Inline pH Measurement for the Food / Dairy and Beverage Industry
A white paper by Endress+Hauser, Inc.

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The Role of pH Control

The ability to monitor and control pH has important applications in many food processing operations. Food quality and freshness are often directly related to pH. Sanitary procedures such as chilling, cleaning, and disinfecting control spoilage (souring) of dairy, meat, and other food products. However, pH measurement is a tool to ensure that freshness and processing are optimized. Some of the food products where pH is important include yogurt, cheese, fruit and vegetable juices, along with sauces, purees, and pastes, and value-added condiments such as taco and spaghetti sauces. Maintaining the proper pH levels in these foods is important for food safety, as well as quality. FDA regulations (CFR 21-114) govern the manufacture of low-acid foods that have been naturally or artificially acidified to a pH of 4.6 or lower to inhibit the growth of C. Botulinum, a bacteria that creates the exotoxin responsible for deadly botulism.

The regulations specify the manner and frequency for measuring and documenting the pH of these foods during manufacturing. It is also important to measure and control pH because taste and product qualities are often closely related to pH. An example exists in the incubation of yogurt. If the incubation is not stopped at the right pH level, the product taste is directly affected.

Controlling pH Today

Conventional lab measurement (manual sampling) is the most common method of monitoring the pH of a process and ensuring compliance to FDA regulations (CFR 21-114). Manual sampling is time consuming and subject to sample contamination. Even small changes in temperature can result in misleading measurement results. Because of these issues, most pH measurements are taken on an infrequent basis - at intervals ranging from 5 minutes to several hours. This makes tight process control difficult to achieve. In addition, infrequent sampling could potentially result in inconsistent product quality, or a waste of product, additives, and preservatives.
In-line Continuous pH Measurement vs. Manual Lab Testing

To illustrate some issues with manual sampling, measurements taken manually in a cheese sauce application were plotted on a graph with in-line measurements. All of the manual sampling measurements were taken by the same operator using the same sensor.

Clearly, the manual measurements have much greater variation, even though the same person was doing the measurements with the same sensor. The in-line measurement not only provides less variation in the measurement, but the benefits of continuous measurement. In-line continuous measurements provide tighter control and faster response to changes, while also helping reduce labor costs. However, conventional glass sensors are generally not accepted in food processing applications because of concerns about glass breakage directly in the process stream.
Non-glass pH Electrode (IsFET)

The liability involved simply does not justify using in-line glass pH sensors. In response to this concern, non-glass pH probes based on IsFET technology have been commercially available for in-line use. However, these probes are sensitive to extreme pH conditions encountered during normal CIP operations. In particular, exposure to caustic materials at elevated temperatures significantly reduces the useful life of these pH sensors. To overcome this problem, Endress+Hauser has developed the CPS 401, a pH sensor based on IsFET technology that can be retracted during CIP operations. The sensor itself can be automatically cleaned while it is retracted. Therefore, this new probe offers an excellent solution for in-line pH measurement.

The mechanical construction of this probe also meets the rigorous process conditions in food applications such as changing temperatures, pressures, and pH values. As a preferred sealing technology, EPDM form seals are used to hold the pH sensitive IsFET sensor element in place. This electronic board contains all necessary sensor connections, and a temperature sensor for temperature compensation. To ensure food safety, a sensor behind the EPDM seal detects moisture inside the sensor. The sensor can immediately be retracted if the seal is compromised.

Another critical requirement in food applications is fast response time. To achieve this, the reference junction's design is key. In the Endress+Hauser probe a fine structured ceramic junction minimizes memory effects due to coating. This allows for response times of a few seconds for tight process control.

A temperature sensor integrated in the front part of the sensor body is used for temperature compensation of the measurement. It is important to avoid errors from temperature effects in CIP applications with hot process phases. The sensor's plastic construction causes an insulation effect that will slow down the temperature compensation. By placing the temperature sensor in the very front tip of the IsFET sensor, this effect is minimized.

1. ISFET (Ion Selective Field Effect Transistor)
2. Chip board
3. EPDM seal
4. PT100 temperature sensor
5. Reference junction
6. Virgin PEEK body
7. Meets 3A sanitary standards and has passed EHEDG test
In order to prove this new sensor’s capabilities, testing occurred in-line in series with a liquid-filled glass pH sensor. The liquid filled glass pH sensor was chosen because this technology provides the fastest response time available today. In addition, it is considered the most accurate. The test was performed in a number of different media, and the results were very consistent. The IsFET was comparable to the glass sensor, responding to pH changes within one to three seconds.

The graph shows the performance of an IsFET non-glass pH sensor used to monitor the fermentation of yogurt. It is again compared to a liquid-filled glass pH electrode, which is considered the most accurate means of in-line pH measurement. It can be seen that the IsFET sensor provides a measurement comparable to that of a high performance liquid-filled glass pH sensor. The control of the fermentation process is not only critical to the quality of the product, but can also be a tool to plan and manage downstream processing or packaging.

