Advances in Materials
2020; 9(2): 35-41
http://www.sciencepublishinggroup.com/j/am
doi: 10.11648/j.am.20200902.13
ISSN: 2327-2503 (Print); ISSN: 2327-252X (Online)

Thermophysical Characterization of an Insulating Bio-material Based on the Macerate of "Néré" (*Parkiabiglobosa*) Pods and Cow Dung

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To cite this article:

Djossou Ayihaou Armand, Adamon Gildas David, Houngbeme Carlos, Houngan Comlan Aristide, Houehanou Ernesto, Vianou Antoine. Thermophysical Characterization of an Insulating Bio-material Based on the Macerate of "néré" (*Parkiabiglobosa*) Pods and Cow Dung. *Advances in Materials*. Vol. 9, No. 2, 2020, pp. 35-41. doi: 10.11648/j.am.20200902.13

Received: May 25, 2020; **Accepted**: June 17, 2020; **Published**: July 23, 2020

Abstract: This work concerns the thermophysical characterization of a bio-eco-material made from cow dung and the macerate of *néré* pods. To achieve this, chemical tests based on tannin concentration determination of four different solutions of néré pods (60 $g.l^{-1}$; 120 $g.l^{-1}$; 180 $g.l^{-1}$ and 240 $g.l^{-1}$) were prepared at 100°C, then brought to the boil for 5 minutes. After three different maceration times (6 h; 24 h and 48 h), the analysis of the solutions obtained using a spectrometer made it possible to select the solutions of 120 $g.l^{-1}$ and 180 $g.l^{-1}$ which offer best tannin concentrations in 24 hours, necessary for making test pieces. Thermal tests based on thermal effusivity and thermal conductivity measurements were then carried out in transient mode, with hot strip method. In order to compare the thermal performance of developed eco-material with that of ordinary insulators, the thermophysical properties of plywood and plaster were also measured. The results obtained showed that for the two dosages of 120 $g.l^{-1}$ and 180 $g.l^{-1}$, the thermal effusivity of eco-insulator varies from 247.732 $J.K^{-1}.m^{-2}.s^{-0,5}$ to 270.732 $J.K^{-1}.m^{-2}.s^{-0,5}$ respectively and the thermal conductivity from 0.082 $W.m^{-1}.K^{-1}$ to 0.080 $W.m^{-1}.K^{-1}$. For the same dosages, the thermal diffusivity varies from $1.106.10^{-7}$ $m^2.s^{-1}$ to $0.881.10^{-7}$ $m^2.s^{-1}$ respectively. A comparative study has shown that the eco-material developed and tested offers better insulating power due to its relatively weak thermophysical properties compared to ordinary insulating materials, namely plaster and plywood.

Keywords: *Néré* Pods, Tannin, Cow Dung, Thermal Effusivity, Thermal Conductivity

1. Introduction

The present study relates to the development and thermal characterization of a bio eco-insulating material based on the néré pods juice and cow dung, with a view to improving thermal comfort and energy saving in habitat through the eco-materials promotion. This approach integrates both an ecological and socio-cultural aspect by taking into account local raw materials made of vegetable fibers in eco-materials development.

When added to plaster, cow dung improves the thermal and mechanical characteristics of material [1]. Mixed with certain local materials such as sand, earth, straw and branches, etc. cow dung serves as a binder to give a solid character and help seal the material. With its biopolymer properties, cow dung acts as an organic adjuvant of animal or vegetable origin when added to the earth material to improve its properties and the durability of the earth structure [2, 3]. The use of cow dung in plaster mortars is widespread in several African countries [4, 5]. In Benin, it is used especially in the north in the Atacora and Donga regions, but also in the south in lakeside villages. The work of Vissac and al in 2012, Peter and al in 2013, Thej Kumar et al in 2015, provide more information on the incorporation of sawdust and ash from cow dung in mortars and concretes [5-7].

