Title – A novel approach to produce a high purity cellulose product from sawdust waste material

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ABSTRACT

Approximately half of the wood processed in the Forestry, Timber, Pulp and Paper (FTPP) sector is accumulated as waste. The transition towards an almost paperless world driven by the rise of digital media has resulted in a decline in traditional paper markets, prompting the FTTP sector to reposition itself, and expand their product offerings by unlocking the potential of new value adding opportunities from remaining wood waste components to generate revenue. In this study, a novel approach to produce a dissolving wood pulp (DWP) grade from sawdust was developed. DWP is a high purity cellulose product used in several applications such as pharmaceutical, textile, food, paint and coatings industries. The proposed approach demonstrates the potential to eliminate several complex processing stages which are associated with traditional commercial processes to produce high purity cellulose products such as DWP, thereby making the process less chemical, energy and water intensive. Chemical characterisation of the cellulose product thus far has shown properties similar to commercially available products, making the proposed process a promising and viable option for the production of DWP from sawdust, a waste material from wood processing industries.

Keywords: Biomass, dissolving wood pulp, cellulose, chemical treatment

1 INTRODUCTION

The concept of a “green economy” encourages industries to employ revolutionary transformative technologies to eliminate waste generation by exploring the development of new value chains. According to Sithole (2017), the low timber utilization rate of around 47% is a broad challenge faced by the Forestry, Timber, Pulp and Paper (FTPP) sector since approximately 53% of the wood in the form of bark, chips and sawdust goes to waste.

The FTPP sector contributes to about 1% of South Africa’s GDP (Tradingeconomics, 2020) and approximately 4.5% to its manufacturing GDP, making it a vital sector in the economy (Sea, 2020). The diminishing annual demand for forest products can assumingly be attributed to the decline in the demand for wood products such as paper which is no longer required as much today.

Although there are no recent figures available, in 2010 it was estimated that forestry and wood processing in South Africa generates about 4-6 million tons of wood waste per annum, mainly as residues in the plantations and as sawdust and offcuts at the saw mills and pulp mills (Timberwatch, 2010). From all the sawmills in SA, 440 006 tons per annum of sawdust waste was generated from 218 saw mills (Stafford and Lange, 2018). More stringent government legislation will drive industry to consider processes that curtails waste accumulation and favors a more sustainable environment.

The abovementioned scenarios coupled together encourages the urgency for the industry to employ revolutionary change that that will prove to be environmentally superior to existing processes, enabling the industry to continue its legacy in the future.

Dissolving wood pulp (DWP) is a high purity cellulose product that is produced from wood chips (Potgieter, 2018). Such products are valuable for commercial applications for the production of end-user products such as pharmaceuticals, textiles like viscose, and microcrystalline cellulose (Sappi, 2019). Extensive research has been conducted over the years to produce DWP in alignment with pulping mills efforts for technological innovations to fractionate or convert lignocellulosic materials into a wide range of products and by-products for more effective utilisation of renewable feedstocks as opposed to accumulating waste (Lehto and Alen, 2014). Existing processes used to produce DWP from wood chips are time consuming, complex, and energy, water, and chemical intensive (Liu et al., 2016).

The study focused on developing a novel process to produce DWP from sawdust (a waste material) and with more favorable processing conditions which will address the drawbacks of the existing processes. It is anticipated that this will increase revenue by lowering the capital intensity (processing costs and time) whilst still rendering the support of global best practices to reduce waste to landfill and to rather turn waste into wealth, giving the South African producers of DWP a competitive edge.

2 LITERATURE

2.1 Dissolving wood pulp production

The DWP demand has shown a buoyant increase and is the basis of new forest products. The dissolving pulp industry exhibited prospective growth in developing countries in recent years. In 2018, the global revenue generated from DWP amounted to $9.3 Billion, this being a 6.5% increase from the previous year.

Dissolving pulps across the world are produced mainly by two conventional processes; acid sulphite (AS) process and the prehydrolysis kraft (PHK) process (Astuy, 2016, Strunk, 2012, Yang et al., 2018). Each of these chemical pulping processes follow different reaction mechanisms. However, they are analogous to selectively eliminate the lignin without extensive degradation of carbohydrates, and also remove extractives and other chromophoric structures in residual lignin.Both processes yield different qualities of dissolving wood pulp which then determines its applicability in other industries (Christoffersson, 2005, Jahan et al., 2008).

