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The Cellulosic Biorefinery: Coproduct Extraction from Biomass

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Current Biofuel Industry

This nation is experiencing an unprecedented effort aimed at increasing its energy independence for a number of worthy reasons: replacing fossil fuels with biofuels dampens the need to import oil from politically unstable oil-producing countries; in certain situations, biofuels recycle carbon; and, U.S. rural areas, where biorefineries operate, benefit from economic revitalization. Grave consequences, in terms of rural exodus and political unrest, may affect this nation if total or at least partial energy sustainability is not attained within a short time-frame.

Currently the United States uses 140 billion gallons of gasoline and diesel annually. Approximately 7 billion gallons of ethanol and 450 million gallons of biodiesel were produced in 2007. This already met the federal mandate for 2012 specified in the Energy Policy Act of 2005. This phenomenal growth of the corn to ethanol industry has been coupled with the generation of copious quantities of byproducts. More than one third of the corn that is processed to ethanol ends up as a byproduct, either dried distillers grains with solubles (DDGS) or dried distillers grains (DDG). The sale of DDGS or DDG is an important component of the corn to ethanol process, as up to $0.10 per liter of ethanol produced, depending on sale price, is garnered by the biorefinery. Currently, corn to ethanol byproducts are used as animal feeds to the beef, dairy, swine and poultry industries and also are being investigated as aquaculture feed (Rosentrater, 2007). Because corn to ethanol byproducts are high in fiber and low in starch, they are also being investigated for their potential use in human foods (Rosentrater, 2007). This work is indicative of the complex nature of the corn to ethanol processing industry, illustrating that to be profitable (as in the petroleum industry) many products must stem from the processing plant.

There are currently 146 corn to ethanol plants in operation and another 61 under construction. In 18 months, the estimated production capacity of these 207 corn to ethanol plants will be 13.7 billion gallons. The corn to ethanol industry is undergoing phenomenal growth, owing to a demand for liquid fuels, known processing technology, and the benefits from existing infrastructure with respect to corn cultivation, postharvest technology, and manutention. Although the growth of the corn to ethanol industry is unprecedented, if all the corn produced in the United States were converted to ethanol, about 40 billion gallons of ethanol could be produced, which is far less than the 140 billion gallons or so required yearly by the U.S population.

Upcoming Biofuel Industry

To substantially increase the quantity of biofuels, cellulosic materials will need to be harnessed as a feedstock for liquid fuel conversion. Conversion technologies for cellulosic biomass are centered around either the hydrolysis of cellulose and hemicellulose in biomass, followed by fermentation of the resulting sugars to ethanol (Lynd *et al*., 2002); the gasification of cellulose, hemicellulose and lignin to produce synthesis gas (syngas), followed by the conversion of CO, CO_2 and H_2 to ethanol or other alcohols by fermentation or by catalyst-based processes (Brown, 2003); or the conversion of organic compounds in biomass through fast-pyrolysis to a dark-brown liquid, which can then be combusted for energy (Brown, 2003).

Depending on the conversion technology, 10-25 million tons of dry biomass feedstock are required to produce 1 billion gallons of liquid fuel. Recently, it was reported that cellulosics, such as switchgrass, can produce as much as six times more renewable energy than non-renewable energy consumed to produce the biomass (Schmer *et al*., 2008). Such promising numbers show that renewable fuel production from cellulosic crops is feasible, especially as oil prices are drastically on the rise.

Approximately one billion dry tons of biomass feedstock will be required annually to ensure that the United States can produce up to 30 percent of its liquid fuel demand from renewable resources (Perlack *et al*., 2005). On average, forest

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resources will generate 368 million dry tons per year, while agricultural resources, including energy crops, will contribute 998 million dry tons per year (Perlack *et al*., 2005). Current mandates require 21 billion gallons of cellulosic ethanol be produced by 2022. This will require more than 250 million dry tons of biomass. Nonetheless, back of the envelope calculations indicate that a 50 million gallon liquid fuel production facility, capable of producing 80 gallons of ethanol per ton of biomass, will require 1,838 tons of dry biomass per day. Assuming a biomass yield of 8 tons per acre, approximately one 50 million gallon liquid fuel production facility will draw biomass from an area of 122 square miles (67 miles x 67 miles at a 3 percent density). It is important to note that these numbers are only speculations and no 50 million gallon or more commercial plant has been constructed. However, with DOE funding, six production scale refineries will become reality in the near future (USDOE, 2007). Soperton, Georgia will soon be home to the first commercial cellulosic ethanol plant, setting the stage for the essential infrastructure needed in handling the 2,000 ton per day or so of required feedstock.