Néré pods contain tannin which plays an important role in eco-insulating materials development. Tannins are dispersing agents for fine particles and modify the mixtures plasticity. They are also capable of forming chemical bonds with active sites, which in particular increases the compressive strength of materials thanks to its properties as a natural adjuvant. Sorgbo in 2013 and then in 2016 showed that néré pods or their decoction mixed with clay induce plastic behavior which promotes good adhesion during the formulation of concrete [8, 9]. Keita's work in 2014 showed that the incorporation of néré pods in the clay + sand mixture makes it possible to increase the material mechanical resistance [10]. Similar results were obtained by Banakinao in 2017 on earth-based bricks, boiled skins and banana leaves as well as néré tannins [11].

It is deduced from the results of these various studies that the cow dung and pods combination or with other materials is of great interest for the building sector.

2. Characterized Materials

The developed and characterized eco-insulation consists essentially of plant materials: cow dung and néré pods macerate.

2.1. Néré Pods

Néré pods (*Parkiabiglogosa*) used in this study come from néré fruits harvested in north Benin more precisely in Parakou city during april 2018 month. The fruits are cleared to recover the pods. These pods are then dried in sun until dry before being reduced to small pieces as shown in Figure 1.

Figure 1. *Néré pods cut into small pieces after drying.*

2.1.1. Tannin Extraction

The tannin extraction consists in preparing four different néré pods solutions (60 $g.l^{-1}$; 120 $g.l^{-1}$; 180 $g.l^{-1}$ and 240 $g.l^{-1}$) at 100°C temperature, which is brought to cooking for a 5 minutes period. The tannin concentration of each solution was finally determined at three different maceration times (6 h, 24 h and 48 h). The procedure for obtaining the macerate of néré pods is as follows:

1. 500 ml of distilled water are brought to boil in a container;
2. we introduce the néré pods mass corresponding to each dosage (respectively 30 g, 60 g, 90 g, 120 g), weighed using a precision balance;
3. mixture obtained is then brought to cooking for a 5 min period, then poured into a closed bottle and kept for 6 h, 24 h and 48 h (Figure 2).

Figure 2. *Tannin extraction from néré pods.*

2.1.2. Tannin Concentration Determination

Tannin concentration determination was made as follows [12]: to 01 ml of vanillin (mixture at equal volume of 8% hydrochloric acid at 37% in methanol and 4% of vanillin ($m.v^{-1}$) in methanol), 200 µL of néré pods macerate are added. The mixture is then maintained at 30°C for 15 min. Depending on the maceration time, the absorbance (optical density) at 500 nm was finally determined using a spectrophotometer of Spectrumlab 752S type.

2.2. Cow Dung

Cow dung was collected in wet state after defecation in a cow house. It was then dried in sun and then in an oven at 60°C for 24 hours. It was finally reduced to powder using an appropriate mill (Figure 3).

Figure 3. *Cow dung powder.*

2.3. Test Pieces Making

The test tubes of each concentration (120 $g.l^{-1}$ and 180 $g.l^{-1}$ of néré pod) were made from a mixture of 55 g of cow dung and 82 g of macerate. The mixture is then introduced and compacted in a mold with two inlets, of parallelepiped geometry and (5 x 3.5 x 3) cm^3 dimensions, making it possible to obtain two test pieces by casting (Figure 4). After demolding, all test pieces were finally stored in sun before being placed in oven at 50°C temperature, for 48 h period until constant mass for each sample.

Other plaster and plywood test pieces, 2 cm and 3 cm thick, were also made as shown in Figure 4.

