THERMAL AND ABSORPTION PROPERTIES OF UNBURNT CLAY SAMPLES

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Abstract. Unburnt clay might be used as a building material for construction of both economic buildings and even dwelling houses. This is especially relevant in rural areas as thus energy is saved, employment of local inhabitants is increased and building costs are reduced. The purpose of this work is to investigate the dependence of the density of unburnt clay samples on the technology of their production – extruding pressure and absolute humidity of the forming mass; also dependence of heat conductivity and water vapour absorption capability of unburnt clay samples on the absolute humidity of the forming mass while using 40 MPa pressure. It is also aimed to investigate the dependence of heat conductivity of the samples on absolute humidity of these samples during measurements. Another purpose is to determine relevance of absolute humidity of the forming mass and absolute humidity of samples during the period of investigation on heat conductivity. Dependence of density of formed and dried samples on the technology of their production was established. Hypothesis for explanation of this dependence was suggested. Dependence of samples' heat conductivity and water vapour absorption on the technology of their production and absolute humidity of samples during the period of investigation were determined. A model for explanation of this dependence was suggested. Prediction of heat conductivity pursuant to the forming mass of unburnt clay samples and absolute humidity of these samples was done applying linear regression. Significance of the samples forming mass and absolute humidity of samples on heat conductivity was determined.

Keywords: clay, density, heat conductivity, absorption capacity, linear regression.

Introduction

Clay as a building material in prewar Lithuania was mostly used for construction of farming buildings. Stables, barns, storehouses and even dwelling houses were built from unburnt clay. Clay is found in almost every rural area and therefore construction from unburnt clay was significantly cheaper comparing to burnt clay bricks. Healthy air humidity and good microclimate forms in earthen buildings by itself. Earthen houses are fairly warm if the thickness of walls and the mix of clay with organic supplements were properly chosen.

Clay is an eco-friendly material as it does not emit toxic substances and it is not hazardous for health. Therefore, clay may be used for construction of dwelling houses. There are four and five storey buildings with main inner walls made of unburnt clay bricks in Vilnius old town.

Clay was used for construction not only in Lithuania. All buildings were earthen in ancient Mesopotamia rich by clay, sand and canes. Certain pyramids and tabernacles were built from unburnt clay bricks in Egypt. Well-preserved earthen dwelling houses, castles and even churches built in the eighteenth century have remained in Germany and France till nowadays. Many dwelling houses are built from clay in Afghanistan, Egypt and other Asian and African countries.

Growing costs of both energy and fuel make to search for methods to reduce production costs of building materials and to increase their quality. Building made of unburnt clay with various supplements may be one of such methods.

Even industrialized countries quite widely started building dwelling houses of clay using production systems of modern bricks and refusing burnt clay. Dwelling houses made of unburnt clay blocks with 5 % cement before 1985 are found in France between Lyon and Grenoble till nowadays. Approximately 50 one and two-storey kit houses are built in Kassel, Germany. There are houses made of unburnt clay bricks extruded by manual press near Brussel. Kit houses are built of clay kneaded with straws in Europe. Such outer walls are non flammable, have good insulating properties and are easy to plaster or decorate in any other way. It is worked on trying to produce large wall panels. A high school with a testing laboratory for experimental construction of local materials including clay was found in Kassel, Germany. A center for revitalizing earthen buildings was established in Grenoble, France. The company of Peter Brechenbach in Germany produces light earthen panels with organic fiber supplements and other products of unburnt clay since 1984. These products are also exported abroad.

On purpose to revitalize application of unburnt clay in construction works new investigation is necessary to identify the composition of materials with a defined project and operation properties. Heat conductivity can be subsumed to the most important properties because unburnt clay is mostly used for construction of outer walls.

Unburnt clay is composed of small particles with hollows and capillaries between the particles. Clay has various degree of humidity and heat transfer in the clay occurs due to the following phenomena: conduction, convection, vapour latent heat transmission and radiation from one particle to another.

Conduction is a phenomenon where heat transfer from one particle to another occurs due to inter contact of the particles.

