VAISALA

Liquid concentration measurement in chemical and textile fibers

eBook for chemical and textile fiber fabricators and processors

www.vaisala.com

Table of contents

Introduction

This eBook provides information on process optimization and in-line measurement instrumentation for fiber making and textile industries.

It gives a brief overview of different man-made fibers and fiber forming technologies. The eBook also takes an in-depth look into the most common fiber and textile processes where in-line refractive index measurement technology can be applied, with a particular emphasis on the automatic and precise control of various raw materials and solvents.

Examples of benefits, such as energy and material savings in different processes, are provided. A short section dedicated to process refractometers and their measurement principle is also included.

The eBook concludes with a summary of generic applications in chemical industry process units.

Contact our expert team to discover our full offering and discuss how we can help you to improve your process and applications.

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An overview of chemical fiber processing and fabrication

What are chemical fibers?

Chemical fibers are fibrous materials produced from organic and inorganic raw materials in a chemical process. The organic materials may be natural or synthetic polymers, while the inorganic compounds include glass, metal, basalt, quartz, and other composites.

Man-made fibers should be distinguished from natural fibers such as silk, cotton, and wool. The chemical composition, structure, and properties of man-made fibers become significantly modified during the manufacturing process.

This eBook focuses on process and quality control applications in the industrial manufacturing of man-made fibers, including those made from both organic, e.g. cellulose-based natural, polymers and synthetic polymers.

We walk through the chemical fiber making and spinning processes and, in the final chapter, introduce process control applications in textile wet treatments.

Man-made fibers made from natural polymers

Natural fibers consist of polymers: regenerated compounds such as cellulose and protein. Some man-made fibers are derived from naturally occurring polymers, e.g. rayon and acetate from cellulose polymers.

- Viscose rayon
- Cuprammonium rayon
- Cellulose acetate
- Lyocell
- Modal
- Other natural polymers such as alginic acid can be made into sodium alginate fibers in the fiber-forming process.

Man-made fibers made from synthetic polymers

Synthetic fibers are made from the synthesized polymers of small molecules. The compounds that are used to make these fibers come from raw materials such as petroleum-based chemicals or petrochemicals.

- Polyester
- Rayon
- Nylon
- Polyolefin
- Acrylic and modacrylic

Typical fiber end uses

- Clothing
- Home textiles
- Industrial uses: electric wire casing, canvas, etc.

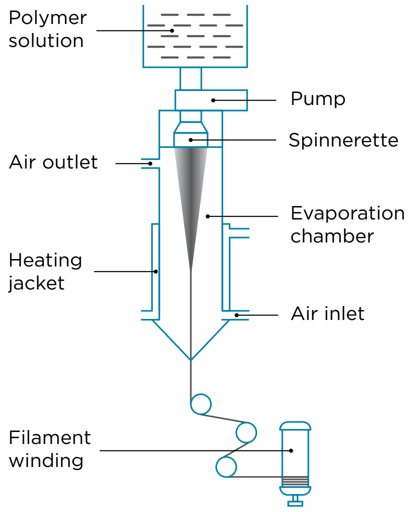
Fiber spinning technologies

Before polymer can be converted into fiber it must first be converted into a liquid or semi-liquid state, either by being dissolved in a solvent or by being heated until molten. This process frees the long-chain molecules from close association with one another, allowing them to move independently. The resulting liquid is extruded through small holes in a device known as a spinnerette, emerging as fine jets of liquid that harden to form solid rods with all the superficial characteristics of a very long fiber or filament. This extrusion of liquid fiber-forming polymer, followed by hardening to form filaments, is called spinning.

Several spinning techniques are used in the production of man-made fiber, including wet and dry solution spinning and melt spinning.

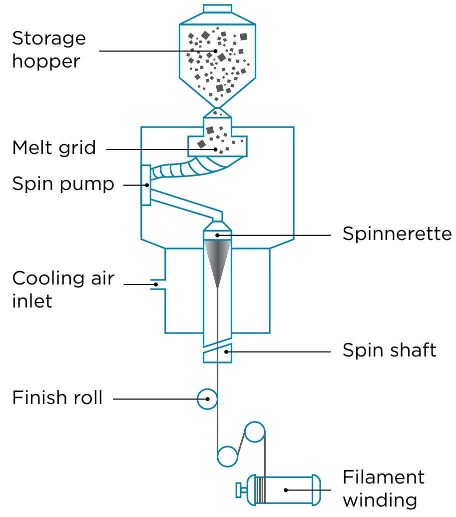
Dry spinning

The fiber is made in a heated tower. The solvent evaporates, leaving behind the fiber.



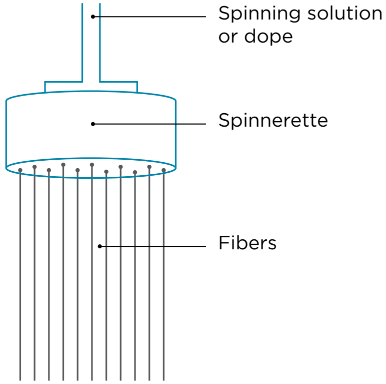
Melt spinning

Viscous melted polymer is extruded through a spinnerette. The filament is then solidified using cold air.



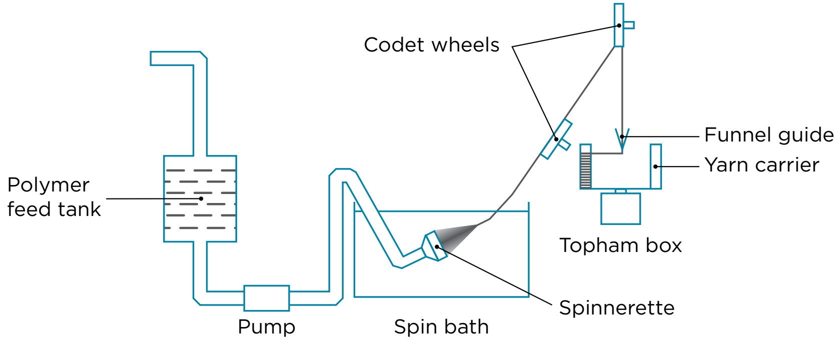
Polymer solution (spinning dope)

A small thimble-shaped metal nozzle with fine holes through which a spinning solution is forced to form a filament.



Wet spinning

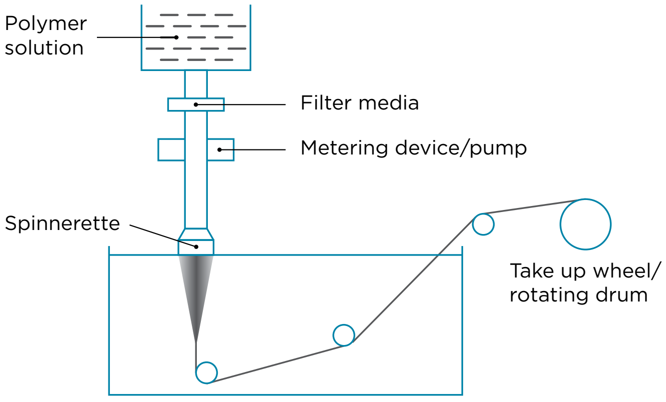
The fiber is made in a bath. The solvent diffuses out of the extruded material creating a solid fiber.



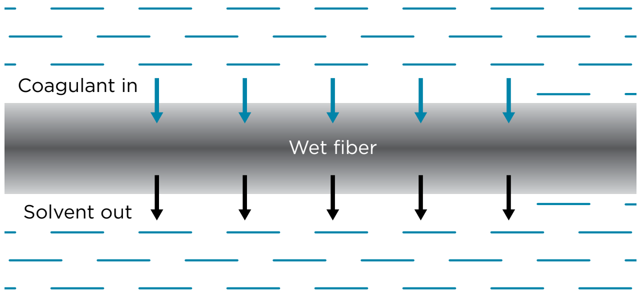
Fiber formation

Dry and wet spinning operations require a spinning solution which is made by dissolving the polymer in a solvent; this solution is also known as the dope. In dry spinning, the fiber is then formed by evaporating the solvent from the dope into the surrounding air. In wet spinning the dope solution is passed through a spinnerette and the fiber is formed as follows:

- **desolvation**, where the non-solvent material in the spinning bath diffuses into the filament and the solvent diffuses out of it,
- **chemical precipitation**, where a chemical reaction coagulates and hardens the filament, or
- **ion-exchange between the dope solution and bath**, which results in a non-soluble fiber component.



Wet spinning process schematic.



In wet spinning the dope solution precipitates and forms a solid fiber as it is submerged in a chemical bath.

A green approach accelerates fiber and textile process optimization

Global challenges of fiber and textile manufacturing

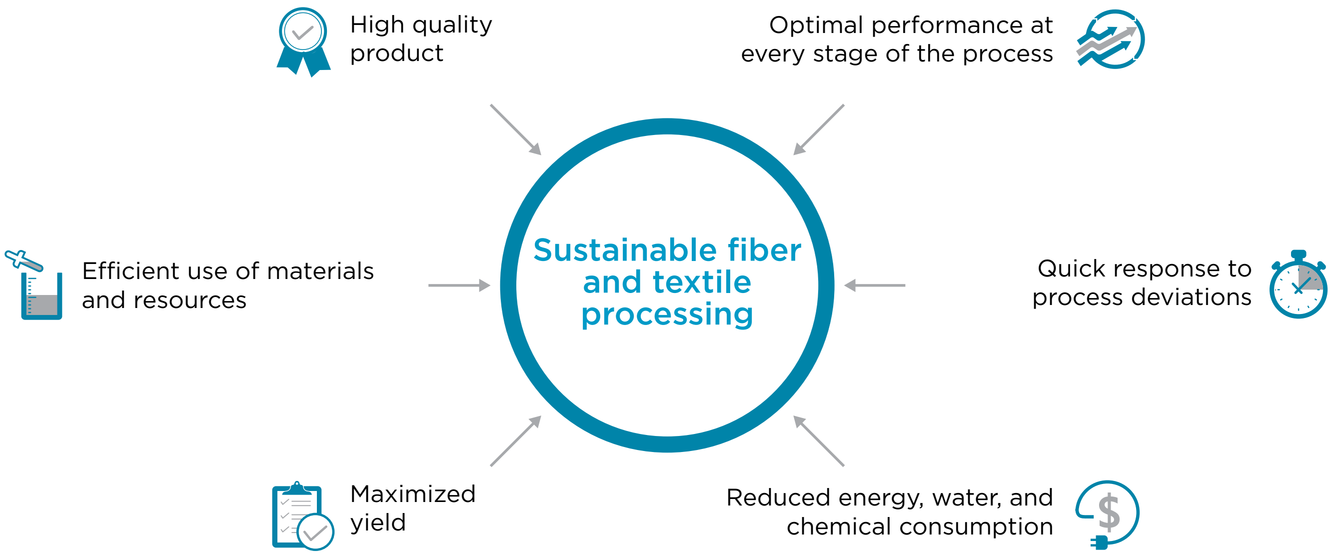
The demand for fibers and yarn is expected to grow dramatically over the next decade, thanks to the global increase in both population and prosperity. In general, wet fiber and textile processing is heavily dependent on the excessive use of chemicals in the form of solvents, finishing agents, and other related auxiliaries. Wet textile processing also requires a huge amount of water at each step of every process, starting from preparatory treatments until final finished goods.

