

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.

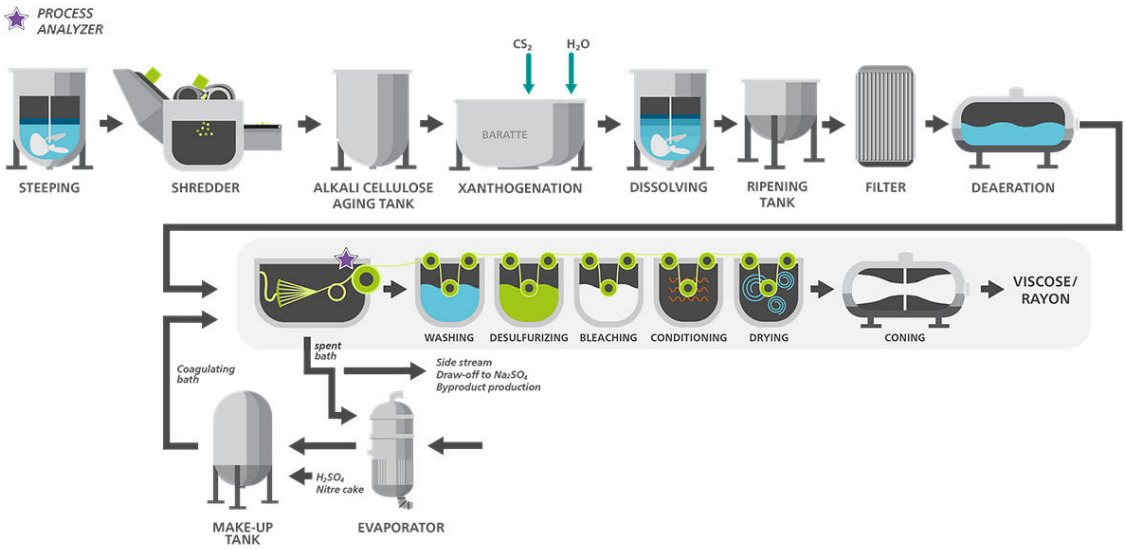


Figure 1. Illustrated process diagram of viscose (rayon) production (adapted from [4]).

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



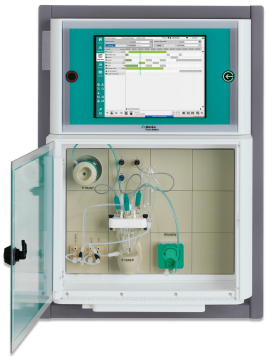
2060 Process Analyzer

2060 Process Analyzer 是一种在线湿化学分析仪,适用于无数应用。此过程分析仪提供了一个新的模块化概念,由一个称为«主机柜»的中心平台组成。

主机柜由两部分组成。上部包含触摸屏和工业计算机。下部含有柔性取样部,其中放有用于实际分析的硬件。如果主取样部容量不足以应对分析挑战,那么主机柜可以扩展为多达四个额外的取样部机柜,以确保有足够的空间来应对具挑战性的应用。附加机柜的配置方式使每个取样部机柜可以与具有集成(非接触式)液位检测的试剂柜组合使用,以增加分析仪的正常运行时间。

2060 Process Analyzer 提供不同的湿化学技术:滴定法、卡尔费舍尔滴定法、光度测定、直接测量和标准添加入法。

为满足所有项目要求(或满足您的所有需求),可提供样品预处理系统,以确保分析解决方案可靠。我们可以提供任何样品预处理系统,如冷却或加热、减压和脱气、过滤等。



2035 Process Analyzer Potentiometric

适用于电位滴定和离子选择性测量的 2035 Process Analyzer 过程分析仪,可使用专用电极和滴定剂进行分析。此外,该版本的 2035 Process Analyzer 过程分析仪还适用于使用万通高性能电极进行离子选择性分析。这一准确的标准溶液技术是处理复杂样品基质的靠谱方法。

此电位分析款型的分析仪可提供当前市场上所有测量技术的准确结果。滴定法作为常用的分析方法之一,具有远超过 1000 种应用可供使用,能分析数百种成份,从酸/碱元素直到电解池中金属浓度,可用于几乎任何行业。

滴定法是目前使用广泛的绝对化学方法之一。该技术简单易行,无需校准。

可用于此配置的部分滴定选项:

- 电位分析滴定
- 使用光纤技术的比色滴定
- 基于卡尔·费休滴定法测定水份