Convection is a phenomenon where heat transfer occurs due to particles' inter-confusion. This phenomenon takes place in substances of gaseous and liquid phases. Water and water vapour, air transfer heat from one place to another while moving in the hollows and capillaries.

Vapour latent heat transmission is a phenomenon when vapour latent heat is absorbed from environment while water vaporizes. The composed water vapour is transferred to another place due to convection or diffusion. After condensation of water vapour occurs, vapour latent heat is transmitted to the material.

Radiation from one particle to another is a phenomenon when heat is transmitted during radiation without inter-connection of particles. Due to this phenomenon heat is also transmitted in vacuum.

Heat transmission mostly happens due to conduction, however other patterns of heat transmission may assert depending on the state of the material.

It was observed from the results given in references that earlier most attention was paid to estimation of strength and shrinkage during desiccation and there were very little data about investigation of heat conduction.

Results of unburnt clay investigation are given in this article. These results can be used while producing new building elements for construction of various buildings.

The purpose of the work – to investigate the dependence of the density of unburnt clay samples on the technology of their production – extruding pressure and absolute humidity of forming mass; also the dependence of heat conductivity and water vapour absorption capability of unburnt clay samples on absolute humidity of the forming mass using 40 MPa pressure. It is also aimed to investigate the dependence of heat conductivity of the samples on absolute humidity of these samples during the measurements.

Methodology of investigation

Easy melting clay from Šatijai clay quarry in Kaunas district was used for production of samples. This clay is used for production of ceramic bricks. The composition of clay used for investigation is as follows: 9 % of particles the size of which was 1.0…0.05mm; 60.63 % of 0.05…0.005 mm particles; 30.37 % of particles smaller than 0.005 mm. No particles larger than 1mm were found in the investigated clay. Humidity of plasticity and fluidity for this earth were respectively 19.67 and 40.1%, and the plasticity index is equal to 20.43 %. According to the latter factor the researched earth is middle plasticity clay.

Clay plates with borders of 100x100mm were produced for investigation. The thickness of the plates was from 7.5 to 13 mm (depending on extruding pressure and humidity of forming mass). A digital analytical scale of 0.01 g was used for weighting both raw materials and samples. The necessary amount of water was measured with a glass beaker of 1ml accuracy.

The extruding form used for samples' production consists of two pistons and a clamp. The clamp elements (four plates) are connected by bolts which are unscrewed after extruding the sample and the clamp is dismantled. A hydraulic press ПГ-100 was used for extruding samples.

While producing clay samples the extruding pressure varied from 3 to 40 MPa and humidity of the forming mass varied from 1.7 % to 19.9 % in respect of dry clay mass. The same amount of dry clay is used while producing the sample. The minimum (critical) pressure at which samples can be extruded from clay with 1.7 % humidity is equal to 20 MPa. The critical samples' extruding pressure declines as humidity of the forming mass increases. The critical pressure declines to 10 MPa when humidity of the forming mass is more than 3 %. The extruded samples were kept in dry environment at 18-20 ˚C temperature for approximately one month until the humidity of samples almost equaled. Then samples were kept at 40 ˚C temperature. After the weight did not change any more, the samples were placed into hermetic vessels to avoid absorption of humidity from air.

For further investigation of properties of unburnt clay samples the samples were dried at 105 ˚C temperature for two days and then cooled in hermetic vessels until 20 ˚C. After weighting and measuring the dimensions of the samples their density was calculated. Afterwards the samples were kept in the thermostat at 32 ˚C above water for 9 days until the weight of the samples no longer increased. The samples were weighted every 24 hours.

Dependence of heat conductivity on the sample's humidity during investigation was also researched. 10 such measurements were done for every sample every 24 hours. On purpose to achieve higher accuracy of results, average measurement results of three samples formed at the same conditions (40 MPa extruding pressure and the same humidity of the forming mass) were taken.

Heat conductivity of samples was measured using calorimetrical method. The measurements were done using a gauge of heat conductivity FOX200 which was created by the company *LaserComp* according to ASTM C518-91 "Standard test method for steady-state heat flux measurements and thermal transmission properties by means of the heat flux meter apparatus".

