Application Note AN-PAN-1010

Online analysis of sulfuric acid and zinc sulfate in the viscose process

Viscose is a versatile material employed in various industries. In addition to its widespread use in textiles, it plays a crucial role in automotive components like tires and belts.

To optimize viscose production and maintain product quality, rigorous process control is essential. A critical component of this control is the precise determination of sulfuric acid (H_2SO_4) and zinc sulfate ($ZnSO_4$) during the wet-spinning process.

This Process Application Note demonstrates the use of the 2060 TI Process Analyzer or the 2035 Process Analyzer for online potentiometric titration and colorimetric analysis of H_2SO_4 and $ZnSO_4$, respectively. These online process analyzers continuously monitor sulfuric acid and zinc sulfate to ensure optimal concentrations in the wet-spinning process of viscose manufacturing.

INTRODUCTION

Viscose, often called rayon, is the original man-made fiber. Created from regenerated cellulose materials like wood pulp and cotton linters, its market size is expected to reach 40.26 billion USD by 2032 [1,2].

Viscose is gaining popularity through a growing demand for sustainable fashion. Its soft, breathable, and absorbent qualities make it a comfortable and eco-friendly alternative to cotton and polyester.

In the first production step, the wood pulp is immersed in sodium hydroxide (NaOH) to convert it to alkaline cellulose (Figure 1). After pressing and shredding, the alkaline cellulose is aged to depolymerize. A solution of carbon disulfide (CS_2) is added to form cellulose xanthate. The resulting crumbs are dissolved in NaOH to obtain a viscous solution called viscose.

After ripening, filtering, and degassing, the viscose solution is pumped under pressure through metal spinnerets submerged in a spin bath. The spin bath contains sulfuric acid (H_2SO_4) to acidify the cellulose xanthate, sodium sulfate (Na_2SO_4) for rapid coagulation, and zinc sulfate ($ZnSO_4$) to cross-link the cellulose molecules.

Many types of viscose fibers can be made by changing various process conditions and adding chemicals. The final steps are drawing, washing, and bleaching.

To optimize the wet-spinning process (Figure 1, purple star), it is crucial to measure the acid and zinc concentration 24/7. Traditionally, total sulfur and byproduct sulfur have been quantified through a laborious gravimetric process involving sulfate precipitation [3]. However, the technical expertise, time, and space required for this method have limited its practical application. Thus, the implementation of rapid and reliable analytical techniques is essential for effective process control.

Metrohm Process Analytics offers several options to measure the critical chemical components in the viscose spin bath. The 2060 TI Process Analyzer (Figure 2) is integrated to measure the sulfuric acid and zinc concentration simultaneously. This analyzer plays a vital role in closed-loop control. This increases product throughput and yield while minimizing chemical consumption.

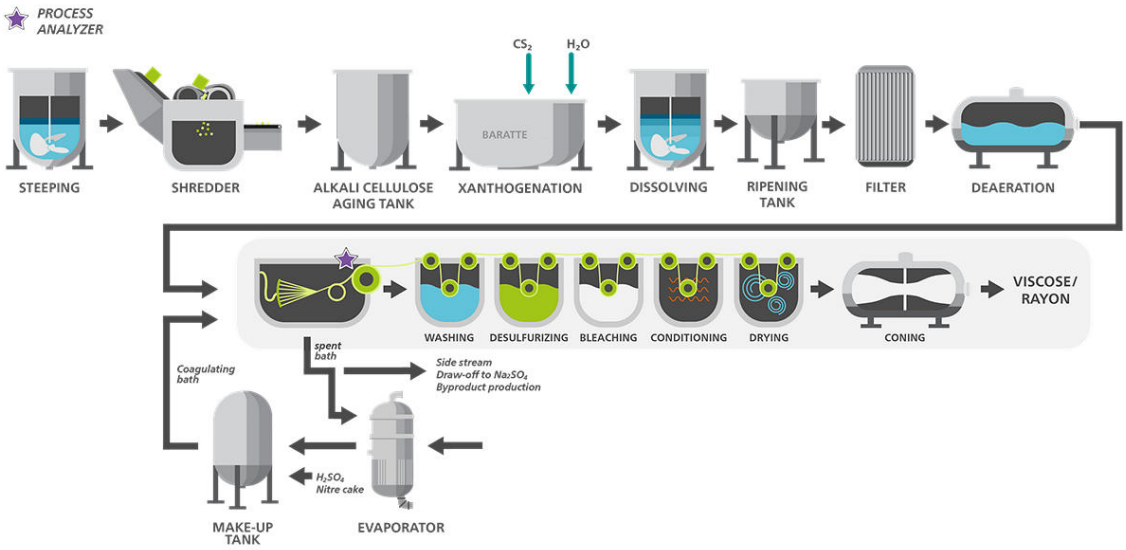

Figure 1. Illustrated process diagram of viscose (rayon) production (adapted from [4]).

APPLICATION

Sulfuric acid and zinc sulfate are analyzed using potentiometric titration and colorimetric measurement, respectively. The 2060 TI Process Analyzer can perform both analyses simultaneously: potentiometric titration for H_2SO_4 and colorimetric measurement for $ZnSO_4$. Results are automatically validated against known standard solutions to ensure compliance with pre-set control limits (see Table 1). For single-parameter analysis, the 2035 Process Analyzer is available in two dedicated versions: Potentiometric for H_2SO_4 and Photometric for $ZnSO_4$.

