Integrating a Biorefinery into an Operating Kraft Mill

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Kraft pulp and paper mills have several advantages for serving the emerging biorefinery industry as a source of raw materials. This review examines technologies for producing liquid biofuels, chemicals, and advanced materials from woody feedstocks to generate new sources of revenue. Market pull comes in part from government policies that drive substitution of petroleum-based products with biobased equivalents. Kraft mills have ample networks to supply feedstocks, whether these are forest residues or byproduct side streams. Pulp mills are well suited to expand sufficiently to accommodate production of value added platform chemicals that are in demand because of brand owner sustainability commitments.

Keywords: Kraft pulping; Biorefinery; Biofuels; Biochemicals; Pyrolysis; Hydrothermal treatment; Enzymatic hydrolysis; Platform Chemicals

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INTRODUCTION

Integrated biorefineries are potentially attractive options for enhancing the long-term viability of the pulp and paper industry as a balanced source of value-added products. Traditional kraft paper mill biorefineries have long produced methanol, lignin, tall oil fatty acids, rosins, and turpentine as byproducts that go into a wide range of chemical platforms and internal use (Ragauskas *et al.* 2006; Thorp *et al.* 2011; Bruycker *et al.* 2014; Jobes 2015). Government sponsored research and subsidies for biofuels stimulated efforts in the pulp and paper industry to explore a wider range of options for producing chemicals. The scale and maturity of the kraft pulp industry in biomass logistics and operations can be used to leverage a biorefinery industry (Van Heiningen 2006; Amidon and Liu 2009; Zhang *et al.* 2011; Zhu *et al.* 2011; Christopher 2012; Lundberg *et al.* 2013; Wertz and Bédué 2013; Alen 2014; Hakovirta 2014; Lundberg *et al.* 2014).

A growing demand for energy, scarcity of materials, and volatility in commodity markets are seen as opportunities for growth in the pulp and paper industry through a biorefinery strategy (Hämäläinen *et al.* 2011; Pätäri et al. 2011, 2016; Hansen and Coenen 2015; Hansen 2016; Panwar *et al.* 2016). Key challenges come from the risk associated with early stages of development and the need to engage in collaborative efforts across unfamiliar industries. The history of using forest products to create chemical and transportation fuel supply chains has been reviewed by several authors (Luque *et al.* 2008; Manzer *et al.* 2013; Guo *et al.* 2015; Klein-Marcuschamer and Blanch 2015). The last few years have seen a new wave of biorefining (Runge 2013; Vasara 2013).

Several projects have demonstrated paths to value from biomass, creating a need to sort out those where an integrated biorefinery at a kraft mill has some advantages. Value-added products from marginal side streams that replace petroleum-based feedstocks meet the aims and objectives of sustainability and economic development (Bugge *et al.* 2016). Fostering niche markets that arise out of underutilized or damaged forest resources will call for strategies of close collaboration and innovation across the value chain (McCormick and Kautto 2013; Lamers *et al*. 2014; Hansen 2016; Van Lancker *et al.* 2016).

RESEARCH AND COMMERCIAL DEVELOPMENT CENTERS

US and European initiatives focusing on biorefinery platforms support several well-funded centers of research and consortia. Early reviews of the founding years of these research center initiatives can be found in a US Department of Agriculture (USDA) publication (Rudie 2009) and the European Joint Biorefinery Vision for 2030 (Annevelink and van den Oever 2010).

Progress by the three US Department of Energy (DOE) Bioenergy Research Centers over the last 7 years (Joint BioEnergy Institute (JBEI Berkeley), Great Lakes Bioenergy Research Center (GLBRC Madison), and Bioenergy Science Center (BESC Oak Ridge)) has been reviewed recently (Peters 2014; Klein-Marcuschamer and Blanch 2015; Papoutsakis 2015; Slater *et al.* 2015). Key research areas addressed were biomass supply, crop optimization, deconstruction of biomass, and conversion of biomass to fuels. Further research will focus on combining biochemical processes to produce intermediates that can be catalytically upgraded to biofuels and platform chemicals. Fermentation technology is emerging to yield a commercial path to platform chemicals and the biobased polymers (Papoutsakis 2015; Shen *et al.* 2015).

The USDA National Institute of Food and Agriculture (NIFA) invested $156 MM in seven regional advanced biofuel centers across the US (*Sustainable Advanced Biofuels across the United States* 2015). The Northwest Advanced Renewables Alliance (NARA) developed a process for converting wood residues to jet fuel while taking into consideration the sustainability of the entire supply chain (*Northwest Advanced Renewables Alliance (NARA). 3rd cumulative report* 2015). Reviews of biomass conversion projects for advanced biofuel development by the USDA Agricultural Research Service focused on residues and energy crops (Orts and McMahan 2016; Steiner and Buford 2016). A survey by the USDA Forest Service reviewed their research on pretreatment technologies for conversion of wood to ethanol, biomass gasification, and fuel pellet technology (Rudie *et al.* 2016).

BIOREFINERY PLATFORMS

Integrated biorefinery platforms applicable to kraft mills can be broadly divided into two categories: (1) combined heat and power and (2) chemicals and materials (Jansson *et al.* 2015). Products, processes, and feedstocks overlap between these categories. Future kraft mill biorefineries are expected to serve markets in composites that use nanocellulose or lignin-based carbon fiber, synthetic transportation fuels (synfuels), biobased polymers from sugar or syngas carbon, and value-added chemicals.

Biomass gasification to produce a synthetic gas (H_2 + CO syngas) has been used to power lime kilns (Manning and Tran 2015). Fischer-Tropsch catalytic processes have been used to reform syngas into a broad range of hydrocarbons and yield excess heat that can be used to power an integrated pulp mill (Haikonen *et al.* 2011; Ljungstedt *et al.* 2013; Isaksson *et al.* 2014). Cleaned syngas can also be used as a carbon source in fermentation to give platform chemicals and biofuels (Nanda *et al.* 2013; Karatzos 2014; Shen *et al.* 2015).

Chemical platforms fall into several categories: sugars from hemicellulose and cellulose, phenolics from lignin, biogas from waste treatment, tall oil and turpentine byproducts, and pyrolytic liquids and gasses. Pyrolytic gasses and liquids can be formed by either fast pyrolysis in a dry state or hydrothermal liquefaction in water (Amidon *et al.* 2008; Thorp *et al.* 2011; Lundberg *et al.* 2013; Hu *et al.* 2016; Isikgor and Remzi Becer 2015; Taylor *et al.* 2015; Thorp *et al.* 2015; Wikberg *et al.* 2015; Matsakas *et al.* 2016; Wilson and Lee 2016).

