MEASUREMENT AND INFORMATION SYSTEM FOR PH AND TEMPERATURE VALUE MONITORING OF GRAPE MUST IN THE WINE PRODUCTION PROCESS

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This research paper describes the utilization of experimental measurement and an information system based on an ATmega328P microcontroller and a 16-bit A/D converter for application in the process of measuring and determining the pH value and temperature of the grape must during wine production. The system also contains a WiFi module enabling the measurement values to be monitored wirelessly via a local WiFi network or the Internet. Measurement of the pH value and temperature of grape must represents the basic steps in wine production. During the wine fermentation process, the pH value changes, while the temperature is maintained at 16 degrees, and this process was monitored in the wine cellar of the agricultural farm Milosavljević in Svrljig, Serbia. In addition to the proposed system, a professional pH meter was used as a reference measuring device, Eutech CyberScan pH 510, which was calibrated in a national laboratory. Measurements over the period of 10 days are presented. A comparison of the obtained measurement results with those obtained from a professional pH meter is also presented.

Keywords: measurement and information system, pH value, temperature sensor, grape must.

Introduction

The kinetics of wine fermentation, as well as those of other alcoholic and non-alcoholic beverages, involve intricate biochemical reactions, where pH variations and temperature are fundamental elements. Throughout fermentation, the pH of the must generally decreases due to the production of organic acids, resulting in the stability and sensory profile of the final product. Temperature regulation is equally critical, as elevated temperatures may impair yeast activity, while suboptimal temperatures can hinder fermentation. Systematic monitoring of these parameters facilitates precise control over the fermentation process, ensuring optimal quality in wine and other fermented beverages. Effective management of these factors contributes not only to the enhancement of organoleptic attributes but also to improved energy efficiency, minimized waste, and increased process stability. Advanced models for fermentation simulation offer deeper insights into these dynamic processes, enabling the development of control mechanisms that enhance both production efficiency and the quality of the beverages [1-5].

The fermentation process of grape must involves complex biochemical transformations, marked by distinct stages characterized by pH and temperature fluctuations. Initially, fresh grape juice exhibits high acidity, with pH values typically below 3.5. As yeast activation begins, sugars are metabolized into ethanol and carbon dioxide, leading to the onset of intensive alcoholic fermentation. This phase is accompanied by a gradual increase in temperature due to exothermic reactions. The pH level may experience a slight rise due to the buffering effects of potassium and the dissociation of organic acids. As fermentation progresses, ethanol accumulation inhibits microbial activity, and the pH stabilizes toward the end of alcoholic fermentation. Subsequent malolactic fermentation involves the conversion of malic acid into lactic acid by lactic acid bacteria, resulting in a further rise in pH and a smoother sensory profile of the wine. Temperature control during these stages is crucial for ensuring optimal yeast activity and preserving the biochemical integrity of the must [6-11].

One of the most common chemical measurements conducted in process control is pH measurement. Usually, as part of the quality control in a production line in almost every industry, pH measurement can be conducted either inline, online, at line, or offline. In any alcohol technological process, determination of the pH value or range is of immense importance [12]. A chemical shall be highly effective at a particular pH [13]. pH or acidity is used to express the level of acidity or base that is possessed by a substance, solution, or object. Normal pH has a value of 7, while a pH value > 7 indicates that

