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DISTRIBUTED MEASUREMENT SYSTEM FOR BEER ELECTROCHEMICAL PROPERTIES MONITORING

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Abstract

Monitoring the properties of beer at the production stage is one of the key factors affecting the quality of the final product. The chemical composition, the reactions that take place, and the microorganisms that enable its production change the electrochemical properties of beer. Therefore, monitoring them on an ongoing basis can contribute to increasing convenience and certainty as to the correctness of the entire process. One of the methods that allows measurements of the electrical properties of matter *in situ* is impedance spectroscopy. To support beer production, especially in craft breweries, an attempt was made to design, launch, and test a system that is highly scalable and universal, as it can be used both for measurements and process control. The Distributed Measurement System presented in the article fulfils all the aforementioned conditions. The laboratory measurement results were compared with those obtained by using a specialised system for electrochemical impedance spectroscopy.

Keywords: Impedance Spectroscopy, Distributed Measurement System, Internet of Things, MQTT message broker, VPN connection.

1. Introduction

Beer is a low-alcohol beverage obtained by alcoholic fermentation of beer wort. It is produced from malt, usually barley or wheat, water, and hops using yeast. Due to its high microbiological stability, beer has been recognised as a safe and sustainable beverage for centuries. Factors that inhibit the growth of microorganisms in beer are a high concentration of carbon dioxide and low levels of oxygen, low pH, the presence of iso-α-acids from hops, ethyl alcohol, and low levels of carbohydrates and amino acids. Despite this, microbial contamination is an ongoing problem in the brewing industry [1, 2].

Craft beer is much more exposed to microbiological contamination because it is usually not pasteurised and/or microfiltered, as is the case with beers from large brewing concerns. In addition,

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craft breweries are usually not equipped with microbiology laboratories where the production process can be continuously monitored. Therefore, non-pasteurised beers have a relatively short shelf life, and their production is associated with a high risk of technological process failure [3].

The methods of detection and identification of beer contamination caused by microorganisms can be divided into traditional and rapid methods. Traditional methods, including cultures on selective media and microscopic examination, are widely used in the first approach to detection and identification of microorganisms. However, a major disadvantage of these methods is that they are time-consuming. In addition, there is no universal microbiological medium that can detect all species of microorganisms that contaminate beer. Another problem is the detection of anaerobic microorganisms [4, 5].

Rapid detection methods are physical, biochemical, and molecular, which require much less time than traditional methods. In addition, they are characterised by greater accuracy in the identification of microorganisms and ease of execution [6].

One method of microbiological control of food products is the impedimetric method, which uses impedance measurements as a measure of metabolic activity of microorganisms. This method significantly reduces the time of analyses and reduces the amount of work and materials, which is extremely important in a situation where a quick assessment of microbiological quality of a product is needed [7].

All biological materials exhibit dielectric properties. This is directly related to their molecular structure and the fact that during growth, bacteria transform uncharged or weakly charged chemical compounds (high-molecular compounds such as proteins, carbohydrates, or lipids) into highly charged ones (mainly low-molecular amino acids and organic acids). This causes a change in the electrical properties of the culture environment. There is a decrease in the impedance value, *i.e.* an increase in admittance. Measurements of either or both values can be a tool for detecting the presence of microorganisms or their metabolic activity in the environment tested. Based on the measurement of the detection time, that is, the time counted from the start of the test to the appearance of a significant change in the impedance graph, the number of microorganisms in a given volume of material can be estimated [8, 9].

Given the relevance of the problem, the authors decided to use the impedance spectroscopy method to study the properties of beer. The method has so far found many applications, including measurements in countless areas. The continued interest of researchers in the development of the method is an indication of its actuality, as new ways to generate the measurement signal are being developed [10]. Impedance spectroscopy can also be used to characterise food [11]. However, so far it has been a different approach from that presented by the authors in this manuscript due to the single measurement point or sample preparation prior to measurement. Impedance testing of food products at only one point is relatively well described in the literature. The authors have also had the opportunity to conduct such studies [12, 13]. However, multi-point impedance testing of foods appears not to be a popular research topic. Based on previous experience in single-point impedance testing and in the use of distributed measurement and control systems with success [14, 15], it was decided to develop a system combining both topics.