Material platforms are based on upgrading hemicellulose, cellulose, and lignin to higher value materials. Xylans have shown oxygen and mineral oil barrier performance in packaging materials (Johansson *et al.* 2012; Mikkonen and Tenkanen 2012; Laine *et al.* 2013; Le Normand *et al.* 2014). Nanocellulose has broad applications in packaging materials for surface smoothness, bending stiffness, and barrier performance (Lavoine *et al.* 2012; Li *et al.* 2015a; Osong *et al.* 2015). Reports have shown active development of new pilot facilities for producing microfibrillated cellulose and nanocrystalline cellulose for exploratory development in the paper industry (Miller 2015; Nelson *et al.* 2016). A recent review covered a wide range of uses for cellulose nanofibrils (CNF) and cellulose nanocrystals (CNC) in advanced material applications such as biosensors, optoelectronics, and organic solar cells (Zhu *et al.* 2016). The optical and mechanical properties of CNF and CNC combined with biodegradability, biocompatibility, and ease of functionalization provide a unique path to sustainable materials. Nanocellulose has broad potential in biomedical applications (Lin and Dufresne 2014). Conversion of lignin to carbon fiber is active area of research (Gellerstedt *et al.* 2010; Strassberger *et al.* 2014; Chatterjee and Saito 2015; Beckham *et al.* 2016).

BIOREFINERIES IN THE PULP AND PAPER INDUSTRY

The pulp and paper industry explored several technologies for producing transportation fuels and sugar feedstocks over the last 10 years with limited success, mostly from upgrading traditional byproducts or focusing on sulfite mill (dissolving pulp) sugars from spent liquor (Magdzinski 2006; Mendell *et al.* 2011; Lai and Bura 2012; Rødsrud *et al.* 2012; Anon. 2014; Rueda *et al.* 2015; Zhu *et al.* 2015; Rueda *et al.* 2016). Production of ethanol from sugars from sulfite liquors in the US was reported at 11,000 gallons per day in 1987 (Zerbe 1991). The Tembec Témiscaming sulfite mill in Canada had a capacity of 15 to 18 million liters of ethanol per year, roughly the equivalent of US production in 1987 (Magdzinski 2006; Williamson 2013).

Early work in kraft mill biorefineries focused on an indirect thermal process that transforms black liquor to gasses that can be recombined to transportation fuels using Fischer-Tropsch (FT) catalysis (Larson *et al.* 2006; Haikonen *et al.* 2011; Ljungstedt *et al.* 2013; Bajpai 2014; Isaksson *et al.* 2014; Mesfun *et al.* 2014; Mesfun and Toffolo 2015) . FT catalysis has been long established in the petroleum industry on a large scale, but only recently has it been considered viable on the small scale of a typical biorefinery through the invention of micro-channel reactors and fluidized bed steam reforming (Connor 2007a,b; LeViness *et al.* 2013). These technologies have been used for non-kraft pulp mills without adequate chemical recovery systems. Micro-channel FT reactors are now in development for Red Rock’s wood chip to jet fuel facility in Oregon (LeViness *et al.* 2014).

Selection of a pulp mill biorefinery design depends on analysis of pathways that factor in the supply chain, process integration technologies, and product and market access (Hytönen and Stuart 2012; Dansereau *et al.* 2014; Cambero *et al.* 2015; Kim and Dale 2015; Kokossis *et al.* 2015). While the drivers for change toward a biorefinery solution in the forest products industry have been recognized, paths to commercial products are not well developed (Hänninen *et al.* 2014; Marinova *et al.* 2014; Novotny and Laestadius 2014; Pätäri *et al.* 2016). There is a large array of potential technical and commercial paths without clear agreement on an optimum (Nanda *et al.* 2013; de Jong *et al.* 2015; Jungmeier *et al.* 2015; Maronese *et al.* 2015; Zhao *et al.* 2015). Analysis of potential forest biorefineries have been done based on SWOT analysis (strengths, weaknesses, opportunities, and threats) for several feedstock and process pathways (Chambost and Stuart 2007; Wagemann 2012; Kretschmer 2014). SWOT analyses are recommended as a method for identifying a path to value by showing unique strengths and the competitive environment (Chambost *et al.* 2007).

A SWOT analysis shown in Table 1 highlights the general benefits and challenges of having a biorefinery integrated with a kraft pulp mill. Integration offers a cost saving in shared energy resources, biomass supply lines, creating value from underutilized waste streams and manufacturing expertise. A more specific SWOT analysis is needed for each type of feedstock, process technology, and product combination, producing an array of SWOT matrices that define platforms and success factors (Kretschmer 2014).

Table 1. SWOT Analysis of a Biorefinery Integrated with a Kraft Pulp Mill

Strengths	Weaknesses
● Kraft pulp mill infrastructure and networks exist for utilizing woody biomass on a large scale. ● There is an efficient recovery system for generating heat and power. ● Lignin isolation pathways have been commercialized in integrated pulp mills. ● Markets in the chemical industry for kraft pulping byproducts are well established. ● Fractionation of lignocellulose into hemicellulose, cellulose, and lignin can be done with equipment that is compatible with the scale and environment of a pulp mill. ● Mill residues and side streams can be used for feedstock or energy. ● Higher density of wood and year-round harvest compared with crop residues gives better supply predictability and lower logistics cost.	● Wood sugar and lignin products that qualify for higher margin markets have not been developed at the scale needed to justify investment. ● Most of the development efforts in lignocellulosic biorefineries have been customized for agricultural residues. ● Environmental burden of a biorefinery at a pulp mill may exceed what the site can handle. ● Market entry will require a high cost of capital and uncertain process feasibility, thus requiring extensive demonstration plant experience. ● Production scale may require a large portion of products for biofuels in order to get higher margin and smaller volume intermediates. ● A high energy demand for wood chipping, drying, and grinding to fine particles is needed for efficient conversion to chemicals. ● There will be additional need for waste treatment support. ● There will be increased load on the recovery system.

Opportunities	Threats
● Pyrolysis and hydrothermal liquefaction technologies to produce bio-oils are well suited to utilize the full value of wood without the need for fractionation. ● Biorefinery technology can improve the value and efficiency of biomass utilization from forest lands. ● Biorefineries may be able to convert pulp to materials of higher value when pulp production is needed to balance the mill. ● Integrated biorefinery technology may be used to off-load a recovery system. ● Biobased polymers that use oxygenated intermediates could be more easily obtained from woody biomass than petroleum feedstocks. ● The quality of fiber obtained after removal of some hemicellulose can be matched to suitable new pulp, paper or board grades. ● Societal goals of sustainability can be addressed by using derivatives of wood components. ● The plant can serve several niche markets (bio-based surfactants, adhesives, solvents, oxygen barrier films).	● Potential alternate supply chains may emerge from energy crops for the same products. ● The business case may be more compelling for sourcing low cost forest residues or Municipal Solid Waste (MSW), leaving pulp mills out of the value chain. ● Large investments in second generation biofuels and sugars from crop residues and their slow rate of commercialization may deter further interest in forest product biorefineries. ● There is a need for commercial and market support of a multi-product biorefinery to justify investment. ● The high cost of collecting bulky wet biomass makes forest products less competitive with low cost petroleum as a feedstock for making transportation fuels and platform chemicals.