The chemical composition of the raw material is a predetermining factor and should be considered for production of dissolving pulp when selecting an appropriate pulping process (Chen et al., 2016, Shahzad, 2012, Jahan et al., 2008, Jardim et al., 2018, Li, 2016).According to Li (2016), wood still remains one of the widely used raw materials for dissolving pulp production . The principle of chemical pulp processing targets two main areas, namely removal of hemicelluloses and lignin. The former usually is carried out by some sort of pre-treatment process to target the lignin-hemicellulose matrix and improve the accessibility of cellulose, whilst the latter usually occurs during pulping/cooking (Othman, 2015). The primary aim of the pre-treatment stage is to disrupt the rigid crystalline structure of cellulose, and increase its structural accessibility and chemical reactivity (Ocwelwang, 2017). Lignin removal is important because it imparts a yellowish brown colour to the pulp which is undesirable property of the dissolving pulp since a high level of brightness and cellulose purity is important in the production of cellulose based products such as viscose and acetate fibres (Bodhlyera et al., 2015). The extracted waste liquor with higher concentration of impurities such as lignin and hemicelluloses and higher temperature is sent to the chemical recovery station for recovering the organic or inorganic matter in the waste liquid (acid and alkali recovery process).

Chemical treatments are applied to pulps to make it a suitable candidate for high purity cellulose derived end-user products. The resultant pulp emanating from either of the pulping processes requires further delignification to render it viable to produce a high purity dissolving wood pulp. Bodhlyera et al. (2015) mentions that the most common process employed by industry responsible for producing a dissolving wood pulp of such a standard, irrespective of the pulping process used, is the bleaching process (Bodhlyera et al., 2015). The bleaching process plays a vital role and assists in obtaining a quality pulp with certain whiteness, cleanliness, purity and excellent physical and chemical characteristics thus increasing its application potential.

The PHK process leads as the desired process, accounting for approximately 56% of the world DWP produced whilst the AS process accounts for 42% (Chen et al., 2016, Strunk, 2012, Bi et al., 2021, Rodrigues et al., 2018). The PHK process follows a combined process of pre-treatment (pre-hydrolysis) and alkali kraft cooking (Chen et al., 2016). The hemicelluloses are removed during the initial prehydrolysis stage which is followed by kraft cooking and a multi-stage bleaching process to achieve a high purity dissolving pulp. During prehydrolysis, a significant amount of the hemicelluloses and a small amount of the cellulose are hydrolysed into short chains.

2.2 Dissolving wood pulp (DWP) characterization and its end usage

Dissolving pulp is characterized by high cellulose content, high brightness and low macromolecular polydispersity (PDI) (Chen et al., 2019). DWP contains a high alpha cellulose content (> 90%), a low hemicellulose content (3-6%) and trace amounts of lignin and other impurities (<0.05%) (Dladla, 2018, Chen et al., 2016). The viscosity of the pulp subsequent to the cooking process is often about or below 1100 ml/g, and between 400-600 ml/g post final bleaching (Wennerstrom and Bylund, 2017). According to Jesus et al. (2013), cellulosic pulps should satisfy the requisites of high α-cellulose content (≥ 91%), Kappa number less than 1, intrinsic viscosity (450-550 ml/g), a low amount of hemicelluloses and extractive compounds, and finally a high reactivity (> 60%), to be considered as dissolving pulps.

During dissolving pulp manufacturing, the controlled variable is the degree of polymerization (DP) of the cellulose because it gives an indication of the average length of the polymer chains (Shahzad, 2012). Dissolving pulp is the main feedstock for manufacturing regenerated cellulose such as viscose, lyocell and cellulose derivatives. DWP undergoes a variety of manufacturing processes that are used for the production of various cellulose based end-products (Ocwelwang, 2017, Schwaiger, 2019, Alam and Christopher, 2017). These manufacturing processes are classified according to the different alpha grades used to produce these cellulose derivatives. The versatility of dissolving pulp in a variety of economic and technical areas is due to its functionality, recyclability, and, most importantly, its biodegradability (Małachowska et al., 2020, Schwaiger, 2019) . The end user products of dissolving pulp are widely used in industrial products such as textiles, tyres, coatings, paints, tobacco products and food, as well as pharmaceutical products (Liu et al., 2016). Other applications include rheological modifiers in products such as lipstick, fillers in fat-free yoghurt, tablets and washing powders and micro crystalline cellulose (MCC) which is used as a binder in pharmaceuticals and as a thickener in food (Sappi, 2019).

Removing lignin and hemicellulose yields high purity DWP (91–98% cellulose) (Sappi, 2019). Lower grade dissolving pulps are believed to exhibit a cellulose content of approximately 90% whilst the medium grades possess a cellulose content of about 94%. The highest grade is said to have a cellulose content of approximately 96% or more (Liu et al., 2016). Chunilall (2009) suggests that 90%, 92 -94% and 96% α-cellulose are respectively utilised for the production of microcrystalline cellulose (MCC), viscose, and cellulose acetate.

