

PRODUCTION OF HIGH VALUE DISSOLVING WOOD PULPS FROM SAWDUST WASTE MATERIAL

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KEY FINDINGS

Dissolving wood pulp (DWP) is a high purity cellulose product used as precursor for production of cellulose acetate, cellulose ethers, microcrystalline cellulose (MCC), viscose and lyocell fibres. Typically, DWP is produced from wood chips using high energy, water, and chemical intensive processes such as pulping and bleaching. In this study, a novel low temperature process (60 °C) was developed to produce a DWP grade from both hardwood and softwood sawdust originating from a sawmill, and pin chips originating from a chemical pulp mill that processes a mixture of hardwood species. Based on the properties of the sawdust pulps, they were then converted to MCC, a commodity widely used in everyday products in the pharmaceutical, food, cosmetics, paint and coatings sectors. Overall MCC yields ranged between 32-42% based on the starting sawdust used. The degree of polymerization for the synthesized MCC ranged between 257-398. Fourier transform infrared (FTIR) analysis of the synthesized MCC compared well to commercially available MCC. ISO brightness of the synthesized MCC ranged between 79-86%, whilst the crystallinity indices ranged between 76-84%. In addition, the molecular weight of the synthesized MCC was found to be comparable to commercial MCC at 339 g/mol. In the context of the biorefinery concept, the study contributes to efforts to transition from a linear to a circular economic model, and beneficiation of waste streams from biomass processing industries such as sawmills and pulp mills may lead to opportunities to diversify product offering, increase revenue streams, decrease operating expenses such as waste disposal, divert waste from landfill and overall improve both the financial and environmental sustainability of these industries.

INTRODUCTION

In South Africa, although deforestation is not of concern, approximately half of the wood processed in the Forestry, Timber, Pulp, and Paper (FTTP) sector goes to waste (Herbst, 2013, Phillips, 2017). Waste biomass generated from sawmills includes tree bark, sawdust, and wood shavings. According to Stafford and Lange (2018), approximately 440 000 tons per annum of sawdust waste is generated by approximately 218 sawmills in South Africa, posing a significant challenge for waste disposal. Disposal of sawdust by landfilling is not a sustainable option due to additional costs associated with transporting waste to landfill, maintaining landfilling sites, and opening new landfill sites. In addition, impending regulations may prohibit the disposal of organic waste in landfills. The idea

surrounding utilization of materials commonly viewed as waste becoming a resourceful commodity has emerged in the field of sustainable development. Biomass waste can be classified as renewable material resources and as secondary material resources. The necessity for cleaner production technologies, as well as the need to hamper rapid global deforestation has stimulated renewed interest in waste beneficiation (Andritz, 2020). Moreover, the overall decline in the FTTP sector due to the decrease in the demand for traditional wood, pulp, and paper products, and other challenges facing the industry such as energy and water shortages, have forced the industry to explore alternative revenue-generating opportunities to sustain the industry.

Sawdust, an abundant lignocellulosic biomass, is readily available as a by-product from wood processing industries, including sawmills and pulp mills. The production of high value products such as dissolving wood pulp (DWP) from sawdust waste offers several benefits. Firstly, it provides a sustainable solution for the disposal of sawdust waste, which is typically burned or discarded in landfills. Secondly, it reduces the demand for virgin wood pulp, which helps to conserve natural resources. Finally, it offers a cost-effective alternative to traditional production methods, which can be expensive and resource-intensive (time, energy, water, and chemicals).

In this study, DWP was produced from hardwood and softwood sawdust using a low temperature process. Based on the properties of the resulting sawdust pulps, they were converted to microcrystalline cellulose (MCC), a high purity and partially depolymerised form of cellulose. MCC is widely used in everyday products such as food, pharmaceuticals, cosmetics, paints and coatings. Conventional methods of producing MCC rely on dissolving pulps derived from various raw material sources such as wood, cotton, bagasse, and other plant materials, with wood being the preferred choice. However, the high production costs associated with producing MCC from wood poses a significant challenge. Moreover, the dependence on plant-based materials for raw material resources threatens sustainability, and particularly expensive and limited raw material resources like cotton, impose constraints on meeting the potential demands and production of MCC.

The project addresses some of the challenges associated with transitioning to a green economy and is closely aligned to several National Government initiatives such as the Bio-Economy Strategy, the Industrial Policy Action Plan, and Forestry Masterplan. The project also addresses Priority 3 in the action plan of the National Strategy for Sustainable Development that calls for optimisation of biomass processing industries whilst ensuring economic growth with reduced environmental impact, i.e. “...resource efficient, low carbon, and pro-employment growth path.” Overall, the study seeks to contribute to the development of a circular economy model while meeting the increasing demand for sustainable materials.

