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Measuring the effective specific heat of building materials[1]

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Abstract

Thermal effects accompanying chemical reactions and phase changes in building materials at elevated temperatures are analyzed using a model of an n-component chemically reacting mixture. The concept of an effective specific heat, c_{ef}, is introduced which accounts for these two effects, and a nonadiabatic calorimeter is used in measuring c_{ef} of selected building materials. The analysis of different types of building materials leads to an identification of several characteristic $c_{ef}(T)$ functions. For compact, thermally stable materials such as concrete, the effective specific heat increases monotonously with temperature, which is analogous to homogeneous materials, e.g., metals. The behavior of light macroporous materials varies depending on the type of material and it is not possible to generalize about the character of the $c_{ef}(T)$ function. One characteristic group is represented by materials containing organic compounds which exhibit dramatic changes in the effective specific heat in the temperature range ~ 300–600°C when the burning temperature is reached. The effective specific heat of materials undergoing structural changes and/or chemical reactions due to increasing temperature, e.g., porous concrete, Porfix, etc., may either decrease with temperature or have an "oscillatory" character.

Keywords: Specific heat; Building materials; Chemical reactions; Phase changes

1. Introduction

Many building materials contain components undergoing chemical reactions not only in the production phase but also after their incorporation in a building structure

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[1] Dedicated to Takeo Ozawa on the Occasion of his 65th Birthday.

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[1]. The hydration and carbonation of cement-containing materials can be considered as a typical example of such reactions [2–4]. The occurrence of chemical reactions can change the thermal behavior of a particular material since heat can be produced or absorbed depending on the type of reaction. These processes are accentuated with increasing temperature when the reactions are faster, and for some material components an increasing risk of burning arises.

A representative mathematical description of transport phenomena in building materials at higher temperatures, which is particularly important, for instance, in the solution of fire-protection problems, includes formulation of a relatively complicated problem of continuum mechanics and thermodynamics. Generally, it can be solved using the theory of chemically reacting mixtures [5], and formulating a set of balance equations for both the mixture and the particular components, which has to be solved with the help of material relations derived by means of nonequilibrium thermodynamics.

Solution of large sets of differential equations obtained in this way was not very frequent in practical applications, until now, for several reasons: analytical solutions are mostly not available, numerical solutions can present problems with computer memory and reasonable CPU time, and in addition, many material parameters entering the equations after applying the laws of nonequilibrium thermodynamics are very difficult to measure. Therefore, most authors have looked for consistent simplifications that make the solutions easier, while keeping the characteristic features of the described process.

In this paper, we introduce the concept of the effective specific heat, c_{ef}, of building materials, which also includes thermal effects due to the chemical reactions and/or phase change processes, and employ a nonadiabatic calorimeter developed earlier for measuring c_{ef} as a function of temperature.

2. Mathematical model

For an n-component chemically reacting mixture, the balance equations of mass, momentum and internal energy, together with the mass balance of a component α can be written in the form (see Refs. [5, 6])

$$\frac{\partial \rho}{\partial t} + \operatorname{div} \rho \vec{v} = 0 \tag{1}$$

$$\frac{\partial \sum_{\alpha=1}^{n} \rho_\alpha \vec{v}_\alpha}{\partial t} + \operatorname{div}\left(\sum_{\alpha=1}^{n} \rho_\alpha \vec{v}_\alpha \otimes \vec{v}_\alpha \right) = \operatorname{div} \sum_{\alpha=1}^{n} \mathbf{P}_\alpha + \sum_{\alpha=1}^{n} \rho_\alpha \vec{E}_\alpha \tag{2}$$

$$\begin{aligned} \frac{\partial \sum_{\alpha=1}^{n} \rho_\alpha u_\alpha}{\partial t} + \operatorname{div}\left(\sum_{\alpha=1}^{n} \rho_\alpha u_\alpha \vec{v}_\alpha \right) &= \sum_{\alpha=1}^{n} \mathbf{P}_\alpha : \mathbf{D}_\alpha - \operatorname{div} \vec{j}_Q \\ + Q - \sum_{\beta=1}^{n-1} \vec{k}_\beta \vec{\omega}_\beta - \sum_{\beta=1}^{n-1} \frac{1}{2} R_\beta \vec{\omega}_\beta \vec{\omega}_\beta & \end{aligned} \tag{3}$$