Figure 4. *Molding and demolding of three characterized insulating materials.*

2.4. Densities and Water Contents of Test Pieces

The wet and dry densities of samples were determined using the mold method, in accordance with NFP94-053 (1991) standard. Its principle is based on determination of volume, wet and dry masses of samples and using (1). The water contents were finally determined in accordance with NFP94-050 (1991) standart using (2). Its principle is based on samples steaming, at a 50°C temperature for 24 hours period, necessary to obtain a constant mass of each sample.

$$\rho_i = \frac{m_i}{V_i} \qquad (1)$$

with i = wet/dry

$$w = \frac{m_h - m_s}{m_s} \qquad (2)$$

with m_h = wet mass and, m_s = dry or anhydrous mass

3. Thermophysical Characterization Method

The thermal tests were carried out with hot strip method. It makes it possible to measure in transient mode, thermal effusivity with hot plane principle and thermal conductivity with hot wire principle. The choice of this method is justified among other things, by the simplicity of the experimental device implementation, the results precision and the fairly short experimentation time (less than 180 s) [13, 14].

3.1. Hot strip Principle

The hot strip method uses a rectangular and flexible electrical resistance, at center of which is placed a thermocouple made of thin wires. The heating element is inserted between plane surfaces of two identical material samples (Figure 5). A 6.3 V voltage is applied to the terminals of heating element. Temperature is measured at center of resistance, which avoids having to take into account the heat losses by electric wires at one end of resistance [13, 14]. Samples dimensions are such that the disturbance caused by the flow level imposed on the probe does not reach any of their external faces during the measurement duration (assumption of the semi infinite medium). The resistance ratio length/width is chosen so that heat transfer at its center may be considered as bidirectional during a minimal time 180 s [13, 14]. The smallest dimension of samples must be greater than 1.5 times the total tape width.

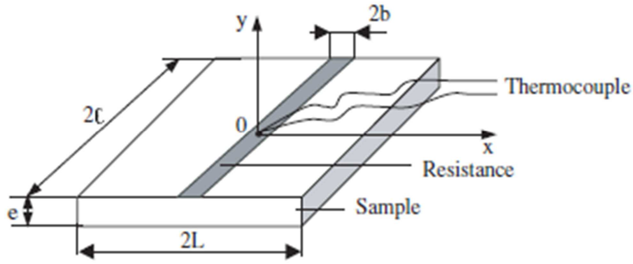

Figure 5. *2D hot strip model.*

3.2. Hot strip Modeling

A 2D hot strip model is presented in Figure 5. The temperature increase T_s (x, y, t) at coordinates point (x, y) of hot strip checks (3) during the time t_2 when heat transfer at this point remains bidirectional (infinite hot strip):

$$\frac{\partial^2 T_s(x,y,t)}{\partial x^2} + \frac{\partial^2 T_s(x,y,t)}{\partial y^2} = \frac{1}{a}\frac{\partial T_s(x,y,t)}{\partial t} \qquad (3)$$

with the following boundary conditions:

$$\text{at } y = 0:\ -\lambda S \frac{\partial T_s(x,y,t)}{\partial y} = -\varphi_0, \text{ if } x < b \qquad (4)$$

$$\text{and } -\lambda S \frac{\partial T_s(x,y,t)}{\partial y} = 0, \text{ if } x > \qquad (5)$$

$$\text{at } x = 0:\ -\lambda S \frac{\partial T_s(x,y,t)}{\partial x} = 0, \text{ by symmetry} \qquad (6)$$

$$\text{at } x = L:\ T_s(L, y, t) = 0, \text{ semi-infinite medium hypothesis in (ox) dirction} \qquad (7)$$

$$\text{at } y = e:\ T_s(x, e, t) = 0, \text{ semi-infinite medium hypothesis (oy) direction} \qquad (8)$$

Where b is half-width of hot strip (m), e is the thickness of the sample (m) and L is the half-width of the sample (m).