METHODOLOGY

Hardwood (HW) and softwood (SW) sawdust was obtained from a local sawmill and sieved to particle size <1180 µm. Mixed hardwood (MHW) pin chips were obtained from a local chemical pulp mill and reduced to sawdust using a Wiley mill. Delignification of the

sawdust was carried out using a novel approach at low temperature (60°C), followed by bleaching to produce a high purity, high brightness cellulose pulp. Chemical characterization was conducted after each processing stage, facilitating the evaluation and monitoring of important pulp quality indicators such as degree of polymerization (DP), crystallinity index (CrI), ISO brightness, and hemicelluloses and lignin contents. Due to the relatively low cellulose DP, further processing of the bleached sawdust pulps to viscose was not possible, and the focus shifted to production of microcrystalline cellulose (MCC). All processing stages of delignification, bleaching, and acid hydrolysis were optimized at laboratory scale using design of experiments (DoE) techniques before scaling up to pilot scale using a 32 L bioreactor. Characterization of the synthesized MCC included carbohydrate content determination by high-performance liquid chromatography (HPLC), thermogravimetric analysis (TGA), functional group analysis by Fourier transform infrared (FTIR) spectroscopy, cellulose crystallinity by X-ray diffraction (XRD) analysis, and molecular weight analysis using liquid chromatography-mass spectroscopy (LC-MS).

MAIN RESULTS

Optimum conditions were identified for delignification of the sawdust to produce unbleached pulps. These were: 60-70 °C and 3-6 h for the three sawdust samples using a novel chemical process (not disclosed). These conditions were significantly milder than current industry processes where temperatures can reach up to 180 °C during the pulping stage. Purification of the pulps were achieved using conventional industry bleaching processes, whilst optimum acid hydrolysis conditions for synthesizing MCC were 4 M HCl at 80 °C for 5, 60, 120 minutes for the MHW, HW, and SW bleached sawdust pulps, respectively (Figure 1). As seen in Table 1, the synthesized MCC exhibited a DP <400, high brightness (79-86%), and low hemicelluloses and lignin content. These results compared well to commercial MCC available off the shelf and MCC produced in the laboratory using industrial 91α DWP.

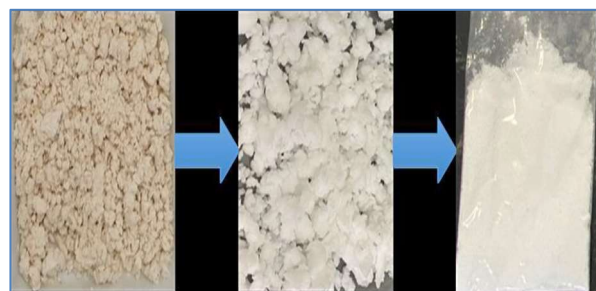


Figure 1: transition showing unbleached sawdust pulp, bleached sawdust pulp and synthesized MCC (spray dried).

Table 1: Summary of properties of synthesized MCC and comparison to commercial MCC.

	Commercial MCC	91α MCC*	HW	SW	MHW
Acid hydrolysis reaction time (mins)**	-	120	60	120	5
Yield (%)	-	-	42	41	30
pH	7.35	7.23	6.72	6.75	5.08
Molecular weight (g/mol)	339	339	339	339	339
Particle size (µm)	4-100	7-1100	4-1100	5-1100	9-1100
Crystallinity Index (%)	89	87	84	84	76
Loss on Drying (%)	4.5	7.90	10	23	15
Viscosity (ml/g)	142	129	131	147	102
Degree of Polymerisation	383	343	347	398	257
Lignin (%)	0.30	0.67	0.28	0.51	0.32
Monosaccharides**					
Arabinose (%)	nd	nd	nd	nd	nd
Galactose (%)	nd	nd	nd	nd	nd
Xylose (%)	2.10	nd	nd	nd	nd
Mannose (%)	nd	nd	nd	1.83	nd
Glucose (%)	95.7	97.5	87.5	83.6	78.1

*91α MCC produced in the laboratory using commercial 91α DWP; **Acid hydrolysis using 4 M HCl at 80 °C; **nd - not detected (less than detection limits)

FTIR analysis (Figure 2) showed good correlation of the synthesized MCC with commercial MCC.