Several reviews give a detailed view of how biorefineries may be deployed in a kraft mill (Ragauskas *et al.* 2006; Hamaguchi *et al.* 2012, 2013; Hakovirta 2014; Mariano 2015). Detailed descriptions of biorefinery plans and early commercial development by location and technology are available (Pytlar 2010; Mendell *et al.* 2011; *Cellulosic Biofuels - Industry Progress Report 2012-2013* 2012; Tuuttila and Joelsson 2012; Bacovsky *et al.* 2013; Bergström and Matisons 2014; Laqua 2015). An updated summary of current biorefinery activities in the forest products industry is provided in Table 2. Dissolving pulp mills are not included.

Table 2. Biorefinery Efforts in the Chemical Pulping and Recycled Fiber Industry

Company	Partner(s)	Technology	Products	Integration
UPM		Hydrotreating and distillation of crude tall oil	BioVerno renewable diesel	Kaukas mill site in Lappeenranta, Finland
UPM	Valmet, Fortum, VTT	Pyrolysis of wood	Bio-oil	Fortum heat and power plant in Finland

UPM	Renmatix	Plantrose supercritical water hydrolysis of wood residues	Wood sugars and clean lignin	
UPM	ValChem consortium	SEKAB's CelluAPP® technology for wood to sugars and Metabolic Explorer's sugar to MPG	bio-MPG (mono propylene glycol) for polyester resins	
Stora Enso		Microfibrillated cellulose	Lightweighting liquid packaging	
Stora Enso	Virdia (acquired)	Lignocellulose to sugars by acid hydrolysis	Xylose from bagasse, xylitol sweetener	Raceland, LA sugar plant
Stora Enso, Domtar	Valmet	Lignin separated from black liquor by the Lignoboost process: cool the black liquor, precipitation of lignin by acidification with CO_2 to pH 10, dewatering, re-slurry while conditioning to pH 2-3 with H_2SO_4, more filtration, washing and dewatering to remove ash	Dried lignin as a fuel to replace natural gas use in the mill	Sunila pulp mill, Finland, Plymouth, NC (BioChoice lignin)
Metsa Group		All-side-stream full utilization in an efficient pulp mill	Fertilizers from ash, biogas from sludge, bio-composites and textiles from pulp, biofuels from bark, sulfuric acid, and methanol	1.2 B Eur Äänekoski bioproduct mill to be built in Finland
Cascades	American Process (API)	Hemicellulose extraction of wood chips to replace carbonate pulping using API Greenbox++	Sugars fermented to ethanol, nanocellulose side stream generated and added back to the web to restore strength	Norampac – Cabano corrugating medium plant in Canada

SCA		Black liquor to biofuels and chemicals		Pilot plant at containerboard mill in Obbola, Sweden
Nippon Paper		TEMPO oxidized CNF made from pulp and finely defibrated	Cellulose nanofiber (CNF) production	Ishinomaki Mill in Miyagi, Japan
Fibria	Ensyn	Wood pyrolysis	20 MM gallons per year renewable fuel oil for RIN credits in the US	Pulp mill in Aracruz, Brazil
Nordic Paper	RenFuel	Refined lignin from black liquor to an oil (Lignol), conversion to biodiesel	Bio-diesel and bio-gasoline	Pulp mill in Bäckhammar, Sweden
Canfor	Licella Fibre Fuels	Catalytic hydrothermal conversion of wood fiber waste	Biocrude oil feedstock for direct substitution in a petroleum refinery	Pulp mill in Prince George, BC
West Fraser	FP Innovations, NORAM Engineering	Lignin separation from black liquor by the Lignoforce process: oxidation of reduced sulfides to improve filtration, acidification with CO_2 to coagulate lignin, acidic press washing by flushing through with H_2SO_4, and flash drying	Lignin as a natural adhesive in engineered wood products	Pulp mill in Hinton, Canada
Norske Skog	Arbiom, Deinove	Saccharification and fermentation	Sustainable chemical intermediates produced by *Deinococcus* bacteria	Newsprint mill in Golbey, France

Figure 1 shows some of the multiple options for extracting value from the process flow of a biorefinery integrated with a kraft mill (Hamaguchi *et al.* 2012). The main paths to fuels and chemicals are through wood residue, bark, pins, fines, or hog fuel that can be de-ashed, dried, and ground. Ground wood residues can be converted to bio-oil through pyrolysis or hydrothermal liquefaction. Bio-oil made from sawmill residues by Ensyn's pyrolysis technology has been developed for industrial heating oil applications and partial substitution of petroleum (up to 5%) at a refinery. Ensyn calls this product Renewable Fuel Oil (RFO). Pyrolysis oils made from wood or lignin are beginning to find commercial applications as biofuels (Anon. 2014b). Recent reviews have covered pyrolysis technology development by sponsor, commercialization stage, feedstocks, and type of unit (Meier *et al.* 2013; Nanda *et al.* 2013).

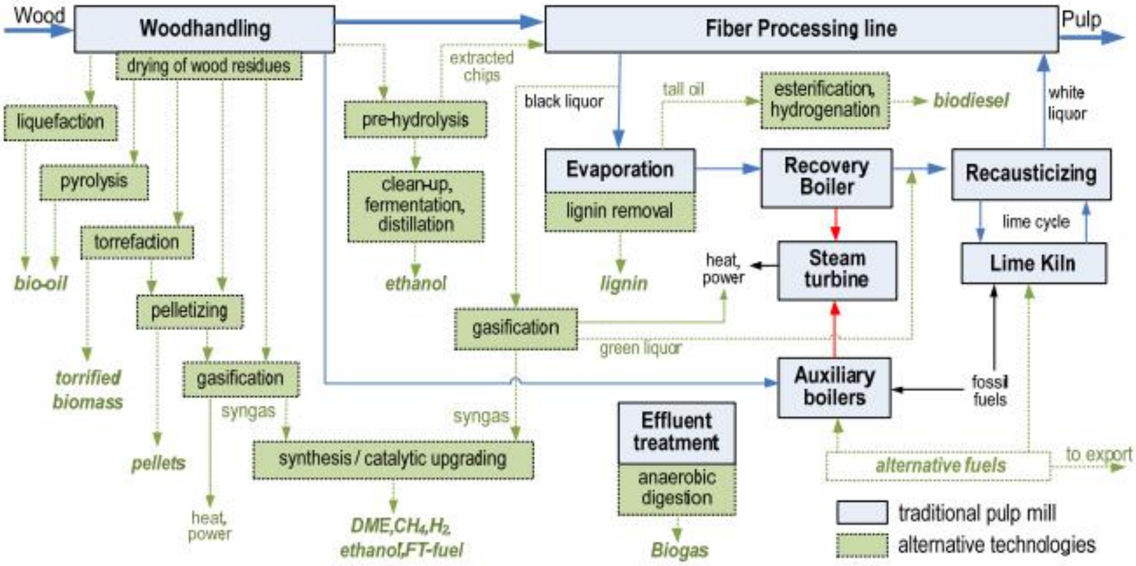

Fig. 1. Process flow of a kraft mill with an integrated biorefinery (source: Hamaguchi *et al.* 2012)