3 EXPERIMENTAL PROCEDURE

3.1 Sawdust characterization

Two different species of wood was selected for evaluation; Pin chips (from the wood chip screening process) was obtained from a local pulp mill. The wood processed by the mill is a mixture of 53.02% Eucalyptus dunnii, 37.80% Eucalyptus grandis and 10.15% clone timber). The pin chips were milled into a finer sawdust particle using a hammer mill followed by a wiley mill. The second sample was a softwood (Pinus Patula) sawdust sample collected from a local saw mill. A shaker with several screens ranging in sizes from 0 to 1180 µm (0, 53, 150, 250, 425, 530, 710, 1180 µm) was used to determine the particle size distribution of the sawdust samples. 100 g aliquots of sawdust samples were screened for 30 minutes in duplicate. The preparation process is shown in Figure 1. The sawdust remaining on each screen was weighed. The saw mill sawdust samples were screened to a particle size of below 1180 µm. The starting wood materials and pulps were chemically analysed using the Technical Association of the Pulp and Paper Industry (TAPPI) standard methods.

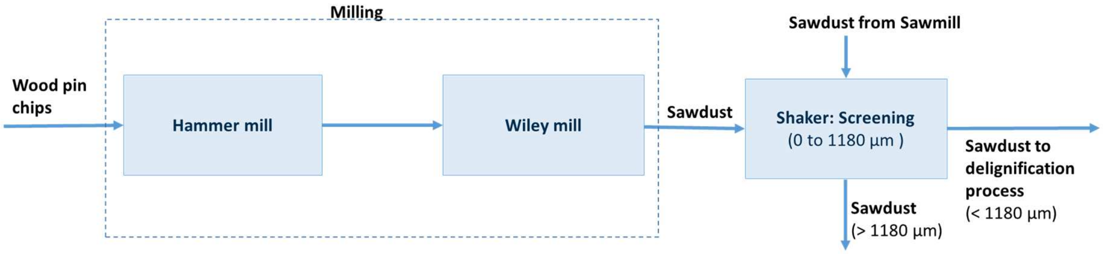
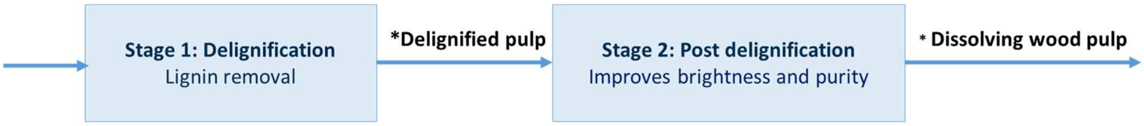


Figure 1: Process flow diagram of raw material preparation process

3.2 Dissolving pulp experiments

The screened sawdust samples were subjected to a two-stage non-disclosed process (Figure 2). The first stage involved delignifying the sawdust according to the conditions listed in Table 1. The sawdust samples were weighed according to the liquid to wood ratio (L: W) and the respective dosage of chemical was added in the form of a slurry to the sawdust. The mixture was then stirred for the desired time and temperature. Experiments were conducted in triplicate for each condition specified. The pulps from each step were washed under vacuum filtration following the experiment and left to air dry. Pulp yield was determined using the standard oven moisture technique (TAPPI test method T550-D) and the pulp was characterised in terms of intrinsic viscosity (SCAN method SCAN-CM 15), degree of polymerisation (SCAN method SCAN-CM 15), ISO brightness (ISO 2470) and hemicellulose content via high performance liquid chromatography (HPLC) (TAPPI test method no. T249 cm-85).



*** Characterization of pulps stage 1 & 2:** Viscosity, DOP, brightness, alpha-cellulose, R18 &R10, hemicelluloses (HPLC), ash, lignin (AIL, ASL), pentosans, DCM extractives, calcium and iron (ppm)

Figure 2: Two-stage process pathway for dissolving wood pulp production from sawdust.

Table 1: Summary of processing conditions optimised.

Process step	Parameter	Conditions
Delignification process	Temperature	60-90°C; Δ10°C
	Time	3-6 h
	Chemical concentration	1, 2 and 3M
	Liquid to wood ratio (L: W)	5:1, 7.5:1, 10:1

4 RESULTS AND DISCUSSION

4.1 Sawdust characterization

The particle size distribution of the two sawdust samples are shown in Figure 3. The softwood sawdust sample showed a high composition of particles greater than 1180 μm. The fraction above 1180 μm was removed as it was not compatible with the delignification process due to its low surface area, which resulted in unreacted sawdust particles.