The problem solution is given by equation 9 using successively Laplace transform, cosine finite Fourier transform between x = 0 and x = L, quadripole formalism, inverse Fourier transform, then inverse Laplace transforma by Stefhest method [13, 14].

$$T_s(0,0,t) - T_s(0,0,0) = \frac{\ln(2)}{t}\sum_{j=1}^{n_L} V_j \theta_s\left(0,0,\frac{j\ln(2)}{t}\right) \qquad (9)$$

3.3. Parameter Estimation

Figure 6 shows the hot strip experimental device used. The different properties are determined by comparing the experimental thermogram of hot strip with the theoretical thermograms of hot plane and hot wire (Figure 7). We note that hot strip behaves like hot plane at short times (between 0 and 50 s) and like hot wire at long times (between 80 s and 180 s) [13, 14]; which makes it possible to determine the thermal effusivity at short times and the thermal conductivity at long times with hot plane principle and that of hot wire respectively.

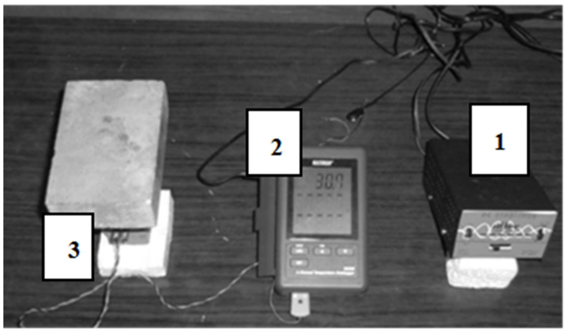

Figure 6. *Hot strip experimental device used.*

(1-stabilized power supply; 2-central acquisition unit; 3-resistance inserted between two material samples)

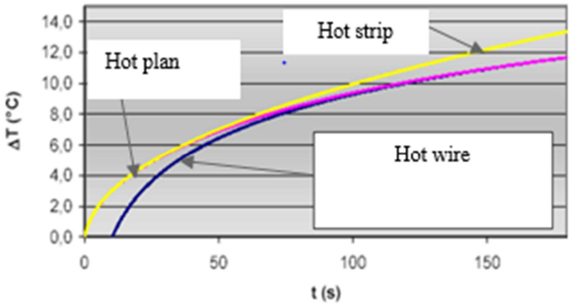

Figure 7. *Comparison of hot strip, hot plane and hot wire models.*

3.3.1. Thermal Effusivity Estimation

The thermogram corresponding to the start of heating between 0 s and 50 s (time interval t_1 when the heat transfer at the resistance center remains unidirectional), is used to estimate the thermal effusivity using hot plane type model with (10) [13, 14].

$$T_s(0,0,t) - T_s(0,0,0) = \frac{2\varphi_0}{ES\sqrt{\pi}}\sqrt{t} + \varphi_0\left[R_c - \frac{(mc)_s}{(ES)^2}\right] \quad (10)$$

Thermal effusivity is obtained by the direct coefficient $\frac{2\varphi_0}{ES\sqrt{\pi}}$ of linear regression $T_s(0,0,t) - T_s(0,0,0) = f(\sqrt{t})$ of experimental thermogram between t_0 and t_1 where the heat transfer is unidirectional.

3.3.2. Thermal Conductivity Estimation

A complete modelization of bidirectional transfer in the samples associated with hot wire model makes it possible to use the entire thermogram between 0 s and 180 s to estimate the thermal conductivity using (11) [13, 14].

$$T_s(0,0,t) - T_s(0,0,0) = \frac{\varphi_0}{4\pi\lambda 2l}\ln(t) + \varphi_0\left[R_c - \frac{\ln\left(\frac{r_0}{\sqrt{a}}\right)}{2\pi\lambda 2l} + \frac{\gamma}{4\pi\lambda 2l}\right] \quad (11)$$

The thermal conductivity is obtained by the direct coefficient $\frac{\varphi_0}{4\pi\lambda 2l}$ of linear regression $T_s(0,0,t) - T_s(0,0,0) = f(\ln(t))$ of experimental thermogram during the time t_2 when heat transfer at the resistance center remains bidirectional.

4. Results and Discussions

4.1. Hot Strip Validation

Thermal effusivity and conductivity of plexiglas, considered as reference material, were measured using hot strip method. Three measurements were performed on two identical samples of plexiglas in same experimental conditions. The mean values obtained are close to literature values (Table 1). These results justify the validity of the experimental measurements and theoretical models developed.