2. Impedance spectroscopy of beer

Impedance spectroscopy (IS) is a measurement method based on a very simple principle, *i.e.* measurement of the impedance of samples in the frequency domain. It can provide measurement results relatively quickly depending on the chosen frequency values. Impedance spectroscopy can be regarded as an alternative to the measurement methods mentioned above. It is used in

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many different areas for detection and measurement because of its large versatility and ease of adaptation to given conditions. In one of the simplest cases, the IS method consists in applying a sinusoidal voltage to the electrode and measuring the flowing current value and the phase shift. Typically, the amplitude of the measurement signal must be small enough (i.e. ≤20 mV) not to trigger additional reactions that could take place in the tested sample. Such conditions are required in the case of *electrochemical impedance spectroscopy* (EIS), which uses the measured impedance values to fit the properties of electric equivalent circuit components. Both static characteristics and phenomena occurring in the measuring cell are modelled by the proposed circuit. Using the measured impedance values in the frequency domain to calculate the permittivity of the test substance is often called dielectric spectroscopy and is mainly used to characterise dielectrics.

In the past, the authors have studied the properties of various materials using IS, including low-conducting liquids such as extra virgin olive oil [12]. The tested samples were filtered and homogeneous; therefore, the results of their impedance measurements did not differ depending on the location of measuring electrodes immersed in oil. Regardless of the depth at which the electrodes were placed, the measured impedance values were the same. In the case of food, homogeneous mixtures are relatively rare. Certainly, this characteristic does not apply to beer in the production stage. Previous studies of the degree of microbiological contamination of beer with the use of IS conducted by the authors concerned the measurement of impedance of the samples only in one place in the measuring cell and were performed on the finished product [13].

In electrochemical impedance spectroscopy, the measured impedance can be expressed as the difference between its real and imaginary parts (1):

$$Z^*(\omega) = Z'(\omega) - jZ'''(\omega), \tag{1}$$

where j is the imaginary unit. Z' and Z'' are often denoted by Z_{re} and Z_{im} respectively for convenience and are the synonym for resistance and reactance. Expressed in this form, the impedance is often presented in a Nyquist plot, becoming a source of knowledge about the substance under test.

In the case of beer, the electric equivalent circuit (Fig. 1) can have the form of a parallel connected resistor R_d with a *constant phase element* (CPE) and another resistor R_s in series [13]. The parameters' values of this circuit can be determined based on the results of sample impedance measurements over a suitably wide frequency range. The most commonly used method for this is to match the experimental data to the proposed circuit using appropriate computer tools. One of the available tools is the pyZwx programme [16], which has been used successfully by the authors in the past.

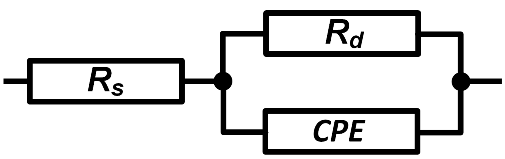

Fig. 1. Electrical equivalent circuit for modelling beer properties.

The elements R_d (resistance) and CPE together describe the diffusion phenomenon. Element R_s represents the series resistance related to the properties and connection of the electrodes. The impedance of the CPE element can be described with the formula (2):

$$Z^*_{\text{CPE}}(\omega) = \frac{1}{(j\omega)^\alpha Y_{\text{CPE}}}, \tag{2}$$

where Y_{CPE} is the admittance value $1/|Z|$ of the *CPE* element at $\omega = 1$ rad/s and α satisfies $0 \leq n \leq 1$. The closer α is to 1, the more similar this element is to an ideal capacitor. Taking into account the equation above, the impedance of the equivalent circuit shown in Fig. 1 will be given by (3):

$$Z^{*}(\omega = \frac{R_d}{1 + (j\omega)^{\alpha} Y_{\text{CPE}} R_d} + R_s. \tag{3}$$

IS is often used for comparative studies. In the case of beer, both the results of measurements of samples in a time interval and the same sample but at different depths can be compared. The recorded differences in the values can be a source of information about the degree of microbiological contamination of beer, as well as the progress of fermentation, maturation process, etc. Extensive knowledge about the electrochemical properties of any sample is necessary for correct interpretation of the results obtained using the IS method. Simultaneous multi-point measurement of beer impedance is not a common topic in published research papers. However, it can be a source of important information about the properties of beer at different depths in the tank in the production stage. Previous studies by the authors' show that the degree of microbiological contamination of beer can result in measurable changes, especially in the value of the Y_{CPE} element in the electrical equivalent circuit [17]. At the same time, the differences between its values at the bottom of the sample and at the surface seem to decrease with increasing contamination [13].

3. Distributed Measurement System

In order to conduct further research on beer quality determination and electrical properties of beer at many points at the same time, the *Distributed Measurement System* (DMS) with *Internet of Things* (IoT) functionality was developed [18]. The Fig. 2 shows the schematic describing the DMS structure.

