Optimization of cellulose dissolution stage in lyocell process as origin for different applications

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The worldwide interest to modify cellulose by means of lyocell process has increased strongly in the last 5 years. Today raw cellulose, regardless of its origin, is available as low cost feed material. It can be processed into staple fibers, filaments and films. The core of the lyocell processing technology is the dissolution besides the spinning step.

Parallel to viscose and cotton, lyocell fibers show a progressive acceptance from the market. The relatively simple adjustment of the properties of the lyocell fibers ensures the provision of qualitative characteristics that previously could not be reached with viscose fibers. The good skin compatibility as well as the high dry and wet tear strength open new and long-range future opportunities for the cellulose fibers.

List AG, Arisdorf/Switzerland further optimized the cellulose dissolution technology, which was introduced in 1992. This succeeded the production of excellent spinning solution qualities, produced from a variety of low cost raw materials. The technology fulfils the current high safety standards.

Development and optimization

In order to increase the application of the lyocell process, it seems necessary to further optimize the dissolution step of Cellulose in NMMO. The optimization aims to improve the economy of the process through the application of a cost-efficient technology.

Reviewing the known processing technologies to date, it is apparent that besides the industrially implemented specialized thin film processing technology, the new List dissolution technology is also available. This technology was developed in close collaboration with the "Institute for Textile and Plastic Research (TITK, in Rudolstadt/Germany). In 1998 Alceru GmbH (Rudolstadt/Germany) and Grasim Industries Ltd. (Nagda/India) implemented the process on a pilot scale with a production capacity in the range of 300-400 tons fibers/year.

List technology in brief

Scale of operation and features:
- Volume: 2.5-16,000 l
- High interfacial renewal rate
- Large cross-sectional area
- Large self cleaning heat exchange surfaces
- Close temperature control
- Narrow residence time distribution
- Operating pressure: 0.001 - ~150 bar
- Processing viscosity: 40,000 Pas

The cellulose dissolution in NMMO takes place in a thick-layer kneader of the type List Discotherm B Conti Fiber. The raw materials cellulose and NMMO are homogeneously mixed in the agitated chamber of the kneader and processed to the final cellulose spinning solution.

In 2000 pilot units of the 3rd generation were installed in the China Textile University (Shanghai/PR China) and in Fraunhofer Institut für Angewandte Polymerforschung (Golm/Germany). Both units have a processing capacity of 50 tons cellulose/year.

The dissolution technology of the 3rd generation aimed mainly for the optimization of the quality of the spinning solution (Fig. 1). In the foreground were the following:
- use of raw cellulose of different origin
- production of spinning solution of constant and high quality
- minimization of the gel formation in the spinning solution filter.

For safety reasons the List cellulose dissolving technology preferably operates at temperatures lower than 100 °C. A major advantage of the low operating temperature and product temperature is the minimization of the discoloration of NMMO and the maximization of its recovery.

The large hold-up of the dissolver provides enough buffer capacity to regulate process fluctuation, if it should occur. Hence, the spinning solution is directly discharged from the kneader in the filter and the spinning pump.

A new development project between TITK and List aims for further improvements of the technology. It will result in the 4th generation of the cellulose dissolution by means of List technology.

At present design work is being executed for production units with capacities 1000, 5000, 10,000 and 15,000 t/year per line, for the production of staple fibers, filaments and films. The spinning solution produced in the List cellulose dissolution unit can be used as a main stream for the production of fibers and as a secondary stream for the production of filaments and films. TITK developed and defined the qualitative parameter "Filter Value (F_P)" to assess the spinning solution. This filter value is derived from the particle analysis

