Thermophysical characterization of a new clay-based construction material from the Atlas region

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Abstract. As part of the valorization of local materials and the search for energy efficiency in the building sector, a thermophysical study was carried out on a new clay-based composite material from the Atlas region reinforced by peanut shells, which constitute an abundant ecological and untapped waste. In this study, a series of samples (100x100x26 mm³) of this composite are elaborated at different mass fractions of peanut shells. The thermal parameters are estimated by metrological methods such as the steady-state hot plane method and the flash method. The results showed, in addition to the lightness offered by peanut shells to these composites, that the thermal conductivity and thermal diffusivity vary respectively from values 0.623 W.m⁻¹.K⁻¹ and 4.8 m².s⁻¹ for raw clay to the values 0.323 W.m⁻¹.K⁻¹ and 3.5 m².s⁻¹ for the mass fraction of peanut shells m = 9%, which give this material the quality of the insulating walls.

1. Introduction

The building industry remains the largest consumer of energy and among the factors that can reduce this consumption is thermal insulation, therefore our contribution is to develop new building materials with improved thermal performance while giving value to local materials.

A lot of research has been carried out on local materials such as cement stabilized clay studied by H. Ezbakhe et al [1]. El Bakkouri et al [2] presented a thermomechanical study of lightweight concrete with cork or with olive pomace. Khabbazi et al [3] carried out an experimental study of the thermal and mechanical properties of a new insulating material derived from cork and cement mortar. Elhamdouni et al [4] have demonstrated the effect of alfa fibres on the thermophysical characterization of a clay extracted from northern Morocco. Mounir et al [5] have worked on the thermal inertia and thermal properties of clay-plastic composites. The same authors [6] carried out an experimental study of the thermal properties of clay and sheep's wool samples, the results showed that the addition of sheep's wool gives the clay a 45% lightness factors. Cherki et al [7] studied the thermal behavior of an ecological composite material based on gypsum plaster reinforced with cork grains, they found that this composite is thermally better than plaster without additives. Lamrani et al [8] highlighted the effect of olive pomace on an extracted clay from southern Morocco. In another work, the same authors studied the thermal performance of a new consolidated plaster-based material with peanut shells, their results showed that the thermal conductivity is reduced from 0.301 W.m⁻¹.K⁻¹ for plaster without additives to 0.141 W.m⁻¹.K⁻¹ for plaster with a mass fraction of 20% of the peanut shells, thus proving the possibility to use this new material as a false ceiling [9].

In this work we are interested in developing and characterizing a new material based on clay and peanut shells which is a renewable natural product and eco-friendly. We have highlighted the influence of the mass proportions of additives on the density and thermophysical properties of the materials produced.

Among the metrological methods used to identify thermophysical parameters, we chose the steady-state hot-plane method developed by Jannot et al [10] to estimate the thermal conductivity of the material and the Flash method to estimate thermal diffusivity [11][12]. Many authors have already
used these methods to characterize the thermophysical properties of heterogeneous composite materials, so these methods have shown each time their robustness for insulating materials.

2. Sample preparation
The materials used are:
Raw clay: it is a local natural material coming from the region of the atlas, it can constitute the binding phase of the composite
Peanut shells: it is an abundant, ecological and untapped waste. The dispersed particles of the peanut shells used have sizes between 2.5 mm and 5 mm.

![Figure 1. Used materials:](image)

The SEM image of a particle of peanut shells (figure 2) shows a microporous morphology that justifies a possibility of air containment improving the thermal insulation of the material.

![Figure 2. SEM image of a peanut shell particle (CNRST-UATRS)](image)

The samples prepared are composed of a clay matrix containing peanut shells. We found, after a few tests, that the mixing rate that allows the clay paste to be prepared with a normal consistency is

\[
\frac{q_w}{q_c} = 0.3
\]  

with \(q_w\) and \(q_c\) are respectively the quantity of water and the quantity of clay.