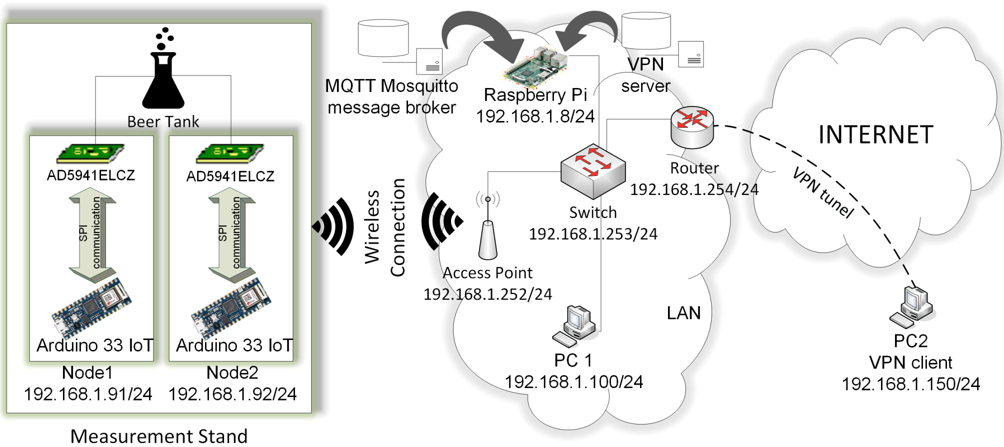

Fig. 2. DMS developed.

The *measurement stand* (MS) is presented on the left side of Fig. 2. There are electrodes immersed in a beer tank. A sinusoidal voltage of specific frequencies is applied, and the resulting value of the flowing current and its phase is measured and converted to impedance values by an Analog Devices AD5941ELCZ evaluation board, including an AD5941 integrated impedance converter. Measurements were made in the frequency range of 0.1 Hz to 1000 Hz with a sinusoidal excitation signal of 100 mV_{p-p}. The electrodes used in the system were the same as those used for the earlier impedance tests of beer [13]. They are symmetrical, made of laminate covered with a gold-plated layer of copper, and were made in the process known from the printed circuit board production. Both AD5941ELCZ boards used in the system are connected to separate pairs of electrodes through the signal conditioning circuit. The connection between the individual MS components mentioned above is shown in Fig. 3.

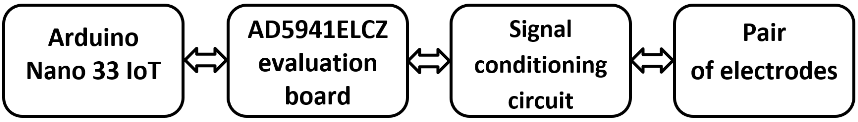

Fig. 3. Interconnection of the components of the measurement stand.

Triggering the measurements and reading the measurement data is handled by the Arduino Nano 33 IoT node. Communication between the Arduino Nano 33 IoT node and the AD5941 system is carried out using a *Serial Peripheral Interface* (SPI). The MS consists of two Arduino Nano 33 IoT nodes. Thus, simultaneous measurement from two pairs of electrodes is possible. The reading of the measurement data is carried out by means of the data flow shown in Fig. 4.

The idea of data flow presented in Fig. 4 requires a series of measurements to be triggered, the broker's *MQ Telemetry Transport* (MQTT) reaction to the user's action consisting in sending a measurement request, the measurement results transfer to the user's application executed by the broker MQTT and data visualisation with the possibility of the subsequent analysis.

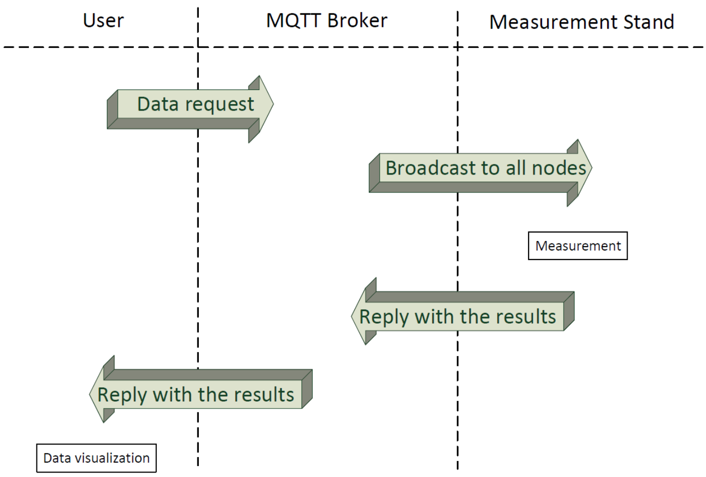

Fig. 4. Data flow in the DMS.

3.1. Graphical user interface

According to the data flow shown in Fig. 4, the user has the option to start a series of measurements. The application's *Beer TANK graphical user interface* (GUI) is shown in Fig. 5.