Using high temperature pyrolysis or hydrothermal gasification, wood is converted to a syngas consisting mostly of carbon monoxide and hydrogen (Brown *et al.* 2014; Li *et al.* 2015c). Prior to upgrading, gases are cleaned of sulfur and nitrogen contaminants that could poison the catalyst in the next stage. Catalytic upgrading of syngas through Fischer-Tropsch (FT) or steam reforming can produce a range of biofuels. One of the products, di-methyl ether (DME) can be used in diesel applications (Wetterlund *et al.* 2011; Ljungstedt *et al.* 2013; Isaksson *et al.* 2014; Ail and Dasappa 2016). Red Rock Biofuels is one of three projects selected by the DOE for producing jet and diesel fuels. A plant capable of producing 12 MM gallons per year is in the planning stage using Velocys small scale micro-channelled FT reactor technology (LeViness *et al.* 2013; Becker *et al.* 2015a; Güttel and Turek 2016).

Gasification of black liquor produces a syngas that can be upgraded to diesel fuels. Black liquor gasification has been studied in great detail and implemented at the Weyerhaeuser mill in New Bern, NC (Landälv *et al.* 2010). Challenges in chemical recovery and sulfur poisoning of the catalysts employed have been responsible for abandoning this technology at kraft mills (Landälv *et al.* 2010). Research on black liquor gasification is continuing in Scandinavia (Naqvi *et al.* 2010; Andersson *et al.* 2015, 2016; Jafri *et al.* 2016).

Hot water extraction (HWE) of wood chips has long been considered as a route to hemicellulose that can be converted to useful polymers or hydrolyzed to fermentable C5 sugars. Under a short burst of high pressure and slightly acidic conditions hemicellulose and cellulose can be selectively hydrolyzed out of the lignocellulose complex to produce separate streams of fermentable sugars in two steps (Colakyan 2012; Silveira *et al.* 2015). Extraction of lignocellulose with sub-critical water has been developed by Renmatix in partnership with UPM and others to yield low-cost sugars and clean lignin.

Autohydrolysis is a process in which hot water extraction can be used to obtain hemicellulose and sugars prior to pulping. Acids removed from hemicellulose and extractive esters catalyze the removal of sugars (Amidon and Liu 2009). Extraction results in wood chips that are better suited for making fluff pulp, specialty papers or dissolving pulp than softwood based kraft pulp for packaging grades due to strength loss (Goyal 2015).

Chemical demand in pulping and bleaching decreases after extraction by autohydrolysis (Chirat *et al.* 2013; Goyal 2015). Increasing hot water extraction of *Eucalyptus* chips measured by a severity factor (*p*) showed that furan degradation products and lignin deposits can accumulate to the point where fermentation of the resulting sugars would not be cost effective (Liu *et al.* 2015). Extraction of *Eucalyptus* chips at low severity factor resulted in removal of the 35% of the hemicellulose and gave handsheets with acceptable paper properties.

Other products such as wood fiber composites, fuel pellets, dissolving pulp, and nanocellulose could be made from hot water extracted wood chips (Hasan *et al.* 2010; Pu *et al.* 2011; Bach and Skreiberg 2016; Zhu and Yadama 2016). Another use for kraft pulp made from hot water extracted chips may be in wood fiber composite pulps such as Södra's DuraPulp that can be used for making formable products (Hasan *et al.* 2010; Melin 2015). Nanocellulose added back to a paperboard furnish made from hot water extracted chips could restore some of the strength properties lost during extraction (Nelson *et al.* 2016).

Utilization of wood sugars to make fermentation products could bring forest products into the mainstream of industrial biotechnology. Surveys of major development efforts in cellulosic biofuels made from 2012 to 2013 by the Advanced Ethanol Council (AEC), the DOE, and others showed more than a dozen significant investments in wood-based sugars and biofuels (*Cellulosic Biofuels - Industry Progress Report 2012 - 2013* 2012; Bacovsky *et al.* 2013; Balan *et al.* 2013; Brown and Brown 2013). Almost all of these companies are now out of business, drifted back to lower cost feedstocks (MSW), or have reverted to early stage R&D.

Pyrolysis-based technology leaders like Ensyn and BTG-Empyro remain with a focus on heating oil made from wood. Recent work has shown that pretreatments and process adjustments can give pyrolysis oils with a high content of levoglucosan, which can be hydrolyzed to glucose, potentially a viable path to wood sugars (Bennett *et al.* 2009; Sukhbaatar *et al.* 2014; Jiang *et al.* 2015, 2016; Wang *et al.* 2016a). Fermentation using sugars from mild sulfite-based pulping technology continues to be developed with potential for repurposed kraft mills to dissolving pulp (Rakkolainen *et al.* 2010; Gao *et al.* 2013; Cheng *et al.* 2015; Zhu *et al.* 2015; Dou *et al.* 2016).

NICHE MARKETS FOR KRAFT MILL BIOREFINERY PRODUCTS

Niche products offer biorefinery operations an opportunity to improve economic feasibility by growing in markets where bio-based feedstocks are valued (Chambost and Stuart 2007; Scott 2016b). Fractionation processes like hot water, cold caustic, solvent, or ionic liquid extractions enable separation of the valuable components of wood (Kenealy *et al.* 2007). Hot water extraction of unbleached birch kraft pulp could separate out enough of the lignin and xylan to allow the pulp to be used in rayon grades (Borrega and Sixta 2013). Use of pH control during hot water extraction can optimize the yield and molecular weight of extracted hemicellulose (Krogell *et al.* 2016). High molecular weight xylans are useful for packaging films that provide oxygen and mineral oil barrier properties (Grondahl and Bindgard 2013). Cold caustic extraction with polyethylene glycol helped preserve the yield, polydispersity, and molecular weight of xylans (Li *et al.* 2015b).