The gauge of heat conductivity is calibrated in LaserComp Inc., using NIST SRM 1450b (Standard information material of National Standard and Technology Institute) and special EPS NIST standard of high accuracy (0.5 %). Calibrations were done at 25 ˚C and 20 ˚C temperature differences directing the heat flux upwards, i.e., bottom isothermal plate of gauge is hotter than upper one, and stored in nonerasable memory of the device. The calibration was done according to ASTM C1132-89 "Standard Practice for Calibration of the Heat Flow Meter Apparatus". Heat conductivity is calculated according to ASTM C1045-90 "Standard Practice for Calculating Thermal Transmission Properties from Steady-State Heat Flux Measurements".

Every isothermal plate has very sensitive sensors of heat flux and temperature. Gauge can work alone or connected to personal computer through RS-232 interface.

General principle of FOX series heat flux gauges is based on one-dimensional Fourier law:

$$q = -\lambda \frac{\partial T}{\partial x}, \tag{1}$$

where q – density of heat flux whose numeric value equals to heat amount running through square unit during time unit;

λ – heat conductivity;

$\frac{\partial T}{\partial x}$ – temperature gradient, when heat flows along x axis.

A flat sample is fitted between two plane isothermal plates, supporting two different temperatures. Then the temperature field of the sample should be constant in the whole volume. Temperature gradient is estimated according to the measured temperature difference $\Delta t = t_k - t_s$ (here t_k, t_s – respectively higher and lower temperature of isothermal plates of heat conductivity gauge) the sample thickness Δx, as in this case the average temperature gradient $\frac{\partial T}{\partial x}$ is equal to $\frac{\Delta t}{\Delta x}$. On purpose to avoid marginal effect errors heat flux sensors are installed in the center of isothermal plates, in the 50x50mm square.

The output signal of the heat flux sensor Q (μV) is proportional to heat flux q:

$$q = \lambda_{kal}(t_{kal}) \frac{\Delta t_{kal}}{\Delta x_{kal}} = S_{kal}(t_{kal}) Q(\mu V), \tag{2}$$

where $\lambda_{kal}(t_{kal})$ – heat conductivity of calibration standard at temperature t_{kal};

$S_{kal}(t_{kal})$ – calibration factor of the heat flux senor installed in isothermal plate at temperature t_{kal}.

The dimension of the calibration factor is $W\cdot(m^2\cdot\mu V)^{-1}$. Each of two flux sensors has its temperature and therefore the calibration factor is estimated according to the factual sensor temperature. Two different sets of calibration factors are measured during calibration. Calibration factors $S_{kal}(t)$ are characteristics of the device. They are used for calculations of heat conductivity during the experiment:

$$\lambda_b = S_{kal}(t_b)Q\frac{\Delta x_b}{\Delta t_b} \quad . \tag{3}$$

The measurements result is average of heat conductivity calculated pursuant to values of heat flux estimated by heat flux sensors of bottom and upper isothermal plates.

The thermal conduction coefficient for heat insulation materials $a=\lambda\cdot(c_p\rho)^{-1}$ is approximately (4–7) $\cdot10^{-7}$ m^2s^{-1} (c_p – sensible heat, when pressure is constant; ρ – density). Fourier number (non-dimensional parameter of heat transmission used to investigate heat flow) $F_0=4at(\Delta x)^{-2}$ is approximately 9-16 per hour for 25.4 mm thickness sample. On purpose to achieve equilibrium temperature and to obtain F_0>>1, a fairly long period of time is required (at least 30 minutes for one 2.5 cm thickness sample). The experimental testing showed that the average size of two heat flux measurement signals achieves equilibrium a few times faster comparing to their individual sizes.

FOX structure. The gauge of heat conductivity FOX200 is composed of a chamber and base with a keyboard and display (Fig. 1).