$$\frac{\partial \rho_\alpha}{\partial t} + \operatorname{div} \rho_\alpha \vec{v}_\alpha = R_\alpha, \quad \alpha = 1, 2, \cdots, n-1 \tag{4}$$

where ρ is the volume mass of the mixture, ρ_α the volume mass of a component α

$$\rho = \sum_{\alpha=1}^{n} \rho_\alpha \tag{5}$$

$\vec{v}$ is the velocity of the mixture, $\vec{v}_\alpha$ of a component α

$$\vec{v} = \frac{1}{\rho} \sum_{\alpha=1}^{n} \rho_\alpha \vec{v}_\alpha \tag{6}$$

$\mathbf{P}_\alpha$ is the stress tensor of a component α, and for the stress tensor of the mixture $\mathbf{P}$ we have

$$\mathbf{P} = \sum_{\alpha=1}^{n} \mathbf{P}_\alpha \tag{7}$$

$\vec{E}_\alpha$ is the force per unit mass acting on the component α, u_α is the specific internal energy (internal energy per unit mass) of a component α, and for the specific internal energy of the mixture u

$$u = \frac{1}{\rho} \sum_{\alpha=1}^{n} \rho_\alpha u_\alpha \tag{8}$$

$\mathbf{D}_\alpha$ is the deformation velocity of a component α, where for a component D^{ij} of the tensor $\mathbf{D}$ we have

$$D^{ij}_\alpha = \frac{1}{2}\left(\frac{\partial v^i_\alpha}{\partial x^j} + \frac{\partial v^j_\alpha}{\partial x^i}\right) \tag{9}$$

$\vec{j}_Q$ is the heat flux, Q the heat power source per unit volume (for instance due to the radiation effects), $\vec{k}_\beta$ is the interaction force per unit volume due to the action of the remaining components on the component β, $\vec{\omega}_\beta$ is the velocity of the component β relative to the component n

$$\vec{\omega}_\beta = \vec{v}_\beta - \vec{v}_n \tag{10}$$

R_α is the production of a component α per unit volume due to the chemical reaction.

We will simplify the general problem by the following assumptions:

1. The mixture is incompressible

$$\rho = \text{const.} \tag{11}$$

(Note that the incompressibility of the mixture does not imply the incompressibility of its components.)

2. The specific external force $\vec{E}_\alpha$ is due to the gravitational field

$$\vec{E}_\alpha = \vec{E} = \vec{g} \tag{12}$$

where $\vec{g}$ is gravity.

3. The interaction forces $\vec{k}_\beta$ can be neglected.

4. The influence of energy dissipation in the balance equation of internal energy as well as the radiation term Q and the terms containing velocities in the second power can be neglected.

Then, we obtain

$$\operatorname{div}\vec{v}=0 \tag{13}$$

$$\rho\frac{\mathrm{d}\vec{v}}{\mathrm{d}t}+\operatorname{div}\left(\sum_{\alpha=1}^{n}\vec{j}_\alpha\otimes\vec{u}_\alpha\right)=\operatorname{div}\mathbf{P}+\rho\vec{g} \tag{14}$$

$$\rho\frac{\mathrm{d}u}{\mathrm{d}t}+\operatorname{div}\left(\sum_{\alpha=1}^{n}\vec{u}_\alpha\cdot\vec{j}_\alpha\right)=-\operatorname{div}\vec{j}_Q \tag{15}$$

$$\rho\frac{\mathrm{d}C_\alpha}{\mathrm{d}t}+\operatorname{div}\vec{j}_\alpha=R_\alpha,\quad \alpha=1,2,\cdots,n-1 \tag{16}$$

