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Optimization of the heat treatment processes of porous thermal insulation materials

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Experimental data on the influence of various factors on the strength and thermal properties of thermal insulation materials is presented. The dependence of the thermophysical and strength properties on the density of the formed material shows that the lower the density and the higher the initial humidity of the material, the better the properties of the final porous material will be. The porosity, thermal conductivity, and strength of the formed thermal insulation material are influenced by the heat treatment modes of the raw materials. Laboratory studies carried out in this paper make it possible to select the temperature conditions necessary for heat treatment of the materials.

The results obtained can be the basis for choosing effective methods for organizing and simulating heat and mass transfer, experimental verification of data, and creating methods for determining the main technical and design parameters of a new material.

Keywords: strength, thermal insulation material, properties, raw material mixture, drying, thermal conductivity, humidity, temperature regimes

Оптимізація процесів термообробки пористих теплоізоляційних матеріалів. *Р.О. Клімов, І.Є. Соколовська, О.Л. Глущенко*

В роботі представлено експериментальні дані щодо впливу різних досліджуваних чинників на міцнісні та теплові властивості теплоізоляційних матеріалів. Як показали залежності теплофізичних та міцнісних властивостей утвореного матеріалу від щільності, чим менша щільність та вища первинна вологість матеріалу, тим кращі властивості буде мати кінцевий пористий матеріал. На пористість, теплопровідність та міцність утвореного теплоізоляційного матеріалу впливають режими термічної обробки сировини. Лабораторні дослідження матеріалів, проведені в цій роботі, дають змогу підібрати температурні режими необхідні для термообробки.

Результати досліджень можуть бути основою для вибору ефективних методів організації та моделювання тепломасопереносу, експериментальної перевірки отриманих даних і створення методик визначення основних технічних і конструктивних параметрів нового матеріалу.

1.Introduction

Porous materials are widely used in various industries. In heat engineering, they are used for thermal insulation in the enclosing constructions of buildings, heat networks, and enclosures of heat engineering equipment. The thermal conductivity of thermal insulation materials should be the lowest; but at the same time, to work effectively, they must be durable and indestructible.

The thermal and mechanical properties of the material are interrelated. They determine the scope of application of the insulation. External load affects material deformation. Temperature stresses, which can lead to the destruction of the material, are of particular importance [1-3].

Table 1 – Variations of the heat treatment factors: X_1 is the process temperature; X_2 is the thermal exposure time; X_3 is the material humidity; X_4 is the material porosity; Y_1 is the modulus of elasticity, MPa; Y_2 is the strength, MPa.

X	-1.414	-1.0	0.0	+1.0	+1.414
X_1	20	100	300	500	580
X_2	0.2	1	3	5	5.8
X_3	24	30	45	60	66
X_4	56	60	70	80	84

The properties of thermal insulation materials are available in the literature, but determining the functional dependence of mechanical and thermophysical properties on the structural parameters of the material remains an urgent task. It has been established that the mechanical properties of the studied porous materials mainly depend on their density [1-7].

In turn, the density also depends on many factors, in particular, on the thermal conditions of the explosion during vaporization. Since the density of the material changes significantly depending on humidity, holding time and heating temperature, accordingly, its strength properties change with a change in density. The positive feature of this dependence is that it can be controlled and therefore predictable.

The main goal of the work is to obtain raw material mixtures for the production of thermal insulation with increased thermal resistance and porosity and to improve the method of producing porous insulation by changing the temperature-time regime in order to improve the mechanical, thermophysical and operational properties [8]. The aim of the paper was to study the influence of various factors on porous thermal insulation materials. The task was to obtain a mathematical model of the heat treatment of porous materials in order to identify the influence of its mode on the mechanical properties of the materials.

2.Experimental

The modulus of elasticity Y_1 and strength Y_2 are taken as technological indicators characterizing the behavior of the material during deformation and subsequent destruction under load. Temperature X_1, time X_2, initial humidity X_3, and porosity X_4 are factors characterizing the heat treatment mode of the material.