Public awareness is growing about the impact of the fiber and textile industry, for example the recognized environmental challenges of organic solvents which tend to be harmful to the environment and have adverse effects on ecosystems and freshwater. Fiber processing industries need to listen and respond to public concerns, as the consequences of inattention to these issues could risk companies' brands, and ultimately even their economic raison d'etre.

To garner public acceptance, the whole industry needs to drive continuous improvement and transform to more sustainable fiber manufacturing. Mitigating the environmental impact calls for actions to optimize processes, for instance in:

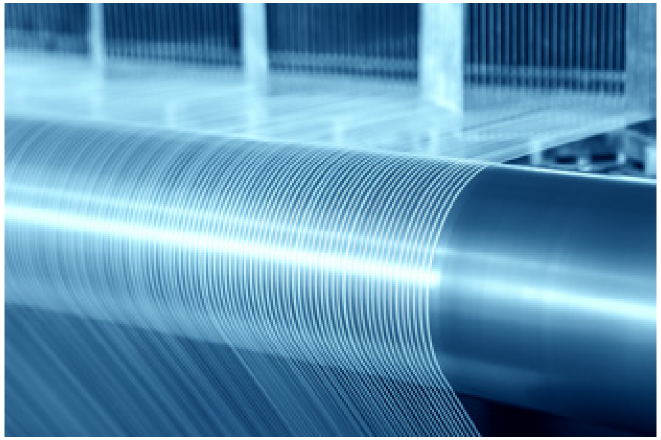
- Chemicals management, water usage, and waste policy
- Energy efficiency
- Environmental efficiency, for instance by maximizing yield – a very important indicator of environmental efficiency in fiber processing
- Material and resource efficiency, for instance more efficient use of solvents via improved product and product precursor recovery through solvent recycling

By improving their environmental performance and processing efficiency, fiber companies can enhance their reputation, meet growing demand, and protect our environment.



What producers can do – the course of future development

An increasing trend towards green chemistry attempts to encourage fiber and textile processors to implement automation and process control to help improve overall process efficiency and ensure high-quality final products while using less energy and reducing emissions and chemical waste. As a part of this greener approach, fiber and textile companies have recently also implemented plans to audit the environmental performance of their supply-chain partners.



“Nothing can be improved until it is measured.”

Measurements help to reduce the use of harmful chemicals

Efficient fabrication using accurate measurement technology presents an immediate opportunity to improve profits. Minimizing waste by recycling valuable solvents lowers the cost of disposal and reduces resource consumption in a sustainable way.

Implementing advanced automation technologies also places new requirements on process monitoring and instrumentation, particularly in chemical raw materials handling.

A process refractometer is an intelligent instrument that can be installed in-line to measure the liquid concentration of chemical raw materials. When integrated into an automation control system, it can dramatically help to improve fiber quality and stabilize production in the following operations:

1. Polymerization

- Follow the progress of the reaction
- Identify the end-point of the reaction in real time

2. Preparation of polymer, dope, or spinning solutions

- Ensure consistent fiber quality with consistent and optimal dope concentration
- Measure the dope concentration before spinning to check and control quality
- Measure and control the dope concentration (e.g. in the Lyocell fiber process), as this affects the dissolution of the polymer and the properties of the fiber

3. Spinning baths

- Measure the concentration of the spinning bath as the coagulant solution changes when the solvent diffuses out of the extruded material
- Control the dope concentration in real time and keep it at the optimal level for high-quality fiber formation
- The concentration of the dope affects the structure and properties of the fiber

Our response – better process control and optimization with precise measurements

Ecological and economical separation and recovery of solvents

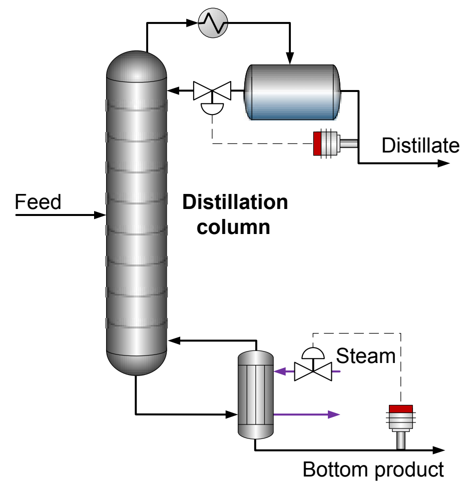
Solvent spinning and fiber formation processes use large amounts of dope solution containing both organic and inorganic solvents, of which 94–98 % are recovered and reused in the manufacturing process for environmental and economic reasons. Typically, recovery systems are used in fiber spinning bath operations as these contain the highest concentrations of solvent and allow for the most efficient and cost-effective solvent recovery. Commonly recovered solvents include:

- Dimethylformamide (DMF)
- Dimethylacetamide (DMAC)
- Acetone
- Di-isocyanates
- N-Methyl-2-pyrrolidone (NMP)
- Dimethylsulphoxide (DMSO)
- N-methylmorpholine-N-oxide (NMMO)
- Inorganic solvents, e.g. sodium hydroxide (NaOH)

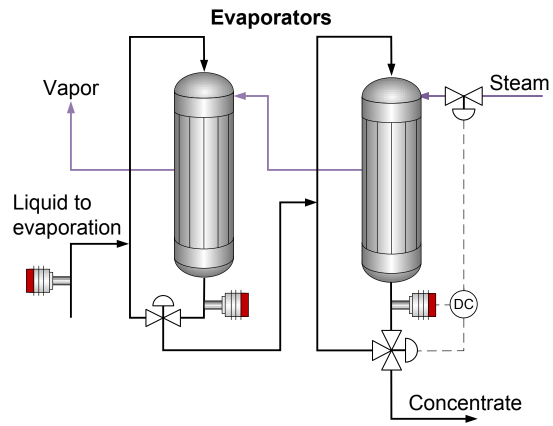
Recovery operations from wet spinning use, for example, distillation and evaporation. Distillation is the separation of the constituent parts of a liquid mixture using partial vaporization and subsequent condensation, taking advantage of differences in volatility. Control over the distillation process is essential to meet product specifications, maximize return on investment and energy efficiency, and limit environmental impact. These goals can be achieved by maintaining the purity of the column's top and bottom products within the specifications.

A refractometer can be used to monitor the concentration of the distillation products in real time. It is installed directly in-line after the column (bottom) or after the condenser (distillate). The refractometer output signal can be used to automatically adjust the column's reflux or boil-up to meet product specifications. In binary systems, the refractometer provides accurate information on product concentrations. In multi-component systems, the control can be based on a property, which is a function of the composition.

The refractometer is an ideal tool for solvent evaporation, as it provides real-time information about concentration changes in a medium. Concentration information at the inlet, outlet, and intermediate stages of the concentrator can be used to optimize the process. For instance, the refractometer output signals can be used to control the heat source flow (steam) for adjustments and to achieve the target concentration. If the concentration of the solvent is below specifications, the refractometer's signal is used to control the valves to either decrease the feed flow to the evaporator or to increase steam flow.



The refractometer is installed in-line after the distillation column (bottom) or after the condenser (distillate) to adjust the column's reflux or boil-up according to specifications, in order to maximize the solvent recovery rate for later reuse in the process.



The refractometer is installed in the inlet, outlet, and intermediate stages of the evaporator to control the heat source flow (steam) for adjustments and to achieve the target concentration of the solvent.

Lyocell (Tencel) process

Regenerated cellulose fibers (also known as rayon fibers) are made by chemically modifying the cellulose contained in wood pulp so that it can be dissolved and spun into fine fibers using a dope solution. Lyocell is a man-made fiber derived from cellulose, better known in the United States under the brand name Tencel. Though it is related to rayon, Lyocell is created using a solvent spinning technique, and the cellulose undergoes no significant chemical change.

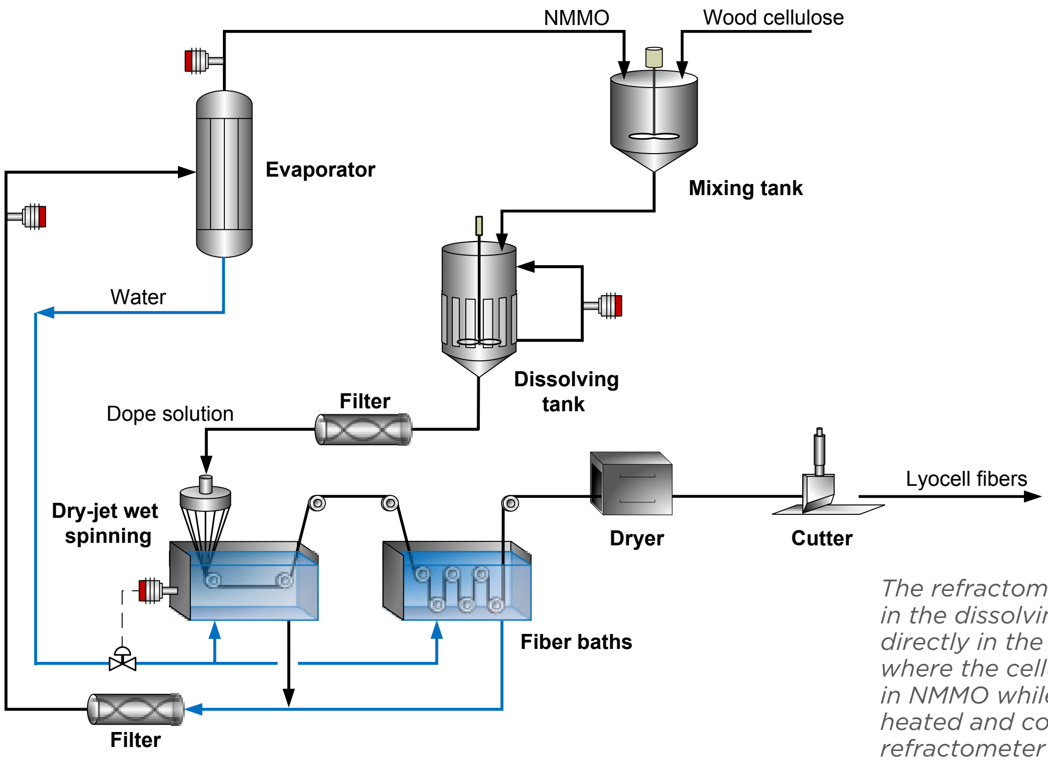
Production process

In the Lyocell process, wood chips are mixed with an aqueous solution of N-methylmorpholine N-oxide (NMMO) to dissolve the cellulose. The concentration and temperature of the solvent play a significant role in the process, e.g. if the water content is too high (above 15 %) the cellulose will not dissolve. The solution is initially prepared to 50-60 % NMMO and then concentrated using evaporation in a heated dissolving vessel to give a dope solution composed of about 76:10:14 NMMO:water:cellulose.

