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PRZETWARZANIE DANYCH W SYSTEMIE INFORMACYJNO-POMIAROWYM DO SYNCHRONICZNEJ ANALIZY TERMALNEJ

Streszczenie: W artykule przedstawiono system do pojemności cieplnej oraz ciepła parowania cieczy organicznych z materiałów niejednorodnych. W omawianym systemie zaimplementowano równoczesną rejestrację utraty masy przez badaną próbkę oraz ciepła, które jest zużywane na wyparowanie cieczy. Uzyskane dane pomiarowe są używane do potwierdzenia poprawności przyjmowanych parametrów procesu suszenia. Zaproponowano algorytm do przetwarzania sygnałów pomiarowych, a także metodologię analizy oraz interpretacji charakterystyk metrologicznych.

Słowa kluczowe: system informacyjno-pomiarowy, metoda synchronicznej analizy termicznej, pojemność cieplna, ciepło parowania

DATA PROCESSING IN SYNCHRONOUS THERMAL ANALYSIS INFORMATION MEASUREMENT SYSTEM

Summary: The work presents a system for studying the heat capacity and heat of evaporation of organic liquids moisture from heterogeneous materials. It implements the simultaneous registration of the mass loss of the sample and the heat that is spent on the evaporation of the liquid, which can be used to justify the choice of drying mode parameters. An algorithm for processing the measuring signal is proposed, as well as a methodology for studying metrological characteristics.

Keywords: information measurement system, method of synchronous thermal analysis, heat capacity, heat of evaporation

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1. Introduction

An effective drying process of raw materials provides for the correct choice of operating parameters of drying, which takes into account the characteristics of a particular wet material. The basis of the justification of the optimal parameters of the drying process is the drying kinetics equation and the Rebinder criterion for the determination of which it is necessary to know the heat capacity of the material and the heat of evaporation of moisture from it, depending on the drying temperature and humidity of the material [1]. In the case of heterogeneous materials drying (food products, plant materials for pellets, raw materials for pharmaceuticals, etc.), the determination of heat capacity is not a problem, since there are standardized research methods, as well as a sufficient number of measuring instruments, using which it is possible to study materials with complex structure with high accuracy of measurements[2-4]. However, the study of the evaporation heat of a liquid from heterogeneous materials is associated with complications, namely, the evaporation of moisture from such materials is accompanied by additional processes (a change in the ratio of the components of the evaporating solution, structural or biochemical transformations of the individual components of the material, the redistribution of free and bound moisture in the material, phase transitions of the individual components, etc.) that affect the actual values of the specific heat of vaporization of a liquid from a particular material. It is very difficult to analytically take into account the influence of each of these factors on the value of the heat of evaporation, therefore, it is the most practical to measure it experimentally.

One of the promising methods for the experimental determination of the heat of vaporization is the method of synchronous thermal analysis (STA) [5]. The essence of the method is to simultaneously measure the change in mass of the sample during drying under isothermal conditions at a given temperature and the amount of heat that is spent on the evaporation of moisture from the sample. The advantages of the method include the possibility of obtaining data on the heat of vaporization of the material at an arbitrary stage of the drying process, rather than the average value for the entire process [6]. Previously, the authors developed a specialized calorimetric device that allows to study both the heat of evaporation of moisture from inhomogeneous materials and their heat capacity [7]. However, the processing of experimental data at the final stages of drying is problematic, which is associated with a decrease in the signal level to the level of background noise. The purpose of this work is to review the problems of processing experimental data to determine the heat of vaporization of liquids from heterogeneous materials and to find possible solutions.

2. The synchronous thermal analysis and calorimetric device application

The method for determining the heat of evaporation of moisture is based on the assumption that under isothermal conditions all the heat that is supplied to the sample is expended either on the evaporation of moisture from it or on the conversion of the components of the sample (for example, biochemical reactions). According to the method, the test sample is placed in the working cell with an isothermal environment of the working chamber, where the mass loss of the sample and the corresponding heat flux passing through the calorimetric cells are simultaneously recorded, as shown in Fig. 1.

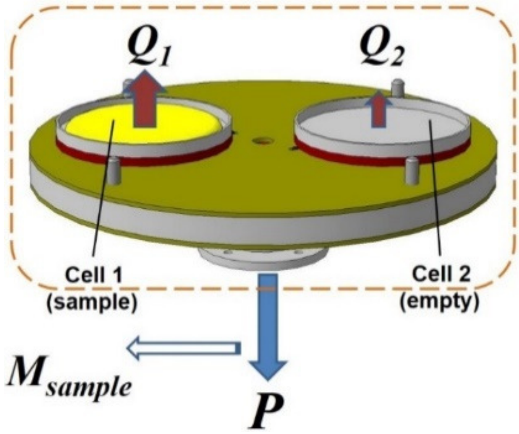

Figure 1. Measurement scheme by synchronous thermal analysis

The measuring part of the device is a thermostatically controlled working chamber, in which a platform with a heater and two identical calorimetric cells with heat flux sensors is placed (Fig. 2).