Importance of Coproducts in Biofuel Industry

As mentioned earlier, coproducts, in the form of DDG or DDGS, are important to the vitality of the corn to ethanol conversion process. Although not usually extracted in the current dry-mill based ethanol industry, corn germ oil and corn fiber oil can also be extracted in the wet-mill process, adding value to the overall corn processing operation (Singh *et al*., 2001). Coproducts are also an important component of the cellulosic conversion process. As shown in Figure 1, McAloon *et al*. (2000) outlined the unit operations for coproduct production in the biorefinery in terms of energy. Beer column bottoms, consisting largely of lignin, will be obtained from the processing of the fermentation solids and will be processed in a triple-effect evaporator before being recovered and combusted in a fluidized bed combustor. Lynd *et al*. (2008) showed through calculations that the thermochemical conversion of fermentation waste products to heat or electricity enhances the economics of the cellulosic biorefinery. Aside from energy production, McAloon *et al*. (2000) reported that the transformation of lignin into

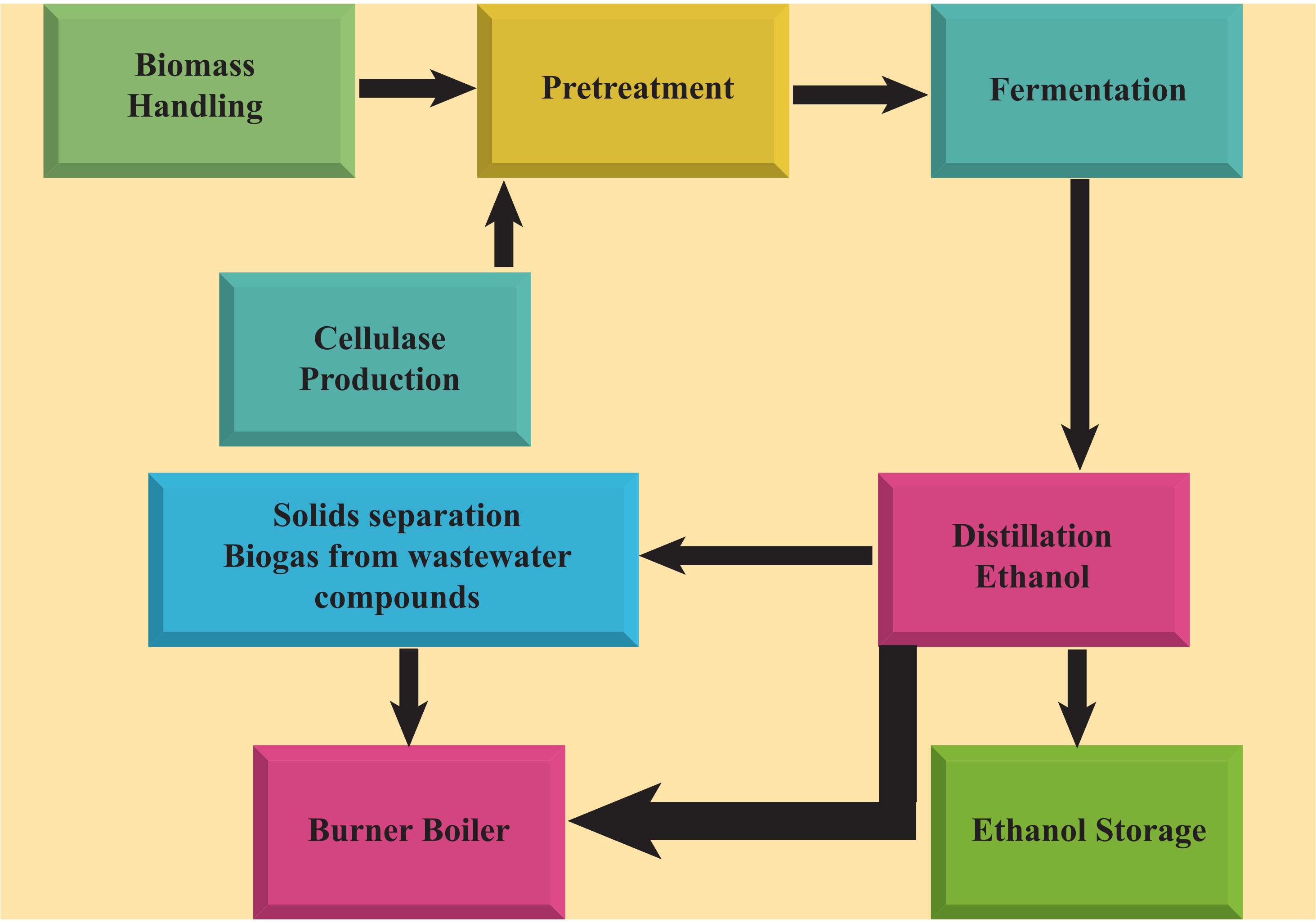

Figure 1. Schematic of Biorefinery

higher-value coproducts is important to the long-term commercial viability of the biorefinery, and that the recovery of interstitial cell matter could also be valuable, but would require significant purification.

Extraction of Coproducts in the Lignocellulosic Biorefinery

As stated by Hess, Wright, and Kenney (2007), the economic competitiveness of cellulosic ethanol production is highly dependent on feedstock cost, which contributes 35-50 percent of total ethanol production costs. In addition to a $30 to $36 per dry ton payment to the producer, Kumar and Sokhansanj (2007) estimate a harvesting, storage, and transportation cost between $40 and $48 per dry ton of biomass, depending if the feedstock is harvested as a bale, a loaf, or ensiled.

In an effort to increase revenues from a given feedstock, valuable phytochemicals could be extracted prior to the biochemical or thermochemical conversion at the site of the biorefinery or a site of close proximity. This extraction step could occur especially if a biochemical process is used because the dry biomass needs to be in contact with water during the dilute acid pretreatment step. Figure 2 shows how a slip stream for phytochemical extraction could be integrated in the biochemical biorefinery scheme. This phytochemical extraction scheme could be nestled within the biorefinery or could be part of a different operation located in proximity to the biorefinery. Phytochemical extraction could also be practiced in a thermoconversion biorefinery on the condition that the revenue obtained from the extraction of the phytochemicals warrants an extraction and an additional feedstock drying step. Either from a biochemical or a thermochemical biorefinery, these phytochemicals could find use in human and animal health care products, cosmetic applications, and as essential ingredients in green cleaning products. According to market research surveys, there is a growing preference among consumers for phytochemicals in the foods they consume, as well as other personal care and household products they utilize. Growth in the use of phytochemicals is predicted in the flavor industry, which includes beverages, confectionery, savory, dairy, and pharmaceuticals (Market Research.com, 2008). It is important to note that for the extraction of coproducts from lignocellulosic biomass to be workable, the