Samples are prepared by incorporating the peanut shells into the clay paste and then poured into molds. After 24 hours they are removed from the molds and left in the open air for 4 days. To remove moisture from the pores of each of these samples, they are dried in a vacuum drying oven at a temperature of 60 degrees. After drying they are weighed and stored in plastic bags.

The macroscopic aspect of samples with different mass contents of peanut shells is illustrated in Figure 3.

![Figure 3. Macroscopic appearance of samples prepared with the proportions 2%, 5%, 7% and 9% of peanut shells](image)
3. Theoretical approach and experimental description of measurement of thermal properties

3.1. Hot plate method in steady state:

This method makes it possible to characterize the thermal conductivity of the samples[10], its description is given in figure 4, where a heating element is sandwiched between the sample and an insulating foam, in order to maximize the flow of heat through the sample, the thermocouples are positioned to measure the temperatures $T_1$ and $T_0$ at the centers of the upper and lower faces of the sample and the temperature $T_2$ at the center of the heating element, the role of the two aluminium blocks, which has a fairly high thermal conductivity, is to reach steady state after a reasonable time.

![Figure 4. Asymmetric hot plate method in steady state: (a) Principle of the method (b) Experimental device](image)

We can write:

$$\phi = \frac{U^2}{RS} = \phi_1 + \phi_2$$

$$\phi_1 = \frac{\lambda}{e}(T_0 - T_1)$$

$$\phi_2 = \frac{\lambda_i}{e_i}(T_0 - T_2)$$

with:

- $\phi$ is the total flow emitted by the heating element by joule effect,
- $\lambda = 0.04 \text{ W.m}^{-1}.\text{K}^{-1}$ and
- $e_i = 10 \text{ mm}$ are successively the thermal conductivity and the thickness of the insulating foam.
- $e$ is the thickness of the sample,
- $R$ and $S$ are successively the electrical resistance and the surface of the heating element traversed by an electric current $I$ under the effect of a voltage $U$ imposed on its terminals.

The combination of these equations leads to the expression of the thermal conductivity of our sample:

$$\lambda = \frac{e}{(T_0 - T_1)} \left[ \frac{U^2}{RS} - \frac{\lambda_i}{e_i}(T_0 - T_2) \right]$$

3.2. Theoretical models of equivalent thermal conductivity of a two-phase composite material

Theoretical models have been developed in the literature for predicting the equivalent thermal conductivity of a composite material consisting of two phases: continuous phase and dispersed phase [13][14][15]. The expressions of the equivalent thermal conductivity for each model are given by the following relationships:

- Series model:
\[ \lambda_{series} = \frac{1}{\frac{1-y}{\lambda_{cont}} + \frac{y}{\lambda_{disp}}} \]  

(6)

- Parallel model:

\[ \lambda_{parallel} = (1 - y) \lambda_{cont} + y \lambda_{disp} \]  

(7)

- Beck model:

\[ \lambda_{Beck} = \sqrt{\lambda_{series} \cdot \lambda_{parallel}} \]  

(8)

- Woodside model:

\[ \lambda_{woodside} = \lambda_{disp}^{y} \cdot \lambda_{cont}^{(1-y)} \]  

(9)

- Maxwell model:

\[ \lambda_{Maxwell} = \frac{(1-y)\lambda_{cont}(2\lambda_{cont}+\lambda_{disp})+3y\lambda_{cont}\lambda_{disp}}{(1-y)(2\lambda_{cont}+\lambda_{disp})+3y\lambda_{cont}} \]  

(10)

with: \( y \) and \( \lambda_{disp} \) are respectively the volume content and the thermal conductivity of the dispersed phase and \( \lambda_{cont} \) is the thermal conductivity of the continuous phase.

