

Experimental and graph-analytical research of thermal properties phase change materials

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ABSTRACT: This article presents the results of an experimental study of materials with a phase transition (PCM) to conserve thermal energy (TES). On a specific example, using the method of T-history, a study of the thermophysical characteristics of sodium acetate trihydrate (CH_3COONa) and a mixture of sodium acetate trihydrates with palygorskite crumb ($\text{Mg,Al}_2\text{Si}_4\text{O}_{10}(\text{OH})\cdot 4\text{H}_2\text{O}$) was demonstrated. The application and effectiveness of the T-history method are described. Analytical methods of processing the results of experimental research are considered and a graphical-analytical method for evaluating the effectiveness of selected substances is proposed. On a concrete example, the use of modern methods of research of thermophysical properties of substances and efficiency of thermal processes using the analysis of a correlation field and an angle of inclination at the graphic representation of pair regression approximated by elementary functions taking into account correlation coefficient is offered.

The proposed graphical-analytical method allows to determine with high accuracy the efficiency of thermophysical characteristics of the selected substances and to conduct a comparative analysis of experimental studies.

KEYWORDS: Thermal energy storage, Method, Efficiency, Heating, and cooling rate.

I. INTRODUCTION

Recently, much attention has been paid to heat savings. Heat conservation is a modern technology for improving energy efficiency and can be used to regulate the instability and mismatch between energy supply and energy demand.

[1]. The principle of operation of heat storage is based on the use of heat capacity. The efficiency of TES depends on the properties of the selected materials that retain heat, on the conditions in

which these devices are used, and on the purpose. The amount of accumulated heat is proportional to the density, volume, specific heat, and temperature changes.

[2]. Heat accumulators can be various substances, such as liquids (water, mineral oils, salt solutions, liquid metals, and alloys) or solids (stone, concrete, sand, brick). Concealed heat storage materials are phase change materials (PCM) such as organic materials (paraffin, fatty acids, esters, alcohols, glycol), inorganic substances (salt solutions, metals, and metal alloys, multicomponent materials).

Conventional heat storage technologies have an energy density of 100 kJ / kg to 200 kJ / kg. Low storage capacity usually prevents their use on a large scale, and the most accessible and widespread substance is water. For example, only 1.0 l of water when cooled to 1 ° C can heat 1 m³ of air at 4 ° C. There is an urgent need to study the selection of the most effective substances for heat storage. The main criteria for the selection of materials are the assessment of their rate of heating and cooling, resistance to overheating and supercooling, affordability, environmental friendliness.

[3]. The solution of multicriteria problems is poorly coordinated with the use of classical methods of variational calculus. Thermal systems are objects with distributed parameters, their dynamics are described by equations in partial derivatives. In real operating conditions, parameters are determined that change in a limited area and require the use of mathematical principles of maximum and dynamic programming. Therefore, the main source of information is the experiment. Processing and analysis of experimental data obtained during the research are aimed at systematizing and establishing different and numerical indicators.

[4]. Statistical, tabular, and graphical methods are often used to process experimental results. The data processing procedure includes the selection of the

approximating function and the definition of its parameters, as well as the calculation of the error of the obtained data. The graphical method of presenting empirical results more clearly than the tables shows the relationship between the indicators of the experiment and the nature of the process. The graphical image allows to compare experimental data with theoretical and to determine the empirical relationship between the values and relationships between the parameters under study.

[5]. Graphic representations of experimental results are usually based on a system of rectangular coordinates. Uniform and non-uniform (functional) scales are used to construct graphs. The construction of the graph includes the following stages, namely: - selection of the scale and construction of the grid; - taking into account the appropriate scale of the graphic image; - postponement of numerical values of the results of the experiment (points) on the coordinate grid; - connection of experimental points with a smooth line; - finding the distribution rule and construction of the trend line taking into account the value of the probability of approximation.

The purpose of the work is a preliminary assessment of the efficiency of the process, namely the determination of the ratio of the amount of heat removed to the supplied; determining the rate of heating and cooling using the proposed method.

