

# METTLER TOLEDO APPLICATION NOTE

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## pH And ORP Monitoring For The Indigo Dye Process

### BACKGROUND

Indigo dye is used for dyeing cotton and other cellulose fibers such as rayon. In its pure form indigo dye is not soluble in water, so the dye bath contains additives to solubilize it.

Sodium hydrosulfite (“hydro”, sodium dithionite,  $\text{Na}_2\text{S}_2\text{O}_4$ ) is present in the dye bath to reduce the indigo dye to its reduced (leuco) form. Sodium hydroxide (caustic,  $\text{NaOH}$ ) is added to the bath to form water-soluble phenolates with the reduced indigo dye.

After the dye has been applied to the fabric, it is re-oxidized to its insoluble form, which makes it resistant to water and other environmental factors.

### THE PROCESS

The properties of the dye are affected by the pH of the bath solution. There are actually two phenolates that can form in solution depending upon the amount of caustic added.

The first is monophenolate, which is predominant between pH 11.0 and pH 11.5. Monophenolate has relatively low affinity for the fabric, which leads to a more thorough and uniform dye penetration.

The second, diphenolate, predominant between pH 12.5 and pH 13.5, has a higher affinity for the fabric and tends to aggregate on the surface of the fabric. This leads to a deeper blue color, a faster rate of dyeing (strike rate), and the ability to be washed down to a faded denim look.

Measurement and control of the dye bath pH can be used to maintain either form of phenolate in order to produce the desired effect.

The oxidation-reduction potential (ORP) must be maintained at a low enough level to keep the indigo dye in the reduced (leuco) form for solubility. The ORP of the bath can also affect the final shade. The typical ORP range is -760 to -860 millivolts, with the actual target ORP dependent upon the desired final shade.

### INSTRUMENTATION

The pH of the dye bath is greater than pH 10, so the pH electrode used must have a high alkalinity glass membrane to minimize the effects of sodium error. The Xerolyt<sup>®</sup> sensor is the electrode of choice. The Xerolyt solid polymer reference system with open annular junction should be used for both the pH and ORP sensors to minimize junction contamination.

Mounting of the sensors can be either in-line using the InFit<sup>®</sup> 761 Stationary Housing, or submersed using the InDip<sup>®</sup> 500 Series Immersion Housing depending upon the particular design of the equipment. In any case, the sensors should be mounted in an area where the measuring solution is homogeneous. When controlling caustic ( $\text{NaOH}$ ) is added, the sensors should be immersed into a well-mixed sample. The lag time between the moment the additives are applied and the time the sample reaches the sensor should be minimized.

The 2100 pH/ORP Analyzer is recommended for this application.

## PRODUCTS

### 2100 pH/ORP Analyzer

- Detachable front panel and plug-in terminals for ease of installation
- All functions accessible through the keypad for increased ease of use
- Continuous sensor and transmitter diagnostics to monitor performance
- FM certification for Class I, Div 1 & 2 Environments and CSA General Purpose Approval
- 3 year warranty

### Xerolyt<sup>®</sup> pH/ ORP Electrode

- Patented Xerolyt solid polymer reference system maintains a stable potential for accurate and repeatable pH measurement and low maintenance
- Open junction eliminates reference clogging and extends sensor life
- High pressure resistance eliminates requirement for pressurizable housing
- Xerolyt solid polymer is particularly suitable for use in emulsions, suspensions, heavily contaminated or sulfide-containing media, and solutions with high concentration of suspended solids

### InFit<sup>®</sup> 761 Stationary Housing

- For vertical or side mounting in bioreactors and pipes
- Wide range of process connections and materials of construction to meet most application requirements
- Steam-sterilizable & autoclavable for hygienic and CIP/SIP applications
- Optional integral cage shields the sensing membrane from accidental breakage

### InDip<sup>®</sup> 500 Series Immersion Housings

- Economic method of sensor installation in open tanks and vessels
- Rugged protective cage protects the sensor against abrasive solids in the process medium