The tensile test data are most often used to determine static mechanical properties [4]. The tensile tests are carried out on a special machine recording a diagram of the sample elongation Δl (mm) versus the effective loading force P, i.e. $\Delta l = f(P)$. The diagram "relative elongation δ (%) versus the stress σ" is plotted based on the tensile diagram.

3. Results and discussion

Variations of the heat treatment factors and data obtained during the experiment are presented in Tables 1 and 2. An orthogonal central composite plan of the second order with a kernel 2^4 was used to build the models [8].

In general, the regression equation for a quadratic model design has the following form:

$$Y(a,X) = a_0 + a_1 \cdot X_1 + \ldots + a_n \cdot X_n + a_{n+1} \cdot X_1^2 + \ldots + a_{2n} \cdot X_n^2 + a_{2n+1} \cdot X_1 \cdot X_2 + \ldots + a_k \cdot X_{n-1} \cdot X_n \quad (1)$$

where a_V are unknown process parameters, estimates of which must be found by processing experimental data; X_V are input controlled or independent variables.

After performing calculations according to the algorithm of the method, the following quadratic models of the dependences of Y_1 and Y_2 on the studied factors X_K, $\kappa=1,\ldots 4$ were obtained:

$$Y_1 = 0.376 - 0.053X_1 - 0.064X_2 + 0.005X_3 + 0.0009X_4 - 0.020X_1^2 - 0.025X_2^2 + 0.042X_3^2 - 0.003X_4^2 - 0.043X_1 \cdot X_2 + 0.005X_3 \cdot X_4, \quad (2)$$

$$Y_2 = 0.05 - 0.0058X_1 - 0.0089X_2 + 0.0008X_3 + 0.0006X_4 - 0.0046X_1^2 - 0.0051X_2^2 + 0.004X_3^2 - 0.0038X_4^2 - 0.0003X_1 \cdot X_2 - 0.0015X_1 \cdot X_3 + 0.0003X_1 \cdot X_4 - 0.0004X_2 \cdot X_3 + 0.0009X_2 \cdot X_4 + 0.0041X_3 \cdot X_4, \quad (3)$$

where X_1, X_2, X_3, X_4 are coded factor values [8].

Table 2 – Obtained values of modulus of elasticity and strength

No.	X_1	X_2	X_3	X_4	Y_1	Y_2
1	+1	+1	+1	+1	0.182	0.021
2	-1	+1	+1	+1	0.361	0.030
3	+1	-1	+1	+1	0.396	0.037
4	-1	-1	+1	+1	0.518	0.077
5	+1	+1	-1	+1	0.155	0.022
6	-1	+1	-1	+1	0.415	0.036
7	+1	-1	-1	+1	0.434	0.040
8	-1	-1	-1	+1	0.428	0.038
9	+1	+1	+1	-1	0.158	0.019
10	-1	+1	+1	-1	0.429	0.043
11	+1	-1	+1	-1	0.443	0.049
12	-1	-1	+1	-1	0.434	0.047
13	+1	+1	-1	-1	0.191	0.026
14	-1	+1	-1	-1	0.389	0.038
15	+1	-1	-1	-1	0.418	0.052
16	-1	-1	-1	-1	0.531	0.077
17	-1.414	0	0	0	0.322	0.048
18	+1.414	0	0	0	0.373	0.052
19	0	-1.414	0	0	0.323	0.045
20	0	+1.414	0	0	0.352	0.051
21	0	0	-1.414	0	0,420	0.055
22	0	0	+1.414	0	0.518	0.075
23	0	0	0	-1.414	0.284	0.039
24	0	0	0	+1.414	0.481	0.062
25	0	0	0	0	0.332	0.028

As can be seen from the dependencies (2) and (3), the time of thermal exposure has the greatest influence on the modulus of elasticity and strength of the material; the next factor is the temperature of the process. Primary humidity and porosity of the material are not as important for the process as the previous two.