The sensor is constructed of FDA Generally Recognized as Safe (GRAS) and 3-A listed materials such as virgin PEEK. The material is virtually unbreakable and withstands handling without damage. The European Hygienic Equipment Design Group (EHEDG) offers standardized tests to prove the cleanability of sensors and transmitters for
the food, beverage, and pharmaceutical industry. The construction shown has passed EHEDG’s tests for clean-ability as well as the device’s ability to be sterilized. The CPS 401 sensor cable is fitted with a watertight connector that simplifies sensor replacement and protects the sensor, ensuring the long-term integrity of the seal, even when the new sensors are installed.

**Complete System Solution**

The IsFET sensor can be universally applied with an in-line, removable Tri-clover connection, or in conjunction with a retractable probe holder. The retractable probe holder is preferred because the probe can be retracted during CIP and kept wet during longer process shutdowns. The basic purpose of a retractable holder is to access the sensor without opening the pipe system and disassembling the sensor for cleaning and calibration. All pH sensors must be cleaned and calibrated regularly to ensure proper functioning. Endress+Hauser’s retractable holders meet sanitary requirements, such as surface finish and cleanability. They also meet 3-A sanitary standards. To complete the in-line pH measurement solution, Endress+Hauser offers a basic transmitter or a fully automated cleaning and calibration system.

The transmitter is easy to setup via a menu-driven interface. Output for control or limit can be assigned for specific applications. The automatic calibration and cleaning system makes maintenance very user friendly. Operators simply need to keep the buffer and cleaning solutions replenished. Fully automated pH measuring systems offer a stand-alone solution with microprocessor-based programming. The system can be programmed to retract the sensor for cleaning and calibration on a time-based interval or at a predetermined pH value. An external input is also available to allow retraction of the sensor on operator command, or from an external control system. Maintenance steps such as cleaning, calibration, and replacement can be performed during process shutdown, or during the CIP cycle when a retractable holder is used. For automatic synchronization to the process, the retraction of the sensor can be accomplished manually, or via pneumatic signals from the automatic cleaning and calibration system. Replaceable seals are used to seal the process towards the moving electrode guide and isolate the electrode. In a fully

**Modular component system**

1. ISFET or conventional Glass pH sensor
2. In-line with triclover connection
3. Retractable probe holder for automated cleaning and calibration
4. Re-usable cable
5. Transmitter/controller
6. Automatic cleaning/calibration unit
retracted position, the electrode resides in a separate cleaning chamber. Cleaning or calibration liquids are connected to the chamber flush ports. These defined and repeatable conditions achieve better cleaning and calibration results. The lifetime of the pH electrode is extended since no manual interaction is necessary. The overall reliability and accuracy of the pH measurement point is optimized with an automated retractable system for better process control.

Potential Cost Savings

It is difficult to put a dollar value on the benefits of in-line pH measurement. Each application must be evaluated on its investment return. However, this solution will offer substantial product quality improvements and a reduction in operating costs in many applications. The following are just a few examples of areas where significant improvements can be achieved.

Example: Cheese manufacturing

With a production rate of 50-pounds per minute, a 30-minute lab sample interval could equal a loss of 1,500 pounds of product worth approximately $1,500 to $2,500 if the pH is not within the permitted values. In-line measurement could eliminate these costs.

Example: Fermentation control

To allow preparation of a packaging line or downstream processing, pH control is critical in the prediction of fermentation end points. Product left too long in a fermentor can have a detrimental effect on the end product. These concerns can be eliminated with in-line pH measurement.

Example: Starter culture for cheese, sour cream, and yogurt

A pH reading is used to quench (stop growth) at the desired pH, allowing the next batch to start. In-line pH measurement offers a potential for one to five additional batches per week. This could translate into an additional production yield totaling $10,000 to $150,000.
Summary

It is a big step for most industrial processing operations to stray from the conventional and well-accepted manual method of pH control. However, like many aspects of business today, change is unavoidable. Individuals should proactively participate and take actions that effectuate positive changes in plant operations.

With pH measurement in regulated applications, several groups of people need to be involved to implement the change in a beneficial way.

- Process engineers need to design the system to work around the in-line measurement.
- Operations and quality managers need to understand the benefits.
- The sanitation team must feel comfortable with the device’s cleanability.
- The lab team must be certain that the measurement data can be used to make process improvements.

When applied correctly, the non-glass IsFET pH sensor will enable better process control and safer operation. The advantages and cost savings will vary, depending on the application and environment. Look for areas where pH is currently not being measured. In-line pH measurement can provide a competitive advantage in the food / dairy and beverage industry.

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