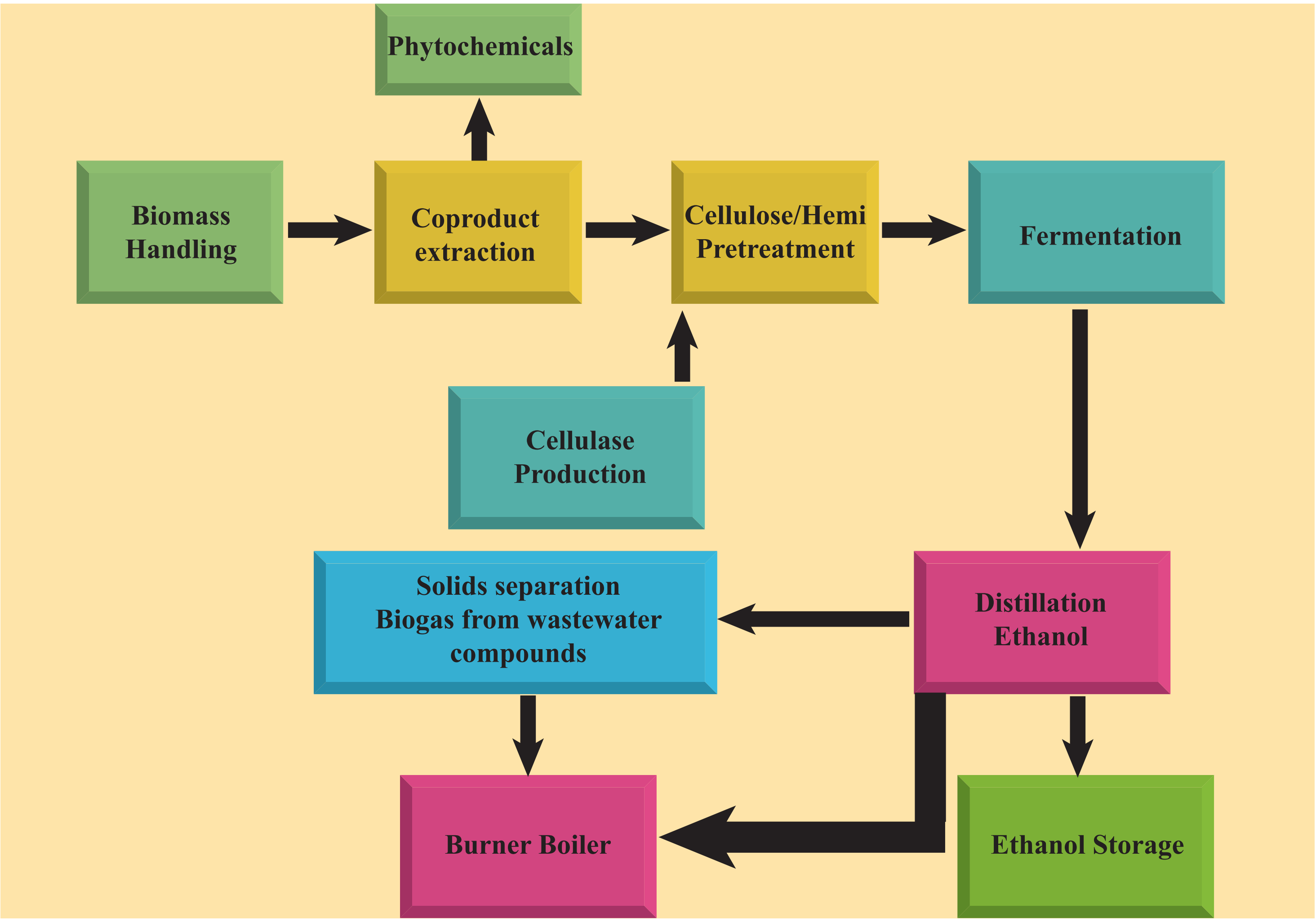

Figure 2. Biorefinery with Coproducts Extraction

extraction step must not hinder the conversion to energy by decreasing yields or adding processing steps.

There is a rich tradition in the phytochemical literature that presents organic solvent extraction schemes for all classes of plant-derived compounds. Scientific journals, such as *Phytochemistry*, *Phytochemical Analysis*, *Journal of Chromatography B*, or *Planta Medica* contain a multitude of articles detailing the required methodology for phytochemical extraction with solvents such as acetone, benzene, or hexane. Although the use of organic solvents is common in the pharmaceutical industry, organic solvent use for the extraction of phytochemicals is costly because of purchase price and inherent handling protocols. Additionally, the use of organic solvents is not deemed 'green' technology because of disposal and other environmental problems. Several alternatives techniques to organic solvent extraction are available for phytochemical extraction, namely supercritical fluids, pressurized liquids, and subcritical water extraction.

The application of subcritical water extraction to phytochemicals is novel as an environmentally compatible "green" technology, and is based on the exposure of the biomass to hot liquid water under pressure. The temperature of the water for extraction typically ranges from 100-180ºC in a pressurized system, well below the critical temperature of water (King, 2006). The use of subcritical water to extract high value phytochemicals permits extraction without concerns about solvent recovery or disposal. Additionally, the extraction of phytochemicals with subcritical water could serve as a biomass pretreatment step in the saccharification/fermentation process, and thus serve an important dual purpose. Thus, the extraction of valuable phytochemicals from biorefinery-destined biomass with subcritical water could harmonize well with the existing biochemical biorefinery.