where the material derivative for an arbitrary scalar or vector quantity A is defined as

$$\frac{\mathrm{d}A}{\mathrm{d}t}=\frac{\partial A}{\partial t}+\vec{v}\cdot\operatorname{grad}A \tag{17}$$

the diffusion velocity of a component α is

$$\vec{u}_\alpha=\vec{v}_\alpha-\vec{v} \tag{18}$$

the diffusion flux

$$\vec{j}_\alpha=\rho_\alpha(\vec{v}_\alpha-\vec{v}) \tag{19}$$

and the concentration

$$C_\alpha=\frac{\rho_\alpha}{\rho} \tag{20}$$

For most common applications in building science, the velocity of the mixture (the barycentric velocity) is small. Therefore, we can use the following approximations

$$\vec{v}\approx 0 \tag{21}$$

and

$$\frac{\mathrm{d}A}{\mathrm{d}t}\approx\frac{\partial A}{\partial t} \tag{22}$$

for an arbitrary quantity A.

Applying Eqs. (21) and (22) to the system of equations (13–16), we can see that Eq. (13) vanishes, Eq. (14) can be considered only as a tool for determining **P** or some of its components (pressure, for instance, when working with fluids), and the system (13–16) is reduced to

$$\rho\frac{\partial u}{\partial t}+\operatorname{div}\left(\sum_{\alpha=1}^{n}u_\alpha\cdot j_\alpha\right)=-\operatorname{div}\vec{j}_Q \tag{23}$$

$$\rho\frac{\partial C_\alpha}{\partial t}+\operatorname{div}\vec{j}_\alpha=R_\alpha,\ \alpha=1,2,\cdots,n-1 \tag{24}$$

The second term on the left-hand side of Eq. (23) includes the amount of internal energy gained or lost by the system due to the chemical reactions, and we can approximate it by an empirical relation in the form

$$\operatorname{div}\left(\sum_{\alpha=1}^{n} \vec{u}_\alpha \cdot \vec{j}_\alpha\right) \approx \Delta H \frac{\partial C_s}{\partial t} \tag{25}$$

where ΔH is the reaction heat per volume unit of the reaction sources, and C_s is the concentration of reaction sources, $C_s = \rho_s/\rho$.

Finally, we can use the approximations

$$\frac{\partial C_s}{\partial t} \approx \frac{\partial C_s}{\partial T}\frac{\partial T}{\partial t} \tag{26}$$

$$\mathrm{d}u \approx c_p \mathrm{d}T \tag{27}$$

where T is the temperature, and c_p is the specific heat at constant pressure, and after substituting Eqs. (25–27) into Eq. (23) we get

$$\rho\left(c_p + \frac{\Delta H}{\rho}\frac{\partial C_s}{\partial T}\right)\frac{\partial T}{\partial t} = -\operatorname{div}\vec{j}_Q \tag{28}$$

The quantity

$$c_{ef} = c_p + \frac{\Delta H}{\rho}\frac{\partial C_s}{\partial T} \tag{29}$$

we will call the effective specific heat.

When phase changes also occur in the reacting system, we have basically two possibilities for the solution [7]: we can model the interface between the phases either by a discontinuity surface or by using the mushy-zone concept. As we are primarily concerned with a generalization of the specific heat for practical applications, we will use the mushy-zone model. Here, the solved system can be described by a single set of equations defined for the whole region, which is due to the general definition of an arbitrary quantity X (see Ref. [7], for details) in the form

$$X = \lambda X_l + (1-\lambda) X_s \tag{30}$$

where X_l, X_s are the quantities X in the liquid and solid phases, respectively, λ is the liquid fraction, defined as $\lambda = 1$ in the liquid, $\lambda = 0$ in the solid, and $\lambda \in (0, 1)$ in the mushy zone, is determined from the phase diagram (see Ref. [7]).