To check the model adequacy and the influence of the factors and their interactions on the indicators, the variance of the experimental errors S_1^2 and S_2^2 for Y_1 and Y_2, respectively, was estimated. For each indicator, four repeated experiments were carried out at the "zero" point $X_1 = X_2 = X_3 = X_4 = 0$. The following results were obtained: 0.300; 0.358; 0.347; 0.319 for Y_1 and 0.023; 0.034; 0.024; 0.026 for Y_2. To estimate the experimental error variance, we used the formula:

$$S^2 = \frac{1}{3}\sum_{i=1}^{4}\left(Y_i - \bar{Y}\right)^2, \quad (4)$$

where Y_1 is the value of the indicator Y in the i^{th} repeated experiment, $\bar{Y}$ is the mean value of Y at the "zero" point. The following results of the calculations were obtained: $S_1^2 = 0.0007$ i $S_2^2 = 0.0000025$.

For estimates of coefficients characterizing the influence of factors and the effects of their interaction, "significance thresholds" were found as $h_i \cdot S$; here S is the root mean square deviation of the observation error, $h_i = t_{cr}(\alpha;\phi)\cdot\sqrt{c_i}$, $t_{cr}(\alpha;\phi)$ is the critical value of the Student's t-distribution for the number of degrees of freedom ϕ and the significance level α. In our studies, $\varphi=3$, $c_1 = 0.05$ for X_V, $c_2 = 0.125$ for X_i^2, $c_3 = 0.0625$ for $X_i \cdot X_j$, $i,j = 1,...4$ [8].

Based on the results of calculations using the above formula, "significance thresholds" for coefficient estimates were established for the significance level $\alpha = 0.05$: 0.021, 0.033, 0.023 for the value Y_1; 0.004, 0.0056, 0.004 for the value Y_2. For the level of significance $a = 0.1$: 0.014, 0.020, 0.015 for the value Y_1; 0.003, 0.0042, 0.003 for the value Y_2.

We excluded factors and their interactions with coefficients smaller (in absolute value) than the specified "significance thresholds"; then for the level of significance $a = 0.1$ the following dependencies were obtained:

$$\hat{Y}_1 = 0.374 - 0.053X_1 - 0.064X_2 - -0.020X_1^2 + 0.025X_2^2 + 0.042X_3^2 - -0.043X_1 \cdot X_2, \quad (5)$$

$$\hat{Y}_2 = 0.05 - 0.0058X_1 + 0.0089X_2 + +0.0046X_1^2 + 0.0051X_2^2 + 0.0041X_3 \cdot X_4. \quad (6)$$

It should be noted that the impact on $Y_1(X_1, X_2, X_3^2, X_1 \cdot X_2)$ is significant with a probability of 0.95, and the impact on X_1^2 and $\%_2^2$ is with a probability of 0.90. Similarly, the effect on $Y_2(X_1, X_2, X_3 \cdot X_4)$ is significant with a probability of 0.95 and also X_1^2 and X_2^2 – with a probability of 0.9, respectively.

The adequacy of the obtained models was tested using Fisher's test. The calculated value of the F-statistic was found by the formula:

$$F_P = \frac{S_{OCT}^2}{S^2}, \quad (7)$$

The residual variance for the obtained models was found as:

$$S_{\text{residual}}^2 = \frac{1}{n-m}\sum_{i=1}^{n}\left(Y_i - \hat{Y}_i\right)^2, \quad (8)$$

where n=25 is the number of tests, m is the number of coefficients in the model.

As a result, for Y_1: $S_{\text{residual.1}}^2$=0.0053 and F_{P1} = 7.56; for Y_2: $S_{\text{residual.2}}^2$=0.00002 and F_{P2}=8.00. Table values of F-statistics for the significance level a=0.05 for Y_1 $F_{\text{tabular}} = F(0.05;18;3) = 8.675$, for Y_2 $F_{\text{tabular}} = F(0.05;19;3) = 8.667$. Since F_p for both models is less than $F_{tabular}$, both models are adequate with a reliability of 0.95 of the true relationship and can be used for technological analysis of the process and forecasting the values of indicators Y_1 and Y_2.

According to equation (6), the graph of material strength Y_2 has a clearly defined maximum when the accepted factors change, which can be seen from Fig. 1. The strength change graph is given for the values of the factors $\%_3 = 0$ and $\%_4 = 0$.