The USDA and DOE (Perlack *et al*., 2005) Biomass as Feedstock for a Bioenergy and Bioproducts Industry study estimates that approximately one billion dry tons of biomass feedstock will be required annually to ensure that the United States can produce up to 30 percent of its liquid fuel demand from renewable resources. Thought was given as to the distribution of the one billion dry tons of biomass feedstock that will be required annually. The U.S. DOE (2006) presented a list of the most plausible energy crops that will be grown in the various regions throughout the United States (Figure 3). Successful bridging of the extraction of phytochemicals to the biorefinery can only occur when valuable phytochemicals are present in targeted energy crops. Not all energy crops fulfill this criterion.

Energy Crops with Potential Coproducts

It is most likely that cellulosic plants in the near future will be using a feedstock supply system that relies on current infrastructure and technologies (Hess, Wright, and Kenney, 2007). The thermoconversion-based cellulosic plant in Soperton, Georgia will be drawing on existing forestry supply logistics. Other cellulosic plants that are being planned will be based on the supply of agricultural residues, like wheat straw and corn stover, which is somewhat supported by existing crop harvesting infrastructure. However, the mid-term 50 million gallon facility will consume 2,000 dry tons of agricultural residues per day and will rapidly exhaust regional residue and waste capacities. To address this supply issue in cellulosic feedstock, energy crops will need to augment the feedstock portfolio. Energy crops will be developed regionally as outlined in Figure 3. Collection, storage, preprocessing, transportation and handling practices, logistics, and infrastructure will need to be developed for specific energy crops (Hess, Wright, and Kenney, 2007). While developing energy crop-specific logistics and infrastructure, particular energy crops can warrant value-added processing for the extraction of useful phytochemicals. A few of these energy crops are discussed below.

Black locust (*Robinia pseudoacacia* L) is a multipurpose tree species that can be used for livestock browse and as an energy crop in the eastern United States. The flavonoid acacetin, present in a whole tree extract of black locust is significantly cytotoxic against a human tumor cell line (Tian and McLaughlin, 2000). A water-soluble lectin, robin, initially discovered in black locust inner bark, is most likely the toxic principle for humans which consume the plant (Hui, Marraffa, and Stork, 2004). Toxalbumins are composed of an alpha chain and a beta chain that is linked by a disulfide bond. The beta chain binds to cell surface glycoproteins where it is transported to the endoplasmic reticulum of the cell. The alpha chain inhibits the 60s ribosomal subunit and prevents protein synthesis. Although currently viewed as toxic, it is possible that an extremely biologically active molecule like robin may prove to have uses in advanced therapies.

Eucalyptus is a native from Australia and its genus comprises more than 700 different species. Interestingly, there are currently more than 45 million acres of this tree planted in 90 countries, making this one the most widely planted 'working' tree in the world. As shown in Figure 3, Eucalyptus is grown in California and Florida; however, there are agronomic trials currently underway to examine its hardiness in the Southeastern United States. Eucalyptus is desirable and widely planted because it is a fast growing and high yielding hardwood. Currently, the genome of Eucalyptus is being sequenced through the Eucalyptus Genome Network project, through U.S. DOE support. From the phytochemical perspective, Eucalyptus contains phytochemicals such as flavonoids (Abd-Alla *et al*., 1980) and monoterpenes (Dayal, 1988). The most famous Eucalyptus-derived phytochemical is the monoterpene 1,8-cinenol, which is an active ingredient in Listerine® mouthwash. Eucalyptus preparations were

Figure 3. Herbaceous and Wood Crop Possibilities as Suggested by the Department of Energy
Source: USDOE, 2006

shown to be active against methicillin-resistant *Staphylococcus aureus* and vancomycin-resistant enterococcus (Sherry, Boeck, and Warnke, 2001). Thus, Eucalyptus would be an excellent candidate to demonstrate the feasibility of subcritical extraction of useful phytochemicals and conversion of the biomass to liquid fuels, indicating that the concept of bridging two unrelated research areas is possible.

