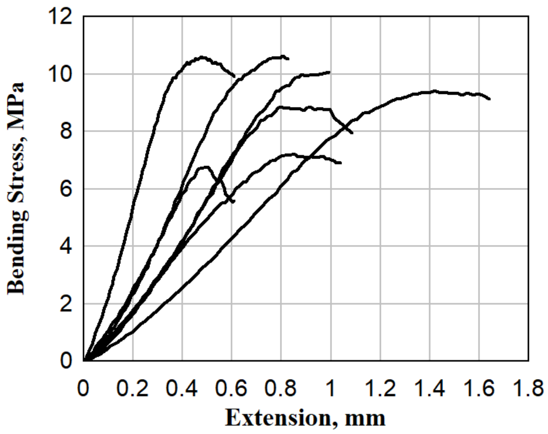

Figure 7. Bending stress (MPa) of un-treated wood pellets

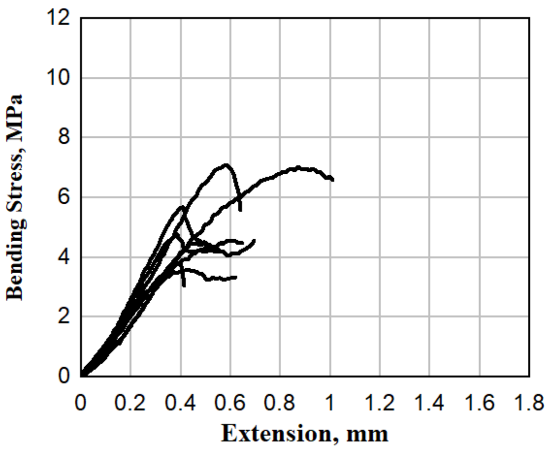

Figure 8. Bending stress (MPa) of coated pellets using 2 minutes pressure release time

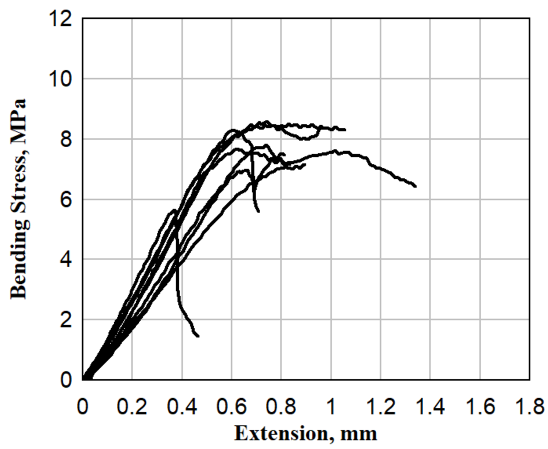

Figure 9. Bending stress (MPa) of coated pellets using 4 minutes pressure release time.

Contact angle

Fig. 10 shows that only a quick release of vacuum pressure in presence of oil (2 min), changed the initial contact angle. Generally speaking, the causes affecting contact angle can be changes in surface roughness or the chemistry of the surfaces.

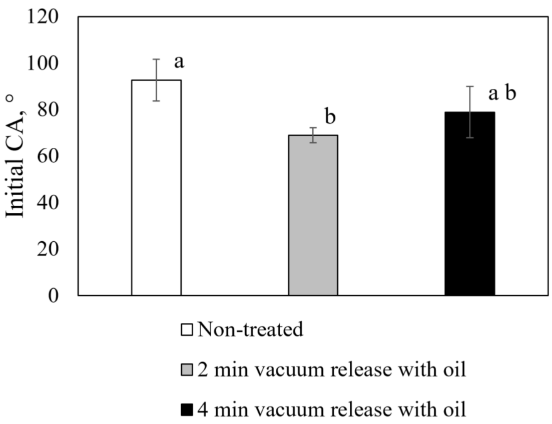

Figure 10. Initial contact angle. Means ± CI that do not share a letter are significantly different ($p < 0.05$).

The effects of surface roughness on contact angle measurements are widely described on literature and they have been described throughout many years [9, 10]. For example, in wooden surfaces, the surface roughness of sanded Beech, Spruce, Birch and Oak have been described as having an effect on contact angle. From that study[11], it was found that the lowest contact angles were found in the samples having the lowest roughness (*R* similar to this study). On our results (Fig. 10), there were no significant differences between the treated pellets. This could be attributed to small changes in roughness and due to the dispersion of the data.

The other cause affecting contact angle is a change in the surface chemistry. Non-treated wooden pellets are rich in lignin while after coating, the pellets are rich in oil at the surface. It is important to mention that both lignin[12] and oil are known to be hydrophobic. Hydrophobicity is desired in wood pellets as it prevents water entering into the pellets. Initial CA can be a good indicator when surfaces do not change during the wetting process, but as previously reported, we have found that pellet surface morphology changes when in contact with a sessile drop of distilled water[3, 6] and consequently it becomes not only important with regard to the initial wetting, but the changes along time.