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the substance has alkaline properties, and a pH value < 7 indicates acidity. pH 0 shows a high degree of acidity, and pH 14 shows the highest degree of alkalinity. Any fermented liquor or alcoholic beverage, such as wine, distilled spirits, or beer, contains ethyl alcohol or ethanol (CH_3CH_2OH) and represents an acidic beverage. In the case of non-alcoholic beverages, the most common acids are carbonic, citric, and tartaric. pH measurement is a more useful parameter in comparison with the titratable acidity (TA) level, although both measurements are very common [14]. The physicochemical characteristics of the juice are necessary to determine for winemaking purposes. To meet wine standards, it is necessary to know specific quality parameters, which represents an old practice in wine production. Depending on the type of wine, pH, TSS, and acidity values differ [15]. Faults in wine are associated with increased pH values that result in an increased potential risk of undesirable microorganisms. [16] Just as time, mass, and dimensions are expressed, the physical state of matter is expressed in the form of temperature in a similar way [17]. In applications involving liquids, temperature measurement is a fundamental aspect, as it is in various industrial processes in industries such as food processing, chemical engineering, pharmaceuticals, and environmental monitoring, controlling liquid temperature is a critical process. Product quality, process efficiency, and regulatory compliance are ensured by precise temperature measurements. Most often, traditional methods of temperature measurement are not suitable for continuous or remote monitoring and often include specialized equipment or manual monitoring of changes in temperature values [18]. The voltage count or pH value display for buffer solutions is performed using a digital pH meter, which has a pH probe to conduct the electrical signals. An internal or sensor electrode and a reference electrode form the pH probe [19]. There are a lot of developed devices with similar characteristics to the one proposed in this research. In the research, they also compared the characteristics of the developed device with a commercial FieldScout pH 110 pH meter or with the laboratory-type PHM83 pH meter, and stated that the error ranged between a minimum of 0.43% and a maximum of 10.1% or a minimum of 1.02% and a maximum of 11.04%. [20]. However, all these developed devices are intended for determining the current pH value, and do not include temperature sensors for determining the temperature value while measuring the pH value. Moreover, an advantage of this proposed system is the possibility of monitoring the changing pH value for a long period due to the possibility of logging data. Another advantage is its ability to connect to the Internet or monitor values locally via a network due to the integrated wireless module, which provides a significant improvement compared to existing devices, including laboratory instruments. With all the improvements, this device is a complete measurement information system that can also be utilized in other areas and other technological processes.

This study presents the development and evaluation of devices designed for comprehensive process monitoring and measurement applicable across various stages of beverage production, with a particular emphasis on alcoholic beverages. The proposed devices are capable of performing precise measurements of key process parameters, including pH and temperature, thereby contributing to improved process control and product quality across the beverage industry.

Materials and methods

For this research, different types of materials and equipment were used. For the calibration of the device, different aqua solutions of the acids and bases with different pH values were used. The materials used for the realization of the experiment were primarily liquids for testing the equipment and electronic components for the implementation of the proposed system. Determination of the value of the electrochemical potential that occurs between the unknown solution outside the glass electrode and the solution contained in the known glass electrode (glass membrane) represents the basis for the measurement of the pH value. Hydrogen ions, which are relatively small and active, will interact with a thin layer of glass bubbles. The electrochemical potential of hydrogen ions, termed the potential of hydrogen, is measured with the glass electrode. The pH sensor measures the electric potential between hydrochloric acid (HCl) at the internal electrode and potassium chloride (KCl), which is a solution in the reference electrode [21]. Figure 1 shows the internal structure of the pH electrode.

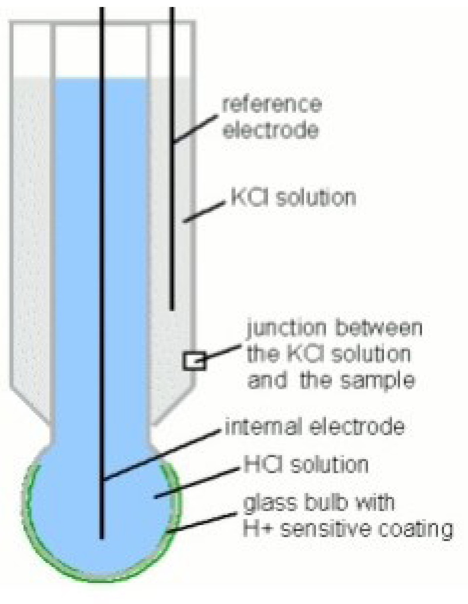

Figure 1. Internal structure of pH electrode [22].