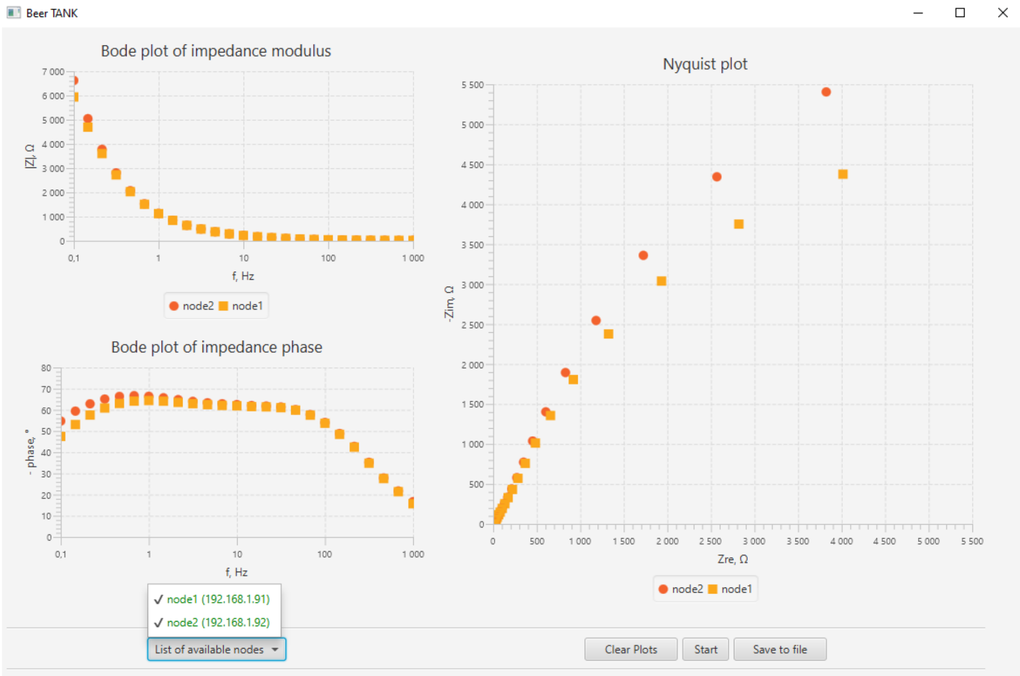

Fig. 5. Beer TANK Application GUI: Node Availability Status, Associated Data, and sample results.

The *Beer TANK* application (Fig. 5) also allows the user to save the read measured values to a file in CSV format for later analysis. Furthermore, it is possible to start a new series of measurements with deletion of previously read data. The connection status with individual nodes in MS is visualised in the lower left part of the application. Based on the colour coding of the nodes in Fig. 5 in the drop-down checkbox list, both *node1* (192.168.1.91) and *node2* (192.168.1.92) are available. In Section 3.3, the mechanism responsible for obtaining information about the status of individual nodes will be described. It is possible to visualise the read data on an ongoing basis on three charts: a *Bode plot of impedance modulus*, a *Bode plot of impedance phase*, and a *Nyquist plot*.

In the current version of the DMS, it is possible to track the values of read data from two nodes. In the future, it is planned to expand the current system to support more nodes.

3.2. OpenVPN

Access to the DMS is possible not only from a local area network (LAN), but also from the Internet. This functionality was obtained with the use of OpenVPN technology. This technology was configured on a Raspberry Pi 2 server (Fig. 2). The corresponding configuration was also done on *Router 192.168.1.254*. In this case, port 1194 configuration for UDP was required.

The solution in the form of OpenVPN technology and hardware (Raspberry Pi 2) has been selected to increase the level of network security and enable scaling of such a solution in the future. In the event of an increased load of requests for measurement data, it will be possible to use commercial services such as AWS IoT, Google Cloud IoT, IBM Cloud IoT, Microsoft Azure IoT, Oracle IoT Cloud Service or others.

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3.3. MQTT broker

At the DMS design stage, an Open Source solution in the form of Eclipse Mosquitto was chosen as the message broker. This solution was chosen because of the possibility of using the mechanisms of publishing and subscribing to messages on different topics. In individual messages, measurement data are transferred on the basis of event handling. Three topics have been defined for the purpose of sending messages. Individual messages with topics and sample JSON values are shown in Listing 1, 2, and 3.