Using all the approximations mentioned in the first part of this Section and taking Eq. (28) as a basis for further considerations, we arrive at

$$\rho\left(c_p + \frac{\Delta H}{\rho}\frac{\partial C_s}{\partial T}\right)\frac{\partial T}{\partial t} + \rho L_m \frac{\partial \lambda}{\partial T}\frac{\partial T}{\partial t} + \rho L_m \sum_{\alpha=1}^{n-1} \frac{\partial \lambda}{\partial C_\alpha}\frac{\partial C_\alpha}{\partial t} = -\operatorname{div}\vec{j}_Q \tag{31}$$

where L_m is the latent heat of melting. Employing again a similar approximation as before, i.e.

$$\frac{\partial C_\alpha}{\partial t} \approx \frac{\partial C_\alpha}{\partial T}\frac{\partial T}{\partial t}, \quad \alpha = 1, 2, \cdots, n-1 \tag{32}$$

we get

$$\rho\left(c_p + \frac{\Delta H}{\rho}\frac{\partial C_s}{\partial T} + L_m\frac{\partial \lambda}{\partial T} + L_m\sum_{\alpha=1}^{n-1}\frac{\partial \lambda}{\partial C_\alpha}\frac{\partial C_\alpha}{\partial I}\right)\frac{\partial T}{\partial t} = -\operatorname{div}\vec{j}_Q \tag{33}$$

where the effective specific heat includes both chemical reactions and phase changes

$$c_{ef} = c_p + \frac{\Delta H}{\rho}\frac{\partial C_s}{\partial T} + L_m\frac{\partial \lambda}{\partial T} + L_m\sum_{\alpha=1}^{n-1}\frac{\partial \lambda}{\partial C_\alpha}\frac{\partial C_\alpha}{\partial T} \tag{34}$$

3. Measuring the effective specific heat

In the previous section we formally included the influence of chemical reactions and phase changes in a single parameter, the effective specific heat c_{ef}. Considering the relative complexity of the definition relations (29) or (34), the accurate measurement of c_{ef} is not an easy task. We have to take into account the parameters of chemical reactions and phase diagrams which, for certain reactions and materials, had not been determined exactly until now, particularly concerning their exact mathematical description.

A natural way to solve these difficulties appears to be the use of an empirical treatment for the c_{ef} determination under specified conditions, and measuring the generalized specific enthalpy h defined as

$$h(T) = \int_{T_0}^{T} c_{ef}\,\mathrm{d}T \tag{35}$$

where T_0 is some reference temperature, e.g., $T = 0°\mathrm{C}$. Substituting Eq. (35) in Eq. (33), we obtain the usual enthalpy formulation of the heat conduction

$$\rho\frac{\partial h(T)}{\partial T} = -\operatorname{div}\vec{j}_Q \tag{36}$$

In practical measurements of the effective specific heat c_{ef}, we can employ the nonadiabatic calorimeter proposed by Toman and Černý [8]. The calorimeter has a mixing vessel with a volume of 2.5 liters and uses water as the measuring fluid. Heat loss of the nonadiabatic system is determined by a calibration. The measuring method itself is based on well-known principles. The sample is heated to a predetermined temperature T_s in a muffle furnace and put into the calorimeter with water. Then, the relation of the water temperature to time $T_w(t)$ is measured, the water being slowly stirred all the time, until the temperatures of the measured sample and the calorimeter are equal. The duration of temperature equilibration ranges from 20 min to 1 h, depending on the thermal conductivity and size of the material being measured.

First, the theoretical equilibrated temperature, T_e, is determined using the heat loss calibration curve. Then, the specific enthalpy, h, related to a reference temperature, $T_0 = 0°\mathrm{C}$, is calculated from the calorimetric equation

$$h(T_s) = \frac{(K_c + m_w c_w)(T_e - T_{wo}) + \Delta m L}{m} + c_n(T_e - T_0) \tag{37}$$

where m is the mass of the sample, T_s is the temperature of the sample prior to being put into the calorimeter, K_c is the heat capacity of the calorimeter, m_w is the mass of the water, c_w is the specific heat of water, T_{wo} is the initial water temperature, L is the latent heat of evaporation of water, c_n is the mean specific heat of the sample in the temperature interval $[T_0, T_e]$, Δm is the mass of evaporated water