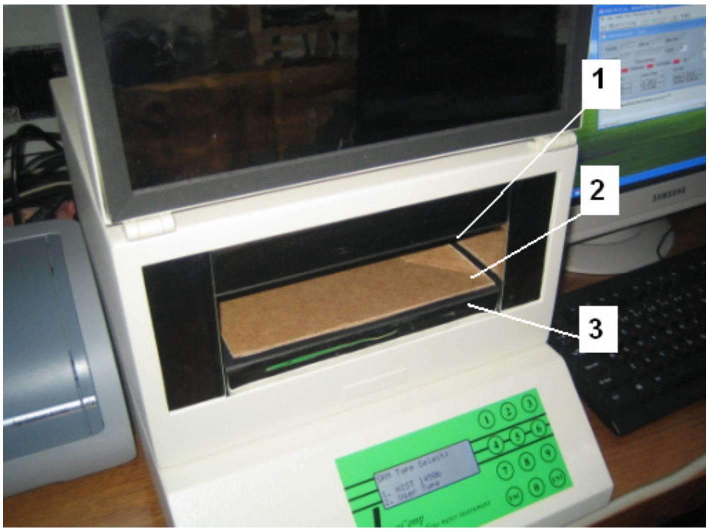

Fig. 1 **Gauge of heat conductivity:**
1 – upper isothermal plate, 2 – sample, 3 – lower isothermal plate

All electronics is mounted in the base, keyboard and display. When the chamber door is opened the sample can be fitted between two isothermal plates. The upper plate does not move while the bottom plate might move upwards and down because of four independently controlled stepping motors. Four digital sensors of accurate thickness control the position of every corner of the bottom plate. When the sample is placed into the device and the chamber is closed, average thickness of the sample is estimated with accuracy of ±0.025 mm.

High accuracy heat flux sensors, created in LaserComp Inc., are near the surfaces of both plates. They are composed of thousand small thermocouples. It ensures high sensitivity of sensors and signals' integration. General sensors' thickness is approximately 1 mm. E type thermocouples are in the center of every sensor. Thermocouples are located near the sample and show accurate temperature of both sample surfaces. The same thermocouples are used for temperature measurement in the center and margins of the plates. 24 bytes analog digital converter (ADC) changes analog signals of thermocouples and the heat flux gauge into digital signals with 0.6 μV resolution. Resolution of temperature measurements is 0.01 ˚C.

Each isothermal plate has its heating-cooling system. Every system consists of central and side groups of thermo-electrical elements. These groups are independently controlled on purpose to eliminate gradients of radial temperature in the plates. If temperature of cooling water is about 18 ˚C or lower temperature of isothermal plates can be independently kept from –20 ˚C up to 75 ˚C with allowance of ±0.02 ˚C.

FOX device has a powerful digital signal processor (DSP). DSP control all processes of the gauge. It helps to keep temperature of isothermal plates with accuracy of ±0.02 ˚C. Specified control signals are periodically sent to the energy output table which properly powers the heating-cooling system of every plate.

DSP control system executes the necessary following actions using special software:

- scanning of signals from heat flux gauges and thermocouples, installed in both plates;
- control correction and stabilization of both plates' temperature;
- estimate if heat equilibrium was achieved using equilibrium criteria;
- control lift-droop motors and estimation of accurate sample thickness;
- show average data and calculations during device calibration or during measurement of sample's heat conductivity;
- data transfer to the main computer or printer through serial interface RS-232.

Accuracy of heat conductivity measurements is 1 %; reiteration of the results is equal to 0.2 %.

Results and discussion

Density of the formed dry samples depends on forming conditions: humidity of the forming mass and extruding pressure. At the constant extruding pressure while increasing humidity of the forming mass density of the formed samples increases, reaches maximum and then declines. Water evaporates while samples desiccate and maximum density values decline and are observed at lower humidity of the forming mass. Density dependence of samples dried at 105 ˚C on the production conditions are given in Fig. 2. Trends of these dependencies are expressed by second degree polynomial $y=ax^2+bx+c$. Dependence of polynomial coefficients on extruding pressure is given in Table 1.

