Development of automated measuring device with magnetic fluid

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Abstract. The article presents theoretical principles of the methods and devices for water activity ($a_w$) measurement. The purpose of the research is the increase of the validity of water activity indicator measurement on the basis of a direct method of material test; the optimization of the structure of an automated measuring device to provide the possibilities for a simultaneous study of several samples (for example, food materials) with various water activity levels. A developed measuring device allows a simultaneous determination of indicators in several samples (8) located circumferentially and includes thermal sensors. The sensors in this device are represented by induction coils. They are installed on both leg pipes (made of non-magnet materials) of a liquid level manometer. The device contains a special silicon organic oil with the pressure of partial steams within $10^{-6}$ Pa (at normal conditions) used as a manometric liquid. While measuring the device is vacuumized and thermostated. The method and device approbation resulted in making calibration charts at various temperatures, approximated with the help of software. The device provides the validity of measurements in water activity range from 0.45 to 1.0.

1. Introduction

The relevance of improving the methods and devices of quality measurement for various materials, including food systems, is without any doubt. The chemical analysis and technologies include the parameter “solution activity” while the food industry has the parameter “water activity”. The indicator $a_w$ correlates with the development of microorganisms in food products [1, 2].

At the device elaboration the authors used the unit designed in our country and further improved as a prototype. The prototype drawback is the presence of a mechanical sensor, significantly reducing the reliability and accuracy of the measuring device which, in its turn, makes the device structure more complicated. In addition, this device allows measuring water activity in reservoirs only in succession.

The measuring device, developed in the research, can be used for the control and measurement in industrial areas with the account of the tool base improvement concept and the improvement of methods&devices properties. The results of water activity measurement provide forecasting the safety of raw materials and products in scientific research in the area of chemical technologies, food industry, retail, etc. [2, 3].
2. Methods of water activity measurement

The purpose of the developed device is improving measurement reliability and accuracy due to a simplified measurement unit design, providing for a possibility of a simultaneous research of a large sample number (up to 8) with various water activity as well as increasing the accuracy of measurements and software data processing.

The principles and methods of water activity measurement $a_w$ are versatile. To determine water activity, the food industry uses the direct or indirect determining of water steam equilibrium pressure in closed systems; certain methods are recognized as official and/or international ones [1, 8]. The authors developed the following methods to determine $a_w$: 1) direct pressure manometric measurement and its varieties [2, 4, 5]; 2) dew point measurement; 3) determining the change of the condenser capacity; 4) determining the change of the electrolyte electrical conductivity; 5) measuring the gyroscopic line length; 6) defining sorbing agent weight increase; 7) measuring temperature changes at establishing the equilibrium in closed systems; 8) defining the change of the freezing point in the experimental system, etc.

At the moment water activity is the key quality (safety) indicator of food products. To control the product quality, the tool base of methods for the analysis of the indicator $a_w$ is improved, for example, the biospeckle laser [7] is reported to be used for testing food materials (starches, pectins, sugars) at various temperatures (from 0 up to 25 °C as well as at negative temperatures). For the production of certain types of products it is suggested using a special microbioreactor to trace the changes of $a_w$ at maturing of biotechnological media in cheese manufacturing [6].

The present research purpose and objectives are resolved due to the reservoirs for the researched products installed at the base of the device intended for a direct (i.e., manometric) determination of water activity. These reservoirs are equipped with thermal sensors connected to a corresponding number of the measuring tubes of a liquid manometer. These reservoirs are located circumferentially. The liquid manometer measuring tubes are made of non-magnetic materials and the sensors (induction coils or electrodes) are installed on them; these sensors are connected to the external registering device. As a manometric liquid magnetic fluids on the basis of silicon organic oils are used. The device is equipped with a vacuum system.

The magnetic fluid application provides for a possibility to increase the measurement accuracy and reliability due to the conversion of direct working fluid movements into an electrical signal. To measure the change of induction, the tubes should be made of non-magnetic materials (glass or plastics). As the basis for magnetic fluids one can use silicon organic oils - polyphenyl methylsiloxanes with partial steam pressures at 20°C within 10-6 Pa.

3. Development of the automated measuring device and the operation principles

Figure 1 shows the diagram of the device containing the reservoirs for the studied product (1), installed in the thermostat, with thermal sensors inside (2). Each reservoir is connected to one of the liquid manometer leg pipes (3). The second free manometer leg pipes are interconnected and also connected to the reservoir for distilled water (4) and the vacuum system (5). Both leg pipes of the liquid manometer (made of non-magnetic materials) have the sensors - induction coils (6, 7) - installed on them. The sensors are situated on the manometer leg pipes and have the length (100-300 mm) providing for the registering of pressure drops within the water activity range 0.5-1.0 at the temperature 0 - 80°C. The liquid manometer is filled with a magnetic fluid on the basis of silicon organic oils (8).