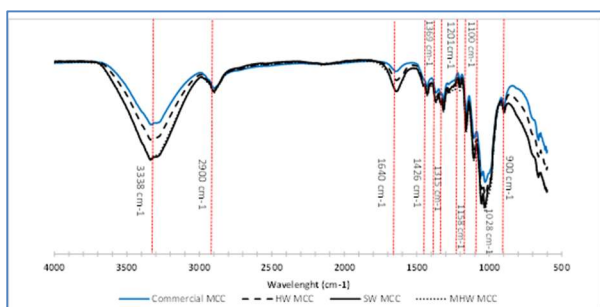


Figure 2: FTIR analysis of synthesized MCC and comparison to commercial MCC.

TGA analysis (Figure 3) showed that after the initial loss of moisture during the early slow pyrolysis stage at 100 °C, the MCC remained unchanged up until 250 °C, at which point fast pyrolysis stage commenced. The onset decomposition temperature of the synthesized MCC compared well with the commercial MCC, and DTG analysis showed that the maximum decomposition temperature of the synthesized MCC also compared well with commercial MCC (325 °C). The decomposition temperature of the synthesized MCC derived from the softwood sawdust was marginally higher at around 350 °C.

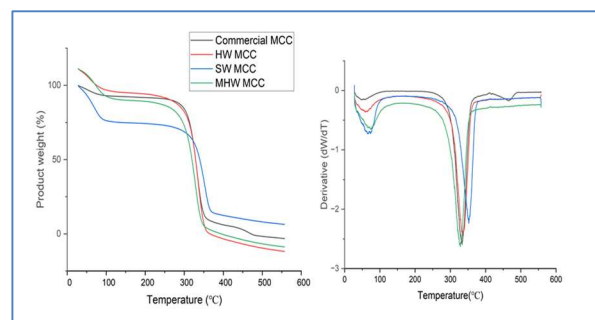


Figure 3: TGA analysis of synthesized MCC and comparison to commercial MCC.

XRD analysis of the synthesized MCC (Figure 4) showed diffractograms that are typical for cellulose I, exhibiting 3 crystalline peaks at diffraction angles of $2\theta = 18^\circ$, 22° and 35° . The crystallinity index (CrI) calculated using the Segal method (Segal et al., 1959) showed values of 76% for MHW and 84% for HW and SW-derived MCC, with the latter closely matching the CrI of the commercial MCC (89%) and MCC derived from 91α DWP (87%).

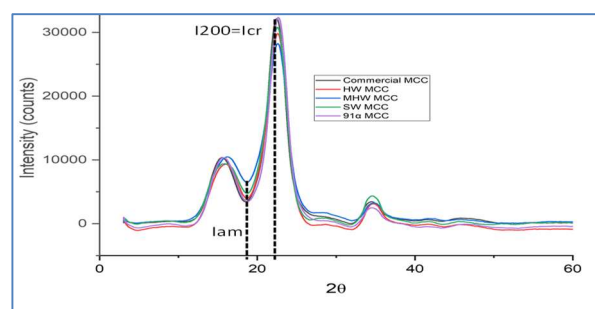


Figure 4: XRD analysis of synthesized MCC and comparison to commercial MCC.

LC-MS analysis of the synthesized MCC showed common peaks appearing at approximately 112, 311, 325, and 339 m/z with the largest peak representing the M+ ion at approximately 339 m/z, therefore corresponding to a molecular weight of approximately 339 g/mol. This is in close proximity with commercial grades which have a molecular weight of 325 and 342 g/mol (BOC-Sciences, 2023, ChemicalBook, 2023).

CONCLUSION AND RECOMMENDATIONS

A novel approach to produce a DWP grade from sawdust was developed, using significantly milder processing conditions compared to industry processes such as pulping and bleaching. Chemical characterisation of the bleached sawdust pulps alluded to its potential as a precursor for production of MCC. The chemical properties of the synthesized MCC compared well to commercial MCC and MCC produced in the lab using commercial 91α DWP. Given that there is a wide range of diverse grades of MCC in the market,

depending on the targeted end-user application, it may be necessary to fine tune properties such as DP, brightness, and particle size to meet specifications.

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Acknowledgments: The authors acknowledge the funding received from the Department of Science and Innovation under the Waste RDI Roadmap.

Disclaimer: The content and views included in this Briefing Note are based on independent analysis and do not necessarily reflect the position of the Department of Science and Innovation or the CSIR.

This Briefing Note is produced as part of the Waste RDI Roadmap Briefing Note Series, an initiative of the Department of Science and Innovation managed by the CSIR. The Note stems from the findings of a grant project funded under the Roadmap, entitled “Production of dissolving wood pulp (DWP) using sawdust waste”.

