

# Continuous dissolution process of cellulose in NMMO

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At the beginning of this century a new dissolution process for cellulose spinning solution was developed. As basis for the new process served the classic cellulosic fiber production process (derivatization of cellulose by carbon disulfide). In the last 30 years or so the conventional viscose process became environmentally critical. Evaluated with regard to its environmental viability it was found to create considerable water and air pollution. This conclusion ignited the research and development of new technologies with less environmental impact.

The classification of performed R&D work shows two main directions:

- Processes similar to the classic viscose process: Hereby the used solvent is changed and the dissolution process discontinuous.
- Processes similar to mass polymerization: Hereby the used solvent is also changed and the dissolution process is continuous. The well known fiber produced with these processes and NMMO solvent is internationally registered as lyocell.

The implementation of the continuous dissolution process of cellulose in NMMO (N-methyl morpholine oxide) has been effected by means of the mixing/kneading technology, and thin film technology.

The main difference of these two technologies is the processing equipment, which is applied for the dissolution of cellulose in NMMO.

## Continuous cellulose dissolution process applying the mixing/kneading technology

The dissolution stage, within the fiber production plant, through the application of the mixing and kneading technology, is realized in two steps. The first step foresees the conditioning of raw materials in a mixer/kneader conditioner. The second step is the dissolution of cellulose in NMMO in a dissolver. Fig. 1 shows the principle concept of the dis-

solution process. Further to the conditioning and dissolution stages the spinning unit and the distillative recovery of NMMO are overall operations completing the principle concept of an integral production plant.

The conditioner, a continuous mixer/kneader, mixes and homogenizes cellulose with NMMO under specified conditions. The homogenized final mixture serves as feed stream to the dissolver. The dissolution step comprises the continuous evaporation of water, which takes place in the dissolver, a kneader/evaporator. Simultaneously the cellulose is dissolved and the processed mass is homogenized to the spinning solution. Following the phase diagram of NMMO process (Fig. 2) it is apparent that the dissolution of cellulose in NMMO takes place only when the water content lays within the shadowed surface of this diagram. Water evaporation is accomplished by both contact heat transfer and dissipated energy. The homogenization is considerably enhanced by shear effects taking place in the process chamber of the dissolver. Both process steps are realized by means of continuous kneaders by List AG, Arisdorf/Switzerland.

## Mixing/kneading technology

List's proprietary technology is synonymous to mixing/kneading equipment for thermal processing of highly viscous materials. The technology provides equipment of large working volumes with optimal combination of intensive mixing/kneading and extensive self cleaning heat exchange surfaces. Fig. 3 shows the position of the technology in respect of application and in relation to other types of processing equipment.

Explicitly the ben-

efits of List mixers/kneaders are:

- processing of heterogeneous product phases in a single unit
- high interface renewal rate ensuring effective mass transfer
- efficacious combination of static and dynamic elements in the process chamber lead to high degree of shear homogenization
- high energy flux and close control of product temperature
- large production capacities in a single unit
- closed, contained design guarantees low operational risk when handling dangerous materials
- product axial conveying rate is independent of agitator speed
- wide range of retention time (10 min to several hours)
- large area to volume ratio.

## Cellulose dissolution process

Cellulose is an interesting polymer produced from a renewable raw material. The development of an alternative cellulose fiber opens new horizons for an economically advantageous and environmental friendly mass fiber technology. This is accomplished through the

