



## Measurement of thermophysical properties of a new bio-composite material using an inverse method.

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**Abstract :** This work aims to develop a new bio-composite material to improve the thermophysical properties and the lightness of gypsum by combining it with date palm fibers improves the energy efficiency in buildings. This composite material is intended to be used in walls or false ceilings. Date palm fibers- gypsum board could be used instead of plaster board as thermal insulator in buildings. By varying the mass fraction of date palm fibers (from 0 to 20%) , an experimental investigation of the thermo physical proprieties of clay reinforced with date palm fibers was mainly performed using the flash method which is coupled to an inverse technique using the Levenberg–Marquardt algorithm

### Keywords:

DPF/gypsum composite, Thermophysical properties, Flash method, Thermal insulation.

## 1. Introduction

The building sector has a big share of the world's total energy consumption due to the increasing demand for buildings. Several attempts have been made to improve energy efficiency in the building sector. In this respect, the enhancement of building materials' thermal properties is of great importance. In fact, the gypsum plaster is used for construction, especially in walls or false ceilings. Some works have already reported on composite materials containing gypsum plaster. [1] studied the thermal properties of a gypsum-based composite material with embedded granular cork using the asymmetrical transient Hot Plate method. [2] carried out an experimental study on the thermal properties of gypsum-based composite materials with microencapsulated Phase Change Material. [3] studied the thermal characteristics of gypsum boards with PCM included. This work is dedicated to improving the experimental set-up and the estimation procedure for a better identification of the thermo physical properties of gypsum plaster reinforced with date palm fibers (DPF).

## 2. Thermophysical property measurement

### 2.1. Composite elaboration

The composite samples were obtained by mixing gypsum and water with different mass fractions of DPF (0, 5, 10, 15 and 20%) with a mean diameter of 2 mm (Fig. 1). The DPF and gypsum powder were mixed together with water at a water–gypsum ratio by mass of ( $w/g=0.6$ ). Then, we stirred continuously at a high speed for 10 min until a pasty mixture was obtained. After that, the prepared mixture was versed into cubic molds (45 x 45 x 8mm). The samples were dried at an ambient temperature for 48 hours in the molds and 28 days after demoulding.

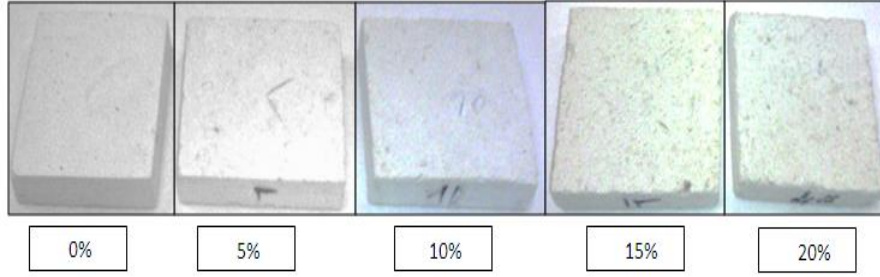


Fig 1: View of gypsum – date palm fibers for different mass fraction of date palm fibers

## 2.2. Flash method

The experimental setup is schematically shown in Fig. 2. It involves a heat source, a sample, an infrared camera and a Hewlett Packard (HP)-based data acquisition system. In this method, the front face of a parallelepiped-shaped composite sample (45 x 45 x 8mm) was subjected to a heat flux density emitted by a halogen Lampe which provides a uniform heat flux density equal to 1 KWm<sup>-2</sup> during 10 s.

The rear surface temperature rise of the sample was measured by an infrared camera. In this method, the front surface of the sample was coated with a thin, black and opaque layer, in order to increase the absorptivity of the radiative heat transfer at the front surface, so that the experimental temperature at the rear face may be easily detected by the infrared camera.

The lateral surface of the sample is insulated. A polyethylene expanded foam (PE) plays the role of a sample holder, which creates an insulating ring around the sample. Regarding that we work at ambient temperature, we assume that the heat losses from both sides of the sample same ( $h_1 = h_2 = h$ ).

The flash method was applied to determine the temperature evolution of the rear face of the sample from solving the heat equation in Laplace space using the quadrupole model. This method is considered to be applicable in a transient regime with only one direction  $x$ . The entire system can be described in Laplace space as:

$$\begin{bmatrix} \theta_f \\ \frac{\psi(1-\exp(-pt_c))}{p} \end{bmatrix} = \begin{bmatrix} 1 & 0 \\ h & 1 \end{bmatrix} \begin{bmatrix} A_1 & B_1 \\ C_1 & D_1 \end{bmatrix} \begin{bmatrix} 1 & 0 \\ h & 1 \end{bmatrix} \begin{bmatrix} \theta_r \\ 0 \end{bmatrix} = \begin{bmatrix} A & B \\ C & D \end{bmatrix} \begin{bmatrix} \theta_r \\ 0 \end{bmatrix} \quad (1)$$

Here  $\theta_f$  and  $\theta_r$  are the Laplace transforms of the front and the rear face temperatures of the sample, respectively.  $p$  is the Laplace parameter.