II. CHOICE OF METHOD AND CONDUCTING EXPERIMENTS

The development of the use of PCM materials for TES requires the determination of their thermal properties. Currently, differential scanning calorimetry (DSC), which complies with ASTM E793 standards, is used to determine enthalpy melting and crystallization. These are other standards that determine heat capacity, strength, and viscosity. These standards work in a general way, as there is no exclusive standard for PCM, and they require more than the use of a device.

[6]. Quality criteria and measurement methods for PCM have been established by the ZAE Bayern Research Centre and ISE Fraunhofer, and since 2008 the RAL brand has been assigned to products that have been tested according to these comprehensive monitoring and testing criteria.

In this work, the T-history method was used to determine the temperature curves, which also gave the specific heat in the solid and liquid phases. This method was used because of its simplicity and low

cost. It should be noted that the T-history method is recommended for use in the study of samples with the phenomenon of hypothermia because with the help of DSC it is impossible to obtain a representative measure of the change in enthalpy with temperature.

[7]. In addition, the sample size used in the DSC is only a few milligrams, while for the T-history method it reaches a few grams, which allows obtaining a representative sample in inhomogeneous materials. However, both methods have their limitations, which create ease of use depending on the application.

In the general sense, determining the amount of heat transfer in heating and cooling is conveniently expressed by Newton's cooling law, which states that: The rate of heat loss of a body is directly proportional to the temperature difference between the body and its environment.

$$Q = h \Delta T \quad (1)$$

Where Q is the local heat flux density [$\text{W}\cdot\text{m}^{-2}$], h is the heat transfer coefficient [$\text{W}\cdot\text{m}^{-2}\cdot\text{K}$], ΔT is the temperature difference [K].

As can be seen, the proportionality constant will be decisive in the calculations, and it is known as the convective heat transfer coefficient, h . Convective heat transfer coefficient can be defined as The rate of heat transfers between a solid surface and a liquid per unit area per unit temperature difference.

$$h = Q / \Delta T \quad (2)$$

The convective heat transfer coefficient depends on the physical properties of the liquid and the physical situation. The convective heat transfer coefficient is not a property of a liquid. This is an experimentally determined parameter, the value of which depends on all variables that affect convection, such as the geometry of the surface, the nature of the motion of the fluid, the properties of the fluid, and the volumetric velocity of the fluid.

An experimental stand was created for the work and an algorithm for experimenting with the study of substances was developed.

The experimental stand consists of an electric heater with a power regulator 1, a heating tank 2 filled with water, in which there is a tank 3 with the test substance, closed lid 4, and a device of the temperature recorder 6, which receives information from thermocouple sensors 5. A computer 7 with reading software that processes and stores information. The scheme of the stand is presented in Figure 1.

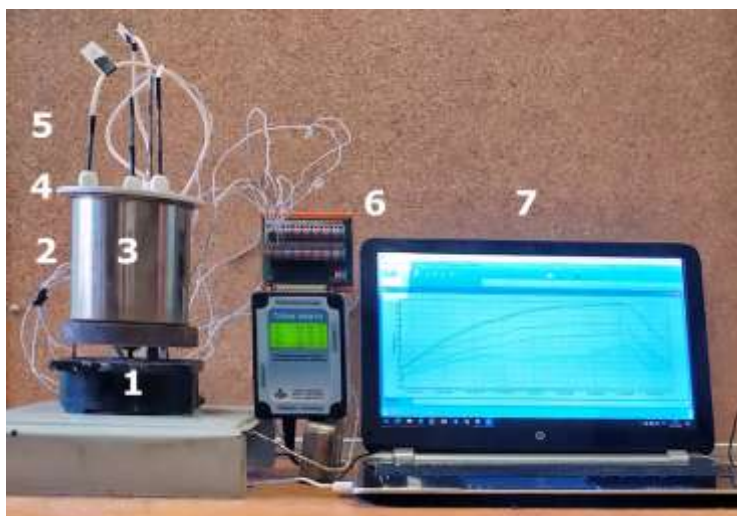


FIGURE 1 - SCHEME OF THE EXPERIMENTAL STAND

The experimental study of the substance was performed in the following sequence:

1. The mass and volume of the substance are determined in advance; the density is calculated.
2. The outer container, filled with water, is heated and the container with the test substance is heated.
3. After heating the test substance to 80 ° C and holding it for 5 minutes, the electric heater is switched off.
4. Cool the substance to 30 ° C at room temperature.
5. Throughout the experiment, the temperature of the thermocouples is recorded through the sensors of the measuring concentrator Triton 6004. The module TRITON 6004TR is a microprocessor device for measuring temperature using resistance thermometers (Pt100, Pt1000, M100, M1000). The received data can be shown on the built-in indicator or transferred to the computer using USB or RS-485. The temperature measurement error is $\pm 0.5^{\circ}$ C. With the help of software, the results are displayed on a computer as graphs of temperature versus time (and with the ability to view tables). The tests are performed at least three times over the heating and cooling cycles of the substance. Conditions that were during the experiments: rated mains voltage 220 V and current frequency - 50 Hz; optimal temperature range - from 30 ° C to 80 °

C, laboratory area - 12.0 m², room temperature 25 ° C.

III. RESEARCH RESULTS

Figure 2 shows the average temperature versus time obtained from a series of experiments for distilled water (1), sodium acetate trihydrate (2), and a mixture of 3 parts sodium acetate trihydrate and 1 part palygorskite (3) respectively [8]. Conventionally, the schedule of the heating/cooling process is divided into three parts - heating, holding, and cooling, respectively. As you can see, water (1) gains temperature faster and cools faster.

Sodium acetate trihydrate (2) heats up more slowly, and at a temperature close to 58 0 C the phase transition of the first kind takes place, due to which the stabilization zone is significantly reduced. During the crystallization of sodium acetate trihydrate, the phase transition is poorly expressed.

The mixture of sodium acetate trihydrate (3) with palygorskite brittle has no pronounced phase transition when heated, but there is a pronounced phase transition during crystallization of sodium acetate trihydrate in the cooling zone.

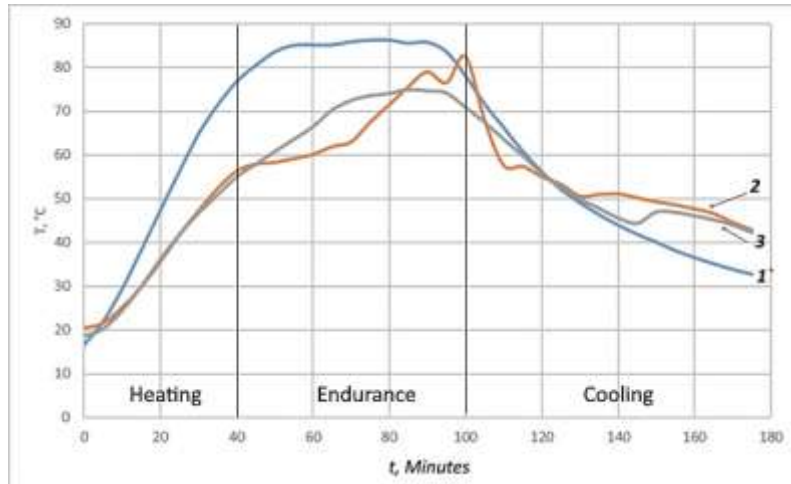


FIGURE 2 - THE PROCESS OF HEATING AND COOLING THE TEST SUBSTANCES

IV. DETERMINING THE AMOUNT OF HEAT RECEIVED.

Let's calculate the amount of heat received during heating and holding, as well as the amount of heat released by the substance after turning off the heat source. Calculations are performed to determine the temperature difference every 5 minutes.

The amount of heat received or released in 5 minutes is determined by the formula:

$$Q = c \cdot m \cdot \Delta t \quad (3)$$

where: c is the specific heat of the substance under study [$J / (kg \cdot K)$];

m is the mass of the substance under study [kg];
 Δt is the temperature difference of the substance in 5 minutes [K].

Graphs of the dependence of the amount of heat supplied for heating and the heat released by the substance during cooling at the time of the process are formed separately.

Figure 3, for example, shows the amount of supplied and removed heat depending on the time of the process of heating and cooling water.

In this way, we have the opportunity to solve problems by the graph-analytical method.