$$\Delta m = m + m_{cw} - \mathrm{m}_s - \Delta m_N - \Delta m_{sc} \tag{38}$$

m_{cw} is the mass of the calorimeter with water before the measurement, m_s is the mass of the system calorimeter-water-sample after measurement, Δm_N is the mass of water naturally evaporated during the measurement (this heat loss is already included in the heat loss calibration curve), Δm_{sc} is the change of mass due to the chemical reaction of the sample with water, e.g., hydrolysis. This latter value can be obtained as $\Delta m_{sc} = m - m_D$, where m_D is the mass of the dried sample after the measurement.

Performing a set of measurements for various sample temperatures T_i, we obtain a set of points $[T_i, h(T_i)]$. A regression analysis of this pointwise given function results in a functional relationship for $h = h(T)$.

Finally, the effective specific heat is calculated in a dependence on temperature using the definition formula

$$c_{ef}(T_i) = \left(\frac{\partial h}{\partial T}\right)_{T_i} \tag{39}$$

4. Experimental results

We have studied several groups of typical building materials:

a) Two heavy concretes, the first with siliceous aggregates ($\rho = 2300\,\mathrm{k\,gm^{-3}}$), the second with calcareous aggregates ($\rho = 2100\,\mathrm{k\,gm^{-3}}$).

b) Three lightweight concretes: boating-clay concrete ($\rho = 1400\,\mathrm{k\,gm^{-3}}$), expanded-perlite concrete ($\rho = 1600\,\mathrm{k\,gm^{-3}}$), and agloporit concrete ($\rho = 1700\,\mathrm{k\,gm^{-3}}$).

c) Four heat-insulating building materials: porous concrete ($\rho = 650\,\mathrm{k\,gm^{-3}}$, 10–15% of recycled liquid sludge), Hobrex E ($\rho = 320\,\mathrm{k\,gm^{-3}}$, mineral wool, 5% of organic substances), Porfix ($\rho = 660\,\mathrm{k\,gm^{-3}}$, expanded perlite, copolymeric bond), and Dekalit P ($\rho = 950\,\mathrm{k\,gm^{-3}}$, asbestos-cement replacement material on the cellulose basis).

The nonadiabatic method described in the previous section was employed to measure the effective specific heat of the materials above in the temperature range $T \in [0°\mathrm{C}, 1000°\mathrm{C}]$. The results are summarized in Figs. 1–4.

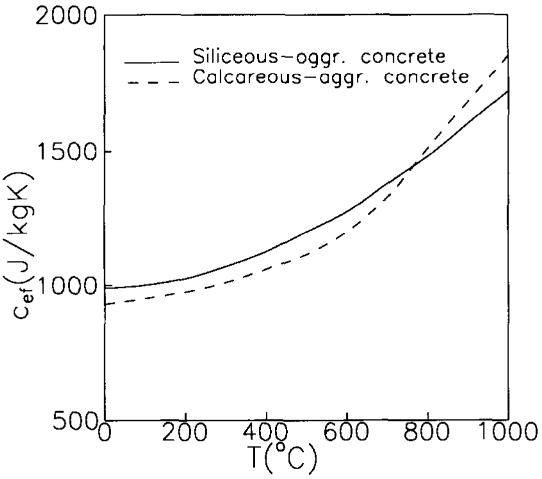

Fig. 1. Temperature dependence of the effective specific heat of heavy concretes.