After dry-jet wet spinning, the coagulating liquid is purified and concentrated to recover the NMMO for reuse in the process, with a recovery rate of up to 99.5 % of the solvent. NMMO is a very costly solvent, and its recovery is essential for the economic viability of the process.

During coagulation, the solvent diffuses out from the doping solution increasing the concentration of the bath. The Vaisala K-PATENTS® Process Refractometer monitors the concentration of the dope solution and the solvent at various stages of the process, helping to:

- Maximize productivity and fiber quality
- Ensure that the correct concentration and complete cellulose dissolution is achieved; undissolved cellulose in the spinning dope results in poor spinnability and lower fiber quality
- Maintain the spinning bath at the optimal concentration through the addition of water
- Maximize the solvent recovery rate for later reuse in the process



The refractometer is installed in the dissolving tank outlet or directly in the dissolving tank where the cellulose is dissolved in NMMO while the solution is heated and concentrated. The refractometer can be installed in large and small pipes or directly in vessels.

Cellulose acetate fiber production

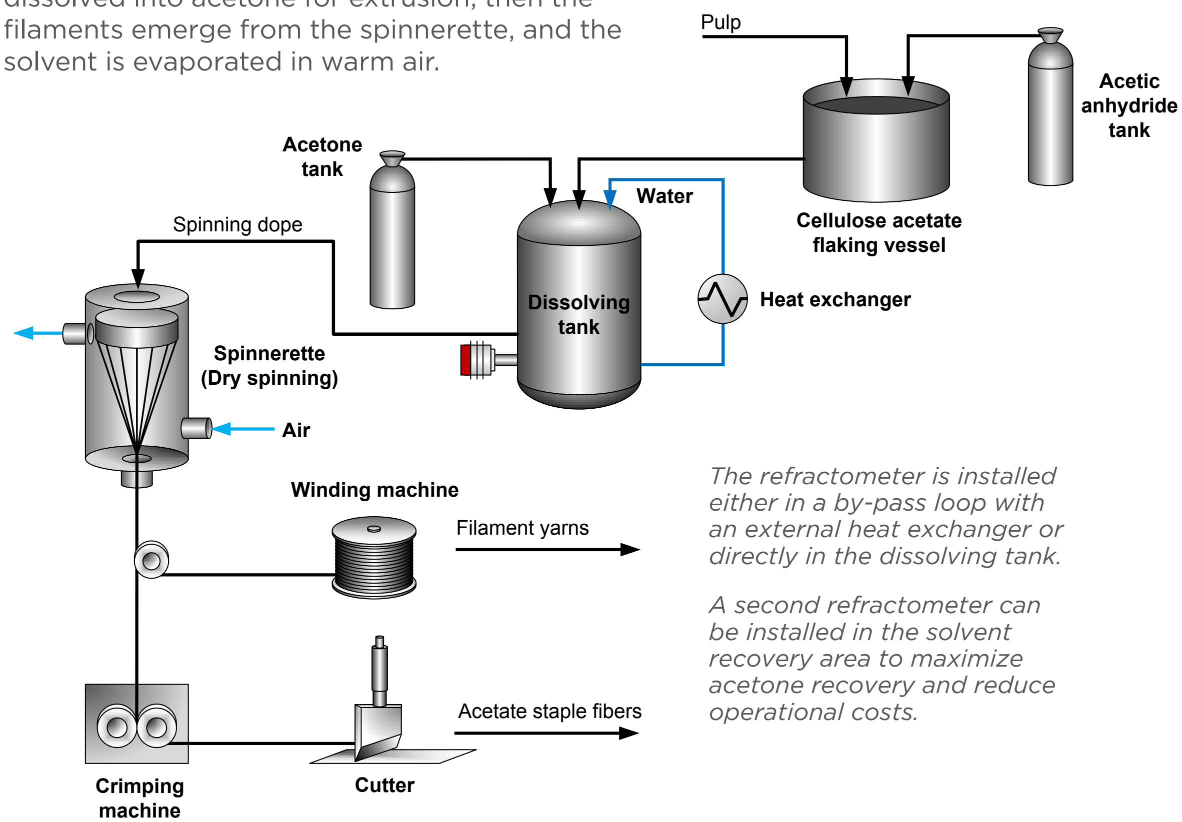
There are two types of cellulose-based fibers: regenerated or pure cellulose (e.g. from the cupro-ammonium process) fibers and modified cellulose (e.g. cellulose acetates and rayon) fibers. Acetate fiber is a synthetic fiber, in which the forming substance is cellulose acetate. When no less than 92 % of the hydroxyl groups are acetylated, the term triacetate may be used as a generic description of the fiber.

Acetate is derived from cellulose by breaking down wood pulp (dissolving pulp) into purified cellulose. Cellulose acetate dope is produced by reacting the purified cellulose with acetic acid and acetic anhydride, whilst using sulfuric acid as a catalyst. The cellulose acetate flakes are dissolved into acetone for extrusion, then the filaments emerge from the spinnerette, and the solvent is evaporated in warm air.

Production process

Purified cellulose from wood pulp or cotton linters is mixed with glacial acetic acid, acetic anhydride, and a sulfuric acid catalyst. The mixture is put through a controlled 20-hour partial hydrolysis to remove the sulfate and the required amount of acetate molecules to obtain the desired properties. In this step a precipitate of acid-resin flakes is created. The flakes are dissolved in acetone and the solution is filtered.

The resulting solution is the spinning dope or spinning solution. The spinning solution is extruded in a column to form filaments, which are dried with dry air. The solvent is recovered and purified for reuse. The filaments are stretched and wound onto beam cones or bobbins to obtain the final product.



The refractometer is installed either in a by-pass loop with an external heat exchanger or directly in the dissolving tank.

A second refractometer can be installed in the solvent recovery area to maximize acetone recovery and reduce operational costs.

Extrusion and spinning

After being formed, cellulose acetate is dissolved in acetone for extrusion. As the filaments emerge from the spinnerette, the solvent is evaporated in warm air (dry spinning) producing fine filaments of cellulose acetate.

The liquid substance of cellulose is forced through a spinnerette. The extruded filament gets solidified by a liquid bath as it emerges from the spinnerette. Filaments are then twisted together to form yarn. As the filaments emerge from the spinnerette holes, the liquid polymer becomes rubbery and then solidifies. This process of extrusion and solidification of endless filaments is called spinning.

While the extruded fibers solidify, or in some cases even after they have hardened, the filaments may be drawn to impart strength.

Instrumentation and installation

Traditionally, the dope has been measured by taking samples and analyzing them in a laboratory. This method is not optimal as acetone evaporates quickly, giving false results. Instead, the Vaisala K-PATENTS Process Refractometer can be used in the dissolving tank to monitor the concentration of the dope solution prior to the fiber spinning.

The refractometer measures and controls the dope concentration in-line. High product quality can be obtained by precisely maintaining the refractive index value within predetermined limits. Hazardous and intrinsic safety approvals are available when required.

Polyamide (nylon) fiber production

Nylon 6-6 is a man-made synthetic fiber resulting from the polymerization reaction of adipic acid and hexamethylene diamines. The name comes from the molecular chains of the two raw chemical components, containing six carbon atoms each.

Production process

The reaction between adipic acid and hexamethylene diamine produces hexamethylene diammonium adipate, commonly called nylon salt. It is essential for the material to be polymerized so that high-quality fibers with very few impurities can be achieved. Different types of nylon can be made using a variety of processes.

In the batch process, the hexamethylene diammonium adipate solution is concentrated in an evaporator and acetic acid is added to stabilize the chain length. After evaporation, the salt solution is heated and the remaining water removed. TiO_2 dispersion is added and the polymerization takes place. After the polymerization is completed, the viscous molten polymer is forced out through the bottom of the autoclave onto a casting wheel and extruded as rapidly as possible.

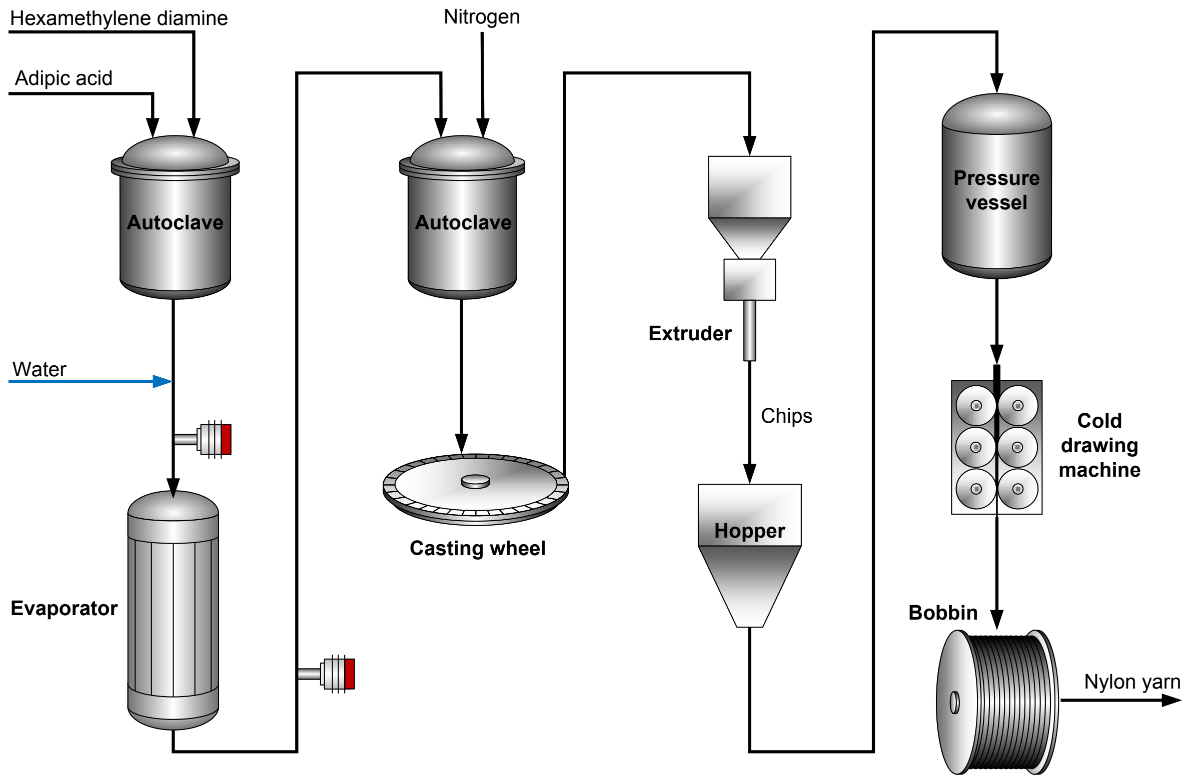
Nylon is also produced using continuous processing, which is more economical for high volume production, whereas the flexibility of the batch process permits more end-product variations.

Nylon 6, or caprolactam, is a polymeric fiber derived from only one constituent: caprolactam. It has a lower melting point than nylon 6-6 but has superior dyeability, elasticity, and resistance to light.

Instrumentation and installation

The Vaisala K-PATENTS Process Refractometer is used to measure and control the nylon salt, caprolactam, and polymer solution concentrations.