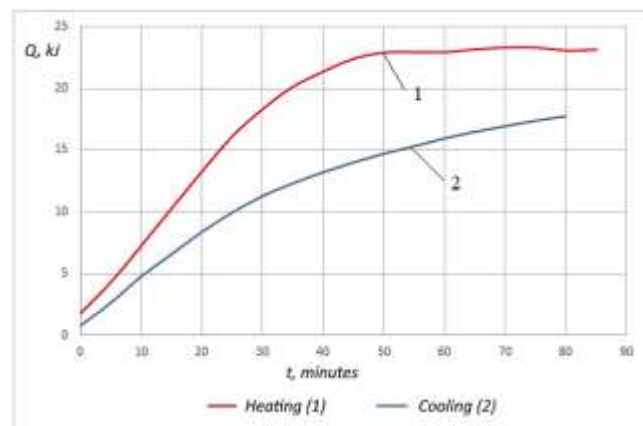


FIGURE 3 - THE AMOUNT OF SUPPLIED AND REMOVED HEAT AS A FUNCTION OF TIME THE PROCESS OF HEATING AND COOLING WATER

V. PROCESSING THE RESULTS OF EXPERIMENTS BY THE PROPOSED METHOD

The proposed method is based on the graphical determination of the efficiency of the test

substance for its evaluation. The sequence of determining the efficiency ratio:

- 1) In Excel, a straight line of the trend is drawn to the constructed graphs of the dependence of the amount of heat on time, the construction of which is based on the method of least

squares. With this method, we have the opportunity to determine the speed of the process of accumulation and return of thermal energy by various substances that accumulate heat.

- 2) For this the angle α_1 and α_2 , at which the trend lines are relative to the x-axis, is determined.
- 3) It must be determined that thermal resistance is an analogy to electrical resistance. Therefore, we introduce the concept of the process efficiency coefficient η - a dimensionless physical quantity that characterizes the relationship between $\text{tg } \alpha_1$ and $\text{tg } \alpha_2$. The efficiency of the process shows how efficiently the process of heating and cooling of different

substances is under the same conditions. The coefficient η takes values from 0.0 to 1.0. In practice, this means that the higher the value of η , the better the technical characteristics of the material that stores heat.

- 4) Calculation of the efficiency of the process according to the formula:

$$\eta = 1 - \frac{\text{tg } \alpha_2}{\text{tg } \alpha_1} \quad (4)$$

- 5) Determination of heating and cooling rate as $\text{tg } \alpha_1$ and $\text{tg } \alpha_2$, respectively.

An example of the result of processing the experiment to determine the efficiency of the process of heating and cooling water is shown in Figure 4.

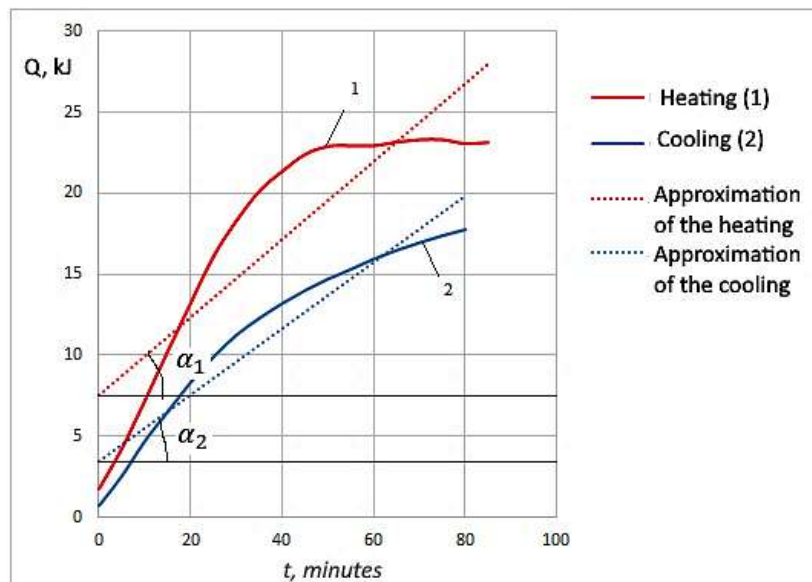


FIGURE 4 - DETERMINATION OF ANGLES α_1 AND α_2 FOR THE PROCESS OF HEATING AND COOLING WATER

At an angle $\alpha_1 = 39^\circ$ and $\alpha_2 = 34.5^\circ$, the efficiency of the complete process is 0.848.

