Applying a diode array process analyzer to the monitoring of MEHQ (a polymerization inhibitor)

Introduction

Acrylonitrile's most important use is in polymerization to polyacrylonitrile, which is used as synthetic fibers in the textile industry. To obtain optimum process operation conditions in the Acrylonitrile manufacturing process, continuous monitoring of several critical components is essential. The on-line monitoring of low levels of MEHQ in Acrylonitrile is described below.

Monitoring MEHQ in Acrylonitrile

MEHQ is a polymerization inhibitor added to Acrylonitrile in controlled amounts to prevent spontaneous polymerization of the Acrylonitrile. Controlling the MEHQ addition process is highly beneficial for a number of reasons.

If in excess, MEHQ might result in difficulties in the polymerization stages. If the MEHQ levels are too low, spontaneous polymerization might occur. The MEHQ is a fairly expensive component. Therefore, the rate of return on an analyzer monitoring the MEHQ on-line can be as low as three months depending on the production levels. UV absorbance spectrophotometry, an accurate and robust analytical technique, was applied to this application.

MEHQ Detection Method

The OMA-517, a diode array process spectrophotometer, was installed on-line, measuring a complete UV absorbance spectrum of the process stream and auto-processing the data to output the MEHQ concentration.Figure 1 shows the absorbance spectra of MEHQ in Acrylonitrile. In order to eliminate baseline interference due to the Acrylonitrile, the 1st derivative of the absorbance spectrum was used (see figure 2). MEHQ in Acrylonitrile Standards were used for calibration. STD of calibration was 0.22 PPM MEHQ. Figure 3 shows the calibration results, comparing lab and pro.

Sampling system

The sampling system's main task is to allow for auto zeroing at predefined time intervals. The natural material for zeroing would have been pure Acrylonitrile, but that could not be used since storing it in the pure form

is extremely difficult. Water would have been the second choice; however, due to miscibility effects between Acrylonitrile and water, interface problems such as coating of windows makes water an unsuitable material for zeroing. Though it is well known that using air for zeroing while running liquid samples is not an ideal solution, mainly due to air bubbles, it was found to be the best solution. The sampling system was designed to minimize the formation of bubbles. A time delay in switching from sample to air and vice versa was set to allow for a smooth transition between the liquid and the gas matter in the flow cell.

Conclusion

MEHQ in Acrylonitrile was monitored continuously by a diode array process spectrophotometer. Air was used for blanking. The MEHQ was measured at an accuracy of ± 0.22PPM. The sampling system was designed so that auto zeroing at predefined time periods using air can be performed.

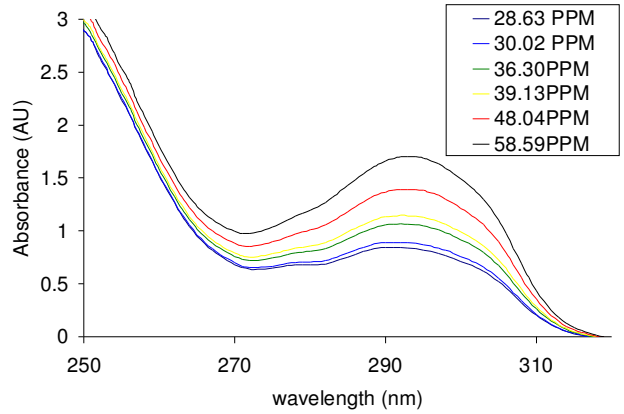

Fig 1: absorbance spectra of MEHQ in Acrylonitrile

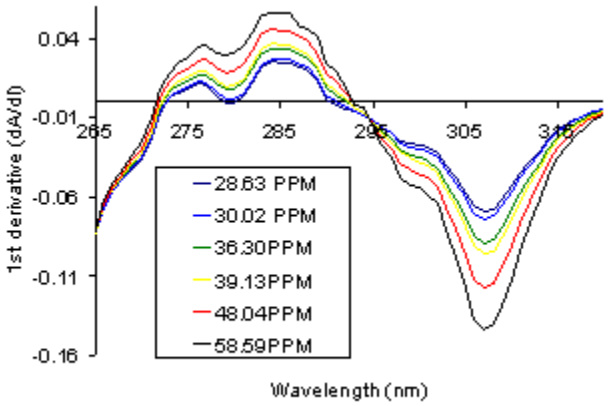

Fig 2: 1st derivative spectra of MEHQ in Acrylonitrile

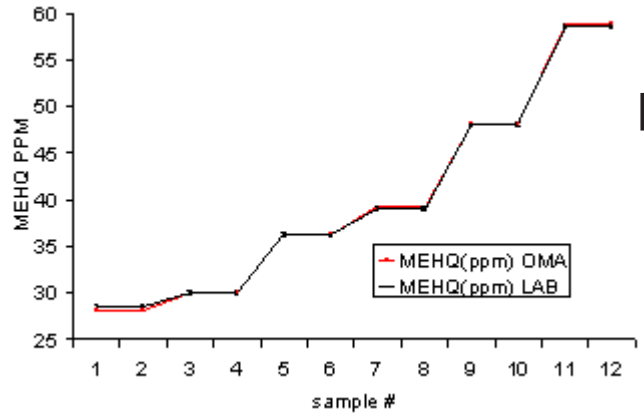

Fig 3: Trends graph of laboratory and diode array results

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