For measuring the solution temperature and providing high-precision temperature data, DS18B20 temperature sensor is used, which is placed in a stainless steel

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case suitable for measuring the temperature of liquids and environments with a high percentage of moisture. In addition, this type of sensor can be used in fields such as healthcare, environmental monitoring, and smart homes. This digital sensor is high-precision, highly integrated, low-power, and widely used. It integrates signal processors, digital interfaces, and temperature-sensitive elements, so it is capable of real-time monitoring of temperature and providing accurate temperature data through a digital interface. The temperature sensor monitors temperature changes in the solution and converts the temperature signal into a digital signal. For transmitting these digital signals, that is, for sending temperature values to the microcontroller, a 1-Wire digital interface [23] is used. Figure 2 presents the block diagram of the DS18B20 temperature sensor.

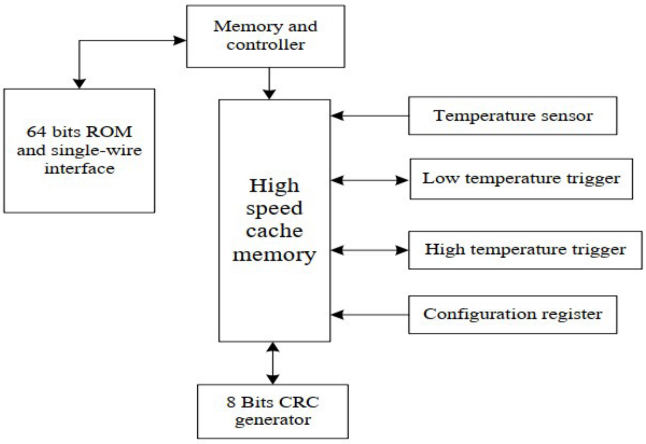

Figure 2. Block diagram of the DS18B20 temperature sensor [24]

For determining the pH and temperature values of the grape must, an experimental prototype microcontroller system was developed, based on an ATMega328P microcontroller with an external 16-bit ADS1115 analog-to-digital converter for conversion of the analog value of the pH from the E-201-C sensor electrode for grape must. This system contains an LCD1602 alphanumeric display for presenting the obtained results. Also, for the temperature measurement the ATMega8A microcontroller is used as a coprocessor, and it takes the temperature value from the digital temperature sensor DS18B20 via One Wire protocol. Communication between two microcontrollers is done by using the UART protocol. Measurement data are recorded on a microSD card with a capacity of 16GB in CSV format, so it is possible to analyze these data in Excel or similar software. Also, this system contains an ESP01 module which is based on an ESP8266 microcontroller, enabling real-time data monitoring over a local wireless network or via the Internet. Thanks to the WiFi module, it is possible to connect this device to a server that hosts artificial intelligence, so complete automation of the process is also possible. For pH value sensing, a pH sensor E-201-C was used. Figure 3 illustrates a block diagram of the proposed system with a connection to the local WiFi network and the Internet.

The proposed system contains more than one main microcontroller, and every chip must have its firmware for normal operation. The flowchart starts with reading the temperature and pH value data. The temperature sensor DS18B20 is connected with the 1-wire protocol. Figure 4 shows the general working principle algorithm for a complete system.

The device testing was conducted during the production of a control batch in the fermentation of Gamay (Gamay) grape must, carried out at the Milosavljević agricultural estate in Svrljig. Conventional phenotyping and genotyping analyses most adequately characterize this variety. One of the top ten most planted cultivars of grapes is the Gamay variety [25].