3.3. Flash method

This method is used to estimate the thermal diffusivity of solids [11][12], its principle is described in Figure 5:

![Flash method](image)

**Figure 5.** Experimental device of the flash method

(a) Principle of the method    (b) picture of the device

A luminous pulse is sent by high power lamps (flash lamps) to the upper surface of the sample. The heat losses on both sides of the sample to its environment are represented by the same convective exchange coefficient \( h \). The lateral sides of the sample are insulated and the luminous flux absorbed by the sample is assumed to be uniform. The temperature evolution over time, at a point on the underside, is monitored by means of a thermocouple or an infrared photodiode.

The modeling of the flash method is based on the resolution of the heat equation in Laplace space. In the case of unidirectional heat transfer (1D), it is written:

\[ \frac{d^2}{dx^2} \Theta(x,p) = \frac{v}{a} \Theta(x,p) \]  

(11)
\( \theta(x,p) \) is the Laplace transform of the temperature rise \( T(x,t) - T_0 \), with \( T_0 \) is the ambient temperature.

The Laplace transform of the temperature elevation on the underside of the sample is determined using quadruples formalism [16] by the expression:

\[
\theta_1(p) = \frac{\phi_0}{p} \frac{(1 - e^{-tp})}{\lambda k \sinh(ke) + 2h \cosh(ke) + h^2 \frac{\sinh(ke)}{\lambda k}}
\]  

(12)

With:

\( \phi_0 \): The thermal flux arriving on the upper surface of the sample \((W \cdot m^{-2})\)

\( \tau \): the short time of the flash (s)

\( p \): Laplace's parameter

\( a, \lambda \) are respectively the thermal diffusivity and the thermal conductivity of the sample and \( k = \sqrt{\frac{p}{\lambda a}} \).

The numerical inversion of the expression is ensured by the algorithm of De Hoog[17] thus giving the theoretical expression of temperature as a function of time \( T_{theo}(t) \).

The Levenberg–Marquart algorithm [18][15] is used to estimate parameters reducing the quadratic error between the experimental temperature recorded at the center of the sample and theoretical expression given by the complete model

\[
\Psi = \sum_{i=0}^{N} (T_{exp}(t_i) - T_{theo}(t_i))^2
\]  

(13)

these two algorithms are programmed in MATLAB

### 4. Results and discussion

In order to take account of the measurement error, three measurement tests are carried out for each sample and the arithmetic mean of these three tests is adopted.

#### 4.1. Density of the samples

Knowing the dimensions and dry mass of the samples, we determined the average density of each sample (Table 1) with a relative error less than 0.3%.

The apparent density of the composite diminishes with increasing fraction of peanut shell which makes it possible to offer lightness to the material produced. On the other hand, the use of the law of mixtures for a two-component medium allows the volume fraction \( y \) of the peanut shells in each sample to be deduced:

\[
\rho_{sample} = y \rho_{peanut \ shells} + (1 - y) \rho_{clay}
\]  

(14)

| Sample | Apparent density \( \rho \) (Kg/m\(^3\)) | Volume fraction of peanut shell \( y \) |
|--------|--------------------------------------|-----------------------------------|
|        |                                      |                                   |

Table 1. Apparent Density of the samples produced and Volume fraction of the additive in each one
Using the steady-state asymmetric hot plane method, three measurement tests are carried out to statistically validate the results. Figure 6 and Table 2 show the variation in the average thermal conductivity of the samples as a function of the shells content.

**Table 2. Results of characterization of the average Thermal Conductivity of the prepared samples**

| Sample | Volume fraction of peanut shell \(\gamma\) | Thermal conductivity \(\lambda\) (W.m\(^{-1}\).K\(^{-1}\)) |
|--------|---------------------------------|---------------------------------|
| Raw clay        | 0                      | 0.623                           |
| A            | 0.072                      | 0.525                           |
| B            | 0.164                      | 0.435                           |
| C            | 0.213                      | 0.373                           |
| D            | 0.335                      | 0.323                           |
Figure 6. Evolution of the thermal conductivity of the material produced as a function of the percentage of peanut shells

From these results, it can be noted that:
- The measurement error does not exceed 3%, which shows the robustness of the method used
- The average thermal conductivity of the samples decreases with increasing shell content, making the processed material more and more thermally insulating.