At that time, $\text{tg } \alpha_1$ and $\text{tg } \alpha_2$ indicate the heating and cooling rate and are 0.8097 kJ / min and 0.6873 kJ / min, respectively.

VI. VI. DISCUSSION OF RESEARCH RESULTS

The values of the angles α_1 and α_2 are determined, the calculated values of process efficiency, heating, and cooling rates of w

ater are summarized in Table 1. This will allow evaluating the process for the substance (in this case water).

Table 1 - Results of calculations

Water	Heating	Cooling
Angle α , deg.	39	34,5
Process speed, kJ / min	0,8097	0,6873
Process efficiency ratio	0,152	
Sodium acetate trihydrate		
Angle α , deg.	41,5	24,4

Process speed, kJ / min	0,8847	0,4536
Process efficiency ratio	0,487	
Sodium acetates trihydrate with palygorskite		
Angle α , deg.	49,4	37,8
Process speed, kJ / min	1,175	0,7757
Process efficiency ratio	0,34	

Also in this way, it is possible to perform a comparative analysis of several substances to determine the optimal according to the requirements.

VII. CONCLUSIONS

1. The use of the T-history method makes it possible to determine with high accuracy the temperature and speed of the process of heating and cooling of liquid and PCM materials.
2. The obtained experimental data show that the greatest efficiency is inherent in sodium acetate trihydrate. The admixture of palygorskite brittle accelerates the process of accumulation and heat transfer. Water accumulates heat almost simultaneously with sodium acetate trihydrate but cools rapidly.
3. Graph-analytical method is proposed for use for preliminary comparative analysis and determination of the efficiency of materials and substances for heat accumulation.
4. It is recommended to use the proposed method for rapid analysis when choosing materials for heat storage.
5. The coefficient of process efficiency must be taken into account when designing thermal energy storage systems. Incorrectly calculated process efficiency can lead to excessive energy consumption and reduced efficiency of the heat storage system.

SOME OF THE ADVANTAGES FROM THE ABOVE RESULTS

- a) Graph-analytical method of evaluating the effectiveness of the process accelerates the determination of the rate of heating and cooling of the substance during experimental research and determination of the efficiency of the process.
- b) The method can be used to perform a comparative analysis of several substances. The method also allows you to perform a preliminary review of comparative analysis.
- c) The efficiency of the process allows you to assess the quality of heat retention by various materials.

REFERENCES

- [1]. Beckman, G. Thermal energy storage / G. Beckman, PV Gilly. - Moscow, Mir, 1987. - 269 p
- [2]. Levenberg, VD Heat accumulation / VD Levenberg, MR Tkach, VA Holstrom. - Kiev, Tehnika, 1991. - 111 p.
- [3]. A.E. Kononyuk Fundamentals of scientific research (general theory of experiment) - In 4 books. - K.1. - Kyiv.: 2011. - 508 p.
- [4]. Experimental research methods. Measurements of thermophysical quantities / Textbook. Pokhodun AI, Sharkov AV Experimental research methods. Measurements of thermophysical quantities / Textbook. - СПб: СПб ГУ ИТМО, 2006. - 87 p.
- [5]. Kuznetsov IN Scientific research. Methods of conducting and design / IN Kuznetsov - 2nd edition, revised and supplemented - M.: Publishing and Trading Corporation "Dashkov and K0", 2006. - 460 p.
- [6]. Materials of phase changes, 2018, Quality assurance RAL-GZ 896. 2018. Available on the Internet: https://www.pcm-ral.org/pdf/RAL_GZ_896_Phase_Change_Material_Edition_March_2018.pdf
- [7]. Lazaro, A.; Gunther, E.; Melling, H.; Hibler, S.; Marin, J. M.; Zalba, B. Verification of T-history installation for measuring enthalpy and temperature curves of materials with phase change. Measurement. Science. Technol. 2006, 17, 2168. [Google Scholar] [CrossRef]
- [8]. Demchenko VG, Falko VY, Experimental study of thermal stability of substances for storage of thermal energy, Thermophysics, and thermal power, 2019, Vol. 41, №2, p.64-71