The refractometer is installed after the evaporator to ensure the target concentration is achieved. Another refractometer can be installed before the evaporator for real-time monitoring of evaporation efficiency. The refractometer output signals can be used for automatic evaporation control, for instance by regulating heat flow or the feed to the evaporator.



Refractometers are installed in the evaporator inlet and outlet to ensure target concentration and monitor evaporation efficiency.

Polyurethane elastic (spandex) fiber production

Spandex is the generic name for a synthetic fiber whose fiber-forming substance is a long chain of a synthetic polymer. It comprises at least 85 % segmented polyurethane. Common trade names for spandex are Lycra, Dorlostan, Spanzelle, and Vyrene.

Production process

Typically, the spandex fiber structure is achieved by reacting di-isocyanates with long chain glycols, which are usually polyesters or polyethers.

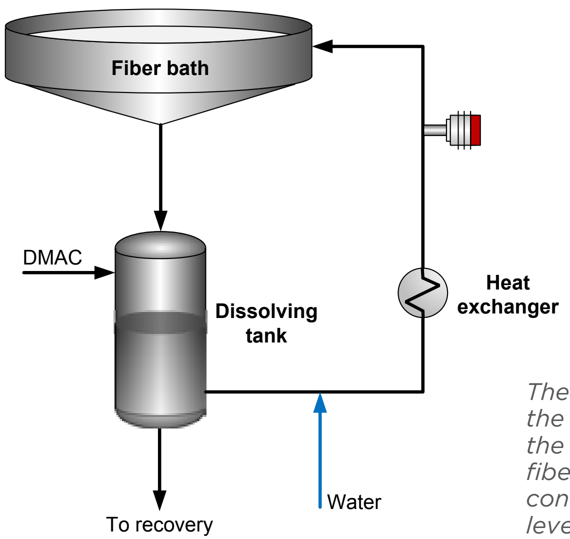
The polymer is dissolved into dimethyl acetamide (DMAC) and then chain-extended or coupled using glycol, diamine, or water. Other solvents can also be used, e.g. dimethylformamide (DMF) or nitric acid (HNO3). The final polymer is converted into fibers using a spinning process.

DMAC is an excellent solvent for a large variety of organic polymers and is widely used for making fibers, adhesives, and dyes.

Instrumentation and installation

The Vaisala K-PATENTS Process Refractometer is ideal for controlling spinning bath concentration.

The refractometer continuously measures the concentration of the bath in order to keep it at the optimum level and avoid a decrease in product quality.



The refractometer is installed in the recirculation line between the dissolving tank and the fiber bath to maintain bath concentration at the optimum level.

Fiberglass production

Fiberglass, or glass fiber wool, is a material made from extremely fine short fibers of glass. These fibers are produced by spinning or blowing molten glass (silica).

Production process

To manufacture fiberglass, an adhesive binding solution is applied to hold the fibers together. These binders are also used as a coloring agent to provide brand identification in the highly competitive insulation materials market.

The fiberglass strands that make up the insulation wool are produced at the front of the glass furnace by flowing a stream of molten glass vertically down through a circular ring. Air is then blown through nozzles to atomize the molten glass into fine strands, which are then sprayed with the binder. The final bonded wool is collected and packaged to be used as insulation. The residual binder waste solution is collected and pumped back to a holding tank, where it settles before being filtered several times to remove particles.

Once the filtering process is complete, a dissolved solids measurement is taken to determine the quantity of resin, ammonium sulfate, phenol, and other components present, so that the filtered residual binder solution can be accurately remixed into the fresh binding solution.

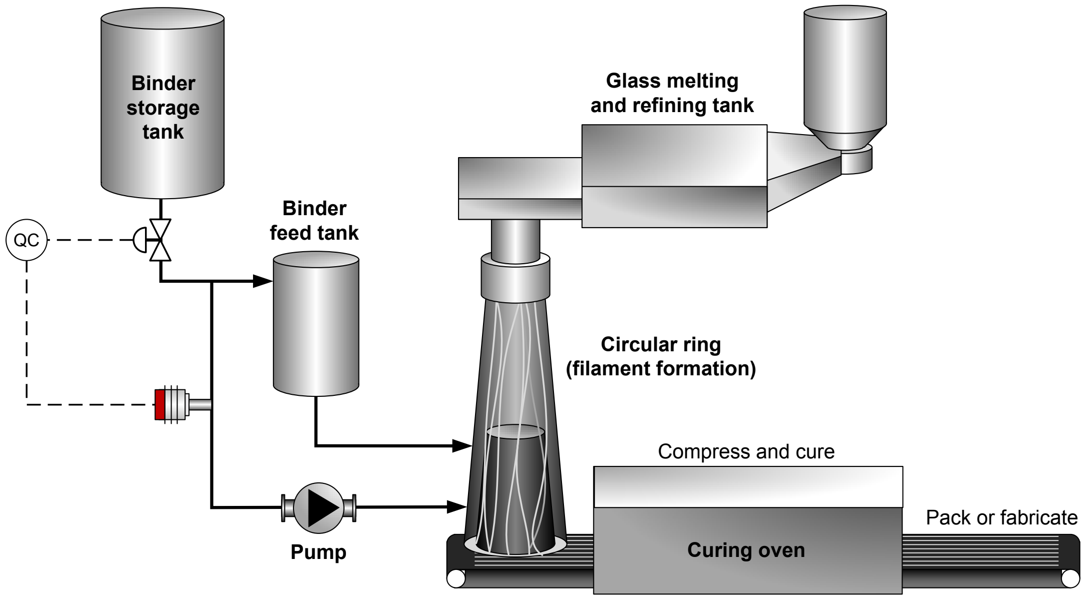
Spent binder solution cannot be released into local sewage systems because the coloring agents are difficult to neutralize, so it is collected and recycled.

Instrumentation and installation

The Vaisala K-PATENTS Process Refractometer is used to determine the amount of dissolved solids in the spent binder solution to control the spiking of fresh binder. Knowledge of the concentrations in spent solution can be used to calculate the precise amount of new binder required to maintain the correct formulation, reducing waste and production costs. The binder is a water-soluble acrylic substance which has direct correlation to the refractive index and binder concentration.

The refractometer is mounted in the recycling line of the binder feed tank. The output signal from the refractometer is used to control the addition of fresh binder to the static mixer in order to maintain the final wash-coat concentration at approximately 8-10 %b.w. The process temperature is between 32°C and 40°C (90-104°F).

An automatic high-pressure water prism wash system is recommended due to the adhesive nature of the binder.



The refractometer is installed in the recycling line between the binder storage and feed tanks to control the addition of fresh binder to the static mixer or to maintain the final wash coat concentration.

Production of polyacrylonitrile (PAN) precursor for carbon fiber

Carbon fiber is a long, thin strand of material that contains over 90 % carbon by weight. The raw material used to make carbon fiber is known as precursor.

Carbon fibers are obtained through the pyrolysis of an appropriate precursor fiber. Polyacrylonitrile (PAN) is the predominant precursor for carbon fiber. About 90 % of the carbon fiber produced is made from PAN and the remaining 10 % is made from rayon or petroleum pitch.

PAN-based carbon fiber is widely used in many industries such as aviation, aerospace, sporting goods, and construction.

Production process

The first step in the production of PAN-based carbon fibers is spinning the PAN co-polymer to form the fibers. Because PAN decomposes before melting, a solvent-based spinning or dope solution is needed in order to be able to spin the material into a fiber. Commonly used solvents are dimethyl sulfoxide (DMSO), dimethyl formamide (DMF), dimethyl acetate (DMAc), and sodium thiocyanate. The concentration of the dope solution is usually 15-25 wt-% and it should be carefully controlled to ensure it can form filaments of high mechanical strength.

Different methods can be used for spinning, such as dry-jet wet spinning or wet spinning. However, only wet-spun PAN fiber is used as a precursor for carbon fiber, as it contains a co-polymer (e.g. itaconic acid) that helps the carbonation process.

In wet spinning, the dope solution is passed through a spinnerette into a coagulation bath to form filaments. The fibers solidify when the solvent diffuses away, leaving behind the PAN fibers.

The spinning step is important because the internal atomic structure of the fiber is formed during this process. The quality of the final fiber depends on different process parameters, such as the composition of the dope and the temperature and concentration of the coagulation bath.

After the fibers are formed, they are further treated by stretching, stabilization, carbonation, and graphitization. The final carbon fiber is then ready for use in different applications. The solvent-water mixture from the coagulation bath is sent to the recovery area where the water and solvent are separated. The recovered solvent is used again in the dope preparation to save on operating costs.

Instrumentation for the production of polyacrylonitrile (PAN) precursor for carbon fiber

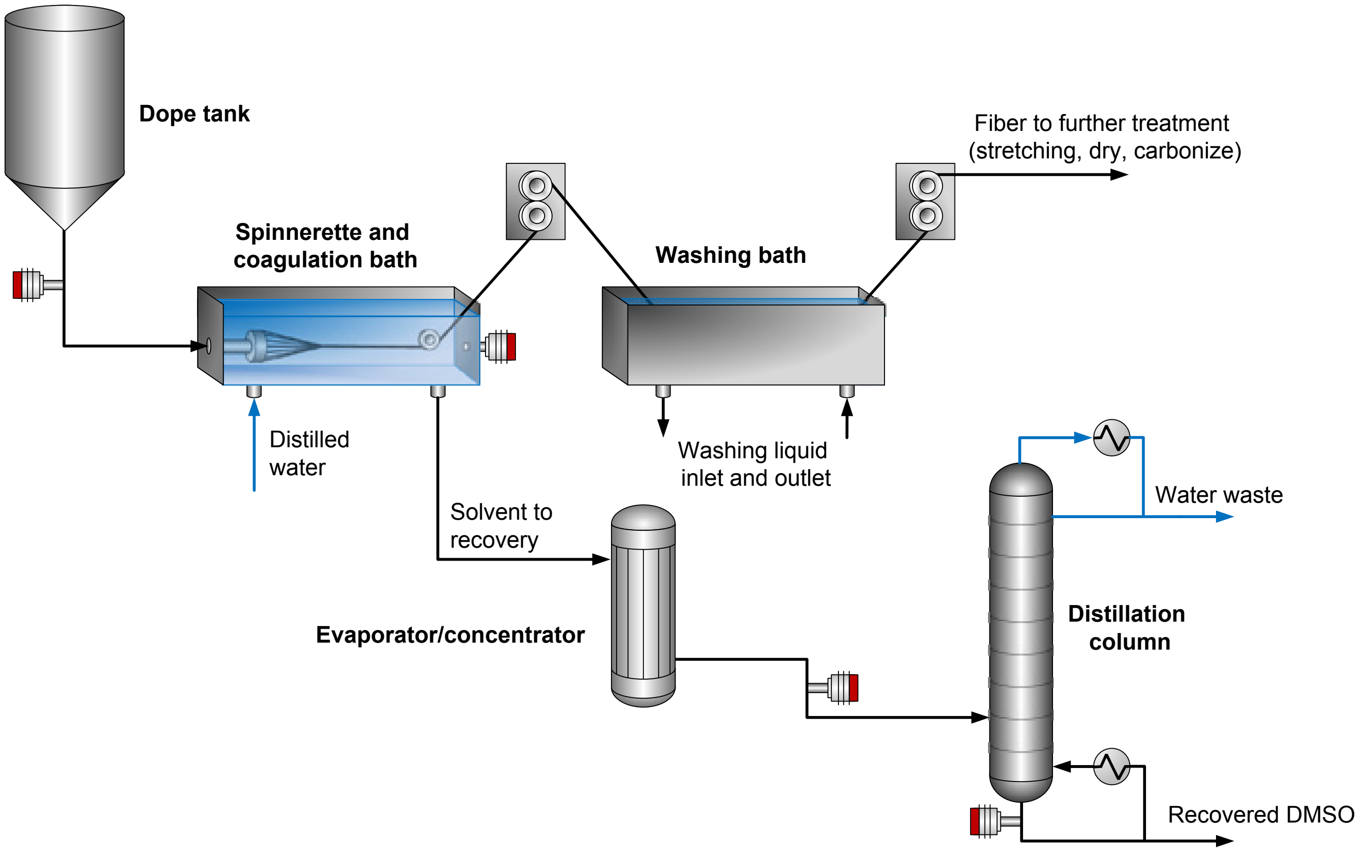
The Vaisala K-PATENTS Process Refractometer monitors in-line the concentration of solvent at different stages of the process.