Table 1

Polynomial coefficients of density dependencies on samples' forming conditions for samples dried at 105 ˚C temperature

Forming pressure, MPa	*a*	*b*	*c*	R^2
40	-2.1548	35.703	1818.8	0.9831
30	-1.9114	35.715	1768.2	0.9747
2 0	-1.7201	36.927	1705.2	0.9845
15	-2.0119	46.15	1623.4	0.989
10	-1.9265	49.67	1529.7	0.9813
5	-1.6821	54.433	1343.4	0.9703
3	-2.0809	84.722	894.84	0.9881

When extruding pressure declines maximum density is observed at higher humidity of the forming mass and maximum density values decrease. This can be explained by the fact that water reduces friction between clay particles. As humidity of the forming mass at constant pressure increases clay particles get closer and porosity of samples declines. It causes growth of sample density. As humidity of the forming mass further increases more pores are filled with water which is not compressible. After humidity of the forming mass is increased even more the state where all pores are filled with water is reached and excess of water is removed from the sample during sample extruding together with small clay particles. Humidity of the forming mass when two density dependencies on extruding humidity concurs can be considered as critical at which all pores are filled with water. The higher the humidity of the forming mass, the less the approach of clay particles and more small particles are removed with water. These two factors increase the sample's porosity and reduce density of the extruded sample.

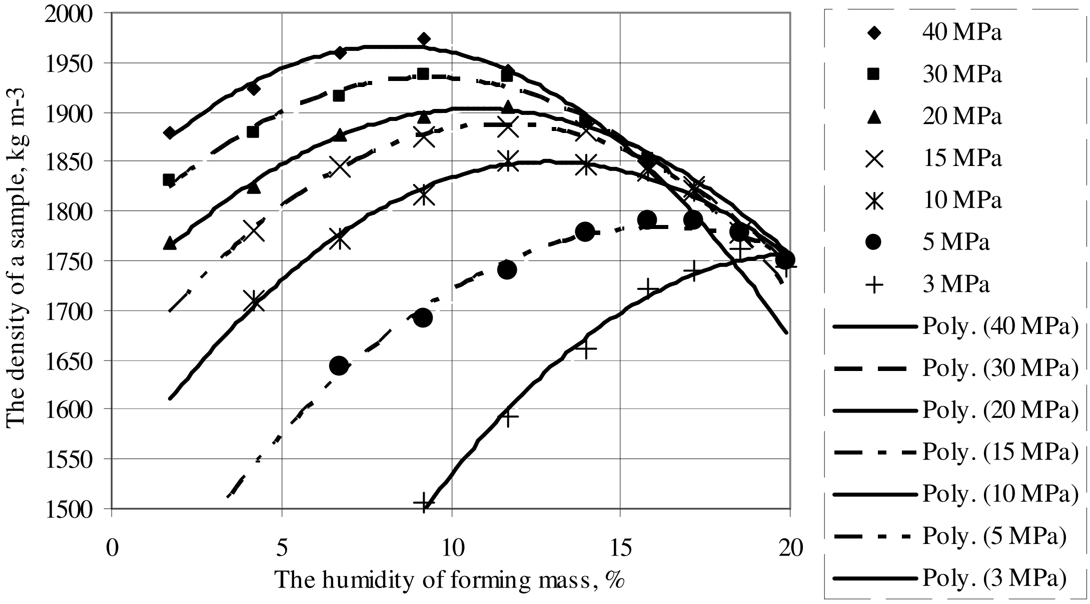

Fig. 2. **Density dependence of dried samples on the humidity of forming mass at various extruding pressure**

Dependencies of heat conductivity of the sample extruded by 40 MPa pressure on humidity of the forming mass at various sample humidity during investigation are given in Fig. 3.

