

Current Status

As world consumption of textile fibers expands with the rapid growth of Asia and other developing countries, sources of fibers other than cotton and rayon must be developed and brought to market to meet demand. Cellulose fibers (Lyocell fibers) can meet these expanding needs because of the specific qualities and characteristics of the fiber, as well as the vast availability of the raw materials.

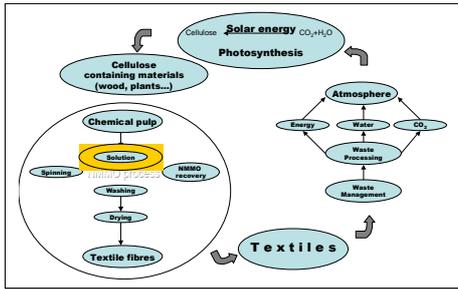


Fig. 1 – LYOCCELL Process

In 1992 the Thuringian Institute for Textile and Plastic Research (TITK) approached LIST AG in Arisdorf, Switzerland about the development of a commercial Lyocell process based on research performed at TITK since the 1980s. Working in conjunction LIST and TITK developed a successful Lyocell mixing and dissolving process at both the pilot plant and semi-industrial scales. In light of the expected growth in demand for these cellulose fibers, aspects for the future of this technology show great potential.

After six years of joint development efforts between LIST and TITK, the first two continuously operating, pilot plants with LIST Lyocell Dissolving systems were installed in 1998 in Europe and in Asia. Since their initial start-ups, each plant has operated successfully with on-going process optimization and continued efforts to increase both process and operational knowledge with the focus on commercialization of the process technology.

In 2000 two additional pilot plants were installed in research and development institutes in Europe and in Asia. In 2003 the first commercial semi-industrial LIST Lyocell Dissolving system was ordered for installation in Asia with start-up scheduled for mid-2005.

Current Situation of Development

Single Step Cellulose Dissolving

In contrast to the huge market for economical man-made fibers for textiles, there are some applications in special, small demand, niche applications for films, filaments, and fibers. For these applications a multi-step process is not economically feasible. Consequently, LIST and TITK developed, and patented, a single-step dissolving technology.

Throughout the process development, both TITK and LIST gained tremendous knowledge of the physical cellulose dissolving process. With annual capacities below 1000 metric tons per year, the LIST single-step dissolver can be applied for an efficient and economical process solution.

A New Generation of LIST Dissolver

Beginning with the LIST DISCOTHERM B CONTI as a base equipment platform, the LIST Lyocell Dissolver has evolved through several generations of optimization. Each generation has improved the process efficiency and performance. The last generation is being currently fabricated.

Increasing Capacity per Line

The current design maximizes the operating capacity per line and guarantees the highest economy and lowest costs per kg fiber. Today the LIST Cellulose Dissolving System is able to produce up to 10'000 t fiber /year and line. The production capacity is depends on the cellulose content in the spinning solution and the composition of the used solvent.

Specialized Auxiliaries

The thixotropic nature of the cellulose pre-mix and the operation of the LIST dissolver under vacuum, demanded for a specialized auxiliaries. LIST designed a specialized feed system able to continuously feed the pre-mixed cellulose from atmospheric environment into vacuum. Further more, LIST optimized the process of

discharging the cellulose dope from the Dissolver combining degassing of the product, pressure build-up for material transport, and discharge from vacuum into over pressure conditions. These designs are integral parts of the complete LIST process solution.

Simulating the Dissolving Process for scale up

LIST possesses process simulation routines for the design of the complete range of plants starting from the pilot scale. The results are the required equipment sizes, the energy consumption, and the investment cost estimates. These process models are invaluable tools for the on-line process simulation and for evaluating the impact of process variables on the operation of the units.

As the market demand for Lyocell fibers increases, efficient and safe manufacturing processes must be in place to meet the production requirements. LIST has many years of experience in the design of cellulose pre-mixing and dissolving equipment and continues to develop and optimize its technology in this field.

$$Q_{\text{water evaporation}} = Q_{\text{mechanical}} + Q_{\text{contact}}$$

$$m_{\text{water}} \cdot \Delta H_{\text{water}} \quad \text{Shear effect} \quad A_{\text{heated}} \cdot \Delta T$$

Fig. 2 – Main Parameters for LYOCCELL Dissolving



LIST LYOCCELL DISSOLVING SYSTEM

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Process Flexibility and Process Safety

LIST AG and the Thuringian Institute for Textile and Plastic Research (TITK), working in conjunction since 1992, developed a specialized Lyocell Dissolving Technology with two process goals:

1. production of the highest quality spinning solution for the production of fibers, films, filaments, and other special technical textiles, and
2. minimization of operational hazards inherent to the use of N-methylmorpholine-N-oxide (NMMO)

The LIST process consists of two process zones within the single dissolving unit. The first zone is the suspension zone where heat transfer is controlling. The second zone is the dissolving zone characterized by the increase of the viscosity of the spinning solution. The supply of energy in this zone is controlled by mechanical energy input. Cellulose would only dissolve at a specific water and solvent concentration and product temperature. As a consequence, the control of these parameters must be very accurate in order to produce excellent quality spinning solution as well as to guarantee the safe operation.

