Concentration Measurement of DMAc and DMF

Relevant for: Pharmaceutical, plastic, agrochemicals, dyes, coatings and textile fibers

DMAc and DMF are organic solvents commonly used in chemical synthesis of pharmaceuticals and agrochemicals and in the manufacturing of artificial fibers and artificial leather. The concentration of the precipitant or solvent recovery stream is a key parameter and provides best data for quality control.

1 Introduction

A variety of chemical reactions are best carried out in N, N-dimethylformamide (DMF) or N, N - dimethylacetamide (DMAc). With the high dissolving power characteristic, they are used in the production of pharmaceuticals, plasticizers, adhesives, synthetic leathers, fibers, films, surface coatings, inks and dyes.

The interest in these polar solvents is because of their high dissolution power of various classes of substrates and their capacity to solvate anions. Both DMF and DMAc are implied in a multitude of reactions in giving one or more of their own atoms to build the framework of the synthesized products.

Both are miscible with water and many other organic solvents.

Anton Paar's inline refractometer L-Rix 5100 has proven to be well suited for the continuous concentration monitoring of DMAc and DMF and can be directly installed into the production line or tank to measure concentration (%) in real-time.

2 Use cases for concentration monitoring

Membranes and filters for dialysis

Innovative membranes or filters filtrating the patient's blood can reduce the risk factors for these diseases. An important parameter for the effectiveness of membranes is their performance, which can be set by the composition of the precipitant. The precipitant - depending on the application – consists of different active substances, in this case a commonly used solvent DMAC (dimethylacetamide), in combination with water. The concentration ratio is an important quality parameter.

In particular the refractive index (RI) is suitable for measuring the concentration of DMAC in water as shown in Figure 2.

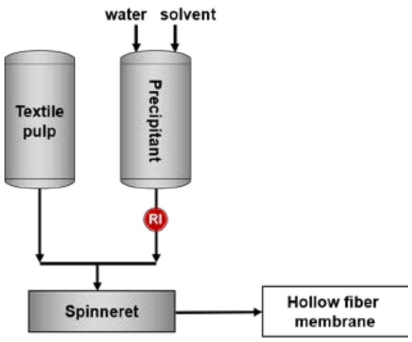

Figure 1: Production of fibers for membranes and filters

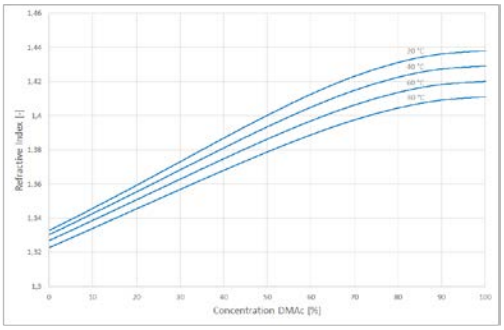

Figure 2: Concentration DMAc versus RI

Production of precursors for carbon fibers

Polyacrylonitrile (PAN)-based carbon fibers are used as important reinforcement materials in many applications. PAN as a precursor polymer is used in 90 % of the world's carbon fiber production. Since the carbon fiber inherits the characteristics of the precursor fibers, they play an important role in carbon fiber quality.

In the process the acrylonitrile in powder form is dissolved in an organic solvent to form the precursor "dope," with a consistency of maple syrup.

The most common solvent used in industry is DMF as it is also used in the polymerization step, allowing the polymer to be directly fed into the spinning line.

After polymer synthesis, the polymer solution with about 25 % of polyacrylonitrile is prepared for spinning.

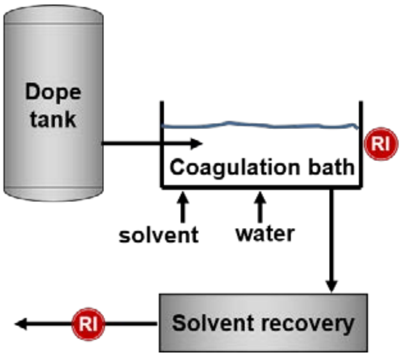

Figure 3: Production of PAN for carbon fibers

In a process called wet spinning the dope is immersed in a bath of solvent and water (coagulation bath) and extruded through holes in a spinneret. The polymer coagulates to solid fibers, which can be conducted to the washing and stretching step, before they are dried and winded up.

The coagulation bath temperature and concentration are the most important factors that decide the desired fiber microstructure. Low solvent concentration promotes rapid solvent extraction, resulting in thick skin and the formation of macro-voids. A high concentration of solvent in the coagulant gives a denser microstructure, but the solvent extraction is slow.

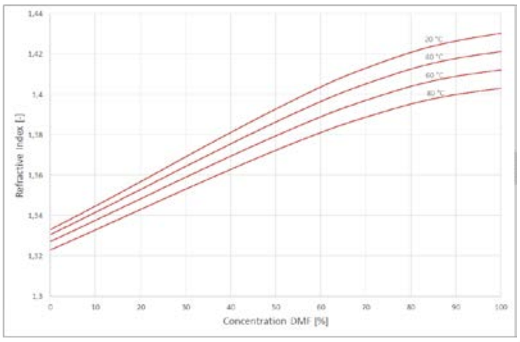

Figure 4: Concentration DMF versus RI

3 Measurement Setup

The Anton Paar solution for concentration measurement of DMAc and DMF consists of the L-Rix 5100 Inline Refractometer which is installed directly in the line or tank and measures the refractive index and temperature continuously.

Figure 5: L-Rix 5100 Inline Refractometer

The application-specific concentration calculations are carried out in the mPDS 5 or with the Pico 3000 (optional also with HMI) evaluation units. The results can be displayed and transferred to a PLC or to the Davis 5 data acquisition and visualization software.

Alternatively, the L-Rix can be connected to a Pico 3000 RC housing for remote control (for a single production line).

Specifications of L-Rix 5100:

Refractive Index	1.3100 to 1.5400 Accuracy: nD ±0.0002 (equivalent to ± 0.1 % mass) Repeatability: nD ±0.0001 (equivalent to ± 0.05 % mass)
Process temperature	-20 °C to 120 °C CIP/SIP up to 145 °C for 30 minutes
Ambient temperature	-20 °C to 60 °C
Pressure range absolute	100 mbar to 16 bar (10 bar @ >120 °C)
Communication (using Pico 3000)	Analog/Digital, Modbus RTU, PROFIBUS DP, PROFINET IO EtherNet/IP

4 Benefits

The reliable and accurate L-Rix 5100 enables

- Precise monitoring of the DMAc or DMF concentration in real-time
- Improved end-product consistency
- Optimization and control of the wet spinning process
- Stable measurement not influenced by bubbles
- Optimization and control of the recovery process
- Direct comparison with lab reference method

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