Ionic liquids are typically molten salts at temperatures less than 100 ^{o}C. The salts are generally composed of an organic cation and inorganic anion. Ionic liquids can be tailored to dissolve the components of wood selectively (Xie *et al.* 2009; Li *et al.* 2010). Ionic liquids can be used to produce regenerated cellulose fiber at higher yield and better strength than conventional Lyocell fiber due to less polymer degradation (Sixta *et al.* 2015). Ionic liquid pretreatments proved useful in dissolving hemicellulose to give a higher yield in saccharification (Viell *et al.* 2013). Hemicellulose films obtained from ionic liquid pretreatment gave better oxygen barrier properties than those prepared from cold caustic extraction (Laine *et al.* 2016). Reductive amination of aldehydes obtained from degradation products of lignin and hemicellulose could be used to make tertiary amines that form the cationic component of low cost ionic liquids (Socha *et al.* 2014). Finding effective low-cost ionic liquids and recovering them in high yield are major challenges in commercializing their use in biomass fractionation (Baral and Shah 2016).

Material substitutions of petroleum-based bulk chemicals such as adhesives, solvents, and surfactants can be seen as greener alternatives where sustainable sources have reduced impacts on the environment (Smith *et al.* 2014; Diorazio *et al.* 2016). The Australian company Circa Group developed a process to convert wood waste and other lignocellulosic materials to levoglucosenone. The latter compound then can be hydrogenated to dihydrolevoglucosenone (Cyrene^{TM}), a polar aprotic solvent similar to n-methylpyrrolidone (NMP) and sulfolane (Clark *et al.* 2015; Duncan 2015).

Another trend in green solvent development is a move toward reactions in aqueous media rather than petroleum based solvents. To facilitate these reactions, bio-based surfactants are needed. Effective xylose-based surfactants can be made by glycosylation with long chain alcohols using p-toluenesulfonic acid as a catalyst (Klai *et al.* 2015). Sugar-based surfactants are expected to have a significant role in commercial processes due to their improved biodegradability and reduced ecotoxicity (Rojas *et al.* 2009; Spence *et al.* 2009).

Commercial lignin production for uses other than generation of heat and power represents 2% of the total lignin produced. Commercial lignin is fragmented into several niche markets (Zakzeski *et al.* 2010; Strassberger *et al.* 2014; Upton and Kasko 2016). The largest fraction of lignin sold comes from sulfite mills as lignosulfonate. Borregaard LignoTech, TEMBEC, and Domjso Fabiker are suppliers of sulfite mill lignin. Ingevity, formerly MeadWestvaco’s Specialty Chemicals division, has long been a supplier of sulfonated and unmodified kraft lignin under the trade name INDULIN^{TM} (Holliday *et al.* 2007; Lake and Blackburn 2014). Newer sources of kraft lignin coming online are from the Lignoboost and LignoForce processes developed by Innventia and FP Innovations, respectively (Table 2) (Tomani 2010; Kouisni *et al.* 2012; Benali *et al.* 2014).

The Lignoboost process has been implemented at the Domtar mill in NC and the Stora Enso pulp mill in Sunila, Finland. Domtar mill lignin is marketed as BioChoice^{TM}. The LignoForce process is running at the West Fraser pulp mill in Hinton, Canada. Kraft lignins originating from cooking processes to produce low or high kappa pulp are expected to have different properties. Chemical differences in pine lignin between a bleachable-grade pulp (BioChoice) and a high kappa linerboard-grade pulp (INDULIN) have been analyzed (Hu *et al.* 2016). These two sources of lignin are similar, with slightly higher phenolic hydroxyl, enol ether, and stilbene contents in the BioChoice sample.

Kraft lignins have been extensively studied for commercial applications. Examples include renewable aromatics, substitutes for phenol formaldehyde in particle board, asphalt emulsifiers, carbon fiber, reinforced polyurethanes, activated carbon, and cement dispersions (Suhas *et al.* 2007; Homma *et al.* 2010; Li and Ragauskas 2012; Aso *et al.* 2013; Zhao and Yan 2014; Graglia *et al.* 2015; Taverna *et al.* 2015; Sadeghifar *et al.* 2016). In the future, lignin will serve as a feedstock for a wide array of advanced materials (Kai *et al.* 2016; Upton and Kasko 2016).

FEEDSTOCK AVAILABILITY

The Billion Ton study was published by the DOE and USDA in 2005 and updated in 2011 and 2016. The study examined the feasibility of displacing 30% of the US consumption of petroleum by conversion of sustainable and affordable biomass (ORNL 2011). More than one billion tons of biomass would be commercially used to reach this goal, primarily by using agricultural residues, forest resources, energy crops, and municipal solid waste. This is a significant move away from currently available biofuels, which are dependent on food crops such as corn, sugarcane, and vegetable oils. Ultimately, dedicated energy crops consisting of grasses and short rotation woody plants such as willow and poplar will have a major role once these sources have been developed. The feasibility of utilizing resources at this scale is largely dependent on local availability, cost, and prospects for supply over decades (Kim and Dale 2015).

Mills producing lumber, plywood, and pulp generate about 87 million tons of lignocellulosic residues in the form of bark, sawdust, fines, and shavings. Most of these residues are utilized in fiber products, combusted for energy, or as used as mulch. Under-utilized material from primary and secondary mill systems that could be diverted to petroleum replacements are estimated at 7 million tons in the US (ORNL 2011). This does not include black liquor, which is needed to recover pulping chemicals and provide energy for the mill. Outside the mill system, logging residues are expected to contribute 4 to 12 million tons and urban wood waste is expected to be 12 to 20 million tons at less than $60/ton.

Overall, the widely dispersed nature of these materials and relative low availability, make widespread use for conversion to transportation fuels unlikely. By comparison, conventional wood chips from pulpwood resources are not generally available below $80/ton, making conversion to products that can compete with fossil fuels even less likely (*Quadrennial Technology Review 2015: Biomass Feedstocks and Logistics* 2015). Rather, low-cost crop residues supplying integrated biorefineries in the food industry are expected to serve the bulk of the next generation of biofuels and come from non-food feedstocks.

In an effort to overcome many of the economic challenges from sharing wood resources with pellet, pulp, paper, and sawmills, studies have looked at the economic and environmental impact of forest plantations dedicated to biofuel production (Hall and Jack 2009; Jack and Hall 2010). Minimal impact on food production was predicted using marginal land for new forest plantations. These areas would be too hilly to cultivate and may be suitable only for grazing. Using the assumption that one ton of green wood could be used to produce a biofuel equivalent to 100 liters of petroleum, forest plantations on marginal land could be profitable sources of biofuel when the price of oil exceeds $180 per barrel. This amount is within range of the 2008 peak at $147 per barrel. In the long run, dedication of vast areas of new forest land to biofuel production would need to support multiple products other than biofuel to reduce the risk from fluctuations in the price of oil (Tomei and Helliwell 2016).