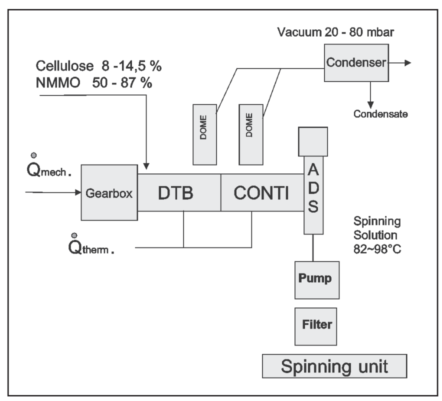

Fig. 2 P & F diagram of the List cellulose solutions-step

Fig. 1 Dissolving unit of the 3rd generation

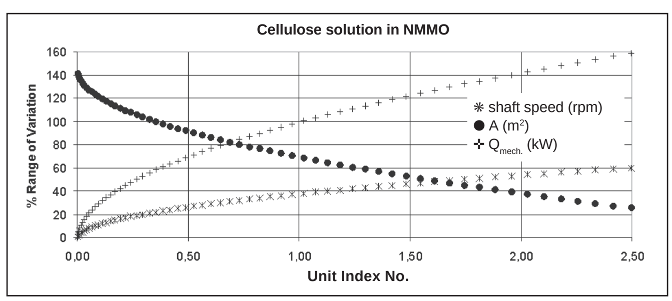

Fig. 3 Process simulation: functions of the process conditions and the apparatus know number

of the spinning solution. It is defined as the quotient of the largest particle diameter X_m and the logarithm of the number of particles N_{10} with sizes equal to 10 µm.

$$F_P = 10 \cdot \frac{X_m}{g\,N_{10}}$$

Solutions with $F_P < 50$ have excellent spinning quality, while solutions with $50 \leq F_P < 100$ designate adequate spinning quality.

Particulars to the List NMMO cellulose dissolution technology

The large volume kneader of the Discotherm B Conti Fiber has proved very flexible with regard to adapting the process for different raw cellulose qualities, but also when variations in the spinning process occur. The large processing volume serves as a buffer tank for the spinning solution. The mixing/homogenization step, which is pertinent for the preparation of the cellulose dope, can be simultaneously used for the addition of additives.

Due to the high flexibility of this dissolution technology, an intermediate buffer tank be avoided. Consequently, the filter and the spinning unit would be installed downstream to the discharge nozzle of the Discotherm B Conti Fiber. It is apparent that the equipment and the space requirements are reduced, which in turn grants economic benefits.

The List cellulose dissolution technology is clearly characterized by the low operating temperature. This offers the advantage that the product is not thermally degraded. Additionally the low operating temperature ensures high safety levels. Up to 80 % of the necessary energy for the dissolution process is supplied by dissipation.

The operating temperature should preferably be under 100 °C. The large hold-up and the large contribution of the dissipation energy to the total thermal duty allow for a simple automatic process control.

For the detailed optimization of the operating parameters, which would best match the solution of any raw cellulose, List provides state-of-the-art pilot units in its test center in Arisdorf/Switzerland. The same pilot units are also available for rental and for purchase.

Process simulation

List developed a process simulation program for supporting the design and optimization of the process. Furthermore, pilot and full scale process data can be fitted. Fitting and interpreting pilot-scale data, the simulation leads to the optimum design of the full scale unit. Using full-scale data the simulation delivers information about the flexibility of the process and the reserves of the plant.

Fig. 3 shows the influence of the speed of revolution of the kneader's agitator on the generated dissipation energy. The combination of those two parameters enables the optimal selection of the processing machine. The basis for the process simulation are the mass and energy balance, equipment characteristic values, caloric properties of the components, shear effects as function of product properties and equipment specific geometric characteristics.

The worth of this cellulose dissolution technology

Taking into account the stringent process safety demands, which are fulfilled through the selection of adequate construction material and the limitation of the operating temperatures, the technology was optimized to produce excellent spinning solutions from practically any cellulose, regardless of its origin.

Through the optimization of the dissolution process and of the kneader it is possible to produce spinning solutions of very good quality without pre-treatment (activation) of the raw cellulose. The spinning solution can be used for the production of fibers, filaments and films.

The really compact technology can optimally be regulated and controlled. It is characterized by its flexibility and easy adaptation on process conditions and product compositions. Low operating temperatures and the short residence time of the processed product minimize the recuperation and rectification costs of NMMO and contribute to the maximization of the economy of this technology.

The integration of the continuous preparation of the cellulose dope in a List Co-Rotating-Processor Conti rounds off the optimization of this technology.

Outlook

List is currently negotiating the extension of the field of application of this technology with new partners.