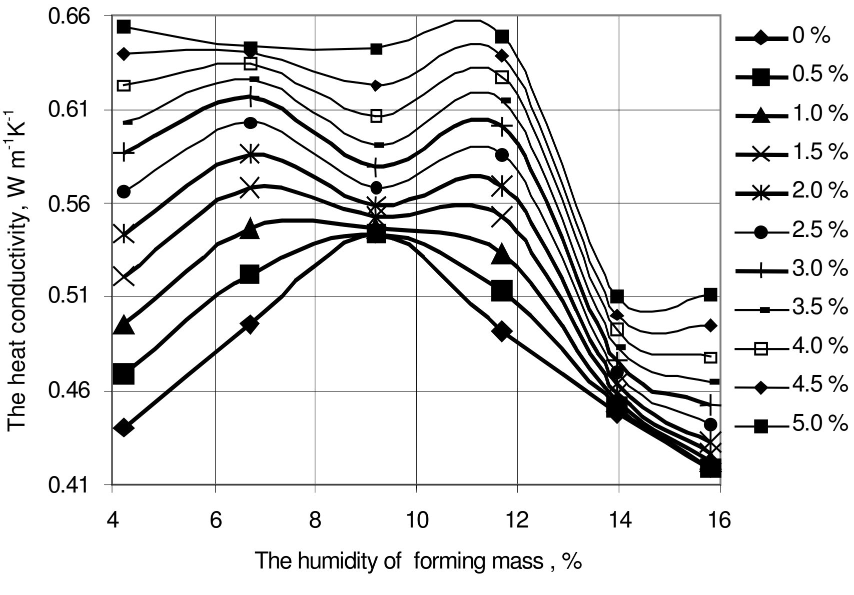

Fig. 3. **Dependence of heat conductivity on humidity of forming mass with various absolute humidity of samples during investigation**

It may be observed that while increasing humidity of the forming mass, heat conductivity of dry samples (0 % curve) increases, reaches maximum and then declines. After comparing these results with dependence of density of dry samples, extruded at 40 MPa pressure, on humidity of the forming mass (Fig. 2) we see that both heat conductivity and density reach maximum values at the same humidity of the forming mass. It does not contravene the known fact that heat conductivity of homogenous materials is proportional to their density. We think that in this case phenomenon of conduction mostly participates in heat transmission. The character of the dependence of heat conductivity on humidity of the forming mass varies as humidity of the samples during investigation increases. When humidity of the samples is less than 1.5 % heat conductivity leastwise changes for the samples, produced from the forming mass whit humidity equal to 9.2 % and 14 % and the supreme change is observed for the samples from the forming mass with humidity equal to 4.2 %, 6.7 % and 11.7 %. When humidity of the samples during investigation is more than 1.5 %, heat conductivity significantly increases for the samples produced from the forming mass the humidity of which is 9.2 %. As humidity of the samples reaches 5 % heat conductivity for all samples extruded from the forming mass the humidity of which was from 4 to 12 % does not depend on humidity of the forming mass and equals to 0.64-0.65 W mK^{-1}. For the samples extruded from the forming mass the humidity of which exceeds 12 % heat conductivity declines as humidity of the forming mass increases. However, heat conductivity of the samples, the humidity of which during investigation is more than 5 %, increases when humidity of the forming mass is more than 14 %. Such motion of dependence can be explained by the following model. At the beginning as humidity of the forming mass increases gaps between clay particles decline and cause increase of heat conductivity due to heat transmission from one particle to another by direct touch. Maximum heat conductivity of dry samples is observed when gaps between the particles are minimal, i.e., when humidity of the forming mass is equal to 9.2 %. As humidity of the forming mass continuously increases water in small gaps does not compress and size of the gaps between clay particles in dry samples increases. It causes monotonous reduction of heat conductivity while humidity of the forming mass increases. Such model is confirmed by dependence of sample's density on humidity of the forming mass. Growth of heat conductivity while increasing humidity of the samples can be explained as follows. For the samples produced from forming mass with humidity equal to 4-7 % the increasing amount of water expands total contact square between particles and partial pressure of water vapour in the pores of the sample. It causes increase of heat transmission due to conduction, convection, diffusion and vapour latent heat transmission. However, water compose plugs in the samples produced from the forming material with humidity values close to the ones at which the maximum sample density is observed and these plugs interrupt diffusion and vapour latent heat transmission. As humidity of the samples during investigation increases the amount of water plugs grows and these plugs join together filling capillaries formed in the samples. It was also confirmed by investigation of samples' absorption (Fig. 4).

