pH Application Note

pH Measurement of Jam and Jelly

InLab Solids Pro-ISM Sensor

pH measurement plays an important role in Jam and Jelly manufacturing processes. The main ingredient, pectin, requires a specific pH in order to solidify and create the characteristic flavor and gel texture of the final product. The production and process controls for jam and jellies are regulated by 21 CFR 150 (Code of federal regulation). Further, since it belongs to the category of acidified foods, they are also regulated by 21 CFR 114, wherein the final products are required to have an equilibrium pH of 4.6 or lower. The conventional pH sensors are generally not able to withstand the pressure of being pushed into a semisolid sample and, moreover, the ceramic junction and membrane shape can pose various challenges in analysis of such semisolid products. METTLER TOLEDO's InLab Solids Pro-ISM is modified to enable easy measurement of pH in such samples with hassle-free piercing. The improved functional features like open junction, solid electrolyte and sensitive pH membrane facilitates faster and more accurate pH measurements.

METTLER TOLEDO

Introduction

Jam and jellies are fruit preserves that enable enjoyment of seasonal fruit throuhout the whole year. These semisolid food products are prepared by combining acidity, sugar and pectin in the correct proportion. High acidity pH is maintained in the product to prevent microbial growth and enhance the color and flavor of the fruit.

Jam and jellies are made from a variety of fruits. Depending on the flavor, either a single fruit or a combination of fruit pulps are used in the making of a jam, while jellies are made from the strained fruit juices. The manufacturing process involves chopping, pasteurization, extraction and cooking of the fruit. Basically, the main ingredients comprises of 1% pectin, 65% sugar and acid concentration to bring the pH within 2.7 - 3.6 range. For a premium batch and flavor consistency, the ratio of these ingredients as per the recipe must be maintained.

Importance of Measurement

Citric acid, tartaric acid and malic acid are the edible acids that are used in the production of jam. Citric acid is the most popular among them. As different fruits have different acidity, pH measurement is very important to regulate the correct acidity. The low pH level helps in the gel formation of pectins and is termed as "sugar-acid-gelling mechanism". For a stable and safe product, the technological aspects of the process has to be followed and are covered under CFR Title 21 Part 150. In case of sugarfree or low sugar jam or jelly products, special considerations have to be taken due to the reduction of the amount of sugar in the final product. To stablize the flavor with such low sugar levels, more fruit content is required along with a reduced acidity. Hence a different balance between sugar/ acidity/ fruit flavor is followed. Preservatives have to be added to preserve the shelf life of such products. All these pH control acidified food products should comply with manufacturing process regulation as mentioned in 21 CFR 114. Also, the person supervising the production needs to be FDA approved as specified in 21 CFR 114.10.

Measurement Challenges

Determination of pH by direct insertion is difficult using a conventional electrode due to the semisolid nature of the sample. Moreover, interaction of the sample with the reference electrolyte is restricted in a ceramic junction based electrode. There is an increased risk of clogging the junction and cleaning it efficiently is difficult. This effects the overall efficacy and accuracy of pH measurement and the user may experience unstable and erroneous results.

The table below outlines the challenges and negative impacts on pH measurement results when using a standard pH sensor.

Sample Challenge	Sample Impact
Semisolid sample consistency with gel matrix	Piercing through the sample is difficult with standard shaped pH sensor. Delicate sensing membranes can have abrasions during measurement.
Sample preparation	Alternate method requires blending the sample into paste for measurement and is time consuming.
Cleaning of sensor after measurement	Sensor fouling due to deposition on the delicate glass membrane, which is difficult to clean. Semisolid nature of the sample can easily clog the reference (ceramic) junction.

Direct pH measurement of the jam and jelly samples needs the pH sensor to pierce through the gel matrix. Conventional sensors are inefficient for direct insertion and the sensing membrane may get scratches during the measurement process. The alternate method includes blending of the sample into a slurry to facilitate proper sample interaction with the sensor. This is time consuming. Moreover, the addition of water may introduce increased scope of errors in pH determination.

The standard sensors have typical ceramic fritted junction that may get clogged easily on being used in samples with semisolid gel matrix as in jams and jelly. Moreover, the outflowing aqueous electrolyte from such junction gets restricted and cannot readily mix with the sample components to provide a stable signal. The increase in time to reach an equilibrium pH using conventional pH electrode, leads to high response time and inaccurate results. A sensitive pH glass membrane can help in such situation.