Listing 1. Predefined message for the topic BeerTank/NodeInfo/.

```
Topic:
BeerTank/NodeInfo/
Value:
{
"status":"online",
"IPAddress":"192.168.1.91"
}
```

Listing 2. Predefined message for the topic BeerTank/TriggeringMeasurements/.

```
Topic:
BeerTank/TriggeringMeasurements/
Value:
20-05-2024 13:55:23
```

Listing 3. Predefined message for the topic BeerTank/NodeData/.

```
Topic:
BeerTank/NodeData/
Value:
{
"TimeStamp": "20-05-2024 13:55:23",
"Description": "node description",
"IPAddress": "192.168.1.91",
"F": 1,
"R": 485,
"I": -1012,
"Z": 1122.216
}
```

The purpose of defining the topic BeerTank/NodeInfo/ (Listing 1) is to enable sending messages about the status of individual nodes. In this case, it is possible to visualise the state of individual nodes in the *Beer TANK* application (Fig. 5 lower left corner).

After pressing the Start button in the *Beer TANK* application, the current date and time (Listing 2) is saved in the BeerTank/TriggeringMeasurements/ topic. The content modification of this topic triggers a series of measurements on all nodes in the MS that have subscribed to this topic. This approach enables user to trigger measurements at the same time.

Each of the nodes, after reading the measured values, saves the data in individual properties. In this case, the properties and values will be saved in BeerTank/NodeData/. For an example of values, see Listing 3. In addition to the measurement data read from the AD5941 chip, the identifier of the time when the measurement was triggered (TimeStamp) is also saved. Among the properties, the identifier of the node that wrote the data (IPAddress) was also specified. There is an additional field for the description of the node in the property Description.

The *Beer TANK* application was implemented in JAVA in the Eclipse IDE and Web IDEs environment. The Paho library version 1.2.5 was used to communicate with the MQTT Mosquitto message broker. The programming part for the Arduino Nano 33 IoT was done in the Arduino

Integrated Development Environment. The AD5940 Firmware Library was used for communication with the AD5941 chip. Communication with the computer network was ensured using the *WiFiNINA* library. The *PubSubClient* library was used to subscribe and publish the data. DMS validation was performed using the MQTT Explorer tool (Fig. 6).

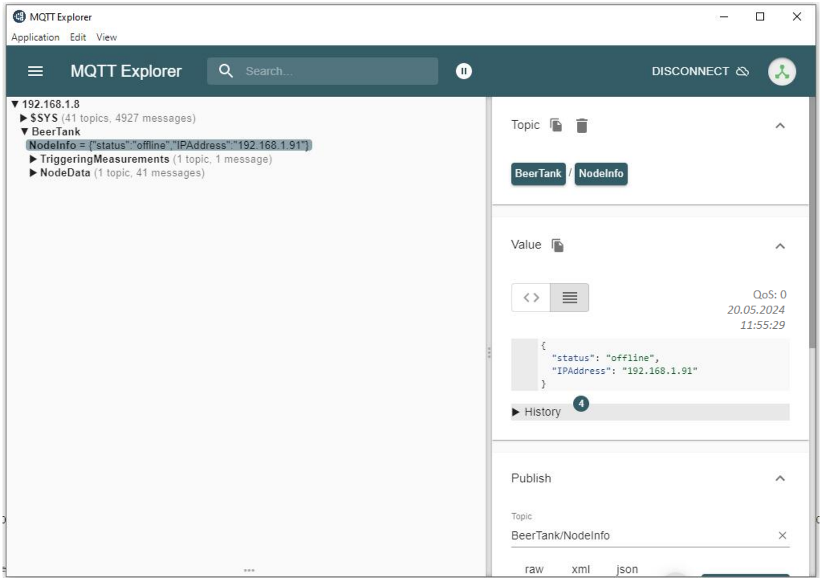

Fig. 6. Tool for testing communication with the MQTT message broker.

The MQTT Explorer tool was also used in the validation phase of the implemented system to verify the reliability of message delivery by determining the Quality of Service (QoS) level [19]. It was established that the QoS level = 0 would be sufficient under the laboratory conditions under which the system was tested. In its assumptions, this level does not guarantee the delivery of messages due to a lack of confirmations. However, the lack of confirmations results in faster performance. If necessary, QoS can be changed in the future at the target site of system implementation.

After confirming the correctness of the DMS, work began on the analysis of the obtained measurement data.

