Impact of Amylose and Amylopectin Content in Starch on Wood Pellet Production

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GRAPHICAL ABSTRACT

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In the production of wood fuel pellets, starch is frequently used as an additive to enhance bonding and durability. This study investigated the effectiveness of four different kinds of starches as additives, each at a concentration of 5% (dry basis), when combined with sawdust from Scots pine (*Pinus sylvestris*). The starches tested included plain wheat flour, hydrothermally treated wheat starch, wheat starch with amylose-like properties, and nearly pure amylopectin obtained from waxy rice flour. All pellets were produced at a die temperature of 100 °C using a Single Pellet Press, with varying moisture contents of 5%, 8%, 11%, and 14% (wet basis). The pellets were evaluated for compression work, back pressure, physical density, hardness, and moisture content. Additionally, chemical bonding was assessed using FT-IR spectroscopy. Compression energy was found to be influenced by moisture content, irrespective of starch utilization, and it decreased with increasing moisture levels, especially between 5 to 8% (wb). The inclusion of starch led to notably higher pellet hardness, with amylose yielding the hardest pellets, 34±3 kg when the moisture content was 11%. Based on this study, it is recommended to use hydrothermally treated wheat flour, as it consistently produced high-quality pellets.

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Keywords: Single-pellet press; Biomass; Starch derivatives; Amylose; Amylopectin; Moisture; Quality

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INTRODUCTION

As society strives for a sustainable future that is free from fossil fuels, the use of lignocellulosic biomass for energy production and the creation of alternative products, such as platform chemicals, is gaining global attention. This is due to its renewable nature and economic viability. Lignocellulosic biomass can be derived from wood, such as sawdust or other residues from wood and paper mills, or non-wood biomass, such as agricultural residues, straw, *etc.* Both types can be transformed into value-added products or fuels in the form of pellets. The use of biomass fuel pellets has seen a notable increase worldwide, particularly in Europe and most notably in Great Britain. In 2022, 46.4 million tons of pellets were produced globally (WBA, 2023). This surge is a result of policy changes and industry collaboration aimed at achieving coal-free electricity production. Wood fuel pellets, made from woody biomass waste such as sawdust or shavings, have emerged as a highly successful renewable fuel source for heat and power production. This success is

largely due to their numerous beneficial characteristics, including high density, higher calorific value, low moisture content, and the relative ease of transportation and storage (Tarasov *et al.* 2013). The depletion of fossil fuels and the urgent need to reduce greenhouse gas emissions have led to a robust growth in the global use of wood fuel pellets in recent years.

Despite being in operation for over 40 years, the pellet manufacturing industry continues to refine its processes. It occasionally grapples with challenges related to handling bulky biomass, including pretreatment methods (such as sorting and grinding), managing dust formation, and preventing self-ignition incidents during storage (Siwale *et al.* 2022). The understanding of the processes occurring within a wood pellet during production remains limited. This limitation arises from the numerous variables that impact the bonding properties in biomass pellets. These variables are associated with both the feedstock species and the technical parameters during the production process (Mani *et al.* 2006; Stelte *et al.* 2012). Given these complexities, it becomes a priority to produce pellets that meet standard requirements (ISO 17225-2:2014), such as durability, using existing equipment and main raw material species, even when the raw material changes. Consequently, an economically viable solution is needed. Currently, pellet production companies are addressing these issues by using various types of additives when a change in raw material is necessary.

The use of additives in wood pellet production is a widespread practice globally (Kuokkanen *et al.* 2011; Berghel *et al.* 2013; Tarasov *et al.* 2013; Larsson *et al.* 2015; Li *et al.* 2022). According to the standard ISO 17225-2 (SIS 2014), an additive is defined as a material that can be used in pellet production up to a maximum of 2% (by weight, as received) of the total pressed mass. Some of the most frequently used additives include starch, lignosulphonate, dolomite, corn flour, potato flour, and vegetable oils (Stelte *et al.* 2012; Ståhl *et al.* 2016). These additives not only enhance the physical qualities of the pellets, such as durability and hardness, but they also improve storability and material handling. Additionally, they can make the pellet production process more energy-efficient (Ståhl *et al.* 2012).