4.3. Comparison of conductivity with theoretical models of equivalent thermal conductivity

The figure 7 shows the plot of the curves obtained by applying the theoretical models: series, parallel, Beck, Maxwell and Woodside as well as the experimental curve of thermal conductivity obtained by the hot-steady-state method.

Figure 7. Thermal conductivity of the composite developed as a function of the volume content of peanut shells compared to theoretical models

When observing the curves in the figure, we can already see that the experimental results lie between the lower limit (series model) and the upper limit (parallel model) of thermal conductivity. It is clear that the prepared samples are not represented by the maxwell model because the peanut shells are not spherical, whereas the geometric woodside model is the closest to representing these samples, reflecting the geometric mean of a random distribution of additives in the clay matrix.

4.4. Practical interest of elaborate composites

On the basis of the experimental results obtained, the following two elements can be introduced to value each composite in comparison with the raw clay:
- Lightness factor of the composite compared to the raw clay:

\[ F_{\text{Lightness}} = 100 \times (1 - \frac{\rho_{\text{composite}}}{\rho_{\text{clay}}}) \]  \hspace{1cm} (15)

- Energy saving that can be obtained using two walls of the same thickness and subjected to the same temperature gradient, one consisting of the composite and the other consisting of pure clay:
The preceding expressions are applied to evaluate the results obtained of the factor of lightness and energy saving for the different elaborated samples compared to clay only (figure 8).

\[ E_{\text{Saving}} = 100 \times \left(1 - \frac{\lambda_{\text{composite}}}{\lambda_{\text{clay}}} \right) \] (16)

Figure 8. Lightness factor and energy saving of the different materials compared to the raw clay for each mass percentage of peanut shells

We notice that when we increase the percentages of peanut shells, these two parameters increase and the energy gain approaches 50% for a mass fraction of 9%.

4.5. Thermal diffusivity

The flash method allows to identify the parameters: Thermal diffusivity \( a \), thermal flux \( \phi_0 \) and the heat exchange convection coefficient \( h \), by minimizing the quadratic error between the experimental curve and the theoretical curve (Figure 9) given by the complete model and programmed under MATLAB. The results of thermal diffusivities obtained are summarized in Table 3, the measurement uncertainty of the thermal diffusivity is about 3%. The residue, defined as the difference between the two curves, is almost flattened as shown in Figure 9, which confirms that until the date \( t = 1400 \) s the 1D model is still valid. Figure 10 illustrates the reduced sensitivity curves for the three parameters to be estimated. Analysis of these curves shows that thermal diffusivity can be reliably estimated at a time around 300 s, while the other two parameters can only be estimated at longer times.

Table 3. Results of characterization of the average thermal diffusivity of the prepared samples

| Sample  | Volume fraction of peanut shell \( y \) | Thermal diffusivity \( a.10^7 \text{m}^2\text{s}^{-1} \) |
|---------|---------------------------------------|-----------------------------------------------|
| Only clay | 0                                    | 4,80                                          |
| A       | 0,072                                 | 4,34                                          |
| B       | 0,164                                 | 3,90                                          |
| C       | 0,213                                 | 3,74                                          |
| D       | 0,335                                 | 3,23                                          |
Figure 9. Experimental and theoretical thermogram after minimization, corresponding to the sample D

Figure 10. Curves of the reduced sensitivities of the three parameters to be estimated corresponding to the sample D

Conclusion

This study underlined that the thermal insulation quality of clay, as a traditional building material, is significantly improved by the addition of peanut shells, thus offering better energy efficiency. However, this work has focused on the influence of additive content, which opens up a possibility to study the impact of the internal structure of the composite (the shape of the additives and their dispersion in the binder) on its thermal behavior. It will also be interesting to carry out an in-depth study on the optimization of the mechanical and acoustic properties involved in the choice of building materials.

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