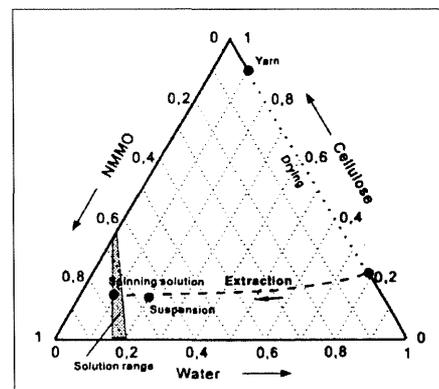


Fig. 2 Phase diagram of NMMO process

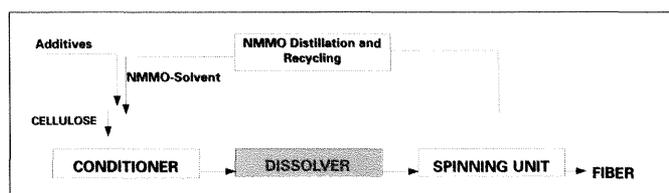


Fig. 1 Block diagram of the continuous dissolution process of cellulose in NMMO

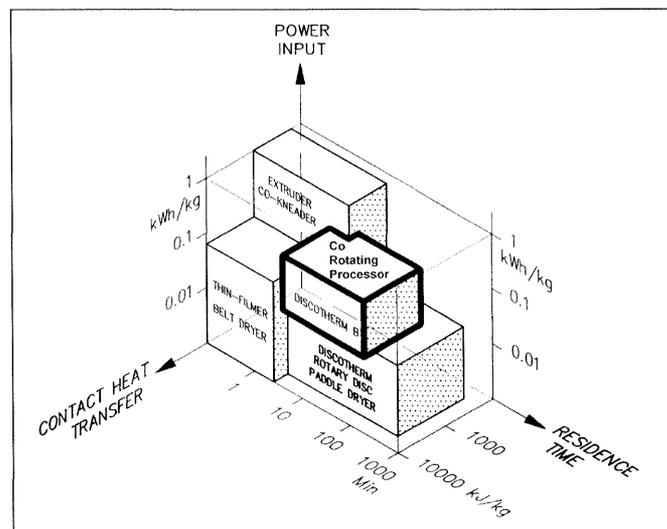


Fig. 3 Positioning of List technology

vertical integration of the new process, i.e., from the raw material to the final fiber, and the limited use of consumables. An important parameter influencing the economy of the new process is the cellulose content in the spinning solution. The application of List processing equipment enables the production of cellulose spinning solutions with a cellulose content of 18% by weight and higher.

The cellulose dissolution in NMMO for the continuous production of a cellulose spinning solution by means of high viscosity processing technology comprises two process steps:

*1. Continuous conditioning of the feed materials in the List Co-Rotating-Processor (CRP, Fig. 4) featuring:*

- mixing and homogenization of cellulose and NMMO, two raw materials of heterogeneous consistency
- activation of dope under atmospheric pressure and temperature in the 80-85°C range
- mixing the dope with additives, whenever and as required
- constant quality of the conditioned product stream with regard to its composition
- quick, effective thermal conditioning of the mixture avoiding thermal degradation of the feed material.

A major comparative advantage of the continuous conditioning step is the possibility to dye the feed stream, thus saving the necessary post dyeing operation.

*2. Continuous dissolution of cellulose in the Discotherm B - Fiber (Fig. 5) featuring:*

- water evaporation till the dissolution conditions of water concentration and product temperature have been reached (see shadowed surface on Fig. 2)
- dissolution and homogenization under vacuum, enhanced by the shear effect of the internal geometry of the dissolver, at product temperature of 90-120 °C (energy supply by contact heat transfer and friction)

- high cellulose content in the spinning solution, thus anticipating low energy consumption for the necessary water evaporation
- minimization of the necessary NMMO quantity for the dissolution of cellulose, thus simultaneously minimizing the costs of its distillative treatment.

The comparative advantages of the continuous dissolution step are:

- reduction of energy spent for water evaporation
- costs savings for the distillative treatment of the NMMO solution
- increase of production capacity per unit volume of installed processing equipment and unit weight of NMMO, due to higher cellulose concentration in the spinning solution
- reduction of investment costs due to the size reduction of peripheral units, which deal with water condensation, distillative treatment of NMMO and its recycling.

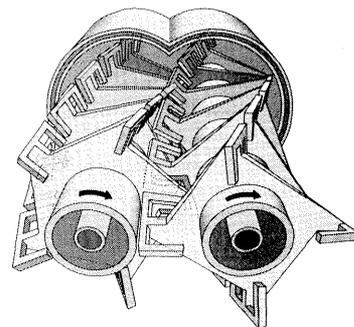
From Fig. 6 it can be deduced that between the conditioner (CRP, Fig. 4) and the dissolver (Discotherm B-Fiber, Fig. 5) there exists an agitated buffer tank. This intermediate stage is necessary in order to ensure the continuous operation between two consecutive stages operating at different pressures. The conditioner is operating under atmospheric pressure, while the dissolver works under vacuum. The sealing item between these two stages is the twin piston valve, which is installed as discharge device of the buffer tank and simultaneously as feed device to the dissolver. Further it can be seen that the conditioned mixture of cellulose/NMMO is discharged from the conditioner into the buffer tank by gravity. The spinning solution, which is a highly viscous material, is discharged from the dissolver by means of a process-tailored twin screw.