The designed, programmed, and assembled DMS was tested under laboratory conditions. This allowed us to eliminate variables that affect impedance measurement, such as ambient temperature. In this case, it was continuously monitored and kept at 23.5 ± 0.1°C. It was also necessary to verify the obtained findings using specialised equipment. For this, a Princeton Applied Research laboratory EIS system was used. It consists of a 263A potentiostat/galvanostat, a 5210 dual phase lock-in amplifier and PowerSINE PC software. The system diagram is shown in Fig. 7. At lower frequencies, the measurement voltage values are generated by the potentiostat. At frequencies above 10 Hz, the 5210 phase-sensitive detector is used to generate the measurement voltage and perform measurements. The impedance of the samples was measured in the two-electrode configuration. This means that the electrode was connected as recommended by the manufacturer to the three-electrode interface of the 263A potentiostat, i.e. with one lead to the working electrode terminal and the other to the counter-electrode and reference electrode terminals. The system is controlled by PowerSINE software, which communicates with the instruments via a GPIB bus. Each series of impedance measurements begins with automatic calibration across all ranges, both

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of the potentiostat and the lock-in amplifier. Subsequently, measurements are initiated at the highest established measurement frequency. To enhance measurement accuracy, samples from three full periods of the excitation signal were collected and averaged by the system in this case. It is worth noting that the averaging mentioned is a user-selectable option of the PowerSINE application. Impedance values were measured with both the DMS and the commercial dedicated EIS system using the same pairs of electrodes and the beer sample. Immediately before each measurement series, both systems were calibrated accordingly. The reference system calibrated itself based on procedures implemented in the firmware by the manufacturer, which were triggered by the authors prior to the measurements. In the case of the DMS, the calibration was based on a procedure proposed by Analog Devices and was based on measuring the impedance of the reference resistor.

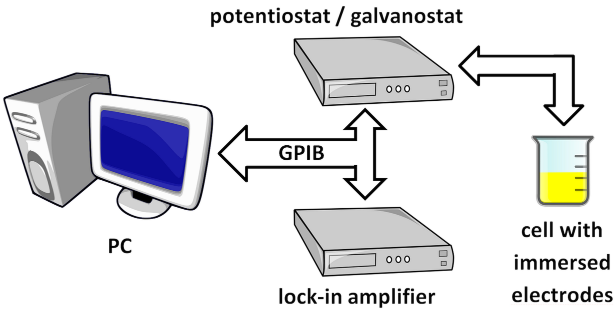

Fig. 7. Diagram of the reference system used in the research.

A sample of beer with a volume of 1 litre was tested in a glass flask. Two pairs of electrodes were immersed in the sample 20 cm apart; one was placed at the bottom and the other just below the surface. In previous studies, it was observed that such a distance may be sufficient to detect differences in the measured impedance of beer that is not stirred during measurements. Next to a flask there were two nodes, both consisting of the AD5941 impedance converter and the Arduino 33 IoT. The Raspberry Pi 2 and access point/router were in the same room during the experiment.

The recorded difference between the values measured with one and the other pair of electrodes is something expected and variable over time. Its characteristics and changes can be a valuable source of information, among others, on the degree of microbiological contamination of beer. The DMS made it possible to see the mentioned differences. Experimentally collected measurement data in the form of graphs created in the *Beer TANK* application are presented in Fig. 5. The aforementioned differences are especially visible in the Nyquist plot (Z_{im} values) on the right and the phase angle in the lower left corner of the figure. Although the EIS system used for verification allows for measurement of impedance with only one electrode at a time, it was possible to perform comparative tests. The time needed to change the electrode connected to the system and perform the measurement was short enough to ensure that the sample's impedance remained unchanged.

Figures 8, 9, and 10 show the results of impedance measurements of microbiologically uncontaminated, moderately contaminated, and significantly contaminated beer, respectively. A significant decrease in the measured impedance values can be observed with increasing contamination, but it does not necessarily have to be related to this phenomenon alone. In addition, there is also a noticeable difference between the values measured at different electrode locations, which again occurs in each case. These differences become less visible as beer becomes more contaminated. It is worth noting that, in terms of quality of the final product, it is crucial to detect contamination as early as possible so that appropriate action can still be attempted.