THERMAL TREATMENTS

Fractionation of wood or pulp into useful chemicals has been categorized by the types of processes employed, primarily thermal or chemical. In the thermal process, the whole feedstock is liquefied together to produce water-soluble hydroxy acids, phenolics, and insoluble bio-oil containing a wide array of ethers and alcohols. The main thermal processes are fast pyrolysis in the absence of oxygen or hydrothermal liquefaction in hot compressed water at sub-critical conditions.

Pyrolysis is non-specific and gives a complex mixture of products that can be used as fuels or further purified to value added chemicals. Use of catalysts, acid pre-treatments, and a hydrogen atmosphere help improve the value of the product mix as fuels (Pittman and Steele 2006; Meier *et al.* 2013; Brown *et al.* 2014; Onarheim *et al.* 2015; Pedersen and Rosendahl 2015; Ramirez *et al.* 2015). Fast pyrolysis methods require drying and grinding wood to particles that can be efficiently fluidized in a gas stream.

Hydrothermal liquefaction favors wet feedstocks, for which drying would be cost-prohibitive and there is less of a need for fine particles (Xu and Lancaster 2008; Huang and Yuan 2015; Knez *et al.* 2015; Wikberg *et al.* 2015). Hydrothermal liquefaction can be used to hydrolyze lignocellulose to C5 and C6 sugars and lignin sequentially (Pandey and Kim 2011; Colakyan 2012). Papermill sludge and black liquor can be readily converted to bio-oil for fuel or chemical platforms by hydrothermal treatment (Xu and Lancaster 2008; Knez *et al.* 2015; Huet *et al.* 2016).

CHEMICAL AND ENZYMATIC TREATMENTS

Fractionation of wood or pulp by chemical and mechanical processes can be used to selectively process major polymeric components separately, usually in the sequence of hemicellulose, cellulose, and lignin. Once separated, each polymer can be degraded to monomeric units to yield cleaner and higher value platform chemicals as compared to thermal routes alone. Kraft mills already separate lignin from fiber by a chemical process, so these mills should be well suited to separate the three main components of wood to useful feedstocks. The challenge has been to keep making quality pulp and paper while producing chemicals from wood feedstocks (Yoon and Van Heiningen 2008; Hamaguchi *et al.* 2013a). It may be necessary to find new markets for the type of pulp produced when supporting a biorefinery (Lundberg *et al.* 2013a).

Chemical pretreatments of wood chips that can be used to fractionate wood components prior to kraft pulping have been reviewed extensively (Lehto and Alén 2015; Liu 2015; Kim *et al*. 2016). Three types of cost-effective pretreatments were examined with two goals: to obtain fermentable sugars and to preserve the quality of the kraft pulp. Pretreatments include hot water, dilute acid, and alkaline extractions. In general, hot water or dilute acid produce sugar monomers while degrading pulp quality (Liu *et al.* 2009; Saukkonen *et al.* 2012). Recovery of hemicellulose from hardwoods is favored by alkaline extraction. Softwood hemicellulose rapidly degrades under alkaline conditions due to peeling reactions. Green liquor pretreatment to obtain fermentable sugars from hemicellulose has been studied and modeled as a natural biorefinery opportunity for kraft mills (Mao *et al.* 2008, 2010; Lundberg *et al.* 2012; Phillips *et al.* 2013; Andrew *et al.* 2014). Prehydrolysis and extraction of hemicellulose under the right conditions may improve the bleachability of pulp and reduce loading on the recovery evaporators.

Severity of chemical pretreatment determines the level of fermentation inhibitors that are formed with sugars. Inhibitors include phenolics, lignin complexes, acetic acid, furans, and aldehydes (Boucher *et al.* 2014; Ko *et al.* 2015; Mechmech *et al.* 2015). Enzymatic hydrolysis after chemical pretreatment is inhibited by lignin complexes that compete for cellulase binding sites. Strategies are needed to produce sufficiently refined sugars from wood that are competitive with corn dextrose as a feedstock for fermentation to platform chemicals. Cost-effective removal of inhibitors will be a key part of developing commercial processes for wood sugars (Ajao *et al.* 2015; Mechmech *et al.* 2015; Nwaneshiudu and Schwartz 2015). Simple processes such as over-liming have been effective in detoxifying acid hydrolysates of wood chips for ethanol production (Mendes *et al.* 2011).

Pretreatment technologies for converting lignocellulose to useful chemical intermediates such as C5 sugars, C6 sugars, and lignin have been the subject of a few books (Bajpai 2013; Wyman 2013; Mussatto 2016). Biorefinery plants using wood may compete for resources with kraft mills in the same way pellet mills do today. The kraft pulping and bleaching processes could be used as a pretreatment process if the end products, delignified fiber, and lignin are intended as a source of sugars and bio-aromatics (Yu *et al.* 2011; Buzała *et al.* 2015a,b; Pinto *et al.* 2016). Lower-cost sources of wood such as forest residues could be used if pulp mill capacity allowed. Modifications of normal pulping operations to accommodate pretreatment technologies would be more of a natural fit for pulp mills than a greenfield biorefinery (Stoklosa and Hodge 2015).

MECHANICAL PRETREATMENTS

A range of mechanical and chemi-mechanical pretreatments for converting biomass to particle sizes amenable to fractionation of wood polymers has been reviewed (Balan 2014; Barakat *et al.* 2014). Methods to couple pulverization with enzymatic hydrolysis in a continuous stream at high solids was proposed to improve cost and streamline the process. The use of conventional mechanical refining was examined as an alternative to chemical pretreatments for hydrolysis of wood to sugars with lower production of fermentation inhibitors (Jones *et al.* 2013; Dou *et al.* 2016; Park *et al.* 2016). Refining coupled with mild pretreatment (steam or dilute acid) reduced overall conversion costs and the level of fermentation inhibitors at equal sugar yield. Mechanical pulping tools are part of the technology Leaf Resources developed for producing sugars from wood by acidic pretreatment and glycerol-based organic extraction (Sabourin 2015; Alex and Alan 2016). A related mechanical alkaline glycerol pulping process called AlkaPolp was developed to give lignin free pulp that can be readily hydrolyzed by enzymes to fermentable sugars (Hundt *et al.* 2015).