Starch is widely used in various industries around the world, ranging from food and feed production to paper and pellet manufacturing. Industries that produce starch not only generate native starches from a variety of crops, such as wheat, oat, maize, and potato, but also create different derivatives of starch. These derivatives are native starches that have been modified through heat treatment or a chemical process such as oxidation to produce a starch tailored for a specific application. The more a starch derivative is modified, the more expensive it becomes. Furthermore, starches can be soluble in either hot or cold water depending on the modification performed. This could influence the choice of pretreatment method, whether using steam or cold water, in pellet production when incorporating starch. The efficiency of a binder is influenced by the characteristics and properties of starch. A starch binder possesses good ductility, excellent adhesion properties, self-curing properties, and non-hygroscopicity (Mohd *et al.* 2016). Identifying a kind of starch or starch derivative suitable for pellet production involves not only finding a starch or starch derivative that enhances the production process and the quality of the pellet, but also one that is relatively inexpensive. This is because the ultimate goal is often to burn the pellets, and the cost of raw materials represents one of the largest expenses in pellet production.

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The findings of conducted tests on two starch products and two lignosulphonate products in full-scale pine pellet production revealed that these additives primarily affect the durability and the number of fines (Samuelsson *et al.* 2014). Moreover, moisture content has emerged as the most crucial parameter linked to both bulk density and the power supply of the pellet press. Potato starch and potato residue have been tested as binding materials in pellet production, and they were shown to have a positive impact on pellet durability and compactness (Kuokkanen *et al.* 2011). A review demonstrated that all starches increased the mechanical durability of the pellets up to 7%. However, using more starch resulted in drier pellets and reduced durability (Tarasov *et al.* 2013). Using pea starch as an additive at 0.6% (wt) allowed for a reduction in the energy required for fullscale wood pellet production in a plant. This included reducing the amount of steam conditioning and lowering the steam temperature, while maintaining pellet quality (Ståhl *et al.* 2019). Additionally, the use of cassava starch, at 5% (wt), in wood pellets resulted in increased durability and a decreased amount of fines (Larsson *et al.* 2015). Ståhl *et al.* (2012) found that native wheat, potato, oxidized potato, and corn starch increased the pellets' durability and reduced electricity consumption. When more expensive and modified starches were used, the results showed even better outcomes regarding increased durability and energy efficiency. Almost every study has shown results of increased pellet quality and decreased energy need when starch additives are used. However, only a few studies have used modified starch derivatives. The characteristics and properties of starch indeed influence its effectiveness as a binder. Native starches, however, have numerous drawbacks that can restrict their application and industrial use (Waliszewski *et al.* 2003). To improve the structure and binding capability of starch when used as a primary binder, it can be modified through physical or chemical methods (Amini *et al.* 2013). It seems clear that starches increase durability and have the potential to decrease energy needs during production. However, only one of these studies evaluated starches based on their specific content.

Starches hold a unique position within the group of carbohydrates, as they are composed of two polymers: amylose, a mostly linear polysaccharide, and amylopectin, a large, highly branched polysaccharide (Frodeson 2019). The proportion of amylose to amylopectin varies among different types of starches. For instance, waxy maize can have an amylose content as low as 1%, regular corn starch contains 25 to 28% amylose, wheat starch contains 25 to 29% amylose, while high-amylose barley can contain as much as 37% amylose (Fredriksson *et al.* 1998). This wide range underscores the diversity of starch compositions and further highlights the need for research to understand the optimal amylose to amylopectin ratio for pellet production since this ratio influences the physical properties of the starch. During the process of gelatinization, starch granules swell and form gel particles. Generally, starches enriched in amylopectin swell to a greater extent than starches with high amylose content. The linear amylose diffuses out of the swollen granules and forms the continuous phase outside the granules. Research has demonstrated that waxy maize starch exhibits a unique crystalline structure and thermodynamic properties, distinct from those of high-amylose maize starch. As the amylose content rises, the crystallinity typically diminishes, since the amylose is found within the amorphous part (Gérard *et al*. 1998; Matveev *et al*. 2001). Waxy starches typically also swell more than their normal-amylose counterparts, and amylose is thought to act as a restraint to swelling (Fredriksson *et al.* 1998). The amylose and amylopectin content will affect the properties

of starch such as gelatinization, paste viscosity, gel stability, and solubility (Builders *et al.* 2014). In comparison to amylopectin, amylose polymers have a reduced surface area and a greater quantity of intramolecular hydrogen bonds. Consequently, due to enhanced molecular adhesion, diminished starch swelling, and a decelerated enzymatic digestion rate, it can either remain undegraded or undergo slow degradation by α-amylase (Matveev *et al.* 2001).