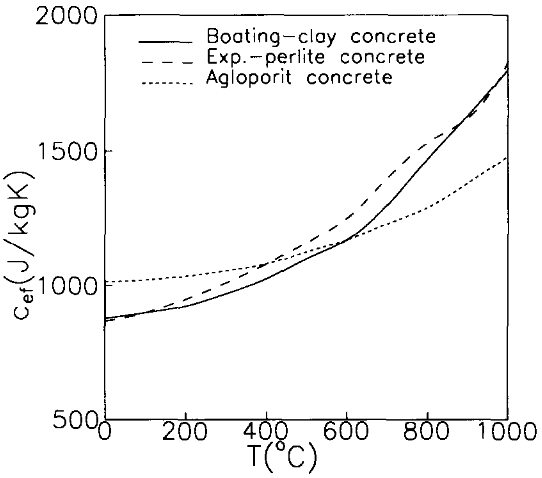

Fig. 2. Temperature dependence of the effective specific heat of selected lightweight concretes.

Fig. 1 shows the dependence of the effective specific heat of the two heavy concretes on temperature. Apparently, c_{ef} for both increases with temperature over the whole temperature range of study, as expected for compact, thermally stable materials. A similar situation is seen in Fig. 2 showing the three lightweight concretes containing only inorganic substances. Also here, $c_{ef} = c_{ef}(T)$ are increasing functions for $T \in [0°C, 1000°C]$. In addition, a comparison of Figs. 1 and 2 reveals that the $c_{ef} = c_{ef}(T)$ functions for heavy and lightweight concretes are very close to each other.

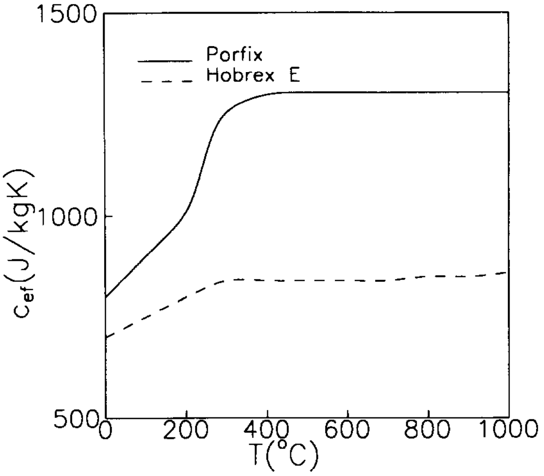

Fig. 3. Temperature dependence of the effective specific heat of selected heat-insulating building materials: Porfix and Hobrex E.

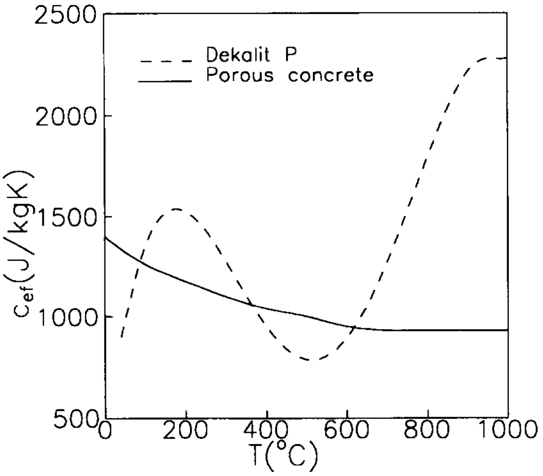

Fig. 4. Temperature dependence of the effective specific heat of selected heat-insulating building materials: porous concrete and Dekalit P.

Fig. 3 shows a different view. The heat-insulating materials Porfix and Hobrex E exhibit an increasing character of the $c_{ef} = c_{ef}(T)$ function only in the lower temperature range, up to 300°C. For higher temperatures, the increase in the specific heat stops, and c_{ef} remains almost constant up to 1000°C. The probable reasons for this are structural changes and chemical reactions of the organic substances present in the higher temperature region which can compensate the natural increase in specific heat with temperature. The lower content of organic compounds in Porfix and Hobrex E (less than 5%) makes it possible for them to avoid dramatic changes in the $c_{ef} = c_{ef}(T)$ functions.