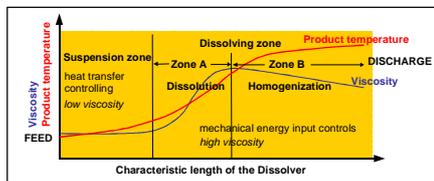


Fig. 3 – Temperature and viscosity profile in Dissolver

Process Flexibility

The LIST Lyocell Dissolving Technology has a significant advantages over other Lyocell technologies in its process flexibility. As a function of the LIST equipment design, the process can easily be adjusted to different cellulose raw materials without sacrificing the high quality of the spinning solutions produced or the rate of solvent recovered and recycled back to the process.

Adjusting of swelling and dissolving time

The large volume LIST Dissolver allows flexibility in extending or reducing residence time as necessary for the raw material used. In addition, another key aspect of the LIST technology is the independence of shaft rotational speed to product throughput. Shaft speeds can be adjusted without impacting the capacity of the LIST unit. This feature provides incredible control of the

process energy balance.

Different raw materials

Ultimately, this process flexibility results in the ability of the LIST Lyocell Dissolver to make the same high quality spinning solution – and high quality fiber – from cheaper raw materials that have different cellulose contents and are much more difficult to dissolve.

Additive adding trough dissolving

The larger hold up used for adjusting the swelling and dissolving time and the perfect mixing function of the Dissolver can be used for adding organic or inorganic additives without adding an additional process step.

Solvent Recycling rate >99,9 %

The cost efficiency of the Lyocell process, when compared to rayon, is dependent on the solvent recycle rate. The closed design of the LIST Lyocell Dissolver allows for complete recovery of the NMMO solvent. Because the LIST process operates at lower temperatures, there is less decomposition of NMMO. Therefore, the solvent can be easily recovered via distillation and recycled back to the process with minimal losses.

Process Safety

While process efficiency and flexibility is important in every industrial manufacturing process, operational safety is tantamount to its successful integration into the manufacturing environment. The LIST Lyocell Dissolving process takes this into account and has been designed to address the particular safety concerns surrounding the use of NMMO as a solvent with its temperature controlled run away reaction properties. The LIST system includes highly sensitive temperature and energy input control systems and an automatic emergency process shutdown for a system that puts safety first.

NMMO will begin to decompose at elevated temperatures. The initiation temperature of this decomposition reaction is dependent on the impurities present. With a recycle rate of up to 99%, these impurities eventually accumulate and reduce the decomposition temperature creating an unsafe operating condition. Special studies conducted at a temperature of 130°C showed a rapid increase of pressure to greater than 100 bar pressure as the NMMO decomposed.

Safety Philosophy

Today, stabilizers are used to increase the temperature at which NMMO decomposes, but the reaction cannot be eliminated completely and must be addressed. The LIST system operates at temperatures less than 100°C to ensure that the heating media and the product maintain a large enough temperature difference to the decomposition temperature of NMMO. The system is also outfitted with many temperature sensors that constantly monitor process temperature for safe and stable operation.

LIST – Automatically shut down system

Regardless of the extent of process control, it is always possible that an upset condition could drive the process temperatures into the range where the NMMO decomposition reaction will begin. To address this possibility the LIST Lyocell Dissolving system has an integrated water flooding system. The rapid introduction of water into the process chamber breaks the vacuum and absorbs a tremendous amount of heat thus quickly lowering the product temperature and preventing the further decomposition of NMMO. Because the LIST equipment is oriented horizontally with a fill level of approximately 50%, the water will distribute itself along the entire length of the unit thus preventing hot spots.

Summary

LIST AG has developed a viable process for the production of high quality fibers, films, filaments, and special technical textiles from different cellulose raw materials. The design of the LIST system allows greater flexibility and an increased level of safe operation. As the demand for cellulose-based fibers grows, LIST will continue to develop and expand its technology to meet this demand.

LIST AG continues to develop and optimize this technology focusing on increasing capacity and the use of cheaper, more abundant, cellulose raw materials.