The coefficients  $A_i, B_i, C_i$  and  $D_i$  depend on the thermophysical properties of the material and on the thickness  $e_i$  of the layer  $i$ . They are given by the following equations:

$$\begin{cases} A_i = D_i = \cosh(\alpha_i e_i), & C_i = \lambda_i \alpha_i \sinh(\alpha_i e_i) \\ B_i = \frac{1}{\lambda_i \alpha_i} \sinh(\alpha_i e_i) & \alpha_i = \sqrt{\frac{p}{a_i}} \end{cases} \quad (2)$$

Using Eq. (1), the rear-face temperature  $\theta_r$  can be written as

$$\begin{aligned} \theta_r &= \frac{\psi}{p \cdot C} (1 - \exp(-pt_c)) \\ &= \frac{\psi}{p} \cdot \frac{1}{\lambda a \sinh(\alpha e) + 2h \cdot \cosh(\alpha e) + \frac{h^2}{\lambda a} \sinh(\alpha e)} (1 - \exp(-pt_c)) \end{aligned} \quad (3)$$

Where  $\psi$  is the density of the heat flux.

The variation of the reduced temperature  $T_c(t, \beta)$  in the usual space domain is calculated using the numerical algorithm proposed by Gaver-Stehfest as shown in the following equation [4].

$$T_c(t, \beta) = \frac{\text{Ln}(2)}{t} \sum_{i=1}^n V_i \theta_r \left( \frac{i \text{Ln}(2)}{t} \right) \quad (4)$$

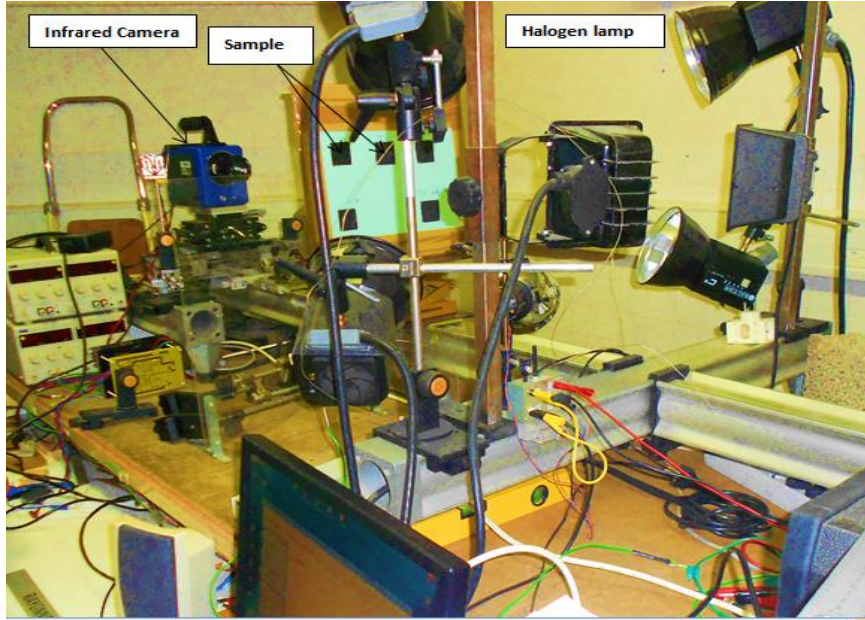


Fig 2 : Experimental device of flash method

### 2.3. Parameter identification procedure

In this study, a parameter estimation technique was applied to estimate the optimal values of both thermal conductivity and diffusivity. The identification procedure consists in finding the set of parameters  $\beta$  that minimize the quadratic error between the measured temperature  $T_m$  and the calculated temperature  $T_c(t, \beta)$ .

$$J(\beta) = \sum_{i=1}^n (T_m(t_i) - T_c(t_i, \beta))^2 \quad (5)$$

The identification of the thermo-physical properties is a nonlinear optimization problem that is solved iteratively using the Levenberg–Marquardt method given by:

$$\beta^{k+1} = \left[ (X^k)^T \cdot X^k + u_k \cdot I \right]^{-1} \cdot (X^k)^T \cdot (T_m - T_c(t, \beta)) \quad (6)$$

The variance–covariance matrix of the estimated thermal property vector  $\beta$  can be approximated as:

$$\text{cov}(\beta) = \sigma^2 (X^T X)^{-1} \quad (7)$$

Where  $X = \left( \frac{\partial T}{\partial \beta} \right)$  represents the Jacobian matrix,  $X^T$  the Jacobian transpose and  $\sigma$  represents the standard deviation of the measurements.