The refractometer is installed directly on the pipe after the dope tank to measure the concentration of the PAN solution before it is pumped into the coagulation bath. The refractometer's real-time information helps to maintain the dope concentration within the desired range and to obtain precursor fibers with excellent properties.

As the fibers are formed and passed through the coagulation bath, the solvent diffuses out and changes the concentration of the bath. The refractometer continuously monitors the solvent concentration in the bath and provides real-time information to keep the concentration optimal through the addition of water.

At the solvent recovery stage, the refractometer monitors the concentration after evaporation and distillation and provides Ethernet and 4-20 mA output signals that can be used as feedback for automated control. The instant and accurate measurement from the refractometer ensures that the target concentration is always achieved with minimum energy consumption.

The refractometer reduces the need for manual sampling and laboratory tests. An automatic prism wash may be needed in this application.



The refractometer is installed in the pipe after the dope tank to measure the solution concentration before it is pumped into the coagulation bath. The real-time measurement helps to maintain the specifications of the dope concentration and achieve high-quality precursor fibers.

A second refractometer is installed after evaporation and distillation in the solvent recovery process to ensure the target concentration is always achieved with minimum energy consumption.

Biopolymer fibers: wet spinning of sodium alginate fibers

Alginates are biopolymers extracted from brown algae species such as seaweed. They are extensively used in biomedical applications, for example wound dressings and fabrics.

Production process

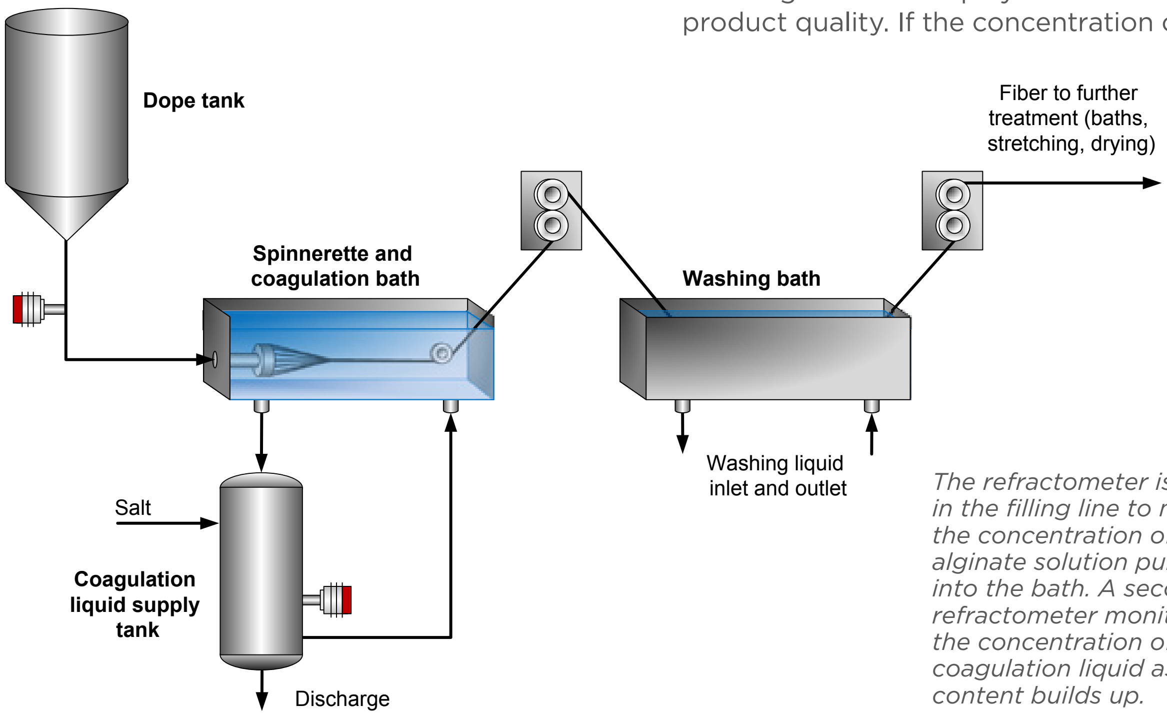
Alginate fibers are produced using a wet-spinning process. It is called wet spinning because the fibers are extruded directly into a solution or bath. The spinning bath is usually a salt solution or a mixture of salts containing metal ions, but it can also be an inorganic acid solution or an organic solvent depending on the end product. The most common salt is calcium chloride, but salts containing zinc, silver, or other bioactive additives are also used.

For the production of the fibers, a spinning dope or spinning solution is prepared by mixing sodium alginate with water to form a homogenous solution with a concentration of 5-10 %. The solution is then spun directly in the coagulation bath through a spinnerette or nozzle. As the sodium alginate makes contact with the salt bath, water is removed from the formed fiber leaving only the biopolymer (alginate) behind.

Coagulation happens when the sodium ions come into contact with the bath's polyvalent ions (e.g. Ca^{2+}). The sodium ions exchange places with the calcium ions to form calcium alginate, which is not soluble in water. The resulting fiber is washed, stretched, and dried to obtain the final product. Additional baths can be used to alter the composition of the fiber.

The concentration of both the dope solution and the coagulation bath play a critical role in final product quality. If the concentration of sodium alginate in the dope solution falls too low neither coagulation nor alginate filament formation take place. In addition, the hemostatic property depends on the alginate concentration.

As the sodium alginate is extruded, the coagulation bath gets diluted and needs to be monitored. Another reason to monitor the bath concentration is that the morphological structure of the fibers is influenced by the composition of the salt.



The refractometer is installed in the filling line to measure the concentration of sodium alginate solution pumped into the bath. A second refractometer monitors the concentration of the coagulation liquid as water content builds up.

Instrumentation and installation

The Vaisala K-PATENTS Sanitary Refractometer provides real-time, accurate, and reliable concentration measurements to ensure the high quality, purity, and consistency of fibers in biomedical applications.

The refractometer is installed in the filling line to measure the concentration of sodium alginate solution pumped into the bath. The concentration of the dope solution is 5-10 % and the temperature is 35-50°C (95-122°F). A second refractometer monitors the concentration of the coagulation liquid as the water content builds up. The water content should be kept below 20 % to achieve high product quality.

The refractometer output signals assist in real-time process control. The concentration of the bath can be controlled and kept at its ideal value using a circulation system where more coagulation agent is added to restore the concentration.

The refractometer is designed to meet pharmaceutical industry standards and regulations, and is the ideal in-line process instrument for the process analytical technology (PAT) framework.

Textile sizing process

The yarn sizing process is vital for eliminating breakages and thereby avoiding stoppages during weaving. Higher quality and a smoother surface finish can be achieved by sizing the strength and abrasion resistance of the yarn. Different types of water-soluble polymers, known as textile sizing agents/chemicals, are used to protect the yarn. Some examples are modified starch, polyvinyl alcohol (PVA), carboxymethyl cellulose (CMC), and acrylates. Mixtures of these and other chemical components are also used.

Application

Before the yarn can be woven, it needs to be strengthened to withstand the stress sustained when weaving on high-speed industrial looms. For this purpose, the yarn is passed through a sizing bath that contains the sizing medium mixed with water and other additives depending on the formula.

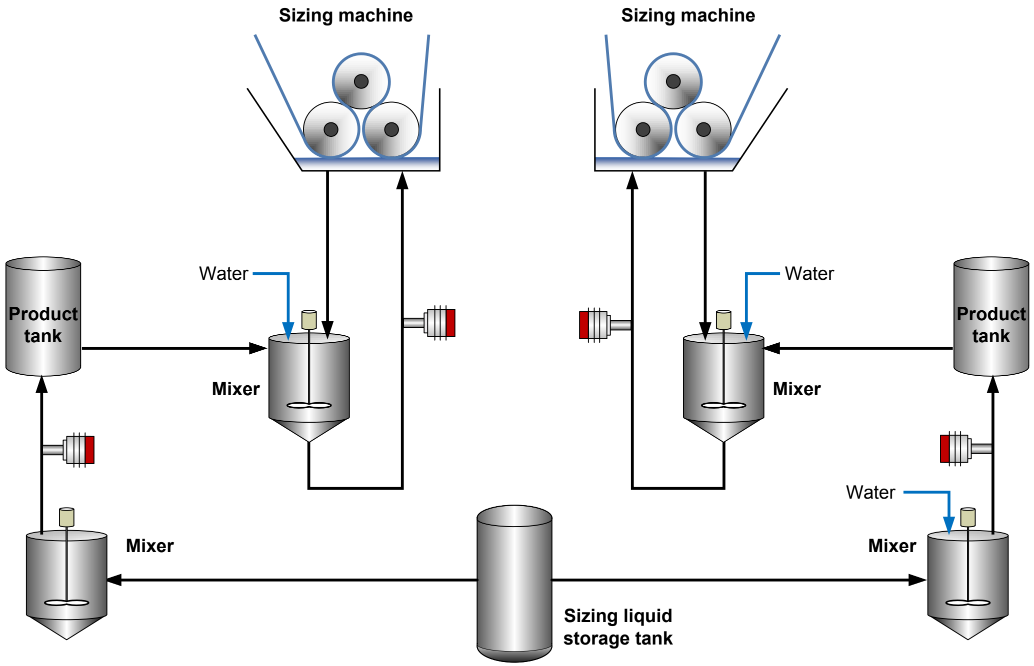
The sizing medium is mixed to achieve the correct level of concentration before loading it into the sizing bath. If starch is used, the mixture must be cooked before it can be used. Usually the cooking or mixing station is close to the sizing bath and may serve several sizing lines.

After mixing, the product is pumped into a product tank. From there, it passes to a final mixing tank, where it is mixed to the required concentration level before being loaded into the sizing bath. The mixing tanks are equipped with a load gauge so a specific quantity of the medium can be mixed for each textile batch. After the initial mixing and cooking, the concentration of the medium is typically 16-18 % and after the final mixing it is at 6-10 %. Recycled sizing medium can sometimes be added to the final mix to adjust the concentration for specific yarns.