Figure 2. The 2060 TI Process Analyzer for monitoring critical chemicals used in viscose/rayon production.

Table 1. Viscose wet-spinning process parameters and concentration ranges.

Parameters	[g/L]
H_2SO_4	0–180
$ZnSO_4$	2.5–2.8

REMARKS

Additional analytical techniques can be employed to optimize the wet-spinning process. For instance, X-ray fluorescence (XRF) can provide real-time monitoring of trace elements like zinc in the spinning solution. Accurate zinc concentration measurement is crucial, as it can fluctuate due to factors including measurement duration, background interference, detector sensitivity, and sample preparation. The 2060 XRF Process Analyzer from Metrohm Process Analytics is fully capable of this online analysis.

CONCLUSION

The Metrohm Process Analytics 2060 TI Process Analyzer and 2035 Process Analyzer - Potentiometric can determine the concentration of sulfuric acid and zinc sulfate in the viscose production process. This facilitates optimized production, improved viscose/rayon quality, and reduced chemical consumption.

REFERENCES

[1] Fibre2fashion. *Global viscose fibre market to grow 6.2% annually by 2026*. https://www.fibre2fashion.com/news/textile-news/global-viscose-fibre-market-to-grow-6-2-annually-by-2026--283880-newsdetails.htm (accessed 2024-08-12).

[2] *Viscose Staple Fiber Market Size | Global Industry Report [2032]*. https://www.fortunebusinessinsights.com/viscose-staple-fiber-market-105431 (accessed 2024-08-12).

[3] Lanieri, D.; Alberini, I. C.; Olmos, G. V.; et al. Rapid Estimation of Gamma Number of Viscose by UV Spectrophotometry. *O Papel* 2014, *75*, 60–65.

[4] Mendes, I. S. F.; Prates, A.; Evtuguin, D. V. Production of Rayon Fibres from Cellulosic Pulps: State of the Art and Current Developments. *Carbohydrate Polymers* 2021, *273*, 118466. DOI:10.1016/j.carbpol.2021.118466

RELATED APPLICATION NOTES

AN-PAN-1004 ABC Titration: Analysis of alkali, carbonate, hydroxide, and sulfide in pulping liquors

AN-PAN-1011 Determination of permanganate absorption number (PAN)

AN-PAN-1035 Automated online analysis of indigo, hydrosulfite, and other parameters in textile dye baths

BENEFITS FOR ONLINE PROCESS ANALYSIS

- **Optimize product quality** and increase profit with fast response times for process variations.
- **Fully automated diagnostics** – automatic alarms for when samples are out of specification parameters.
- **Avoid unnecessary costs** by measuring multiple parameters in your process stream simultaneously.

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CONFIGURATION

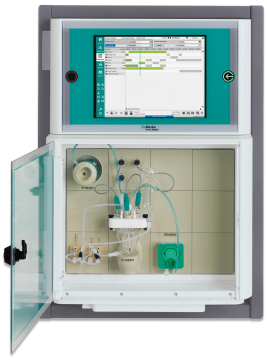
2060 Process Analyzer

The 2060 Process Analyzer is an online wet chemistry analyzer that is suitable for countless applications. This process analyzer offers a new modularity concept consisting of a central platform, which is called a «basic cabinet».

The basic cabinet consists of two parts. The upper part contains a touch screen and an industrial PC. The lower part contains the flexible wet part where the hardware for the actual analysis is housed. If the basic wet part capacity is not sufficient enough to solve an analytical challenge, then the basic cabinet can be expanded to up to four additional wet part cabinets to ensure enough space to solve even the most challenging applications. The additional cabinets can be configured in such a way that each wet part cabinet can be combined with a reagent cabinet with integrated (non-contact) level detection to increase analyzer uptime.

The 2060 process analyzer offers different wet chem techniques: titration, Karl Fischer titration, photometry, direct measurement and standard additions methods.

To meet all project requirements (or to meet all your needs) sample preconditioning systems can be provided to guarantee a robust analytical solution. We can provide any sample preconditioning system, such as cooling or heating, pressure reduction and degassing, filtration, and many more.



2035 Process Analyzer - Potentiometric

The 2035 Process Analyzer for Potentiometric Titration and Ion-Selective Measurements performs analyses with dedicated electrodes and titrants. Additionally, this version of the 2035 Process Analyzer is also suitable for Ion-Selective Analysis using Metrohm high performance electrodes. This accurate standard addition technique is ideal for more difficult sample matrices.

The potentiometric version of the analyzer offers the most accurate results of all measuring techniques available on the market. With far more than 1000 applications already available, titration is also one of the most used methods for analysis in almost any industry for hundreds of components varying from acid/base analysis to metal concentrations in plating baths.

Titration is one of the most widespread absolute chemical methods in use today. The technique is straightforward with no need for calibration.

Some titration options available for this configuration:

- Potentiometric titration
- Colorimetric titration with Fiber Optic Technology
- Moisture determination based on the Karl Fischer titration method