Even though starch is commonly used as an additive in wood pellet production, there is a knowledge gap regarding whether it is the amylose or the amylopectin in the starches that contributes to the increased bonding mechanisms in the pellet. Therefore, the kind of starches used in this study contain varying amounts of their main constituents, amylose and amylopectin. The purpose of this study was to enhance our understanding of how various kind of starches and the ratios of amylose and amylopectin within these starches influence the mechanical properties of pellets. The study aimed to examine the compression work (W_{comp}) and backpressure (F_{max}) during pelletization, as well as the density, hardness, and bonding properties of the pellets. This was done by adding four different kinds of starches to pine sawdust. These starches included plain wheat flour, a hydrothermally treated wheat starch, a wheat starch resembling amylose, and a nearly pure amylopectin derived from waxy rice flour.

EXPERIMENTAL

Materials

The base raw material used in all tests was Scots pine (*Pinus sylvestris*) from the county of Värmland, Sweden. The raw material was prepared for pelletization as described in (Frodeson *et al.* 2019). This process involved sawing the pine log, which had been stored at the felling site for 1 month, into sawdust using a Bosch GCM 8 SJL (Stuttgart, Germany). The sawdust was then ground in a Culatti Mikro Hammer Mill (DFH 48; Limmatstrasse, Zurich, Switzerland) with a sieve size of 2 mm to attain a uniform particle size, to emulate the industrial process. The sawdust was dried in an oven at 50 \degree C for 48 h to ensure a consistent starting point regarding moisture content. The initial moisture content of the pine sawdust and all starches was determined according to ISO 18134-1 (SIS 2015) on a wet basis (wb) as described in (Frodeson *et al.* 2019).

Four types of starches were used as additives: plain wheat flour, hydrothermally treated wheat starch, a wheat starch resembling amylose, and a nearly pure amylopectin derived from waxy rice flour.

- *Wheat Flour*: This is a plain wheat flour with a starch content that is roughly 25% amylose and 75% amylopectin.
- *Hydrothermal Wheat Starch*: This is a water-heat treated wheat starch (*H.t Wheat starch*) where the viscosity of the 7% solution is a maximum of 250 mPa. Through this treatment, the starch is completely gelatinized and the protein is denatured.
- *Amylose*: This is a wheat starch that is debranched through an acid treatment. This procedure primarily cleaves 1,6-glycosidic bonds, resulting in a starch that is more amylose-like than the starting material. Additionally, the Amylose was pregelatinized

through drum-drying. All particles were milled down to less than 250 µm.

• *Amylopectin*: This is a plain waxy rice flour where the starch content is predominantly amylopectin, with only very small amounts of amylose.

All four kinds of starches were mixed with pine sawdust at four different moisture contents: 5%, 8%, 11%, and 14% (wb), as shown in Table 1. Most pellet industries produce pellets at 8 to 11% MC, but since starch is sensitive to water, a low and a high MC was added to the test matrix. Each mixture, with a total weight of approximately 15 g, contained 5% starch addition (db). An amount of 5% starch was chosen to get notable results between the samples, although that it is higher than the standard allows (ISO 17225-2:2014). The mixing was conducted using a 220V Janke and Kunkel Ika-Werk mixer (Ibbenbüren, Germany) at a speed of 60 rpm for 10 min, as further described in (Anukam *et al.* 2019). The production cost is lower for the native starches (Wheat flour and Amylopectin) compared to the modified starch derivatives (Hydrothermal Wheat starch and Amylose), as it involves less production steps.

