Flippr° — an industrial research project in Austria

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ABSTRACT: Flippr° (Future Lignin and Pulp Processing Research) is a cooperative research project on biorefinery issues, funded by three universities and four pulp and paper mills in Austria. Efforts focus on establishing know-how about structural chemistry of cellulose fibers and lignin for more efficient use of these two major raw materials streams. Several subprojects are related to product innovations for use of lignin, fibers, and fines inside and outside the pulp and paper sector. Research toward added-value use is based on existing industrial production processes to achieve rapid realization. In addition to technical developments, ecological and economical perspectives are addressed by respective subprojects. Preliminary results, such as the establishment of quicker fundamental lignin analysis, are the basis for further investigation on lignin use by the industry partners. The method for high throughput analysis is nearly finalized; it will allow screening of industrial lignin sources in a reasonable time. Research on fibers and fines shows that a differentiated view on technological properties opens up a wide variety of process improvements for papermaking.

Application: Research will help companies develop value-added uses of lignin and cellulose using current production processes.

The Flippr° (Future Lignin and Pulp Processing Research) cooperative research project on biorefinery issues is funded by three universities (BOKU University of Natural Resources and Life Sciences, Vienna; University of Graz; and Graz University of Technology) and four pulp and paper mills in Austria. The project will run for 4 years, until April 2017, and has a total budget of EUR6 million. The focus is on establishing know-how about structural chemistry of cellulose fibers and lignin for more efficient use of these two major raw materials streams. The industrial partners acknowledge that fundamental research is a prerequisite to advanced applications and use of cellulose and lignin. Several subprojects include product innovations for use of lignin, fibers, and fines inside and outside the pulp and paper sector. Ecological and economical perspectives are addressed, in addition to technical developments. About 65 staff members are actively involved in Flippr°, including 12 doctoral students; women comprise about 30% of staff.

LIGNIN RESEARCH

Comprehensive lignin analysis has been established at the BOKU University, Vienna, Division of Chemistry of Renewable Resources laboratory. This analysis is aimed at integrating lignin into sustainable, non-energetic applications, thus helping the chemical industries to progressively substitute fossil-based raw materials. A comprehensive knowledge of lignin structure and chemical properties is an absolute prerequisite to any reasonable use, which renders the availability of the analytical methods so important [1-3].

Lignin is one of the major natural polymers. It is an often forgotten fact that lignin is produced in amounts almost similar to those of cellulose upon pulping of wood; it is the major byproduct of the pulp and paper industry. Because of lignin's heterogeneous structure and lack of bulk applications, it has mainly undergone energetic utilization (i.e., it has been burned). Nevertheless, lignin consists of valuable structural units, such as phenols and other aromatics, for which there is generally wide potential for use in the chemical industry [4,5]. Potential applications range from adhesives, binders, and resins over paint components to organic soil conditioners and artificial humic substances — all markets that offer the possibility of using lignin in bulk amounts.

However, before lignin is to be used in sustainable — and thus mostly non-energetic — ways, it is imperative to know its structure. The structure of lignin is much more highly sophisticated than the structure of cellulose. While cellulose structure is constant across plant species, having one binding type between the basic building blocks, lignin structure varies across plant species. It is moreover irregular, comprising several binding modes among the three possible araliphatic phenylpropane–type monomeric units. The structural diversity and variability of natural lignin is further complicated by pulping steps and by differences in pulping processes. To provide consistent quality to customers in the chemical industry, chemical parameters need to be defined that characterize structure and quality of the lignin, and these parameters need to be measured by robust and reliable methods. Only then can reasonable application-oriented development begin. Because most analytical methods to characterize lignin have been developed for natural lignins, these techniques are less apt to be applied to processed lignins (e.g., process liquors, oxidized lignins, lignins in resin applications, lignin gels) that exhibit additional structural and compositional complexity.

BOKU University, Vienna, established a combination of chemical and instrumental analysis methods for in-depth characterization of lignin. In particular, the combination of nuclear magnetic resonance (NMR) spectroscopy (direct 2D and 3D techniques as well as experiments after lignin derivatization) and newly developed, fast wet-chemical and chromatographic methods distinguish this approach from previous ones in the field. The development allowed Austria to catch up with research institutions in countries with traditionally strong domestic pulp and paper industries.

Analysis comprises the fundamental chemical and spectroscopic properties of different lignins as well as characterization with regard to usability in different chemical applications [6]. The approach addresses the needs of the pulp and paper industries at the threshold of becoming modern biorefineries by tackling two problems: (1) the isolation of lignins from black liquors and their in-depth analysis and (2) the development of robust high-speed methods capable of coping with large sample numbers. Thus, the effects of process changes on liquor composition and lignin structure can be followed and quality changes in the lignin can be monitored reliably.