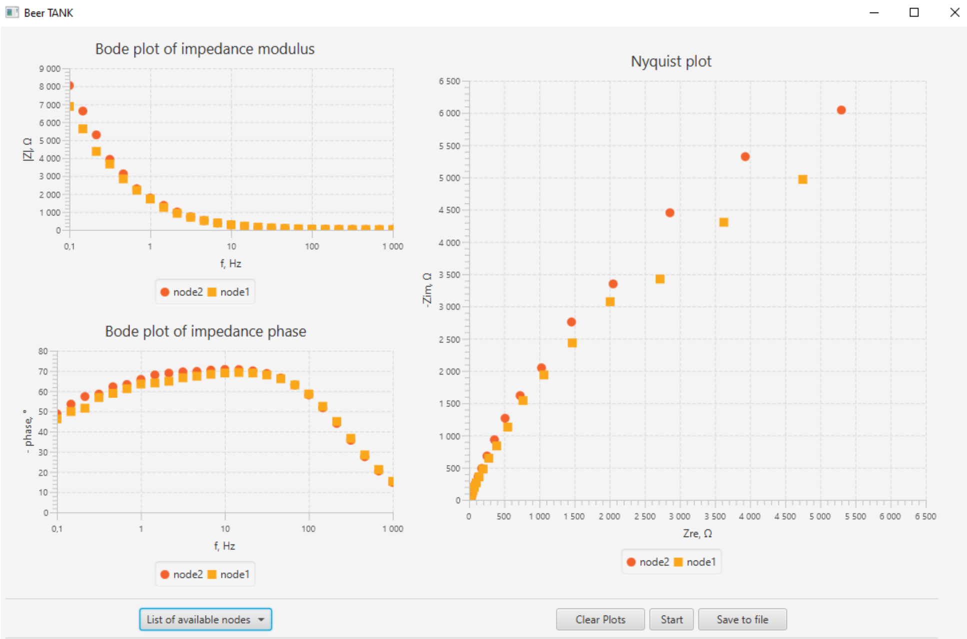

Fig. 8. Application *Beer TANK* presenting the results of impedance measurements of microbiologically uncontaminated beer.

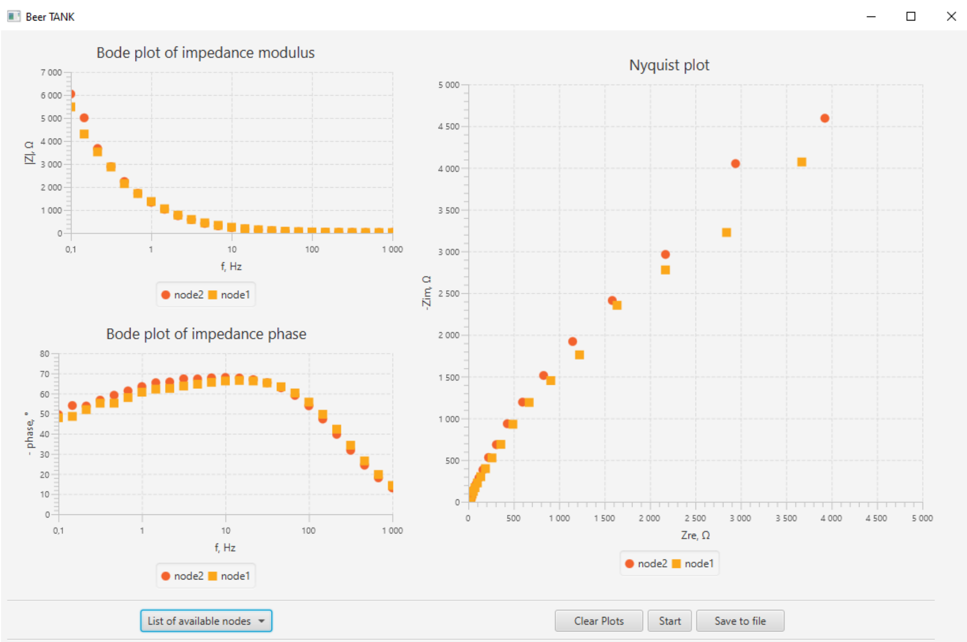

Fig. 9. Application *Beer TANK* presenting the results of impedance measurements of moderately microbiologically contaminated beer.

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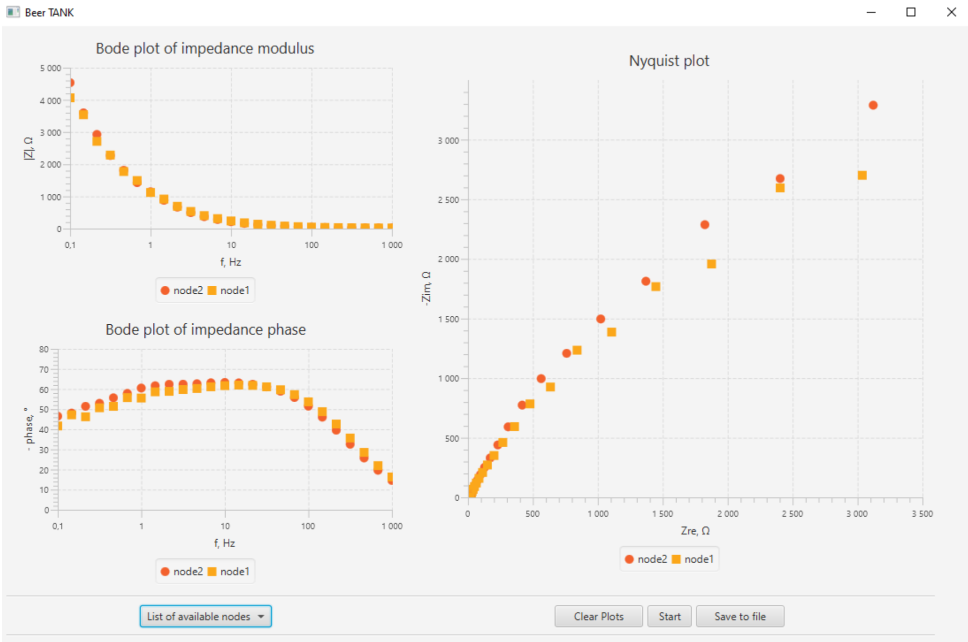