Annual production of grain sorghum in the United States is 10-20 million metric tons. About 12 percent of the grain sorghum produced in the United States is used for ethanol production (Hwang *et al*., 2004). Recently, there has been interest in sorghum as a cellulosic crop that could be used in the lignocellulosic biorefinery. As shown in Figure 3, sorghum grows throughout the Midwest. In addition to being a source of starch and of cellulose, a wax-like material can be extracted from whole kernels and from stalks of sorghum. This wax-like material contains policosanols, which are a mixture of long-chained primary alcohols, comprised mainly of docosanol (C_{22}), tetracosanol (C_{24}), hexacosanol (C_{26}), octacosanol (C_{28}), triacontanol (C_{30}) and dotricontanol (C_{32}) (Irmak, Dunford, and Milligan, 2005; Hwang *et al*., 2004). The policosanol concentration of sorghum can be up to 1,200 mg per kg of sorghum grains (Hwang *et al*., 2004). Policosanols have been reported to improve blood lipid levels, reduce platelet aggregation, ameliorate exercise performance in coronary heart disease patients, and increase muscle endurance (Taylor, Rapport, and Lockwood, 2003). Currently, policosanols are being consumed to reduce low density lipoprotein (LDL) levels, while increasing high density lipoprotein (HDL) levels (Taylor, Rapport, and Lockwood, 2003). Policosanols are currently available as a dietary supplement. Reports suggest that 5–20 mg per day of mixed C_{24}– C_{34} alcohols, specifically C_{28} and C_{30}, lower low-density lipoprotein (LDL) cholesterol by 21–29 percent and raise high-density lipoprotein (HDL) cholesterol by 8–15 percent (Hargrove, Greenspan, and Hartle, 2004). As the clinical significance of policosanols becomes established and the development of organic solvent-free extraction methodology is developed, the extraction of these phytochemicals could be added to the lignocellulosic biorefinery.

Sweetgum (*Liquidambar styraciflua* L.) is a deciduous tree that grows in the southeast United States. The trunk of these trees produces a fragrant resin called styrax, which is used in incense, perfumes, soaps, cosmetics, and medicine. Styrax was reported to contain styrene, vanillin, cinnamic acid, borneol, and bornyl acetate (Willie and Brophy, 1989). Essential oils can be extracted from sweetgum leaves and were reported to contain 30.1 percent of terpinen-4-ol, 18 percent alpha-pinene and 12.8 percent sabinene. *L. styraciflua* essential oil composition is similar to that of Australian tea tree oil, which is used in the herbal industry. It is worth noting that tea tree oil is a player in the $1.9 billion plant-derived chemical industry (Fredonia Group, 2008). With expanding aroma therapies and interest in green cleaning products, an increase in essential oils could be foreseen. Sweetgum biomass could be extracted by subcritical water prior to energy conversion.

Switchgrass, *Panicum virgatum* L., is a warm-season perennial grass that grows throughout the Midwest and the Southeast. Schmer *et al*. (2008) demonstrated that switchgrass can produce 540 percent more output energy than the input energy supplied to grow and harvest the biomass, giving credence to the concept of cellulosic ethanol. Switchgrass is rapidly being developed as an energy crop. During the spring of 2008, the Oklahoma Bioenergy Center sponsored the planting of 1,000 acres of switchgrass near Guymon, Oklahoma that will be used as feedstock by a cellulosic biorefinery in Hugoton, Kansas. In addition, Tennessee through the Tennessee Biofuels Initiative sponsored in the initial year the planting of 720 acres with plans to plant 6,000 acres of switchgrass over a three year period.