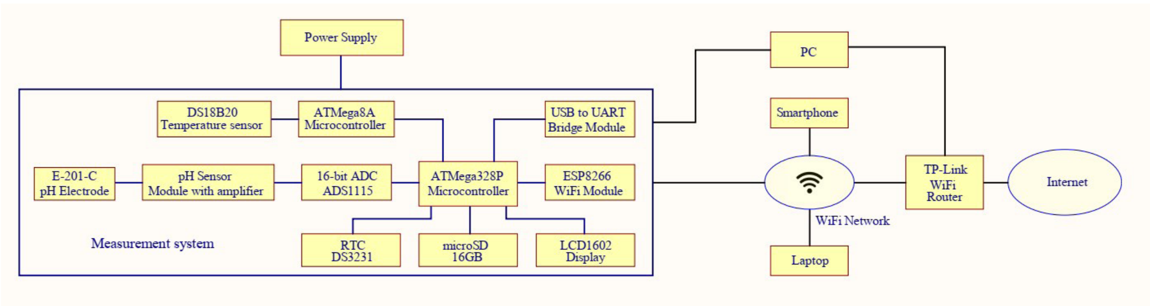

Figure 3. Block diagram of the proposed microcontroller measurement system with network connection

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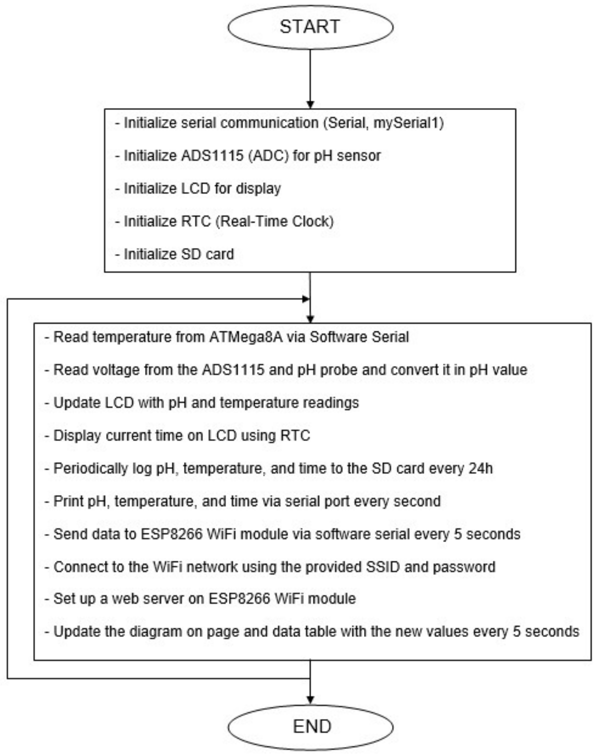

Figure 4. Working principle algorithm of the proposed system for monitoring pH and temperature

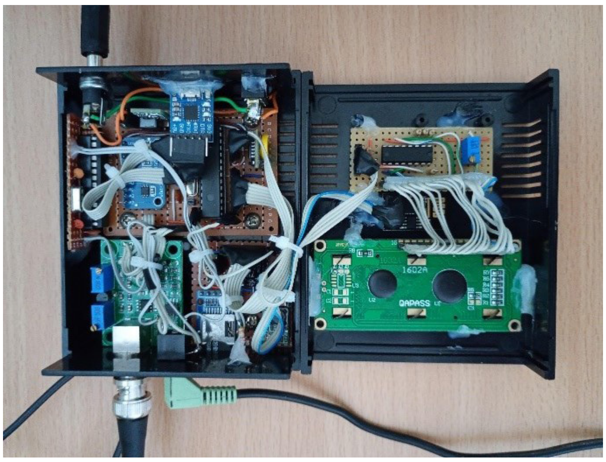

Figure 5. Electronic circuit in a universal plastic box for electronics

Results and discussion

Experimental work was carried out in the laboratory where the proposed experimental system was developed and assembled, and in the wine cellar of the agricultural farm Milosavljević in Svrljig, Serbia, where it was completely tested using a high-precision professional instrument for measurement in the laboratory. The measurement system is partly built from pre-assembled modules and custom-made components, which are soldered onto a universal printed circuit board, including the microcontroller, RTC, and microSD card.Complete electronics for the device are placed in a plastic box designed for electronic devices. On the front side of the case, there are connectors for connecting sensors, a slot for a microSD card, and a power switch, while on the back side, there is a power connector, which is 9 VDC from the external power adapter. Figure 4 presents the electronic circuit of the system in a plastic box, and Figure 5 presents the complete system with connected sensors.