The Pellet Production Process

For all the sample combinations, ten pellets were produced using a Single Pellet Press (SPP). This press is available at the Department for Engineering and Chemical Sciences at Karlstad University in Sweden, and its operation is well-documented in (Anukam *et al.* 2019). Each sample, weighing one gram, was pelletized under optimal conditions as per Table 2. During the pelletizing process, the force was logged three times per second (Frodeson *et al.* 2018). The compression work, denoted as W_{comp} (J), was calculated by integrating the force and distance from the logged data. This was done using the numerical integration trapezoid method, based on the time required to increase the force from 0.5 to 14 kN. The compression work is presented as the mean values from the test series. The maximum force required for the piston to eject the pellet was recorded as the highest force generated. This value, denoted as F_{max} in kN, represents the maximum potential backpressure level that the pellets can generate due to friction between the surfaces of the pellets and the die (Frodeson *et al.* 2018).

Table 1. Moisture Content (wb) of Test Samples - Set Point, Inlet, and Pellet Values

Table 2. Conditions for Single Pellet Production During Pelletizing.

Solid Density and Hardness

The springback effect was evaluated following the procedure outlined in Frodeson *et al*. (2021). Measurements of pellet length and diameter were taken immediately after pelletizing, and again one week later, to calculate the pellet density. A week after production, the hardness of the pellets (in kg) was measured using a KAHL motor-driven hardness tester (K31475-0011, Reinbek, Germany). This tester was equipped with a 3.5 mm spring, suitable for the 0 to 100 kg range. The production results (W_{comp} and F_{max}) were represented as the average of nine pellets. For the quality parameters (density and hardness), the average was taken from six pellets, as three pellets were utilized for FT-IR analysis. Standard deviation is presented for all parameters.

Fourier Transform Infrared (FT-IR) Spectroscopic Analysis

Fourier Transform Infrared (FT-IR) spectroscopic analysis was used to investigate the structural alterations in the pellets. Major functional groups associated with the primary components of the samples, their bonding, and the pellet quality were studied in terms of hardness (Anukam *et al.* 2019). The FT-IR spectra were acquired using an Agilent Cary 630 FTIR spectrophotometer (USA), equipped with a diamond ATR. Subsequently, the spectra processed using MicroLab PC and Resolutions Pro software. Pellet samples from all batches were analyzed at room temperature, covering wavenumbers ranging from 650 to 4000 cm−1. This analysis followed the procedure described by Frodeson *et al.* (2021).

RESULTS AND DISCUSSION

Compression Work and the Maximum Force Needed to Press Out the Pellets

The compression work, denoted as W_{comp} , was determined by the time required to increase the force from 0.5 to 14 kN. The compression process was stopped once it reached 14 kN (Frodeson *et al.* 2018). Figure 1 illustrates the impact of moisture content at 5%, 8%, 11%, and 14% (wb) on the compression work for both the control material and the control material mixed with 5% (db) of respective starch used. It is evident from the figure

that *W*comp was significantly influenced by the moisture content below 8% MC in all samples. Furthermore, *W*_{comp} was notably higher for the lowest MC of 5% (wb) across all test samples. In the context of single pellet processing (SPP), *W*comp showed an inverse relationship with MC, as depicted in Fig. 1. This finding aligns with Nielsen's study (Nielsen *et al.* 2009), where the energy required for compression (*W*comp) decreased with MC for pellet production of beech and pine sawdust at a die temperature of 125 °C. The cited authors suggested that this might be due to the softening of lignin in the sawdust (Nielsen *et al.* 2009). Additionally, it was found that the *W*comp of the hydrothermal wheat flour was significantly lower than *W*comp for all other samples, with the exception of MC at 14% (Fig. 1). If the hydrothermal wheat flour is disregarded, there was no notable variation in *W*comp between the control material and the rest of the starches used (Fig. 1). Therefore, it can be concluded that the compression work in single pellet processing primarily depends on the MC for all materials tested in this study. No notable differences were observed between the amylose and amylopectin starches in terms of W_{comp} .