Since the regression dependences for the modulus of elasticity Y_1 and strength Y_2 turned out to be adequate to the experimental data, this made it possible to use them to control the heat treatment process. The strength Y_2 was taken as the chain option, and the modulus of elasticity Y_1 was included in the constraints. As a result, the following optimization model of the heat treatment process was obtained:

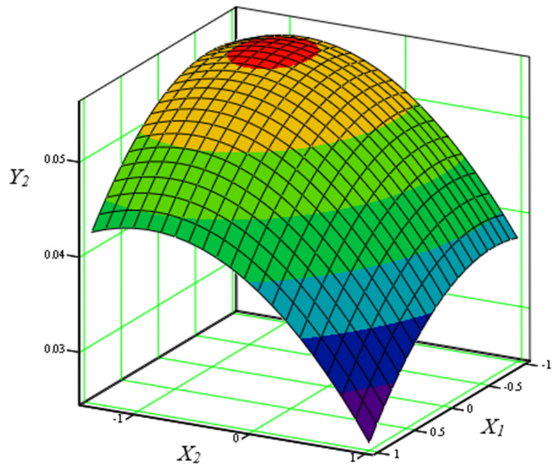

Fig. 1. Graph of dependence of material strength on the factors X_1 and X_2

$$\max Y_2 = 0.05 - 0.0058X_1 - 0.0089X_2 - -0.0046X_1^2 - 0.0051X_2^2 + 0.0041X_3 \cdot X_4, \quad (9)$$

$$Y_1 = 0.374 - 0.053X_1 - 0.064X_2 - 0.020X_1^2 - -0.025X_2^2 + 0.042X_3^2 - 0.043X_1 \cdot X_2 \le 0.45 \quad (10)$$

The Lagrange function was composed to determine the optimal regime of heat treatment in terms of temperature and time:

$$L = Y_2 + \lambda\left(Y_1 + X_5 - 0.45\right). \quad (11)$$

For optimization of values $X_i, i = 1,...5$, the system of equations is used:

$$\begin{cases} \frac{\partial L}{\partial X_1} = -0.0058 - 0.0092X_1 + \lambda(-0.53 - 0.4X_1 - 0.43X_2) = 0 \\ \frac{\partial L}{\partial X_2} = -0.0089 - 0.0102X_2 + \lambda(-0.64 - 0.5X_2 - 0.43X_1) = 0 \\ \frac{\partial L}{\partial X_3} = 0.0041X_4 + 0.84 \cdot \lambda \cdot X_3 = 0 \\ \frac{\partial L}{\partial X_4} = 0.0041X_3 = 0 \\ \frac{\partial L}{\partial X_5} = \lambda = 0 \\ \frac{\partial L}{\partial \lambda} = 0.374 - 0.053X_1 - 0.064X_2 - 0.02X_1^2 - 0.025X_2^2 + \\ +0.042X_3^2 - 0.043X_1 \cdot X_2 + X_5 - 0.45 = 0 \end{cases} \quad (12)$$

As a result of solving (12), a stationary point of the Lagrange function was obtained in the coded values of the variables $X_1 = -0.63$; $X_2 = -0.87$; $X_3 = 0$; $X_4 = 0$; $X_5 = 0.076$. In natural values: $X_1 = 174$

°C, $X_2 = 1.3$ s, $X_3 = 45$ %, $X_4 = 70$ %. According to the obtained model, the indicators are *0.413* for the modulus of elasticity and *0.056* for the strength of the material for a given regime of heat treatment of porous materials.

4. Conclusion

The results of the study show that the time of thermal exposure is the main factor that affects the strength of thermal insulation materials with a porous structure. The intensity of heat and mass exchange processes in wet material is uniquely determined by this factor. The second influencing factor is the processing temperature. Based on the obtained data, the final properties of thermal insulation porous materials, such as strength and elasticity, can be predicted.

This research makes it possible to determine the conditions for the introduction of new technologies for the production of thermal insulation porous materials with predicted properties.

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