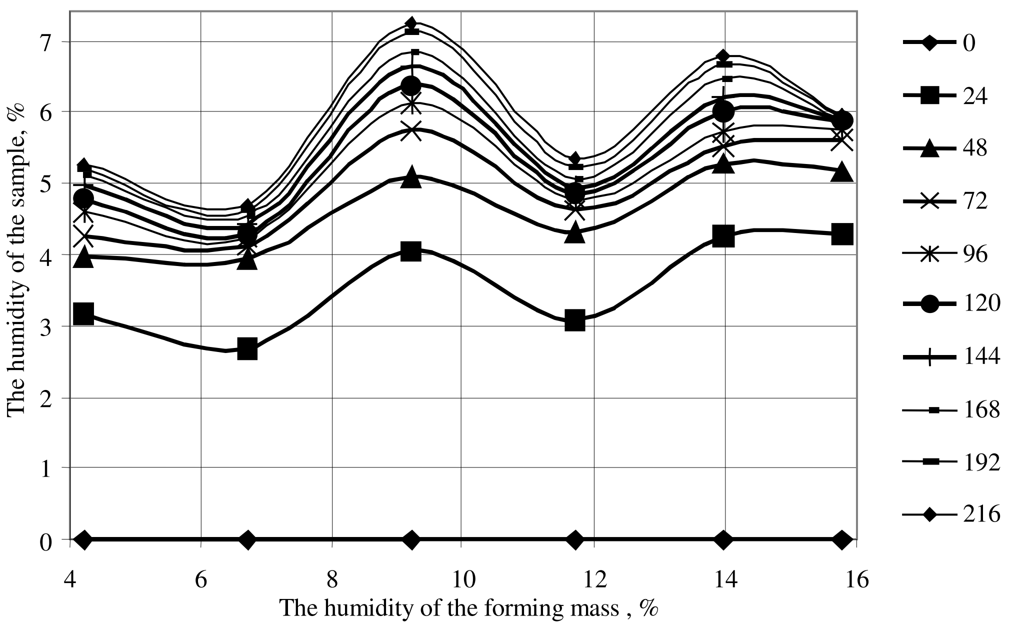

Fig. 4. **Dependence of sample humidity on humidity of the forming mass at various duration of samples' storage above water. Numbers at the curves are sample's storage duration in hours**

It was observed that the samples produced from forming mass with humidity equal to 9.2 % have maximum absorption capability of water vapour. It confirms our statement that capillaries with the minimum diameter form in these samples. Water, occurred in capillaries due to condensation, is sucked deeper into the sample because of maximum additional pressure. This happens because higher additional pressure at water surface appears when capillaries are narrower. It was also observed that the highest absorption of water vapour is during the first 24 hours.

Statistical analysis was accomplished for the obtained data using software package SPSS 15. Dependence of samples' heat conductivity on absolute humidity of the forming mass W_f and absolute humidity of the samples during investigation W_b were analyzed applying the procedure of linear regression. 243 experimental points were used for analysis. The results of the investigation are given in Table 2. The coefficient of determination for the chosen model is equal to 0,735. It is observed from the data given in Table 2 that absolute humidity of the forming mass and humidity of these samples during investigation are statistically significant (p<0.001) while predicting heat conductivity. Pursuant to standardized coefficients it can be stated that humidity of the forming mass (β=–0.551) is less significant comparing to humidity of the samples during investigation (β=0.707). The results of the investigation are expressed by the following linear regression equation:

$$\lambda = 0.588 - 0.012 W_f + 0.028 W_b, \tag{4}$$

wehre λ – heat conductivity, W mK^{-1}.