Fig. 10. Application *Beer TANK* presenting the results of impedance measurements of significantly microbiologically contaminated beer.

The determined deviations of individual impedance values measured by DMS and the laboratory EIS system did not exceed 5% in the frequency range of 50–1000 Hz. However, it is worth noting that deviations in plus or minus were reproducible for given frequencies. This does not affect the comparative data analysis process for several measurement points. The enhanced calibration methodology for the AD5941 impedance converters as well as improving the programme that controls its operation are necessary to correct the quality of measurements made at frequencies lower than 50 Hz. At the time of preparing the manuscript, the achievable measurement error gradually increased as the measurement frequency decreased, exceeding ±40% at frequencies below 1 Hz. It is worth mentioning that such large errors affected less than 5% of the measurements, which is, however, still an all too frequent occurrence. Such error values are unacceptable in the target system and must be eliminated, since a lot of valuable information about beer properties is obtained specifically at low measurement frequencies. If it is not possible to reduce the measurement error, the possibility of developing a proprietary potentiostat based on microcontrollers with suitably accurate ADCs is considered. Figure 11 shows the obtained values of the DMS's measurement errors in the form of error bars and applies to 95% of measurements. In the vast majority of cases, the system has measurement errors approaching ±25% of the value measured at a frequency of 0.1 Hz. The reference points were measurements made with the reference measurement system. A strong dependence of the obtained errors on the measurement frequency is clearly visible.

In the course of ongoing research, several problems have already been encountered with AD5941 chips. These problems, as well as the detailed analysis of the DMS metrological properties, are beyond the scope of this manuscript and are a topic for a future separate study. It is planned to include an analysis of the measurement uncertainty budget and an analysis of the cross-talk phenomenon of Z_{re} components on Z_{im}, as is the case in similar topics [20]. As soon as the problem with excessive measurement errors at lower frequencies is solved, it would be reasonable to add the functionality of identifying the parameters of the equivalent circuit elements to the *Beer TANK* application, similar to the mentioned pyZwx programme. This would allow for a more precise determination of the degree of bee contamination, while in its current form it is possible to use a slightly less informative but still valuable visualisation of impedance measurement results.

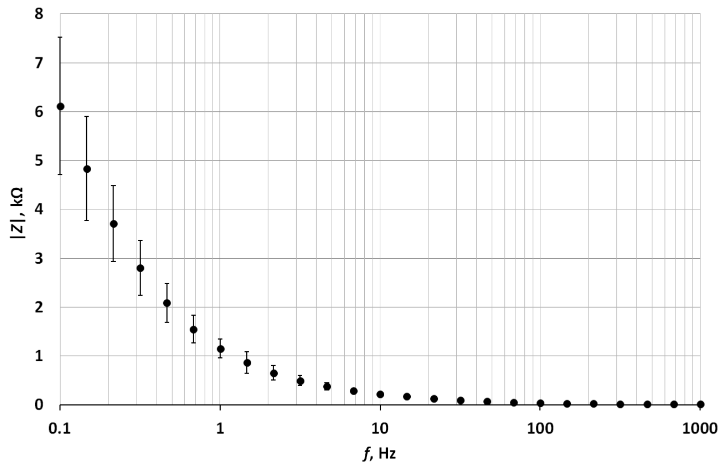

Fig. 11. Measurement errors of the DMS in 95% of measurement cases.

However, measurements taken at higher frequencies can definitely be considered sufficiently accurate, especially for comparative studies. With no additional improvements, the system is already capable of testing the properties of materials whose impedance does not need to be measured at frequencies below 50 Hz.

4. Conclusions

In the paper, the DMS for the beer electrochemical properties monitoring was presented and tested. The reasons why monitoring of beer properties is important were described. Possible methods of their measurement were presented, and against their background a relatively innovative method based on IS was elaborated on. The concept of a system for measuring the impedance of beer in several places at the same time was shown, which was then tested for two measuring nodes. The designed DMS can be used not only to measure the impedance of matter in many places at the same time. Considering its scalability and universality, the authors intend to explore its further practical applications.

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