BIO-ADVANTAGED PLATFORM CHEMICALS FROM WOOD

Platform bulk chemicals that can be made from woody substrates fall into broad categories based on the number of carbons, origin (from polysaccharides or lignin), and can play a role as drop-in biobased equivalents or intermediates. Sugars are preferred feedstock for platform chemicals made through fermentation. A study on technical risk for sugar platforms in a kraft mill biorefinery focused on the challenges of generating added waste water and contamination issues in fermentation (Mariano 2014). Carbon monoxide from synthesis gas (syngas), methane, and carbon dioxide are also potential carbon sources for the fermentation processes that can be obtained from kraft mill waste streams (Shen *et al.* 2015; Drzyzga *et al.* 2015; Bomgardner 2016; Marcellin *et al.* 2016). A large market potential for biobased platform chemicals from sugars exists in fermentation intermediates generated for manufacturing polymers such as bio-PET, bio-PE, and bio-nylon (Collias *et al.* 2014; Becker *et al.* 2015b).

The DOE issued reports on the top value added chemicals that could be made from biomass using sugars, synthesis gas, or lignin as starting materials in 2004 and 2007 (Werpy and Petersen 2004; Holladay *et al.* 2007). An updated market assessment and potential of these chemicals was published in 2016 (Biddy *et al.* 2016).

A review of the most promising opportunities from a chemical and competitive market point of view highlighted multifunctional hydroxy acids that could not be readily made from petroleum products (Dusselier *et al.* 2014). Figure 2 shows a representation of platform chemicals that could be made from biomass, relating to a normalized functional index (F:C) *versus* carbon number. Higher functional numbers represent more highly reactive compounds that are suitable for obtaining biochemical from feedstocks. This guide is useful in sorting out target compounds that can have a niche market that will not be easily served by crude oil feedstocks. These chemicals are considered to be bio-advantaged.

Enzymatic saccharification and fermentation are dependent on pretreatment processes that generate little or no inhibitors during either step. Since woody biomass tends to be more recalcitrant than non-wood plant sources, leading to higher severity of pretreatment, crop residues are often preferred (McCann and Carpita 2015; Taylor *et al.* 2015). Hemicellulose is higher in non-wood residues and is easier to utilize than cellulose as a source of fermentable sugars and furanic chemicals (Cai *et al.* 2014). Waste streams from dissolving pulp mills are an exception, where hemicellulose has been commercially utilized to produce the sweetener xylitol (*Xivia^{TM} Xylitol White Paper* 2012). Enzymatic saccharification and fermentation technologies are being customized for crop residues to give optimal performance and cost on a commercial scale using some processes that can be found in a pulp mill (Schwab *et al.* 2016).

Wood feedstocks have sourcing advantages over crop residues due to the scale and maturity of forest product industries. Kraft mills are well suited to provide a wide range of low cost feedstocks to an integrated biorefinery for production of certain bulk chemicals.

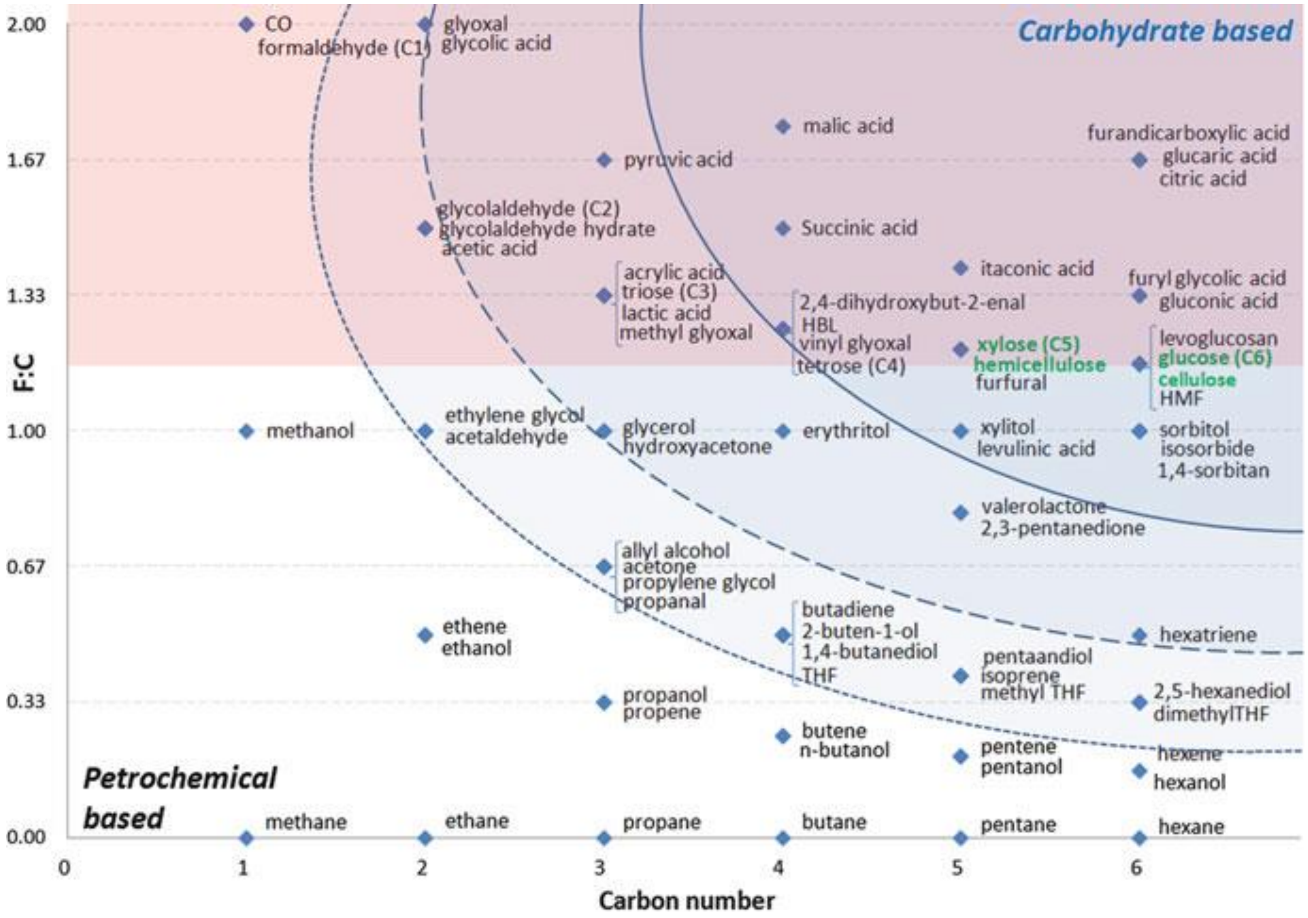

Fig 2. Platform chemicals from biomass based on functional index and carbon number (source: Dusselier *et al.* 2014)

Table 3 lists key platform bulk chemicals that can be generated from lignocellulosic materials (*WP 8.1. Determination of market potential for selected platform chemicals* 2013; Choi *et al.* 2015; Taylor *et al.* 2015). For processes that can utilize commodity sugars such as corn dextrose, the use of lignocellulose as a source of sugars may not currently be cost competitive.