Elaborating these techniques has been a long process. Besides strong international cooperation with leading international experts in lignin characterization, the newest infrastructure and analytical equipment are placed at the disposal of the lignin chemistry group. BOKU University, Vienna assigned a senior scientist to the lignin research area and company partners supported the setup of instrumental equipment. Formally integrated into the so-called lignin platform, chemical and analytical research on lignin has become a mainstay of the activities in Flippr° and a prerequisite to the envisioned non-energetic lignin utilization.

FIBER RESEARCH

The main objective of the fiber research area is to develop opportunities for optimized use of fiber resources within the pulp and paper process as well as potential applications of pulp fibers or pulp fiber fractions outside the standard product chain. The basic approach to gain added-value for a given fiber source is pulp fractionation and targeted, appropriate use of the fractions within the process. The work discussed in the literature has mainly focused on fines and their effect on sheet properties [7-9], bleachability of pulp fractions [10-12], and refining of pulp fractions [13,14].

To better understand the technological effect of a certain fiber or fines fraction, samples will be evaluated according to a multiscale approach — from the fiber level to the properties in the final sheet. For that purpose, a comprehensive morphological, chemical, and physical characterization is needed. A flow cell for morphological characterization is available, providing an optical resolution of 2.5 µm/pixel as well as characterization of fines material in the dry state based on optical microscopy, reflection electron microscopy (REM), and environmental scanning electron microscopy (ESEM). Apart from this morphological characterization based on single fibers and fines, evaluation of cross-sections of paper based on microtome cuts is available also. Fractions of the pulp before sheet forming can be labeled with fluorescent dyes and located in the images.

One possible application of fiber and fines material outside the pulp and paper product chain is reinforcement materials in polymer matrices. To assess the optimum properties of the cellulose nanofibrils (CNF), it is necessary to disintegrate and purify the natural sources. Fines material are of interest because energy needed in the mechanical fibrillation process could be considerably below the level needed to disintegrate coarse pulp fibers as is routinely done.

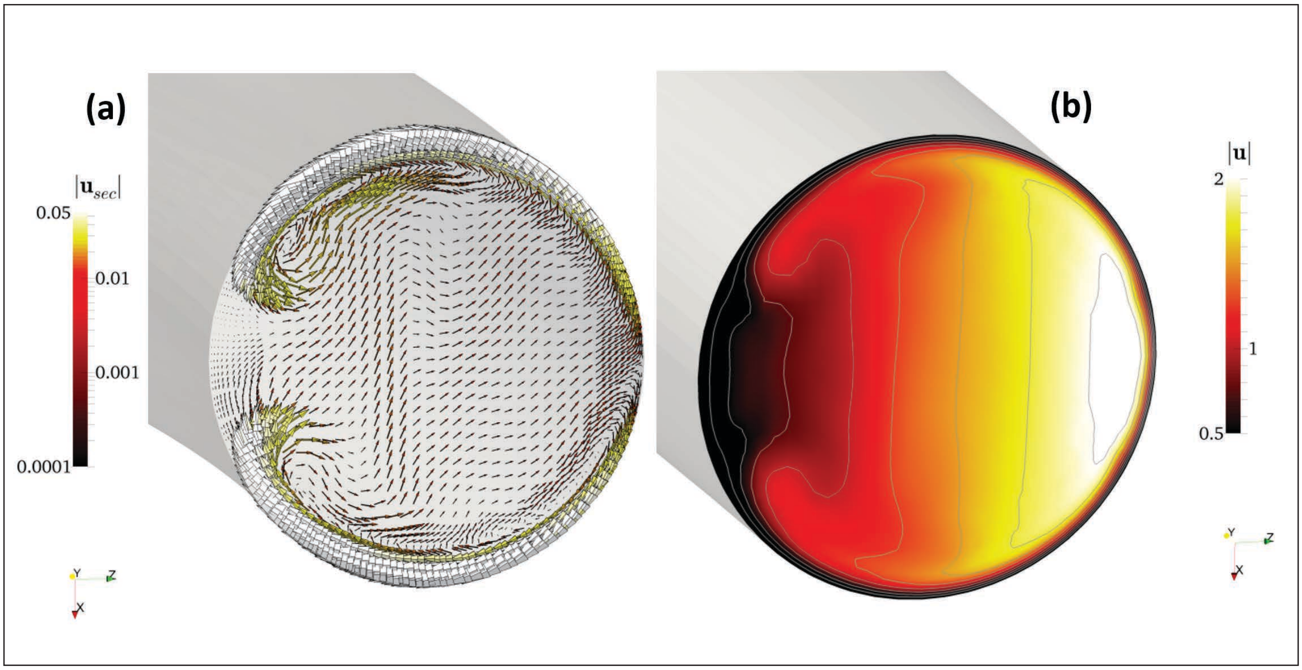
Another approach toward added-value within this research framework is chemical or enzymatic modification of the whole pulp sample or certain pulp fractions. For example, hydrophilization, oxidation, or cationization of pulp fibers has been shown to be capable of increasing the tensile strength [15-17). Cationization also enhances retention of fillers and fines [16,17]. Enzymatic modification has high potential in refining processes by decreasing cellulose crystallinity and increasing fibrillation and fiber flexibility [18-22]. On the other hand, oxidoreductase has potential for attachment of functional molecules and cross-linking of fibers [23-28].