We assume a normal distribution for the measurement errors. The approximate statistical confidence bounds for the estimated thermal conductivity and diffusivity values  $\beta_{\lambda,a}$  are at a confidence level of 95%.

### 3. Results and discussions

#### 3.1 Density and volume fraction

The measurements of the samples' density were made using square-plate samples. We used the Mettler–Toledo™ AT61 delta range balance to measure the mass of the composite samples. A caliper square was used to measure the sample sizes. Then, the density of composite samples  $\rho_c$  is defined as mass divided by volume. Knowing, separately, the densities of gypsum, of date palm fibers and that of the mixture, we can deduce the date palm fibers volume fraction of each compound according to the classical relationship shown below:

$$\phi_v = \frac{\rho_c - \rho_g}{\rho_{\text{dpf}} - \rho_g} \quad (8)$$

with  $\rho_{\text{dpf}}$ ,  $\rho_g$  and  $\rho_c$  being successively the densities of date palm fibers, gypsum and that of the composite. The results are presented in Table 1. We made different samples by varying the mass fraction percentage of DPF in the medium. It can be noticed that reaching 20% of DPF loading, the density of the sample decreased from 1322 to 736 ( $\text{kg m}^{-3}$ ), which presents a decrease of 44.3%. The results reveal that the density decreases with increasing the volume fraction of date palm fibers.

Table 1: Density of samples as function of DPF content

Samples	$\rho_c$ ( $\text{Kgm}^{-3}$ )	$\phi_v$ %
Date palm fibers	276 [5]	-
DPF/gypsum composite		
$\phi_m$ (%)	0	0
	5	9.17
	10	27.5
	15	44.1
	20	56.2

#### 3.2. Thermal conductivity and thermal diffusivity

The Thermal conductivity ( $\lambda$ ) and the thermal diffusivity ( $a$ ) of the gypsum composite with different DPF mass fractions were measured at room temperature using the flash method. The identification procedure is based on the inverse method using the Levenberg–Marquardt algorithm which consists in minimizing the errors between the measured and the calculated temperature at the rear face of the sample. The thermal conductivities

and the thermal diffusivities of the various samples with different mass fraction of date palm fibers are given in Table 2. We notice that the addition of DPF into gypsum matrix reduces the thermal conductivity and thermal diffusivity of the composite. This reduction is expected because the date palm fibers have a thermal conductivity of 0.083 [5] which is lower than that of gypsum matrix. There by, according to Table 2, we can notice that the thermal conductivity of composites filled with 20% of DPF is 61.5% lower than the thermal conductivity of pure gypsum. A similar behavior was obtained by [6] using the same matrix. The authors investigated the impact of granular cork on the thermal conductivity of gypsum. For a 20% mass percentage of granular cork, typical values of the thermal conductivities in [6] are between  $0.12 \text{ Wm}^{-1}\text{K}^{-1}$  and  $0.19 \text{ Wm}^{-1}\text{K}^{-1}$ , while this research shows  $0.19 \text{ Wm}^{-1}\text{K}^{-1}$ . This indicates that the obtained values of the thermal conductivity are close to the cork/gypsum composite. Even with the same fraction of DPF (20%), the thermal diffusivity value decreased by 39.58%. The obtained results indicate an improvement of the thermal properties of the lightweight gypsum samples made with date palm fibers.

To prove the quality of the identification procedure, we plotted, in Fig. 3, a comparison between the measured temperature at the rear face of the sample and the calculated temperature using the newly-identified parameters. The figure shows a good agreement between the two curves. Besides, Fig. 3 shows that the residuals between the measured and the calculated temperature are random, centered on zero and do not present any deviations or oscillations.