Recent developments in commercializing cellulosic ethanol will offer technologies needed to make non-food sources of highly refined sugars more feasible. Virtually all the commercially explored lignocellulosic sugar examples come from agricultural residues. Anellotech uses wood as a feedstock in catalytic pyrolysis to give bio-PET precursors (Tullo 2016). Xylitol comes from xylose in hardwood extract of dissolving pulp pre-hydrolysis liquors.

Sugar production from woody feedstocks as a source of carbon for conversion to platform chemicals is in early stages of commercial exploration (Kobayashi and Fukuoka 2013). Fermentation to chemical intermediates is often more demanding in terms of yield, titer, and contamination than processes to generate ethanol (Papoutsakis 2015).

Table 3. Platform Chemicals with Potential for Sourcing from Lignocellulose

Platform Chemical	**Feedstock**	**Process**	**End Market**	**Bio-Producers**
Succinic acid	Sugars	Yeast fermentation	Polyesters, polyurethanes,	Myriant, BioAmber, Riverdia
Glucaric acid	Sugars	Thermocatalytic hydrodeoxygenation, Nitric acid/O_2 oxidation process	Conversion to adipic acid and bio-nylon polymers, corrosion inhibitors, detergent builder	Rennovia, Rivertop Renewables
Malonic acid	Sugars	Fermentation	Pharmaceuticals, flavors, fragrances	Lygos
Levulinic acid and esters	Lignocellulose	Thermochemical	Additives for food, pharmaceuticals, plastics	GF Biochemicals
2,5-Furan dicarboxylic acid (FDCA)	C6 Sugars	Two step catalytic process, dehydration and oxidation	Poly(ethylene) furanoate (PEF) alternative to PET	Avantium, Corbion
Hydroxymethyl furfural (HMF)	C6 Sugars	Catalytic dehydration, Hydrothermal processing	Levulinate esters, FDCA	Avantium, AVA Biochem
Xylitol	Hemicellulose derived xylose	Hydrogenation	Sweeteners, dental plaque inhibitor	Danisco, Xylitol Canada
Itaconic acid	Sugars	Fermentation	Poly(itaconic acid) detergent builder, scale inhibitor	Itaconix
para-Xylene	Lignocellulose, sugars	Catalytic fast pyrolysis, thermochemical - biochemical hybrid process	Bio-PET for plastic bottles	Anellotech, Virent

A few examples of companies developing a path to sugars from woody substrates can be listed:

- Sweetwater Energy: two-stage hemicellulose and cellulose hydrolysis followed by purification and clarification (Parekh 2015)
- Renmatix Plantrose^{TM}: process for sub-critical water hydrolysis (Colakyan 2012; Colakyan and Jara-Moreno 2015)
- American Process: SO_2-water-ethanol fractionation of wood to sugars (Iakovlev *et al.* 2013; Retsina *et al.* 2014)

- Leaf Resources: acidic glycerol-water fractionation of wood to sugars and lignin (Sabourin 2015; Alex and Alan 2016)
- Arbiom: low temperature phosphoric acid saccharification (Amsallem 2016; Fouache 2016; Scott 2016a)

SUMMARY

Biorefinery platforms available to the pulp and paper industry fall into three categories, thermochemical, biochemical, and materials. Thermochemical platforms are better suited to utilizing the whole tree without the need for fractionating into hemicellulose, cellulose, and lignin. Pyrolysis can be used to convert wood to a bio-oil that can be upgraded to be used in a petroleum refinery or used directly as a heating oil. Wood feedstocks for pyrolysis should be ground and dried. Pyrolysis technology may be adjusted to give a high yield of levoglucosan, which can go into bulk chemical synthetic pathways. Hydrothermal liquefaction of wood gives a bio-oil with a higher heating value than pyrolysis oils, but at lower yield. Pre-drying the feedstock is not needed. Hydrothermal bio-oils are better suited to upgrading to fuels in a petroleum refinery than most pyrolysis oils. Gasification of wood to syngas (H_2 + CO) can be reformed to petroleum substitutes or fermented to platform chemicals.

Biochemical platforms call for fractionation of wood into polysaccharides and lignin. Further fractionation of polysaccharides into cellulose and hemicellulose is needed where downstream fermentation is not efficient with mixed C5 and C6 sugars. Wood is generally more recalcitrant than non-woody sources of lignocellulose, resulting in higher levels of enzyme and fermentation inhibitors arising from the severity of the fractionation process. Unbleached and bleached kraft pulp have an advantage over wood in ease of conversion to fermentable sugars. The low to negative cost of the fiber content in municipal solid waste (MSW) makes this source particularly attractive.

Material platforms can be developed from the polymeric components of wood. Chemically modified hemicellulose or cellulose can be made into films with thermoplastic and barrier properties suitable for packaging applications. Bleached pulp can be highly refined into nanofibrillar cellulose for packaging material reinforcement or advanced materials. Lignin can be chemically modified and converted to carbon fiber or nanotubes.

Several kraft pulp and paper mills are in early stages of planning, piloting, and implementing a biorefinery strategy. Kraft lignin production is expanding with the development of Lignoboost and Lignoforce black liquor separation technologies. Sulfite mills have a long history of producing valuable chemicals from the sugars and lignin remaining in spent liquors. Selection of an appropriate biorefinery strategy can be addressed through a SWOT analysis of available resources, technology readiness, market environment, and customer engagement.

Biopolymer equivalents of petroleum-based polymers have a large market potential for wood based sugars and lignin. Cellulosic ethanol from kraft mill residues is at a cost disadvantage *versus* ethanol from corn starch or sugar cane. Technology to obtain refined sugars from wood for commodity fermentation platforms needs more development. Promising leads are in pre-commercial and pilot stages. Economic viability will depend on the direction of oil prices and sustainability of government subsidies.

OUTLOOK

New pulping and fractionation systems will be developed for the kraft mill system that take advantage of opportunities for better utilization of wood components and their byproducts. Pyrolysis or hydrothermal bio-oils will be upgraded on site to prepare drop-in substitutes for petroleum derivatives using advanced catalysis technologies. Logistic networks will solve the supply chain optimization challenge of collecting forest residues over large areas and converting them to fuel and polymer precursors. Portable thermochemical conversion units show promise for addressing this issue. Fractionation technologies combined with new fermentation technologies will address the issue of inhibitor formation and sugar purity. Advanced materials developed from lignin or nanocellulose will find markets ready for commercialization.

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