Instrumentation and installation

The concentration of the sizing baths needs to be monitored as it can fluctuate due to evaporation and yarn absorption. A Vaisala K-PATENTS Process Refractometer is usually mounted in a circulation loop for adequate flow across the prism. The typical concentration is 5-15 %.

The refractometer is also used to measure the concentration in the product tanks or during mixing in order to ensure correct concentration levels. A steam-cleaning system is recommended for the above-mentioned installation positions.



The refractometer is installed in the circulation loop of the sizing bath to maintain the correct bath concentration. The refractometer is also used to measure concentration in the product tanks or during mixing for the same control task.

Recovery of textile sizing agents by ultrafiltration

Sizing materials, such as starches and water-soluble polymers (polyvinyl alcohol), are used to facilitate the weaving process. The woven cloth is later washed to remove the size, leaving a diluted solution of the sizing material. Ultrafiltration (UF) can be used to recover and concentrate the sizing material for reuse and to produce quality water permeate for discharge or reuse.

Wash water containing sizing materials is harmful to the environment. Sizing materials are also expensive, and it is possible to reuse them several times. This has resulted in a growing interest in UF systems in the textile industry.

Application

Similar to reverse osmosis (RO), UF is a pressure-driven membrane process capable of separating solution components on the basis of molecular size and shape.

Under applied pressure difference against a UF membrane, solvent and small solute particles pass through the membrane and are collected as permeate. Larger solute particles are retained by the membrane and recovered as a concentrated retentate.

The concentration of the sizing agents in the washing liquor is about 20-30 g/l. In the ultrafiltration plant, they are concentrated to 150-350 g/l. The concentrate is recovered and can be reused for sizing, whereas the permeate can be recycled as water in the washing machine. The concentrate is kept at a high temperature (80-85 °C) and does not need to be reheated.

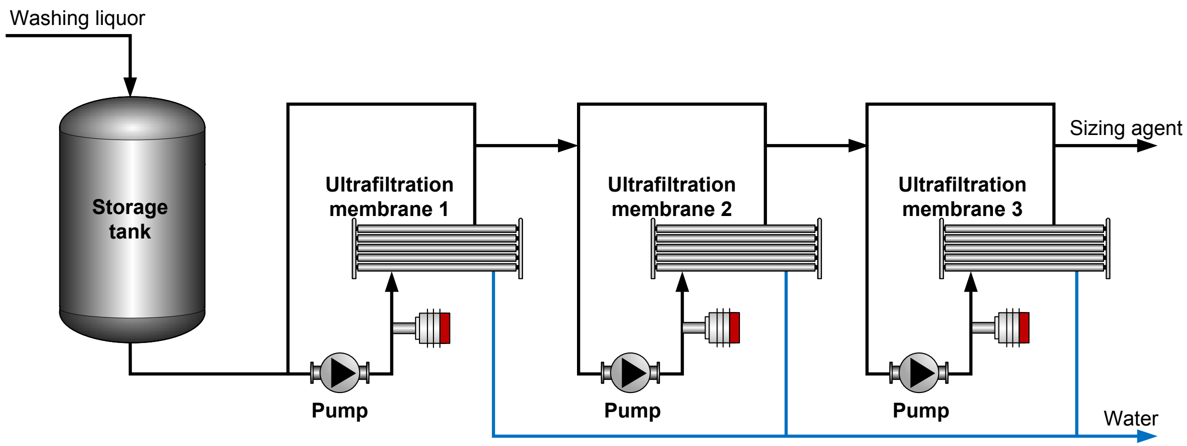
The resultant concentrate is then pumped into a holding tank and the UF system is refilled with the sizing solution.

Instrumentation and installation

The Vaisala K-PATENTS Process Refractometer measures the concentration of the sizing agent as it passes through the UF membranes, providing real-time information on when the required concentration level is obtained.

The refractometer is installed in the circulation loop on the pressure side of the circulation pump. The typical measurement range is 0-15 %.

The refractometer is also used to measure the concentration in the product tanks or at the mixing stage to ensure the correct levels. A steam-cleaning system is recommended for these installation positions.



The refractometer is installed in the circulation loop on the pressure side of the circulation pump. The refractometer is also used to measure the concentration in the product tanks or at the mixing stage to ensure the correct levels.

Wet treatments in the polyethylene fiber antistatic agent process

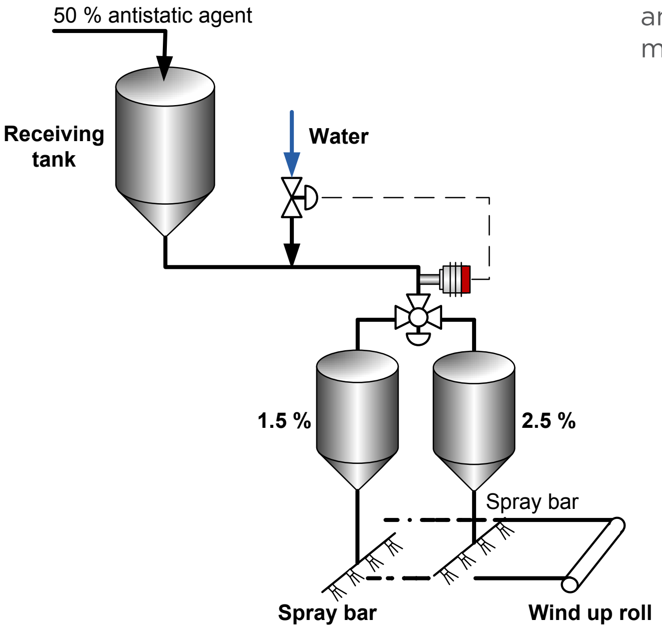
Polyethylene fiber is known for its high strength and durability. The material is used in a variety of applications, including Tyvek® HouseWrap, vehicle covers, envelopes, medical and industrial packaging, and protective apparel.

Application

The polyethylene sheet is made by spinning extremely fine high-density polyethylene fibers that are fused together to produce a strong uniform web.

Inside the sheet, an antistatic agent is applied as a thin microporous film on a coarse fabric consisting of millions of small pores. An isopropyl alcohol-based solution (Zelec®) is used as the antistatic agent.

Before the antistatic agent is applied, it must be diluted to a 1.5 % or 2.5 % solution by adding water.



Instrumentation and installation

The Vaisala K-PATENTS Process Refractometer is used to measure the concentration of antistatic agent after it is diluted and before it is applied to the polyethylene sheet.

The refractometer is installed in a pipe bend to control the concentration of diluted agent before it is pumped into 1.5 % or 2.5 % solution tanks. The antistatic solution is then applied to the polyethylene sheet to form a thin film.

The output signal from the refractometer can be used to control the water feed valve for automated dilution control. The accurate measurement from the refractometer ensures the target concentration is achieved.

Ultrasonic flow meter technology has proved to be inaccurate, unreliable, and maintenance intensive in this application; handheld and laboratory refractometry are time consuming and disruptive. The benefits of the process refractometer are that it is maintenance-free and provides accurate, reliable, and continuous measurement of the agent solution.

The refractometer is installed in a pipe bend to control the concentration of the diluted agent before it is pumped into 1.5 % or 2.5 % solution tanks.

Textile softening bath

Textile softening is an essential finishing process in fabric production. Softening treatment improves the fabric's aesthetic properties and makes further processing easier. A nice, soft feel is often the decisive criteria for buying a textile.

Application

In softening, the fabric is passed via rollers through an immersion bath containing a softener solution.

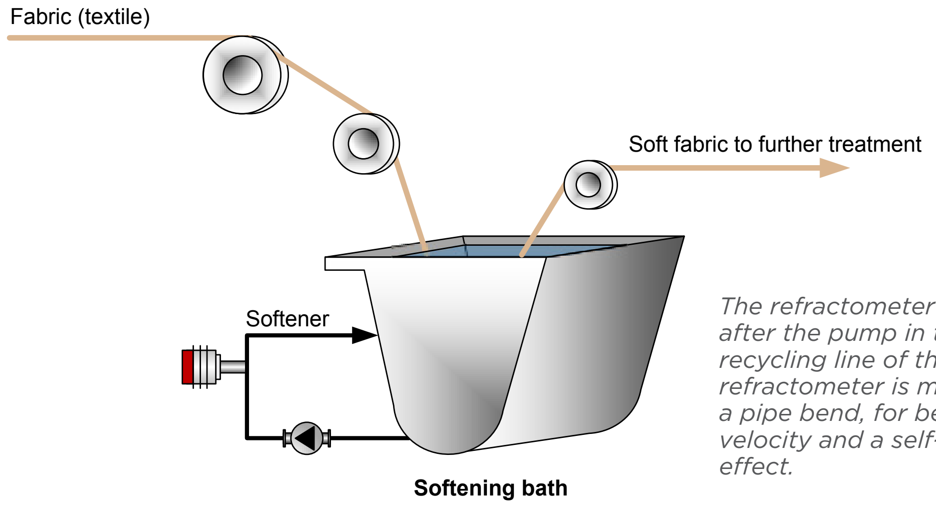
The concentration of the bath must be monitored and kept constant to obtain consistent end-product quality. During the treatment, the fabric may bring some water with it (for example, if it comes from a previous dyeing step), which dilutes the bath. In such cases, the softener needs to be replenished to bring its concentration back to the optimum level. On the other hand, if dry fabric is treated, it will absorb some water and increase the concentration of softener.

Instrumentation and installation

The Vaisala K-PATENTS Refractometer is installed after the pump in the recycling line of the bath. The refractometer is mounted on a pipe bend for better flow velocity and a self-cleaning effect.

The refractometer provides continuous measurement of softener concentration and alerts operators when the concentration needs to be replenished. During the addition of softener, the refractometer also indicates when the optimum concentration has been reached, preventing excess use of material.

The refractometer output signal can be used for real-time control of the softening bath concentration. An automated process ensures consistent product quality and reduces operational costs.



The refractometer is installed after the pump in the recycling line of the bath. The refractometer is mounted on a pipe bend, for better flow velocity and a self-cleaning effect.

Vaisala K-PATENTS Process Refractometer

The Vaisala K-PATENTS Process Refractometer PR-43-G is an ideal all-round instrument for measuring the concentrations of a wide range of chemicals and other liquids in industrial applications.

The PR-43-G refractometer is designed for harsh environments that can be corrosive, abrasive, subject to extreme temperatures, pressures, vibration, contamination, humidity, or dust, or any combination of these factors. It is installed in the main processing line, bypass line, or vessel, either directly with a flange or Sandvik L coupling process connection or via a large variety of cost-effective and easy-to-mount flow cell options. The user interface can be installed locally in the field, remotely in the control room, or in both locations by connecting several user interfaces in a network.

The PR-43-G refractometer has a measurement range of 0 to 100 % and provides an Ethernet or 4–20 mA output signal proportional to the temperature-compensated concentration value for real-time process control. The PR-43-G is factory-calibrated to measure concentration and temperature in standard units.