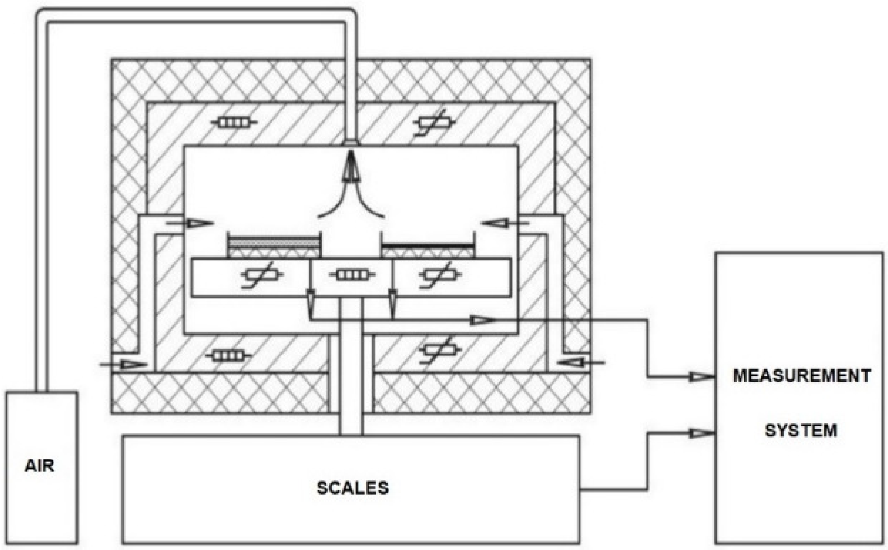

Figure 2. Scheme of a special calorimetric device

The upper and lower parts of the working chamber are also equipped with heaters with independent regulation, which is necessary for reproducing and supporting an isothermal environment. Excess moisture in the air of the working chamber, which is formed during drying of the sample, is removed through the corresponding channel in the lid of the working chamber using a fan. The removed air is replaced by air from the environment, which manages to reach the set temperature before entering the working chamber. The study continues until the equilibrium moisture content of the sample is established under given drying conditions (m_{sample} = const). The temperature range of the research is 30 -105°C.The main recorded parameters are the heat flux through the empty cell and the cell with the sample, the temperature of the calorimetric platform, the mass of the sample, and time parameters. In addition, humidity and the initial temperature of the surrounding air, as well as the flow rate of air passing through the working chamber per unit of time, are controlled. These parameters are recorded every 5 s during the entire drying time of the sample. The duration of the study depends on the characteristics of the test material and drying parameters and can range from 15 minutes to 12 hours.

Data processing in a generalized form was carried out according to the equation:

$$\bar{r}_{ij}(T,m,\tau) = \frac{\int_{\tau_i}^{\tau_j} \left[Q_1(T) - Q_2(T) + Q_{UC}(T) \right] d\tau}{m(\tau_i) - m(\tau_j)} \tag{1}$$

where: $\bar{r}_{ij}(T,m,\tau)$ – specific heat of vaporization in the time interval from the initial moment i to the final moment j;

$Q_1(T)$ – heat flux through the cell 1;

$Q2(T)$ – heat flux through the cell 2;

$Q_{UC}(T)$ – uncontrolled heat flow;

$m(\tau_i) - m(\tau_j)$ – sample weight loss over time from τ_i to τ_j;

3. Experimental data processing

One of the unresolved problems of processing experimental data is the need to divide the entire data set of experiments into separate intervals, the size of which is selected by the operator based on the nature of evaporation, type and weight of the sample. This is due to a decrease in the drying intensity over time, which leads to a proportional decrease in the signals, Δm and ΔQ, as shown in Fig. 3.

Figure 3. The dependence of the mass loss of the sample on the drying time in stationary conditions

For each interval, its own integration step is selected. An integration step that is too small leads to an increase in the noise component of the measurement, especially at the last stages of drying, where insufficient resolution of the analytical balance leads either to a significant distortion of the measurement data or to a division error by zero. On the other hand, too large step of integration leads to loss of information about the possible effects of evaporation of moisture from materials, since the result is the average value of the heat of vaporization over a significant period of time.

Today, data processing is used with manual selection of the magnitude of the integration steps, followed by iterative averaging of the obtained values. Figure 4 and figure 5 shows the initial data on the mass loss of a shiitake mushroom (Lentiula edodes) sample during drying at a temperature of 90°C and the corresponding heat flux. The heat of evaporation from a shiitake mushroom at a temperature of 90°C treated in this way is shown in Fig. 6.