Fig. 1. SPP compression work (*W*comp), including standard deviation, *vs*. moisture content, includes pellets produced from pine (no additive) and pine with 5% (db) starch addition

The maximum force required for the piston to extrude the pellet was recorded as the highest force generated. This value, referred to as F_{max} in kN, represents the maximum potential backpressure that the pellets can create due to friction between the pellet surfaces and the die, which influences the press length of the die (Frodeson *et al.* 2018). Significantly lower *F*max values were observed in the hydrothermal wheat flour compared to the control material and other starches used across all moisture levels (Fig. 2). This result indicates that different starch solutions can impact the full production of pellets in various ways. The fact that F_{max} for the hydrothermal wheat flour was considerably lower than other starches could be associated with reduced energy use during pelletizing when starch is added (Ståhl *et al.* 2012). However, this could also lead to lower bulk density and durability if the press length becomes insufficient. Starches that decrease F_{max} can be viewed as lubrication media. In such cases, it could lead to a situation where the backpressure is not sufficient, resulting in decreased pressure on the raw material and consequently, a reduction in bulk density. This suggests that starches can influence the

process in both positive and negative ways. There was no significant difference in the *F*max value between the control material and the other starch additive mixtures. However, they all exhibited a similar type of *F*max variation with respect to different MC (Fig. 2). Data from all treatments did not show any relationship between respective values of *W*comp and *F*max (Figs. 1 and 2). This result is supported by a previous experiment of single pellet production from different pure substances (lignin, protein, tannin, and various carbohydrates and wood species), which also did not show any relationship between respective values of W_{comp} and F_{max} (Frodeson *et al.* 2018). No notable differences were found between amylose and amylopectin regarding *F*max.

Fig. 2. Maximal friction force (*F*max), including standard deviation, *vs*. moisture content, includes pellets produced from pine (no additive) and pine with 5% (db) starch addition

Pellet density, springback effect and hardness

The results indicated that the use of starch had minimal or no impact on the solid density of the pellets when compared to the control material. As moisture content increased, a decrease in solid density was observed for all tested samples, as depicted in Fig. 3. All kinds of starches exhibited a solid density higher than the reference material, suggesting that the starches slightly influence the solid density. Notably, there was no discernible difference in solid density between amylose and amylopectin. Despite the minor effect of the added starch, Figure 3 illustrates that the inclusion of starch expands the range related to optimal moisture content. In traditional pellet production, maintaining the appropriate moisture content is crucial, as evidenced in Fig. 3. After one week, the density for the control fell much more rapidly than when starch was added, as shown in Fig. 3b. This implies that starch production is likely to yield a wider optimal moisture content. No springback effect was observed between the newly produced pellets and those stored for one week, with all springback effects being below 0.5%.

Fig. 3. Solid density of pellets directly after production (A) and after one week of storage (B), including standard deviation, *vs.* moisture content, includes pellets produced from pine (no additive) and pine with 5% (db) starch addition

The hardness of the produced pellets, a key quality parameter, is depicted in Fig. 4. All mixtures of pine with 5% (db) of starches used yielded harder pellets than those produced from the control pine sawdust, irrespective of the moisture content. This outcome aligns with the enhanced quality parameters, such as durability, as found by (Ståhl *et al.* 2016). The starch types, amylose at 11% MC and the hydrothermal wheat flour at 5% MC, exhibited the highest hardness. Overall, it appears that the hydrothermal wheat flour consistently produces hard pellets, regardless of the moisture content. All the other starches and the control sample decreased in hardness when the MC exceeded 11%. As shown in Fig. 4, Amylose resulted in harder pellets compared to amylopectin, especially at moisture content above 9%. This result agrees with (Builders *et al.* 2014) who stated that if the swelling power is inherently lower, as the swollen granules are prevented in amylose, the tablets get harder. This was not the case for amylopectin, as the granules swelled to a greater extent.

Fig. 4. Pellet Hardness, including standard deviation, *vs*. moisture content, includes pellets produced from pine (no additive) and pine with 5% (db) starch addition

Bonding behavior of the pellets with different starch additives

The FT-IR spectra of the pellet samples, as shown in Fig. 5, provide valuable insights into their chemical composition. Each peak in the spectra corresponds to a specific vibrational mode of a particular molecular bond. The interpretation of these peaks, provided in Table 3, can help identify the functional groups present in the samples.