In Fig. 4, we can see the influence of a higher content of organic substances (10–15%) on the value of the effective specific heat in a higher temperature region, using the example of porous concrete and Dekalit P. The influence of organic sludge in porous concrete is very pronounced in the whole temperature range $T \in [0°\mathrm{C}, 1000°\mathrm{C}]$. The effective specific heat of porous concrete decreases with temperature up to 600°C, then remains constant. The $c_{ef} = c_{ef}(T)$ function of Dekalit P is more "dramatic": the effective specific heat first increases up to 200°C, then decreases rapidly with a local minimum near 500°C, then increases rapidly until 900°C where it reaches a certain stabilized value. These variations of c_{ef} are an apparent consequence of the high content of cellulose in Dekalit P.

5. Discussion

The concept of an effective specific heat was introduced with the primary aim of approaching an understanding of the general processes in building materials taking place at higher temperatures. The method for measuring c_{ef} presented in this paper may be quite useful in estimating the behavior of a material, for instance, in such extreme conditions as a fire in a building structure, without knowing the details of its exact composition.

Using this method, it is possible to estimate the type and velocity of chemical reactions depending on temperature, which may be considered as a first insight in the investigation of a not well-known material. In particular, the minima in the $c_{ef} = c_{ef}(T)$ curves are of special interest because they indicate an exothermic chemical reaction, such as the burning of a component which, from the point of view of its heat-insulating properties, is the most dangerous for a building material.

However, determination of the temperature range in which the chemical reactions are important for the behavior of a specified material at higher temperatures is not very accurate with the c_{ef} concept; it can only serve as a rough first indication of the thermal stability of the material. The reason is that the influences of both chemical reactions and phase changes are only partially expressed by c_{ef}. However, when the experimental conditions are strictly maintained, we can observe indirectly the dependence of the reaction velocity on temperature, and indicate relatively well on the $c_{ef}(T)$ curve the region in which the reaction was fully completed during the time of heating the material in the furnace.

A typical analysis of a characteristic "dramatic" $c_{ef} = c_{ef}(T)$ curve can be demonstrated using the example of Dekalit P in Fig. 4. The first increase in c_{ef} can probably be ascribed to the evaporation of water, a product of an initial chemical reaction. The subsequent decrease is evidence for a culminating chemical reaction which continues after the sample is put into the calorimeter with water. Then, the increase in c_{ef} apparently indicates that the velocity of the reactions increases so that the major part of the reaction is already completed during the heating phase. Finally, the material shows a certain stabilization which may be due to the fact that most reactions are finished and we are actually measuring the effective specific heat of a "new" material, i.e., the reaction products.

The phase change processes of free water (natural moisture content) in the material cannot be detected very well by c_{ef} for materials whose pores are mostly formed by channels, because for temperatures higher than 100°C this water usually evaporates during the heating in the furnace. However, as has been shown in Ref. [9], free water evaporation can easily be detected by measuring the generalized thermal conductivity using a non-steady-state method. Therefore, the concepts of generalized thermal conductivity and effective specific heat can successfully complement each other in studying the behavior of building material at elevated temperatures.

6. Conclusion

A nonadiabatic method was employed to determine the dependence of the effective specific heat of various building materials on temperature. Several groups of these materials can be distinguished from the character of the $c_{ef} = c_{ef}(T)$ function. The effective specific heat of compact, thermally stable materials with only inorganic substances increases monotonously with temperature as in, e.g., the case for metals. The character of the $c_{ef} = c_{ef}(T)$ function of light heat-insulating materials depends primarily on the content of organic compounds which are subject to structural changes and chemical reactions in the higher temperature region. Materials with a lower content of organic substances, typically less than 5%, do not exhibit any dramatic variations of the $c_{ef} = c_{ef}(T)$ function, a typical feature here being the achievement of a constant value for $T \geq 300°\text{C}$. Heat-insulating materials with a higher content of organic substances show very irregular shapes of the $c_{ef} = c_{ef}(T)$ functions, either decreasing monotonously with temperature or having an oscillatory character. Apparently, these types of materials are not suitable for any high-temperature applications and can be used in a building construction as heat-insulating materials only in a lower temperature region up to $\sim 200°\text{C}$.

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