Table 1. *Thermophysical properties of Plexiglas as reference material [15].*

Method	Test N°	$E(J.m^{-2}.K^{-1}.s^{-0.5})$	$\lambda(w.m^{-1}.K^{-1})$
	1	561.068	0.186
	2	562.508	0.181
	3	561.222	0.183
	Mean values	561.600 ± 0.646	0.183 ± 0.002
Literature **[15]**		540.243 ≤ E ≤ 573.323	0.184

4.2. Tannin Content

Figure 8 shows the evolution of the tannin content according to the dosage of néré pods for the different maceration times. Analysis of this figure shows that the best maceration time for néré pods is 24 h with a maximum tannin concentration from 120 $g.l^{-1}$ of néré pods. Beyond 24 h, fermentation was observed in the maceration jars, which could justify the drop in the tannin content observed. This remark was also made by Djimasngar in 2017 [16]. As for maceration time of 6 h, we note that the tannin content remains low for the same dosage compared to 24 h duration. Following these observations, the 120 $g.l^{-1}$ and 180 $g.l^{-1}$ dosages after 24 h maceration period, were used for samples manufacture.

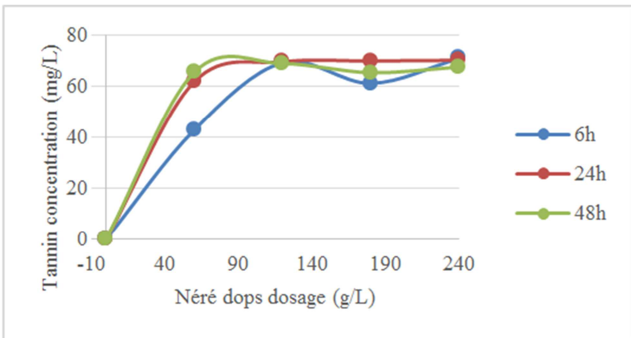

Figure 8. *Tannin content evolution according to néré pods dosage and maceration duration.*

4.3. Samples Density and Humidity

Figures 9 and 10 show respectively test pieces densities and water contents. From figure 9 analysis, it can be noted that from 7^{th} to 28^{th} day, the pieces density decreases from 550 $kg.m^{-3}$ to 390 $kg.m^{-3}$ with concentration of 120 $kg.m^{-3}$ of néré pods and, from 550 $kg.m^{-3}$ to 410 $kg.m^{-3}$ with concentration of

180 g.l^{-1} of néré pods. On the 28th day, the test tubes of 120 g.l^{-1} of néré pods are less dense than those of 180 g.l^{-1} of néré pods whereas they have same density on the 7th day.

From figure 10 analysis, it can be seen that the test pieces made with a concentration of 120 g.l^{-1} of néré pods have a lower moisture content than those of 180 g.l^{-1} of néré pods.