No	Functional group	Wave number $(cm-1)$					
		[Literature]	No	Amylose	Wheat	Amylopectin	H.t.
			additive		flour		Wheat
							flour
1	$O - H$ stretching	$3600 -$	3337	3335	3335	3336	3333
		3300					
$\overline{2}$	$C - H$ stretching	2931	2857	2853	2855	2852	2859
3	$C = O$ stretching	$1700 -$	1715	1720	1718	1722	1725
		1725					
4	CH ₂ symmetric	1458	1463	1446	1443	1456	1455
	deformation						
5	$C - H$ symmetric	$1385 -$	1365	1362	1360	1363	1361
	bending	1375					
6	$C - O$ stretching	$1200 -$	1028	1026	1022	1024	1027
		1800					
$\overline{7}$	$C - O - C$ asymmetric	1149	1162	1164	1161	1167	1163
	stretching						

Table 3. Peak Assignment of the Pellets at 5% to 14% Moisture Content (wb)

As illustrated in Table 3, all samples displayed essentially the same types of functional groups, albeit with transmittance at slightly varying wavenumbers. This supports the similar peak patterns of the infrared (IR) spectra shown in Fig. 5. Notably, the transmittance peaks at around 3333 to 3337, \sim 2860, 1200 to 1800, \sim 1170 (cm⁻¹) suggest that each pellet sample possessed OH, C-H, C-O, and C-O-C functional groups,

respectively. The presence of the C-O-C group is indicative of starch or starch-containing materials (Abdullah *et al*. 2018). The similarity in peak patterns in Fig. 5a could be attributed to the presence of moisture (5%), possibly because the starch content of the pellets requires a certain amount of water to activate the gelatinization process, which often aids bonding during pelletizing (Anukam *et al.* 2019). This is slightly different from the spectra in Fig. 5b, which displays longer peak lengths, indicating higher concentrations of the aforementioned groups. This could be due to the higher moisture content (14%) of the pellets. The differences were more pronounced in the wheat flour and amylopectincontaining pellets at around 3335, 2860, and 1028 (cm⁻¹). This suggests that the high amount of amylopectin in these starches is more sensitive to water (O-H bonds). This observation supports the hardness test results presented in Fig. 4, where the wheat flour and amylopectin-containing pellets exhibited the lowest hardness compared to the other starches used, except for the control material, which showed lower hardness at 14% MC (wb).

Fig. 5. FT-IR analysis results of all pellet samples at (A) 5, (B) 11 and (C) 14 % (wb) moisture content

Moreover, the sample with the lowest peak, Amylopectin, had the highest F_{max} of all test samples, as seen in Fig. 2. In conclusion, the FT-IR analysis indicates that the starchblended pellets have a somewhat comparable chemical structure.

CONCLUSIONS

- 1. The findings underscore some disparities, particularly between starches predominantly composed of amylose versus those predominantly composed of amylopectin. Specifically, amylose-rich starches yielded harder pellets compared to amylopectinrich ones, particularly when the moisture content exceeded 8%. Notably, the inclusion of starch as an additive markedly enhanced pellet hardness compared to pellets produced without additives.
- 2. It is evident that compression work (*W*comp) was influenced by moisture content (MC) regardless of starch utilization, with *W*comp decreasing as MC increases during the Single Pellet Press production. The Fourier Transform Infrared (FT-IR) analysis confirmed the necessity of water to activate starches during pellet production.
- 3. In summary, while the study initially hypothesized that either amylose or amylopectin would offer superior binding properties for wood pellets, the results suggest that it is the combination of these two components that yields optimal effects. Moreover, hydrothermally treated wheat flour emerged as the most effective starch among those tested, despite the need for costly modifications, although further research is warranted.
- 4. Therefore, the recommendation for pellet manufacturing is to utilize hydrothermally treated wheat flour if starch is to be incorporated, as it consistently produces highquality pellets. However, it is acknowledged that the required modifications come with a cost. Thus, from an economic standpoint, plain wheat flour was shown to be a sufficient and the least expensive alternative, producing high-quality pellets at a lower price point.

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