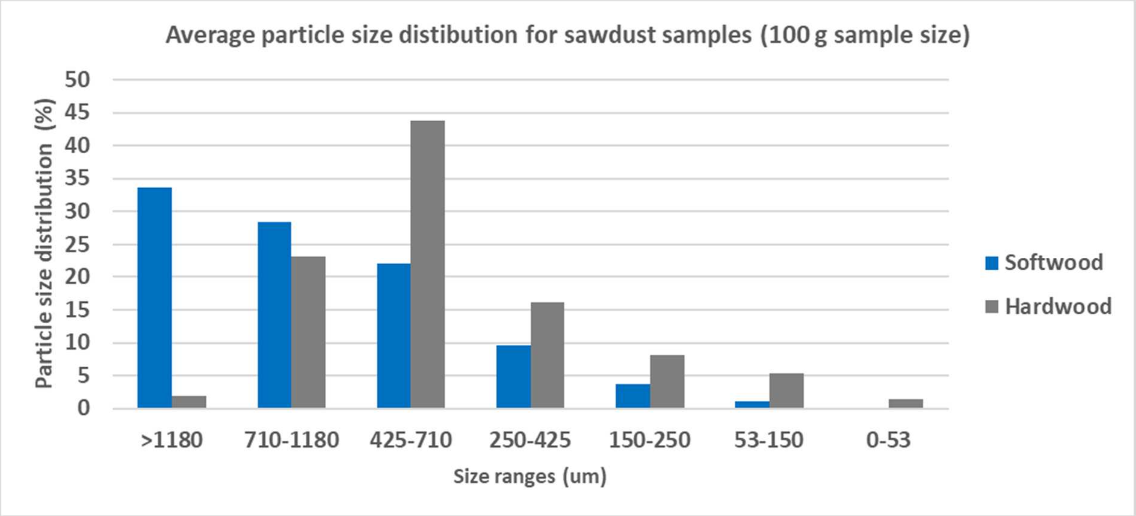


Figure 3: Particle size distribution (PSD) of sawdust species

Each of the sawdust species was chemically characterized (Table 2) to identify the composition prior to treatment. The hardwood sample differed slightly from the softwood species for most of the components. The cellulose and lignin content from all species corresponds to the ranges specified in literature (Cellulose of 40-50% and lignin 15-35%) (Swst, 2017).

Table 2: Chemical composition of different sawdust species used (Dry wood basis)

Analysis	Hardwood sawdust	Softwood sawdust
Solvent extractives (%)	0.40	0.29
Hot water extractives (%)	3.27	1.96
AIL (%)	31.74	30.83
ASL (%)	4.49	2.67
Siefert Cellulose (%)	44.08	41.59
Ash content (%)	0.70	0.17
Monosaccharides		
Arabinose (%)	0.66	0.95
Galactose (%)	0.86	1.94
Xylose (%)	13.07	6.05
Glucose (%)	39.88	42.94
Mannose (%)	0.95	3.09

In terms of the monosaccharide compositions, higher compositions of xylose was present in the hardwood sample as compared to the softwood sample and higher compositions of mannose and galactose was observed in the softwood as compared to the hardwood sample which corresponds to literature (Gladyshko, 2011). The ash composition for the hardwood sawdust also was relatively higher compared to the softwood. According to literature hardwoods contain a high proportion of xylose units and by contrast, softwoods have a high proportion of mannose units and more galactose units (Gladyshko, 2011). This is evident in the results comparing the chemical compositions of the species.

Furthermore, the hardwood sample shows a slightly higher cellulose composition in comparison to the softwood species.

4.2 Dissolving pulp experiments

The two-stage delignification and pulp purification processes showed that a temperature beyond 60 °C with chemical concentrations of 3 M caused effervescence of the product and thus delignification was not achieved. At lower treatment concentrations (1 and 2 M) for temperatures above 60 °C, the conditions were still not effective enough to effect sufficient delignification, leaving a large proportion of unpulped sawdust even up to periods of 6 hours. Optimum conditions were found to be 3 M and 60 °C and L: W (10:1) for the softwood sample and 3 M, 70 °C and L:W (10:1) for the hardwood sample in about 6 hours, where a fully pulped product was achieved with minimal to no rejects. A second set of tests was then done to optimize the reaction time for delignification of each species using the two abovementioned sets of conditions. For the hardwood sample at 70 °C, delignification was achieved in 3 hours. Optimum conditions were established for each species in terms of concentration, temperature and time. A yield from 44-60% and 53% was achieved for sawdust species respectively. The yield obtained was similar to unbleached AS DWP (44.9-45.9%) and slightly higher than unbleached PHK DWP (35-40%) (Dyunyasheva, 2017). PHK DWP is expected to show lower yields due to the prehydrolysis step. Figure 4 shows the transition of sawdust mixture to a pulp as time elapsed during the delignification process with the final stage showing a washed air-dried pulp.