Like sorghum, switchgrass contains policosanols. Oklahoma-grown switchgrass has total policosanol contents ranging from 105 - 182 mg/kg (Vandhana Ravindranath *et al*., 2008), which is less than what is contained in sorghum. However, the composition of individual policosanol alcohols of switchgrass and sorghum differ. Oklahoma-grown switchgrass was shown to contain of 0.4-1 percent of C_{26} alcohols, 10-16 percent of C_{28} alcohols, 35-40 percent of C_{30} alcohols, and 46-50 percent of C_{32} alcohols, while the alcohol distribution in sorghum was 0–1 percent C_{22}, 0–3 percent C_{24}, 6–8 percent C_{26}, 43–47 percent C_{28}, 40–43 percent C_{30}, and 1–4 percent $C_3$2, indicating a lower C_{32} content than that of switchgrass (Hwang *et al*., 2004). It may be possible that future bioactivity-based research shows that the individual alcohol composition of the policosanol dietary supplement plays a role in conferring LDL lowering activity. If such were the case and high proportions of C_{32} are desired, then switchgrass policosanols could be used. In addition to policosanols, switchgrass contains 320 - 400 mg/kg of α – tocopherol if harvested prior to frost (Vandhana Ravindranath *et al*., 2008). It is important to note that the results reported by Vandhana Ravindranath *et al*. (2008) were based on hexane extraction, and this would not be feasible in a cellulosic biorefinery scenario. However, as subcritical water or supercritical extraction methods are developed, policosanol extraction from switchgrass coupled to the cellulosic biorefinery could be possible.

In addition to policosanols, switchgrass contains the flavonoids quercitrin and rutin. By extracting switchgrass biomass with 90ºC water, yields of 184 and of 193 mg per kg of switchgrass were obtained for rutin and quercitrin, respectively (Uppugundla *et al*., 2008). Moreover, 18 µM preparations of both rutin and quercitrin were shown to inhibit the oxidization of LDL by 70 and 80 percent, respectively, as determined the thiobarbituric reactive substance (TBARS) assay (Uppugundla *et al*., 2008). The extraction of switchgrass flavonoids was performed at 90ºC, which is well below the recommended water pretreatment temperatures of 140 and 240ºC, indicating that the extraction of phytochemicals could be harmonized with cellulosic biorefinery operations.

Infrastructure Needs of the Cellulosic Biorefinery

With the implementation of the cellulosic biorefinery comes the movement of large masses of feedstock, where 2,000 to 5,000 dry tons per day will need to be delivered at the doorstep of the biorefiney on a daily basis. Various scenarios for bringing the feedstock from the field to the door of the plant have been explored. Kumar and Sokhansanj (2007) modeled the transportation costs of chopped or ensiled biomass, of round or square bales, or of 2.4 x 3.6 x 6 meter loafs from the field to the cellulosic biorefinery. Of these possibilities, the loafing procedure, at $37 per dry ton, was the least costly. To harvest, transport, grind, and store forage type feedstock, existing machinery could be used and modified. Gathering and postharvest processing of woody feedstocks will most likely draw on technology from the current logging industry. Storage stations will have to be put in place, as the feedstock will be harvested once or twice per season, yet will be converted throughout the year. In regions where feedstocks containing useful phytochemicals are converted to biofuels, subcritical water based extraction facilities could be on-site or off-site from the biochemical cellulosic biorefinery. The phytochemical-exhausted biomass will be wet and could immediately be pretreated, as needed, using a dilute acid protocol.

Conclusion

The economic competitiveness of cellulosic ethanol production is highly dependent on feedstock cost, which constitutes 35-50 percent of the total ethanol production costs. In addition to a $30 to $36 per dry ton payment to the producer, a harvesting, storage and transportation cost between $40 and $48 per dry ton of biomass will also be required, depending if the feedstock is harvested as a bale, a loaf, or ensiled. In an effort to increase the revenues from a given feedstock, coproducts can also be obtained from feedstock during conversion. Thermochemical conversion coproducts are currently incorporated in the biorefinery layout that is proposed by Lynd *et al*. (2008). In addition to thermochemical conversions, valuable phytochemicals could also be extracted with subcritical water prior to the biochemical or thermochemical conversion at the site of the biorefinery or a site of close proximity, thereby adding value to the feedstock. It is important to note that the concept of extracting coproducts from biomass prior to conversion is still in its infancy. Rightfully so, all efforts are currently directed at cellulosic ethanol production. However, as the cellulosic ethanol biorefineries become a reality, it will then become interesting to investigate the production of secondary stream processes, such as coproduct extraction. At that point it will be become critical to generate positive as well as negative information on potential coproduct extraction, so that comprehensive economic evaluation of this process can be prepared.

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