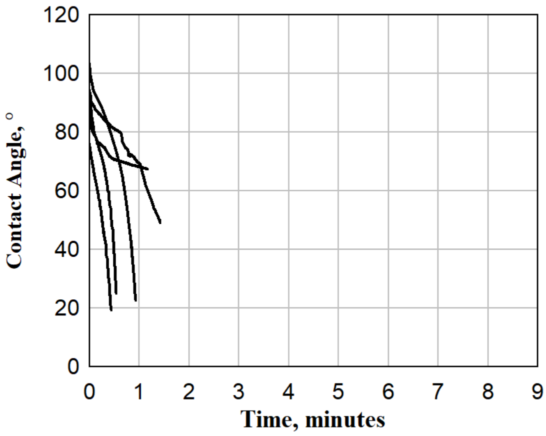

Figure 11. Wettability of non-treated pellets.

Even though the highest initial CA are for non-treated pellets, the hydrophobic effect vanish quickly (Fig. 11) and the water is absorbed in less than 2 min. When comparing Fig. 11 with Fig. 12 and Fig. 13, the first impression is how much longer the water remains in the pellet without been absorbed. Vacuum coating with oil decreases the absorption rate of the 3 μl sessile drop dramatically. When gently vacuum treated pellets with oil, the pellets can hold a sessile water drop for the largest period of time. The sessile drops of water represented in Fig. 13 were not absorbed even after 9 min, but instead they started to evaporate. No marks in the surface of the pellets in contact with water were observed from the group treated with oil and 4 min of vacuum release time. On the contrary, the non-treated pellets presented a large swelling at the surface due to the sessile water penetrating into the structure.

From our previous work[3] on pellets made of mixed oil (5.8 %) with ground wood, a significant increase of water absorption rate was found when comparing with pellets without oil. On the contrary, this study shows a large decrease of water absorption rate when oil vacuum treating the pellets (i.e. in a post forming operation). This can be explained as oil during compaction weakens the bonds and the structure becomes much easier to disintegrate in presence of water.

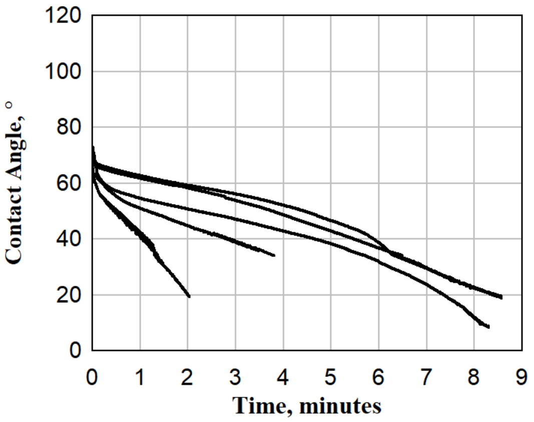

Figure 12. Wettability of coated pellets using 2 minutes of pressure release.

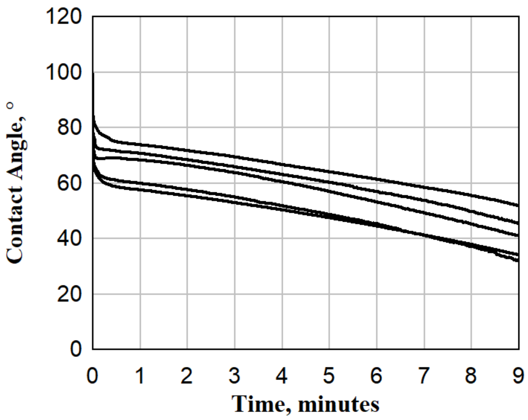

Figure 13. Wettability of coated pellets using 4 minutes of pressure release.

The results from this study show the benefit of using vacuum to coat pellets with oil. Despite of the promising results from this study, further studies should also contemplate leakages of oil after exposure to heat. Solid biofuel pellets can be exposed to heat if they are stored in direct contact with the sun during summer or in their path to the combustion chamber in burners.

CONCLUSIONS

It is concluded that the release time of vacuum of a pellet immersed in oil affects the maximum bending stresses that wood pellets can withstand. A quick release of vacuum (2 min) decreased the strength of pellets, while there was no statistical significant difference in strength when the pellets were treated with slower pressure changes. A similar observation was obtained for the changes of surface roughness. Coating with oil decreases significantly the absorption of a sessile water drop sitting on the pellet. The lowest absorption rates of water were obtained for pellets treated with slow pressure changes during the infusion process.

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