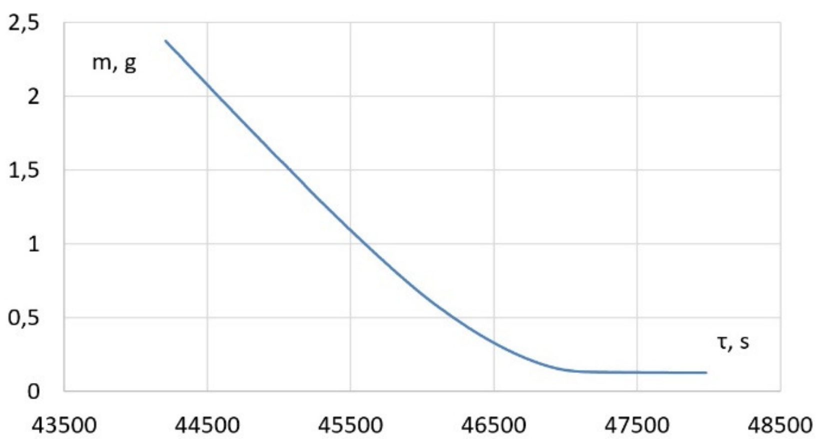

Figure 4. The change in mass of a shiitake mushroom sample during drying at 90°C

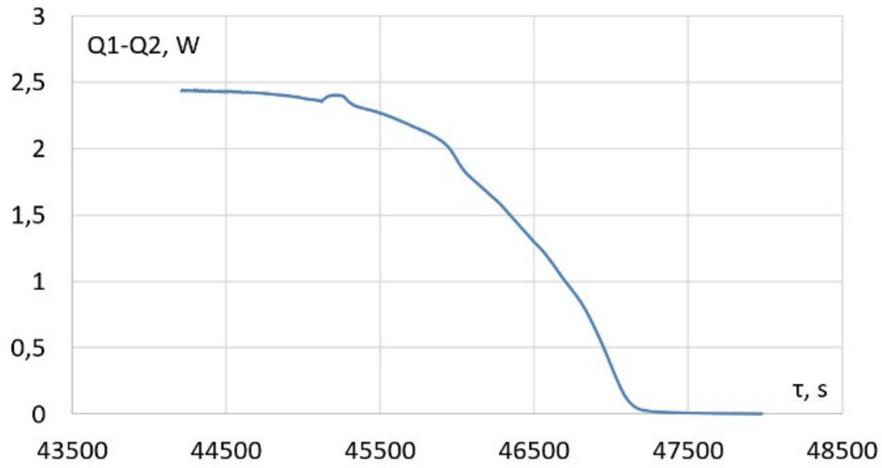

Figure 5. Heat flux that is conductively transferred to the shiitake mushroom sample during drying at 90°C.

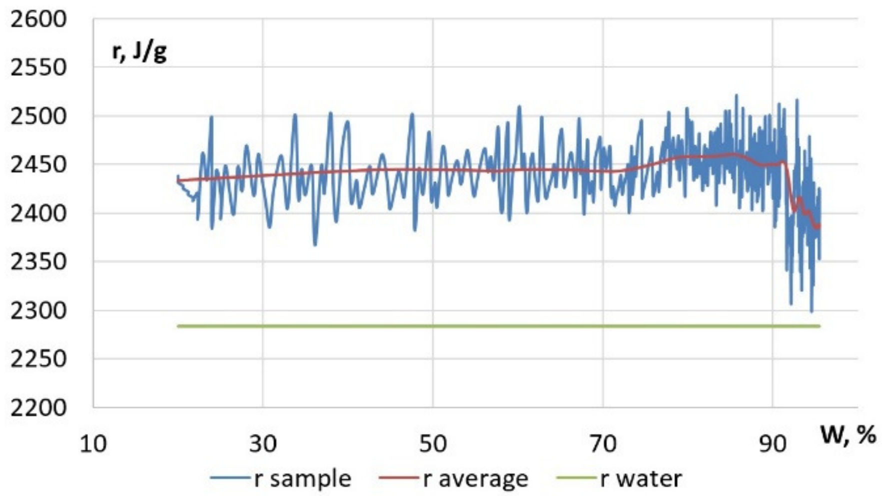

Figure 6. The evaporation heat of the moisture from the sample of shiitake mushroom during drying at 90°C compared to evaporation heat of distilled water at the same temperature.

This method allows to obtain detailed information about the heat of vaporization at all stages of drying the material, however, manual data processing and high requirements for the operator's experience are a significant drawback and require the establishment of a clear criterion for dividing the data into intervals or searching for other approaches to overcome the noise component of the measurement process.

4. Discussion

The main ways to solve the problem of data processing automation can be divided into three areas. The first one is the determination of the criterion for dividing the information series into intervals, which would take into account the change in the drying speed, possible exothermic and endothermic processes in the components of the material under study, as well as the drying parameters. The second is the replacement of experimental data on changes in the mass of the sample and the heat supplied to the material over time with the corresponding mathematical dependencies. This will pre-vent an increase in the influence of the noise component at the final stages of drying, however, for some materials, the kinetics of drying is too complicated to describe the entire process with a single polynomial equation. The third option is direct filtering of the noise component of the results obtained by determining the heat of evaporation based on frequency analysis using hardware or software (for example, Kalman filter[8]).

5. Conclusions

This article formulates the problem of data processing for heterogeneous samples under isothermal drying. To obtain the most complete information, a data processing method has been selected. The method consists in dataset division into integration intervals with the subsequent smoothing. On the one hand, the result of data processing is detailed information about the evaporation heat of moisture from the material at different stages of drying. On the other hand, the high requirements for operator qualifications and a significant share of manual data processing make this data processing option unsatisfactory.

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