- Universal calibration of each sensor: all sensors are freely interchangeable
- Full range nD = 1.3200...1.5300 corresponds to 0-100 % bw
- Process temperature range: -40°C...150°C (-40°F...302°F), for higher temperatures consult factory
- Completely digital system: particles and bubbles do not affect operation or accuracy
- CORE-optics: no drift, no re-calibration, no mechanical adjustments
- Protection class IP67/Type 4X (for outdoor use)
- Hazardous and intrinsic safety approvals available
- Built-in web server allows for configuring, monitoring, verifying, and diagnosing the refractometer via an Ethernet connection
- Fast process temperature measurement using built-in Pt1000 and automatic temperature compensation
- Easy on-site instrument verification within users' own quality assurance system and standard refractive index liquids

"The refractometer's measurement performance is not affected by air or gas bubbles, suspended particles, flow changes, pressure changes, vibrations, or temperature shocks."

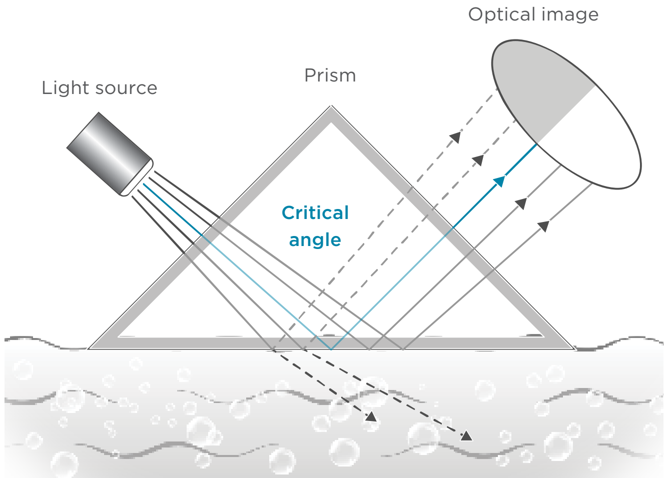
How does the system work?

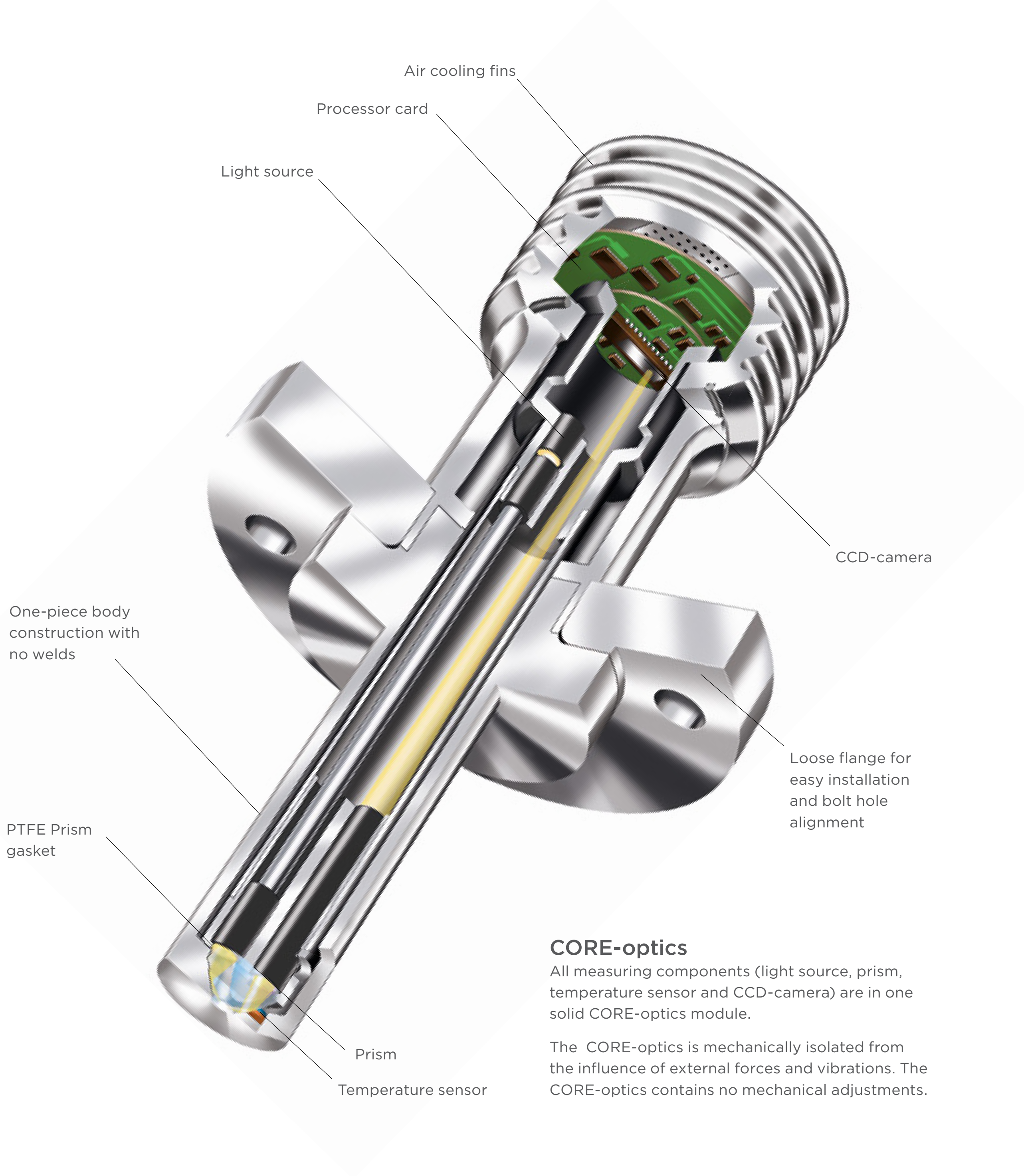
The light source sends light to the interface between a prism and the process solution where the rays meet the surface at different angles. Depending on the angle, some rays undergo a total internal reflection. The rest of the light is refracted into the process solution.

This creates an optical image with a dark sector and a light sector. The angle corresponding to the shadow line is called the critical angle of total internal reflection.

This angle is a function of the refractive index and therefore the concentration of the solution. A CCD camera detects the optical image. The image is then transformed point-by-point into a digital signal. Digital signal processing is used to locate the exact shadow line position and to determine the refractive index (nD).

A built-in temperature sensor measures the temperature (T) on the interface of the process liquid. The sensor converts the nD and T into Brix units and the diagnostics program ensures that the measurement is reliable.





CORE-optics

All measuring components (light source, prism, temperature sensor and CCD-camera) are in one solid CORE-optics module.

The CORE-optics is mechanically isolated from the influence of external forces and vibrations. The CORE-optics contains no mechanical adjustments.

Summary of refractive index measurement and applications in the chemical industry

Refractive index (RI) measurements are performed by determining the critical angle of internal reflection between the fluid being measured and a medium with a known refractive index. A process refractometer measures dissolved material in a liquid, thus the method is very useful for measuring the concentration, density, and purity of almost any chemical or compound.

In the chemical industry, the in-line measurement of concentration is an important parameter. The purpose is to control the raw and in-process materials in order to extract their maximum value and achieve high-quality end products. Unlike intermittent sampling, continuous monitoring can provide instant feedback on changes in the process, and this data can enable real-time and precise process control.

Measurement accuracy and repeatability are important factors so that standard deviation from the set-point can be achieved. The reduction of deviations depends on the process variable and how accurately it can be measured. The concentration can be measured using several methods, and when choosing a measurement technique it is important to consider the advantages and disadvantages of the available options. For instance, in-line density meters are typically side-stream measurements and can be affected by air bubbles and undissolved and suspended particles, whereas these do not affect RI measurement. The refractometer directly indicates the built-in temperature-compensated concentration values.

The performance of all types of chemical refining, manufacturing, and quality-control operations can be dramatically improved with RI technology – even in environments that can be potentially explosive, corrosive, abrasive, subject to extreme temperatures, pressures, vibration, contamination, humidity, or dust, or any combinations of these factors.

In this chapter we introduce typical applications in chemical industry operations that can be improved with precise measurement technology and better process control.

Typical chemical industry applications

Alkalies and chlorine

Chlorine, hydrochloric acid, sodium hydroxide (caustic soda), sodium chloride (brine), sodium hypochlorite, sulfuric acid.

Chemicals

Acetic acid, acrylate, amine oxide, amino acid, ammonium fluoride, ammonium hydroxide, ammonium nitrate, ammonium sulfate, caustic soda, cellulose derivates, citric acid, copper chloride, chromium trioxide (or chromic acid), ethyl alcohol, ethylene glycol, formaldehyde, formic acid, gelatin, glycerol, hydrogen peroxide, iron chloride, lactic acid, lubricating oils, nickel chloride, nitric acid, oleum, polyamides, polycarbonates, polyethylene, potassium compounds, resins, sodium alginate, sodium bicarbonate, sodium dichromate, sodium gluconate, sodium hydroxide, styrene, sulfuric acid, urea, etc.

Plastics, resins, fibers, and synthetic rubber

Acetate, acrylics, adipic acid, caprolactam, cellulose acetate fibers, cellulose triacetate (CTA), cyclohexanol, cyclohexanon, dimethylterephthalate, dimethylformamide, fiberglass binder solution, hexamethylene diamine, nitrile butadiene rubber (NRB), nylon salt, polyamides, polyesters, rayon, spandex, synthetic latex, vinyls. Finishing, coating, and dyeing mediums of textiles.

Salts and sodium compounds

Brine, Glauber's salt, sodium carbonate, sodium chloride, sodium sulfate, sodium sulfite, etc.

Effluent and water treatment

Surface water treatment by chemical precipitation: polyaluminum chloride, sodium hydroxide, sodium hypochlorite. Ammonia removal in underground water treatment: ammonium sulfate. Total organic carbon (TOC) content monitoring in effluent: dissolved organic material.

Other industrial applications

Ion exchange chromatography: regeneration chemicals of ion exchangers. Loading and unloading operations: chemical-interface detection and identification. De-icing preparations, manufacturing, spraying, and recovery: ethylene glycol, propylene glycol. Automotive-grade urea solution, AdBlue (AUS32), diesel exhaust fluid (DEF).

Removal of polycyclic aromatic hydrocarbons (PAH) in green automotive tire production: extender oil. Sugar, starch sweeteners: sucrose, fructose, dextrose. Wood timber treatment and acetylation: acetic anhydride. And more.

The chemical industry is one of the most regulated of all industries. Process safety is mandatory, since hazardous chemicals represent a potential risk to people and the environment. While managing these challenges, chemical manufacturers strive to produce high-quality products that meet customers' specifications. To achieve these goals, accurate process measurements, high process efficiency, low energy consumption, and state-of-the-art process control management are crucial.

How refractive index measurement can help to improve industrial operations

The following chapter introduces how the refractive index can be used in different operations within a chemical processing facility.