The application of this mixing/kneading technology grants several comparatively competitive advantages to the continuous dissolution process of cellulose in NMMO. They are directly deduced from the benefits of this technology and are summarized as follows:

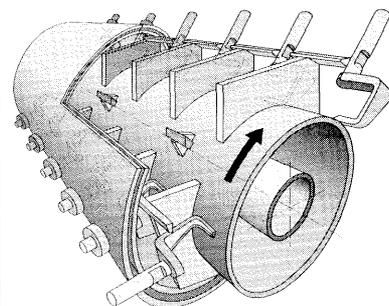
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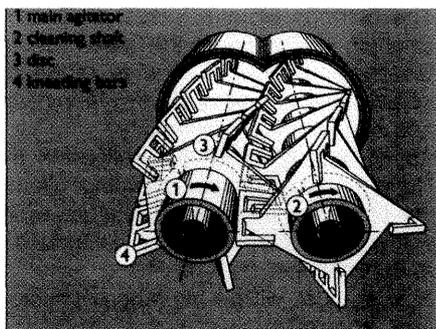


Fig. 4 Co-Rotating Processor (List)

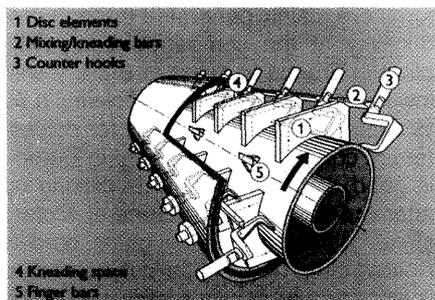
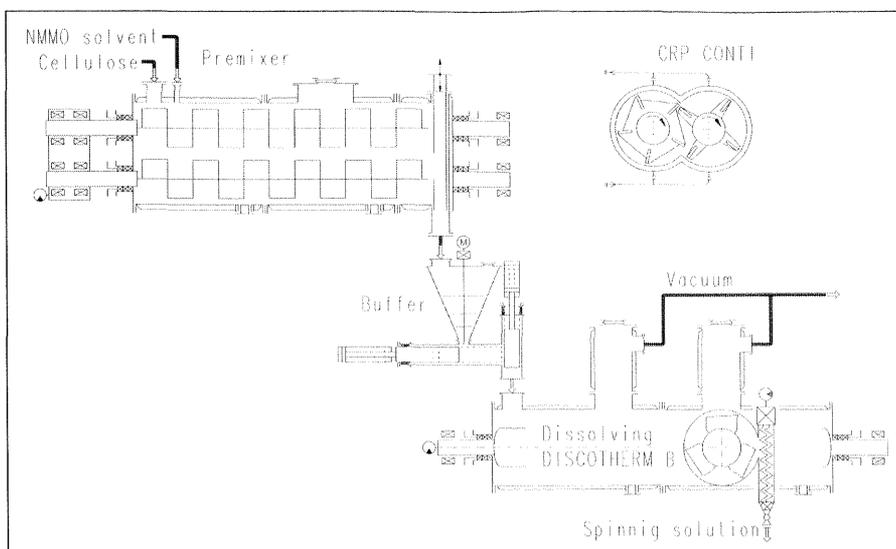


Fig. 5 Discotherm B Fiber

1. Retention time in the 10 min to 2 hours range allowing for
  - operational safety due to the low dissolution temperature ( $< 120^{\circ}\text{C}$ )
  - avoiding solvent decomposition
  - prevention of solvent discoloration
  - flexibility towards the use of various raw materials with different dissolution behavior
  - adjusting the process to meet varying target product qualities.
2. Large volume unit allowing for
  - large production capacities in a single production line
  - good control of fluctuating feed conditions with respect to composition and rate
  - good control of product temperature profile.
  - Therefore, a well adjustable process due to the inertia of the large hold-up.
3. Large surface area to volume ratio allowing for
  - compact installation even for large production capacities.
  - Therefore, investment savings due to low space requirements.

### Concluding remarks

Beside the existing technologies at Courtaulds and Lenzing, TITK (Thüringisches Institut für Textil- und Kunststoff-Forschung) developed an-



**Fig. 6 Process flow diagram of the continuous dissolution process of cellulose in NMMO by means of List mixing/kneading technology**

other technology through the application of List mixing/kneading technology for the production of lyocell fiber, filaments and foils under the trademark "Schwarza Lyocell".

As early as 1992 started in Rudolstadt the first tests with List mixing/kneading equipment. Since 1994 a continuous lab-scale production plant has been successfully operated by TITK. It serves the continuous investigation and optimi-

zation of operating parameters and product formulations.

Mid 1998 Alceru Schwarza GmbH, Rudolstadt, started the operation of a pilot plant with an annual capacity of nearly 300 t fiber. The conditioner and dissolver were delivered by List. The target of this pilot plant is to prove the comparative advantages of Alceru process and to bring it to a worldwide commercialization. □