Figure 1: Sample impact on a conventional pH sensor having a ceramic junction

Direct measurement for reliable pH

InLab Solids Pro-ISM (51344155) is a specialist sensor for measuring the pH of jam and jelly with accuracy and precision. The sensor has a built-in temperature probe and provides Intelligent Sensor Management (ISM) technology, allowing users to accurately capture all critical measurement parameters. It helps in keeping a track of pH analysis including calibration details and sensor performance during the process and batches of final product, and hence contributes to better management of 21 CFR regulations.

The spear-shaped tip is designed for easy piercing into these semi-solid samples. The sensor has a toughened glass shaft that resists breakage. The pH sensing membrane is constructed from low temperature (LoT) glass, which is sensitive enough to yield fast results. The solid XEROLYT®EXTRA polymer reference system instead of a conventional aqueous reference electrolyte takes away the need of frequent refilling of sensor and facilitates an ideal interaction with the sample.

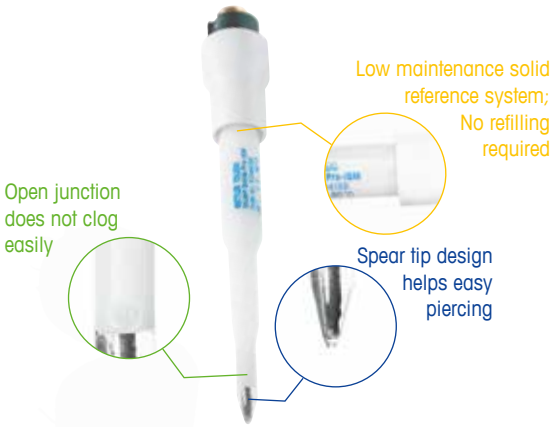

Figure 2: InLab Solids Pro-ISM pH sensor

The open junction replaces the traditional ceramic fritted junction to avoid clogging of the junction and facilitate easy cleaning of the sensor. These features makes this sensor a low maintenance and robust to handle. Overall the design of the sensor simplifies the pH measurement of the semisolid jam and jelly samples and thereby acquiring reliable and hassle free pH results.

Procedure and Method

Calibrate the sensor using buffers that bracket the sample pH range. In this case, a three point calibration using pH 2.00, 4.01 and 7.00 can be done using MT Europe buffers. Record the calibration slope and offset value for the electrode. A slope value of 95 -105 % and an offset of 0 ± 30 mV ensures reliable measurement. 21 CFR 114.90 part elaborates on the methodology to determine pH in such acidified food products.

Figure 3: InLab Solids Pro-ISM sensor to measure the pH of jelly samples

Measure the pH of the jam or jelly sample with InLab Solids Pro-ISM pH sensor by direct immersion of the electrode tip into the sample. Record a minimum of two pH values of a well mixed sample, as mentioned in 21 CFR part 114 protocol. The representative pH reading recorded should be within ± 0.05 pH units.

Results

Typical pH measurement results (in triplicates) for jam and jelly samples using InLab Solids Pro-ISM Sensor are enlisted in the below table:

Sample	Mean pH Value	Std.Dev.	Avg. time (s)
Mixed fruit jam	2.887	0.01	30
Raspberry jam	2.758	0.01	08
Sugarfree jam	3.051	0.01	08
Lychee jelly	3.567	0.01	07

Expert Tips

- After every measurement, thoroughly clean the pH sensor using deionized water. Use mild soapy water if required. In that case, rinse off again using deionized water.
- Do not rub the sensor surface; always dab off the excess water using tissue paper.
- Regular maintenance is very important for prolonging the lifetime of pH electrode. Periodic reconditioning of the electrode in 0.01M HCl is recommended, based on the sensor performance. Frequency of reconditioning would depend upon the number of samples analyzed per day and life of the sensor. An old sensor may need frequent conditioning, compared to a new sensor. Remember to re-calibrate the sensor after reconditioning.
- The pH range for this sensor is 1 to 11 pH units and hence should not be exposed to harsh acidic (below pH 1.00) or alkaline (above pH 11.00) solutions.
- In between measurements or when the electrode is not being used for brief period, it is best to keep the electrode in wetting cap filled with InLab Storage Solution (30111142).
- Never store the electrode dry or in distilled water, as this affects the pH-sensitive glass membrane shortening the lifetime of the electrode.
- Ensure use of correct buffers in the correct sequence. Always use fresh buffers. Check expiry date
- Electrode handling movies on:

YouTube

- Comprehensive range of pH meters, electrodes, solutions, and accessories:
  www.mt.com/pH

References

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