Figure 6. Fully assembled device with connected sensors

Before monitoring the fermentation process, the device was calibrated, and control measurements of the pH values of the prepared chemical solutions were taken. The chemical solutions used to check the measurement accuracy were aqueous solutions of acetic acid (CH_3COOH), hydrochloric acid (HCl), and potassium hydroxide (KOH). The measurement results and relative errors are presented in Table 1.

Table 1. Results of pH solution measurements and relative errors

Measurement Number	Chemical Solution	Results of pH measurement from Eutech CyberScan pH 510	Results of pH measurement from the experimental device	Error(%)
1.	CH_3COOH	1.75	1.78	1.71
2.	CH_3COOH	2.38	2.40	0.84
3.	HCl	0.70	0.71	1.43
4.	HCl	1.15	1.17	1.74
5.	KOH	13.10	13.18	0.61
6.	KOH	12.76	13.12	2.82
7.	KOH	12.20	12.29	0.74

The fermentation process of the proposed system was observed for 10 days. pH value was sampled once per day, and data were stored on a MicroSD card in CSV format. The temperature during fermentation was constant at 16 °C. Figure 7 presents the complete connection of the system in experimental conditions with a connected laptop on the network.

Figure 7. Complete connection of the system in experimental conditions

The measured pH values illustrate the kinetics of the traditional spontaneous fermentation of the wider Gamay (Gamay) grapes, which was carried out at the Milosavljević farm in Svrljig.

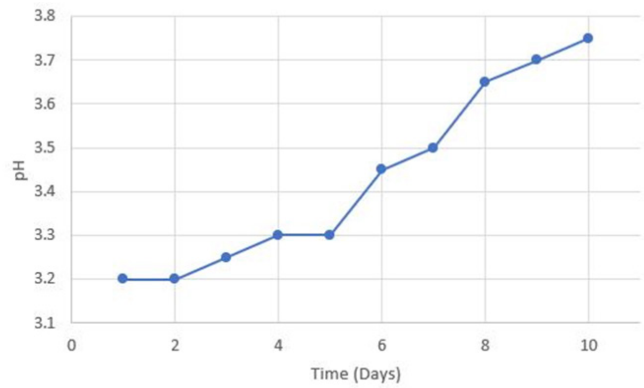

Figure 8. Kinetics of the pH change during the traditional spontaneous fermentation of the wider Gamay (Gamay grapes in an agricultural estate)

The monitored pH values during the first ten days of fermentation indicate dynamic biochemical changes characteristic of wine production and are consistent with literature data. The initial pH of 3.2, typical for fresh grape must with high acidity, remained stable during wild yeast activation (Figure 8). As alcoholic fermentation intensified, a gradual increase in pH was observed, reaching 3.30 in the middle stage. The completion of alcoholic fermentation led to a further rise to 3.45, followed by stabilization at 3.50 before malolactic fermentation. The onset of malolactic fermentation marked a more pronounced increase in pH to 3.65, culminating at 3.75 upon its completion. These results align with reported data, where acid conversion during malolactic fermentation contributes to pH elevation and improved wine stability. The observed pH trajectory underscores the importance of monitoring this parameter to optimize fermentation conditions and ensure consistent product quality in alignment with established technological practices [4, 8, 10, 11].