The pipelines are blocked with magnetic vacuum valves (9, 10, 11, 12). The whole unit is located on a flat round foundation (13) and is placed in the thermostat (14).
Figure. 1. Diagram of the automated device with a magnetic manometric liquid to define $a_w$: 1 - reservoirs for the researched product; 2 - thermal sensors; 3 - liquid manometer; 4 - distilled water reservoir; 5 - vacuum system; 6, 7 - induction coils; 8 - magnetic fluid; 9, 10, 11, 12 - electromagnetic vacuum valves; 13 - foundation; 14 - thermostat

At the device operation the difference between water steam pressure over the product and distilled water causes the change in the liquid level inside the manometer; this change is registered by the induction coil. The electromotive force change causes the electrical signal change. The electrical signal is registered by an external device with the indication in water activity units. The device is calibrated on the solutions of saturated salt solutions with the known water activity values. The device design allows simultaneous studying of several (8) samples as each measuring reservoir has its own manometer.

The reservoir (1) contains the researched products, the reservoirs are sealed. Further, vacuum valves are opened (9, 10, 11, 12). The vacuum system is turned on and pumps air until residual pressure reaches 10-1 Pa. The device is thermostated. The temperature is registered by sensors (2). As a result of the difference in pressures over water and products, the magnetic fluid (8) moves inside the manometer (3), and this motion is registered by the sensors – induction coils (6) and (7) and is transferred to the external device. After the measurement the valves (10) open first and, after pressure equalizing, the valves (9, 11, 12) open simultaneously while the vacuum is released.

The valves (9) serve for disconnecting the device gas medium from the atmosphere in case not all reservoirs are in use. This device advantage is a higher measurement accuracy and reliability.

Magnetic fluid application allows decreasing the device overall dimensions as the density of silicon organic oils is equal to 0.9-1.1 g/cm³, while the oil-based magnetic fluid density – 4.0-6.5 g/cm³. The most reliable way is the use of magnetic fluids for which the criteria $P_m$ and $P_h$ ($P_m$ characterizes a solid substance volumetric fraction with magnetic properties, $P_h$ is the ratio of hydrodynamic concentration and solid phase concentration) shall be close to the values 0.67 and 2.8, correspondingly. Otherwise, one can expect a complicated nature of the dependence of magnetic fluid physical properties on the magnet field, the motion state and the time of liquid storing which is not permissible for measurement devices. The problem of the magnetic fluid free surface sustainability in the magnetic bias field of the excitation coil in case the intensity of a longitudinal homogeneous magnet field is equal to $H = 3.5-7.0$ kA/m while the magnetic fluid magnetization $M_0 = 45-60$ kA/m.
The second option of the device design have the electrodes of the wire with a higher resistance in place of induction sensors inside the manometer leg pipes, in this case pressure drops cause the resistance change registered with the help of a common bridge circuit.

Fig. 2 shows a calibration curve to define the water activity on the liquid manometer data (ΔH, mm) at various temperatures within the interval 15 - 25°C. As the magnetic fluid density is 5 times higher than the one of a silicon organic fluid, the obtained values are correspondingly lower.

The device advantage is the possibility of a remote and continuous change as the registering device is outside the thermostat in which the device is installed. The device is reliably operated in the water activity range from 0.45 to 1.0.

For the unit calibration and obtaining measurement errors the authors used saturated salt solutions with the known water activity values at the temperature 25°C. The thermostat temperature was maintained at the level 25±0.5°C. The water activity values, obtained by the experiment, were compared with the reference ones [5].

The results of computer statistical processing of the experimental data are presented in Figure 3. The figure data show that the accuracy of measurements reduces with the growth in the residual pressure inside the unit and the decrease in the water activity level in the studied material.

The dependence of measurement error on the residual pressure level and the water activity level (s) is expressed by the following formula (1):

\[ s = 0.6233 - 1.300 \cdot a_w + 0.0129 \cdot P_o, \]  

where \( P_o \) – residual pressure, Pa.
Figure 3. Dependence of the device data error (s) on the residual pressure (Po) and water activity (aw)

It is necessary to note that the units of this design do not provide the opportunity to find out the water activity in the products where microbiological processes and enzymatic reactions can occur with gas emission. It neither makes it possible to research the products containing easily volatile components (for example, alcohols, ammonia, etc.) The measurement accuracy is influenced by the water activity level. At the lower values of water activity (lower than 0.45) the accuracy of measurement reduces because of water losses at air pumping.

It is known that a high residual pressure of the gas medium in the unit (more than 79, 300 Pa) leads to a slowdown of the water steam diffusion process while too low pressure (lower than 13.3 Pa) results in the increase of the length of molecule free path, which also influences the rate of sorption - desorption processes and results in the increase of the measurement duration, that is why residual pressure in the unit should be equal to 40-50 Pa.

4. Conclusion
This paper presents principal and technical aspects of the automated unit design for testing materials in terms of water activity. It also provides scientific and practical results demonstrating the prospects for the method upgrade and material (including food products) test instrumentation. The authors identified the advantages of the developed measuring device, in particular, improved technical and metrological characteristics comparing with similar devices. The paper points out some limits of the device application in the research and quality control of various materials.

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