Table 2: Results of thermal conductivity and thermal diffusivity

Samples		$\lambda (\text{Wm}^{-1}\text{K}^{-1})$	$a (\text{m}^2\text{s}^{-1}) 10^{-7}$
DPF/gypsum composite			
$\phi_m (\%)$	0	$0.454 \pm 0.008$	$3.364 \pm 0.02$
	5	$0.398 \pm 0.02$	$3.323 \pm 0.018$
	10	$0.297 \pm 0.007$	$2.965 \pm 0.023$
	15	$0.242 \pm 0.018$	$2.634 \pm 0.012$
	20	$0.196 \pm 0.067$	$2.234 \pm 0.015$

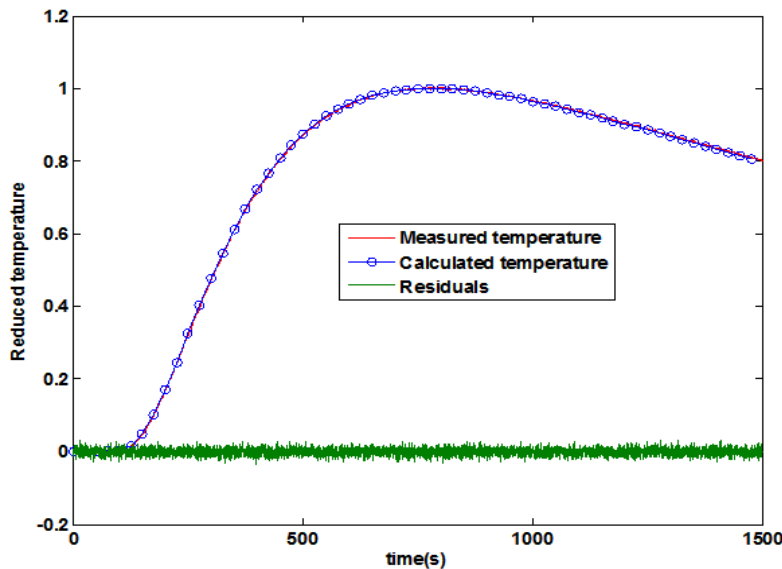


Fig 3: Comparison between measured and calculated temperatures

### 3.3. Thermal effusivity and thermal capacity

The third and fourth parameters are respectively, the thermal effusivity and heat capacity of the composite samples. They can be calculated using the values of the thermal conductivity and diffusivity as follows:

$$b = \sqrt{\lambda \cdot \rho c_p} = \frac{\lambda}{\sqrt{a}} \quad (7)$$

$$\rho C_p = \frac{\lambda}{a} \quad (8)$$

The relative uncertainties on the calculated parameters are obtained using the following expressions:

$$\frac{\Delta \rho C_p}{\rho C_p} = \frac{\Delta \lambda}{\lambda} + \frac{\Delta a}{a} \quad (9)$$

$$\frac{\Delta b}{b} = \frac{\Delta \lambda}{\lambda} + \frac{1}{2} \cdot \frac{\Delta a}{a} \quad (10)$$

Table 3 shows the values of the thermal effusivity and thermal capacity obtained by the results of the identification of thermal conductivity and thermal diffusivity. We can notice that the thermal effusivity of DPF/gypsum samples decreased from 8.93% to 50.45%, with increasing the mass fraction from 5% to 20%.

Table 3: Results of thermal effusivity and capacity

Samples	$b(\text{J m}^{-2}\text{K}^{-1}\text{s}^{-1/2})$	$\rho C_p (\text{J m}^{-3}\text{K}^{-1}) 10^6$
DPF/gypsum composite		
$\phi_m$ (%) 0	782.87±0.018	1.35±0.028
5	691.08±0.029	1.20 ±0.038
10	544.97±0.018	1.00 ±0.03
15	471.84±0.024	0.92±0.03
20	412.94±0.074	0.87±0.082

As compared with the thermal capacity of pure gypsum ( $1.35 \times 10^6 \text{J m}^{-3}\text{K}^{-1}$ ) we can see that the thermal capacity of the DPF/gypsum composite decreased from 3.14% to 36.22% with increasing the mass fraction of date palm fibers from 5% to 20%. We can clearly indicate that the inclusion of date palm fibers not only reduces the densities of the elaborated materials but also enhances the thermal properties.

#### 4. Conclusion

This paper reports the results of an experimental investigation on the thermophysical properties of DPF based gypsum composite. The thermophysical properties measurement was performed using the flash method. The goal in this paper is to evaluate the possibility of using the new composite PDF/gypsum as insulating material to reduce energy consumption in buildings.

A remarkable improvement of the thermal properties of the DPF/gypsum composite is noticed as compared to gypsum alone. Besides, the thermal conductivity and density decreased with increasing DPF concentration in gypsum matrix. Then, the obtained behaviors show that adding 20% DPF produced a composite with  $\lambda = 0.19 \text{W m}^{-1}\text{K}^{-1}$  and  $\rho = 736 \text{kg m}^{-3}$ . Accordingly, it must be kept in mind that the addition of 20% DPF in gypsum allows obtaining a composite with good thermophysical properties compared to other materials available in the literature

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