The pH of the fermentative broth (grape must) was 3.2 at the start of fermentation. During fermentation, i.e., production of ethanol and metabolic processes of yeast, there were slight changes in acidity (pH 3.45). At the end of the alcoholic fermentation, the pH continues to rise slightly to 3.50 and then, due to malolactic fermentation (conversion of malic acid into lactic acid), it rises to 3.75 when, after 10 days of control experimental measurement, the fermentation is stopped. The comparison of the experimental device and professional instrument Eutech CyberScan pH 510 measurement results is presented in the form of the relative measurement error.

During the fermentation process, in addition to measurements taken with the experimental device, periodic checks of pH values were performed using the professional instrument, Eutech CyberScan pH 510. The measurement results obtained over a 10-day period are presented in Table 2, along with the relative measurement errors. During this period, all data were stored on the microSD card.

Table 2. The measurement results during fermentation with the relative error

Measurement number	Time(Days)	Results of pH measurement from Eutech CyberScan pH 510	Results of pH measurement from the experimental device	Error(%)
1.	1	3.18	3.20	0.63
2.	2	3.19	3.20	0.31
3.	3	3.23	3.25	0.62
4.	4	3.27	3.30	0.92
5.	5	3.39	3.30	2.65
6.	6	3.44	3.45	0.29
7.	7	3.49	3.50	0.29
8.	8	3.57	3.65	2.24
9.	9	3.69	3.70	0.27
10.	10	3.73	3.75	0.54

Conclusion

The systems for pH and temperature determination usually provide enough precision with the possibility of measuring other parameters during alcohol production, but they are characterized by high prices and the need for temperature calibration. In comparison with existing solutions, the proposed system provides adequate precision of the obtained results, which is very close to the accuracy of professional measuring instruments, but with a significantly lower price and easier use. During the period of testing the device and the recorded values on the

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MicroSD card, it can be concluded that the device operates normally without interruption, and due to its stability, it can be used in the beer and spirit industry as well as in the production of milk and other non-alcoholic drinks. The implemented system will be employed in future research related to food production processes, because its simplicity enables application in other areas, such as the processing of milk and dairy products. The obtained results have inspired further research focused on the practical application of these devices in the production of alcoholic and non-alcoholic beverages, with particular emphasis on monitoring and optimizing the kinetics of fermentation processes to enhance production efficiency and product quality, especially since their simplicity enables application in other areas of the food and beverage industry.

Acknowledgment

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Izvod

MERNO-INFORMACIONI SISTEM ZA MERENJE pH VREDNOSTI I TEMPERATURE ŠIRE U PROCESU PROIZVODNJE VINA

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Ovaj rad opisuje korišćenje eksperimentalnog merno-informacionog sistema zasnovanog na ATMega328P mikrokontroleru i 16-bitnom AD konvertoru za primenu u procesu merenja i određivanja pH vrednosti i temperature šire tokom proizvodnje vina. Sistem takođe sadrži WiFi modul tako da se merne vrednosti mogu pratiti bežično preko lokalne WiFi mreže ili Interneta. Merenje pH vrednosti i temperature šire predstavlja osnovne korake u proizvodnji vina. Tokom procesa fermentacije vina dolazi do promene pH vrednosti dok je temperatura konstantna na 16 stepeni i ovaj proces je praćen u vinskom podrumu poljoprivrednog gazdinstva Milosavljević u Svrljigu u Srbiji. Takođe, uz predloženi sistem, kao referentni merni uređaj korišćen je profesionalni pH metar Eutech Cyber Scan pH 510 koji je etaloniran u nacionalnoj laboratoriji. Prikazana su merenja tokom 10 dana. Urađeno je poređenje dobijenih rezultata merenja sa rezultatima merenja profesionalnog pH metra.

Ključne reči: merno-informacioni sistem, pH vrednost, temperaturni senzor, šira od grožđa.

CRediT authorship contribution statement:
Đorđe Lazarević: Conceptualization, Writing – Original Draft, Investigation.
Jovan Ćirić: Writing – Reviewing and Editing, Methodology, Visualization.

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