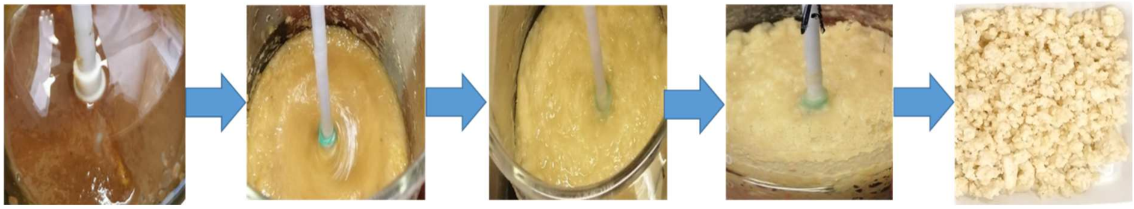


Figure 4: Transition of sawdust to pulp during delignification process.

The pulps were characterized and the results are summarized in Table 3. Both sawdust pulp samples derived from stage 1 showed a low intrinsic viscosity and corresponding degree of polymerization (DOP), between 110-304 mL/g and 283-934 respectively. The low viscosity and DOP were attributed to the possibility of cellulose degradation due to reduced polymer chains as mentioned in literature (Shahzad, 2012). The results suggest that the dissolving pulps achieved to date, without further chemical treatment, would be suitable to target an application that requires a low viscosity pulp such as MCC which requires a DOP < 400 corresponding to a viscosity of 148.03 mL/g. Brightness measurements ranged between 65-66.69% overall. Majority of literature reports on final bleached DWP brightness >85%. However, one source makes mention of unbleached PHK DWP 33.6-42,9%, highlighting that the proposed study pulps have advantage in this aspect and will require minimal improvement in brightness during the subsequent post delignification stage (Sixta, 2006). Lignin concentrations (AIL and ASL) showed significant decreases compared to the original sawdust samples (70-80% reduction), indicating that the delignification process was efficiently removing lignin. Hemicellulose content overall following stage 1 was minimal which suggests that the hemicellulose was also degraded in the stage 1 delignification process. A low hemicellulose content is a key requirement for DWP grades. However, considering the initial raw material hemicellulose composition, separate studies will be undertaken to evaluate the pre-extraction of the hemicelluloses prior to stage 1 delignification for possible beneficiation of the hemicelluloses. Previous studies have shown that removal of hemicellulose prior to the process may aid in delignification, thus requiring milder delignification conditions (Miao et al., 2014, Christopher, 2017, Liu et al., 2013, Koradiyaa et al., 2016) .

Table 3: Results of chemical characterisation of pulps from each sawdust species using optimum conditions

Measurement	**Hardwood sample**	**Softwood sample**
Yield (%)	44-60	53
Intrinsic viscosity (ml/g)	110-180	304
Degree of polymerization (DOP)	283-505.35	934
ASL (%)	1.17-1.53	1.29
AIL (%)	4.27-4.89	5.13
ISO brightness (%)	65-66.69	66
	Monosaccharides	
Glucose	74.59-80.37	90.14
Mannose	0.00	6.74
Arabinose	0.00	0.00
Xylose	2.63-5.15	1.04
Galactose	0.00	0.00

5 CONCLUSIONS

The proposed process has shown significant evidence in consistently producing a low viscosity and high brightness dissolving pulp grade cellulose product by the delignification of sawdust. Optimum conditions were established for pulps with little to no rejects for hardwood and softwood species. Concerns remain around cellulose degradation. However, the current product shows potential as feedstocks for end-user applications such as MCC which can service the pharmaceutical and possibly the food industry at large. Other end-user products such as viscose production is also been researched and considered in the pipeline.

In addition, efforts to extract more value from the sawdust material by applying pre-treatment methods such as prehydrolysis prior to delignification is underway. This will expand the value chain of the biomaterial. The process shows novelty and promising energy and water savings, and in turn, reduced processing costs by achieving a dissolving pulp grade cellulose product using moderate temperatures and reduced times in comparison to conventional processes. The next stages in the project entail developing a proof of concept of end-user products followed by a pilot scale up of the process as well as a techno-economic study.

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