Table 2

The coefficients of linear regression equation for the heat conductivity, which is influenced by the humidity of the forming mass and the humidity of samples

Coefficients / Factors	Non-standardized coefficients		Standardized coefficients	*t*	*p*
	B	Std. Error	*β*		
Constant	0.588	0.015		38.502	0.000
The humidity of a sample	0.028	0.002	0.707	12.095	0.000
The humidity of the forming mass	-0.012	0.001	-0.551	-9.429	0.000

Pursuant to this linear regression equation it can be stated that heat conductivity of unburnt clay samples declines by 0.012 W mK^{-1} after humidity of the forming mass is increased by 1 %. However, the experimental results show that this is not true. On purpose to increase accuracy of the linear regression model the experimental data were divided into two groups according to humidity of the forming mass: up to 9.2 % and above 9.2 %. Humidity of the forming mass, when motion change of heat conductivity dependence of absolutely dry samples on the forming mass was observed, is taken as a division point. The results of the investigation are given in Tables 3 and 4.

Table 3

The coefficients of linear regression equation for the heat conductivity, which is influenced by the humidity of the forming mass and the humidity of samples (the humidity of the forming mass is 9.2 % and less)

Coefficients / Factors	Non-standardized coefficients		Standardized coefficients	*t*	*p*
	B	Std. Error	*β*		
Constant	0.466	0.012		40.398	0.000
The humidity of a sample	0.033	0.002	0.928	20.758	0.000
The humidity of the forming mass	0.004	0.002	0.116	2.601	0.013

Table 4

The coefficients of linear regression equation for the heat conductivity, which is influenced by the humidity of the forming mass and the humidity of samples (the humidity of the forming mass is 9.2 % and major)

Coefficients / Factors	Non-standardized coefficients		Standardized coefficients	*t*	*p*
	B	Std. Error	*β*		
Constant	0.763	0.022		35.356	0.000
The humidity of forming mass	-0.024	0.002	-0.680	-15.189	0.000
The humidity of a sample	0.024	0.002	0.590	13.186	0.000

According to the data, given in Tables 3 and 4, it can be stated that humidity of the forming mass and humidity of the sample during investigation are statistically significant even after distributing data into two groups. When humidity of the forming mass is 9.2 % and less (Table 3), the humidity of the samples during investigation is noticeably more important than humidity of the forming mass (non-standardized coefficients β are equal to 0.928 and 0.116 respectively). Prediction of heat conductivity is done applying the following equation:

$$\lambda = 0.466 + 0.004W_f + 0.033W_b . \qquad (5)$$

It is shown that heat conductivity increases while increasing humidity of both the forming mass and the sample. The coefficient of determination for this model is equal to 0.931, i.e., significantly higher comparing to common case. When humidity of the forming mass is 9.2 % and more, significance of sample humidity during investigation and forming mass humidity for prediction differs marginally. Their non-standardized coefficients β are equal to 0.590 and –0.680 respectively. Prediction of heat conductivity is done applying the following equation:

$$\lambda = 0.763 - 0.024W_f + 0.024W_b . \qquad (6)$$

The coefficient of determination for this model is equal to 0.891, i.e., predictions done applying (5) and (6) are more accurate comparing to predictions done applying (4).

Conclusions

1. Density of samples depends on absolute humidity of the forming mass and extruding pressure. While increasing humidity of the forming mass the density of samples increases, reaches maximum and then declines. When extruding pressure increases density reaches maximum at lessened humidity of the forming mass.
2. Heat conductivity of the samples, extruded by 40 MPa pressure, increases while increasing absolute humidity of the samples.
3. Heat conductivity of the samples depends on humidity of the forming mass as this parameter determines density of the samples and, in our opinion, on the form of pores.
4. The coefficients of friction immovability change when extruding pressure is constant and absolute humidity of the forming mass changes. Therefore, clay particles transfer from the state of larger potential energy to less one saltatorily while humidity of the forming mass increases.
5. The following model of heat transfer is proposed: the main principle in heat transfer is heat transmission from one particle to another, water vapour diffusion and vapour latent heat transmission.
6. Absolute humidity of unburnt clay samples, extruded by 40 MPa pressure, statistically is more significant for heat conductivity than absolute humidity of the forming mass.

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