Besides the mere production of fractions for analytical purposes and technological trials on the laboratory scale (based on standard pulp testing equipment and a purpose-built pressure screen with different mesh sizes down to 100 µm hole diameter), fiber fractionation and separation on a larger scale is also a focus of this research area. Hydrodynamic fractionation devices (HFDs), technology that uses fluid-dynamical effects for size- and shape-based fractionation as in the so-called tube flow fractionator [29,30], are being investigated.

Detailed computer simulations of the respective flow field show the local flow velocities in these devices (**Fig. 1**). A better understanding of these effects is expected to allow implementation of hydrodynamic fractionation on the laboratory scale in a continuous setup.

SUSTAINABILITY RESEARCH

Parallel to the technical research, life cycle sustainability assessment (LCSA) is conducted. This assessment integrates a comprehensive sustainability perspective and provides decision support for the innovation process. It also allows for determining the potential contribution of lignin-derived and cellulose-derived innovations to the emergence of low carbon, sustainable and resilient systems. To consider all three dimensions of sustainability (ecological, social, economic), an LCSA



1. Typical flow structure in a coiled tube flow fractionator: (a) secondary flow in a normal main flow direction plane, and (b) flow in the axial flow direction. Velocities are rendered dimensionless by mean flow velocity; vectors are colored by the magnitude of the secondary flow velocity (IW = inner wall, OW = outer wall).

system is developed that integrates environmental life cycle assessment (LCA), social-LCA (S-LCA), and life cycle costing (LCC) [31-33]. This development is scientifically important, as there is a need for research with respect to the construction of indicators and methods for the social and economic dimensions and their application in the "fuzzy front end" of innovation [34,35]. The results of the LCSA will be useful for the partner companies interested in developing integrated biorefinery systems and for policymakers in the development of new incentives to promote sustainable biorefinery concepts.

INDUSTRIAL PARTNERS

Zellstoff Pöls AG

Zellstoff Pöls AG is one of the biggest producers of elemental chlorine-free (ECF) bleached softwood kraft pulp in Central and Eastern Europe manufacturing a broad range of high-quality papers for packaging applications. The production capacity of the mill is around 430,000 metric tons of pulp and 80,000 metric tons of paper. Besides pulp and paper, Zellstoff Pöls produces heat for 15,000 households; electric power; and renewable bio-based chemicals, such as tall oil and turpentine.

Norske Skog Bruck GmbH

The Norske Skog paper mill in Bruck is running two paper machines producing top-quality newsprint and lightweight coated magazine paper at a capacity of 400,000 metric tons/year. Seventy-five percent of the mill's production is exported. By producing multirecyclable products from renewable raw materials, Norske Skog Bruck is actively addressing resource efficiency issues.

Mondi AG

Mondi Frantschach produces unbleached kraft pulp from 1.2 million CBM of softwood and sack kraft paper as well as specialty kraft paper on three paper machines. Mondi Neusiedler produces a variety of uncoated fine and specialty papers on four paper machines and totally chlorine free (TCF) bleached sulfite pulp. The product range of the mills includes specialty pulps, composite materials, district heating, turpentine, and green energy. The Mondi converting plants provide a range of fiber-based packaging solutions.

Sappi Gratkorn

Paper has been produced at Gratkorn, Austria, for more than four centuries. Sappi Gratkorn's mill is part of Sappi Europe. It produces 990,000 metric tons/year of double- and triple-coated papers, which are used for premium-quality publications all over the world. Sappi revolutionized the mill by introducing one of the largest and most advanced coated fine paper production lines in the world. The mill produces 250,000 metric tons of TCF chemical pulp and has an annual sheet finishing capacity of 875,000 metric tons. About 95% of the mill's production is exported.

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ABOUT THE AUTHORS

Changing consumer behavior and rising competition for wood point toward a need for increased resource efficiency and development of new products. Therefore, there is a rather strong need to go beyond the current situation. Previous research was based on bilateral research activities between scientific partners and companies or cooperative projects targeting manufacturing issues. Flippr° is directed toward solving strategic objectives in a cooperative consortium. This long-term initiative differs from previous projects of the sector in Austria and hence is a specific feature of Flippr°.

The acceptance by all partners that fundamental analysis and research is necessary was an important step. Several discussions in the starting phase of the project were needed to establish that acceptance.

Technical lignins proved to be both the most challenging and most surprising aspect of the research. Lignins are challenging in almost all regards but also yield some pleasant surprises.

The industrial partners will achieve further process information and possibilities for application. In some cases the final results of Flippr° research will be directly implemented. Although some areas are ready for implementation, other topics will need further research.

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