Reactor, reaction degree, and endpoint determination

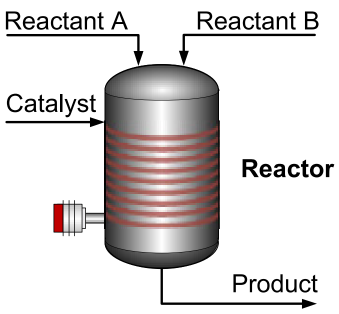
In-line refractive index measurement makes it possible to follow the progress of a reaction in real time until the endpoint is achieved. The ease of end-point determination is the most important advantage of the method. The refractometer's reading can also be used to indicate the correct dosing points for other ingredients.

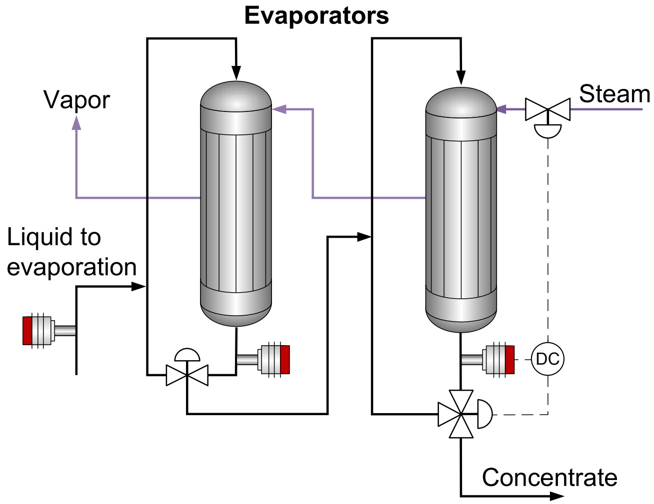
Evaporation

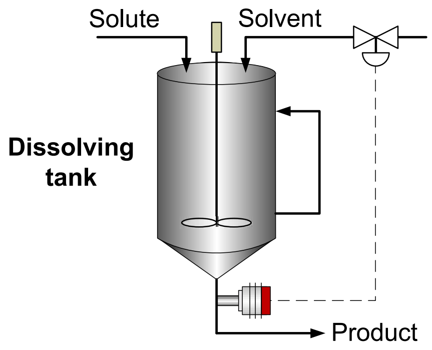
The refractometer is ideal for operations where the liquid is concentrated by evaporation as it provides real-time information of a medium's concentration changes. The concentration information at the inlet, outlet, and intermediate stages in the concentrator can be used to optimize the process. For instance, the refractometer output signals can be used to control the heat source flow (steam), for adjustments, and to achieve the target concentration. If the concentration of the product is below specifications, the refractometer's signal is used to control the valves to either decrease the feed flow to the evaporator or to increase steam flow.

Dissolving tank or vessel

In a dissolving application, the refractometer measures the concentration of the resulting solution while the solute dissolves into water or solvent. It provides instant information on the dissolving rate and the amount of dissolved solids. The refractometer's output signal can be used to automate control of the dissolving operation to consistently achieve the right target concentration. As a result, manual sampling and raw material use can be reduced. Dissolving tanks with stirrers are common in this application. The stirrer can cause vibration or bubbles that are common sources of error for many measurement devices. The Vaisala K-PATENTS refractometer is not affected by undissolved matter, bubbles, or vibration.







Dilution, mixing, or blending

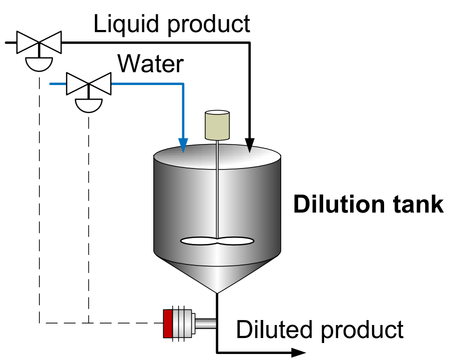
In the dilution process, the product is diluted with solvent (usually water) to reduce its concentration value. The dilution can take place in a tank or static mixer, for example. The dilution process can be controlled by using the refractometer output signal to control the feeding valves. The concentration signal is fed back to the controller to ensure consistently accurate concentration in the dilution or blending process. The Vaisala K-PATENTS refractometer is ideal for this operation as it is not influenced by bubbles that might be formed during mixing.

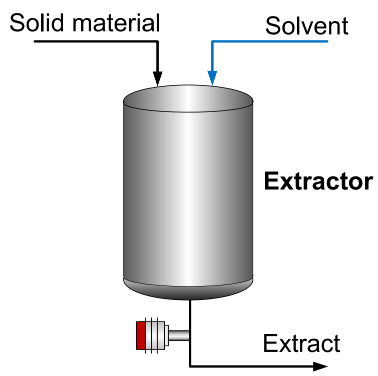
Solid-liquid extraction

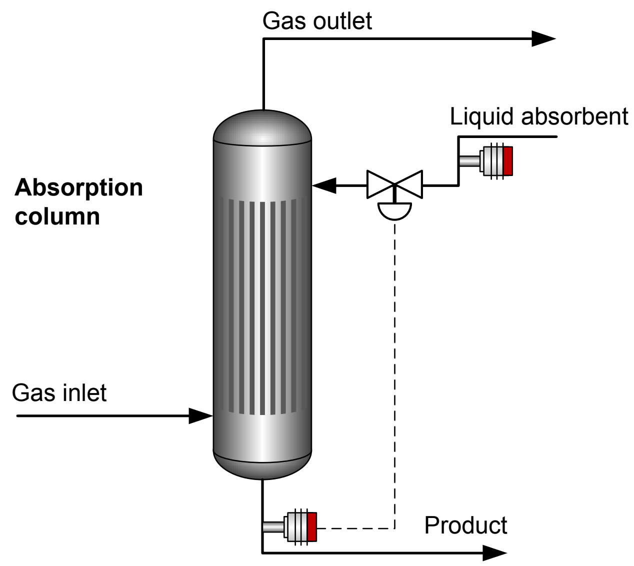
In solid-liquid extraction, the preferential dissolution of one or more of the components is separated of a solid mixture in a liquid solvent. The refractometer is used to detect the amount of extracted substance (dissolved solids) in the liquid after the extraction process. The refractometer measurement is not affected by undissolved solids, only by the dissolved matter – making it the ideal instrument to monitor extraction efficiency. The in-line concentration measurement allows adjustments to be made in real time and helps to maximize efficiency and minimize costs.

Absorbers and wet scrubbers

Absorption operations can be monitored and controlled using the refractometer. In a liquid-gas contactor or wet scrubber, gas is purified by absorbing a solute into a liquid stream. The absorption efficiency can be followed by a refractometer in the outlet of the column. In some liquid-gas operations, the mass transfer is maximized at a certain concentration range for the absorbing liquid. The refractometer is also used to monitor the concentration of the inlet liquid to ensure the optimal absorption conditions and to maximize the separation efficiency.

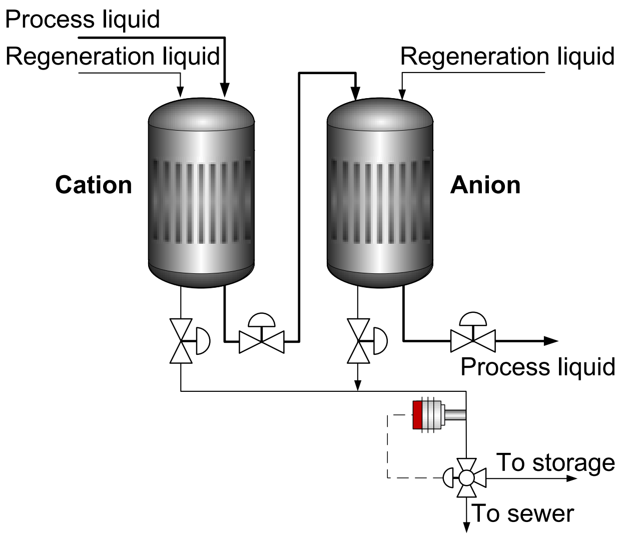






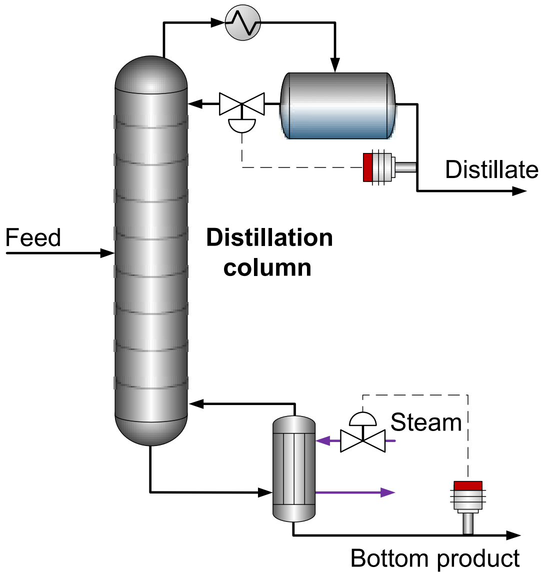
Ion exchangers

Ion-exchange resins are often used to purify liquids. Over time, the resins get saturated with trapped ions and need to be regenerated to improve process efficiency. The refractometer monitors the interface between the product and the rinsing liquid at the outlet line from the ion-exchange columns prior to the regeneration process. Typically, the sensor is installed in the waste line before the divert valve. The refractometer output signal is used to open and close the valves and direct the flow to the process line or sewer.



Distillation

In distillation, a mixture of chemicals is separated into pure components based on their unique boiling points. Control of the distillation process is essential to meet product specifications, maximize return on investment and energy efficiency, and limit the environmental impact. These goals can be achieved by maintaining the purity of the column's top and bottom products within the specifications. The refractometer monitors the concentration of the distillation products in real time. The refractometer is installed directly in-line after the column (bottom) or after the condenser (distillate). The refractometer output signals can be used to automatically adjust the column's reflux or boil-up to meet product specifications. In binary systems, the refractometer provides accurate information on product concentrations. In multi-component systems, control can be based on a property which is a function of the composition.



Interface detection and product identification

Refractive index (RI) is an intrinsic property of liquids and a useful method for their identification. The high reliability and fast response time of the Vaisala K-PATENTS Process Refractometer provides an ideal optical detection method that can be used as a fingerprint for chemical and liquid bulk material identification and interface detection. The RI signal is used, for example, for product-to-product interfaces. This information is necessary for complete automation of chemical loading and unloading operations.

Quality control

As all liquids have a unique RI value, the Vaisala K-PATENTS Process Refractometer can determine the final quality of a liquid product and ensure it is within specifications. Real-time detection allows quick reaction to potential process disturbances and product quality variations.

Building on over 80 years of experience, Vaisala provides observations for a better world. We are a reliable partner for customers around the world, offering a comprehensive range of innovative observation and measurement products and services. Headquartered in Finland, Vaisala employs approximately 1,800 professionals worldwide and is listed on the Nasdaq Helsinki stock exchange.

K-Patents Oy, an industry leader and supplier of K-PATENTS® Process Refractometers, was acquired at the end of 2018 by Vaisala. Following the acquisition, all K-Patents group companies are part of Vaisala.

Contact our expert team to discover our full offering and discuss how we can help you to improve your process and applications.

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