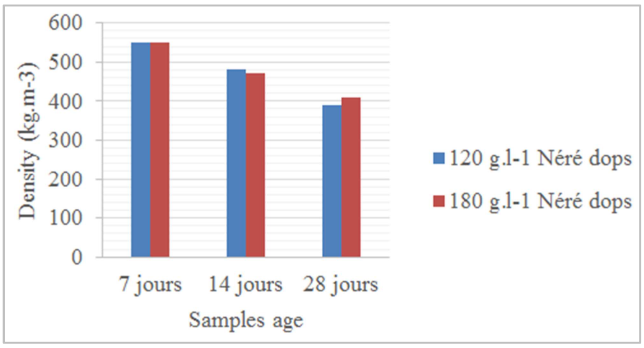

Figure 9. *Samples density as a function of age and dosage of néré pods.*

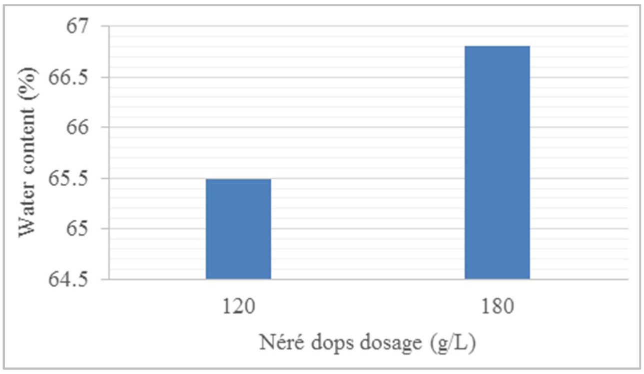

Figure 10. *Samples water content as a function of néré dosage.*

4.4. Thermophysical Properties

Figures 11, 12 and 13 respectively show the thermal conductivity, effusivity and diffusivity of different insulating materials characterized in this study.

The results reported in figures 11 and 12 show that the thermal conductivity and effusivity of material based on cow dung and cowpea juice respectively vary from 0.082 W.m^{-1}.K^{-1} to 0.080 W.m^{-1}.K^{-1} and from 247.732 J.K^{-1}.m^{-2}.s$^{-0,5}$ to 270.732 J.K^{-1}.m^{-2}.s$^{-0,5}$, when néré pods dosage varies from 120 g.l^{-1} and of 180 g.l^{-1}.

Figure 11 shows that thermal conductivity of plywood decreases from 0.133 W.m^{-1}.K^{-1} to 0.126 W.m^{-1}.K^{-1}, when its thickness varies from 2 cm to 3 cm. That of characterized plaster is 0.282 W.m^{-1}.K^{-1}.

In figure 12, it is noted that for respectively same thicknesses, plywood thermal effusivity decreases from 320.594 J.K^{-1}.m^{-2}.s$^{-0,5}$ to 299.457 J.K^{-1}.m^{-2}.s$^{-0,5}$; while that of plaster is 736.867 J.K^{-1}.m^{-2}.s$^{-0,5}$.

Figure 13 shows that the thermal diffusivity of materials based on cow dung and néré pods husk juice decreases from 1.106.10^{-7} m^2.s^{-1} to 0.881.10^{-7} m^2.s^{-1} respectively for néré pods dosage ranging from 120 g.l^{-1} to 180 g.l^{-1}; while that of plywood increases from 1.707.10^{-7} m^2.s^{-1} to 1.763.10^{-7} m^2.s^{-1} for a thickness varying from 2 cm to 3 cm. Plaster thermal diffusivity is 1.463.10^{-7} m^2.s^{-1}.

From results obtained analysis, we note that eco-material based on cow dung and néré pods has the weakest thermophysical characteristics (thermal conductivity, effusivity and diffusivity) compared to plywood and plaster. These weak characteristics show that the eco-material based on cow dung and néré pods is a good insulator, less effusive and less diffusive compared to plywood and plaster characterized. Due to its thermal conductivity, it will better resist heat transfer by conduction. Its relatively low thermal effusivity values show that the material will see its surface temperature increase rapidly, thus absorbing little heat.

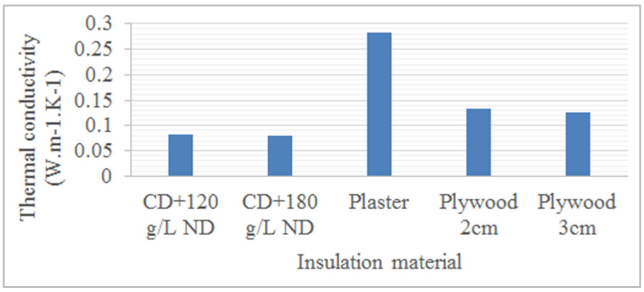

Figure 11. *Eco-insulation based on cow dung + néré pods macerate; plaster and plywood thermal conductivity; (CD = cow dung; ND = néré dops).*

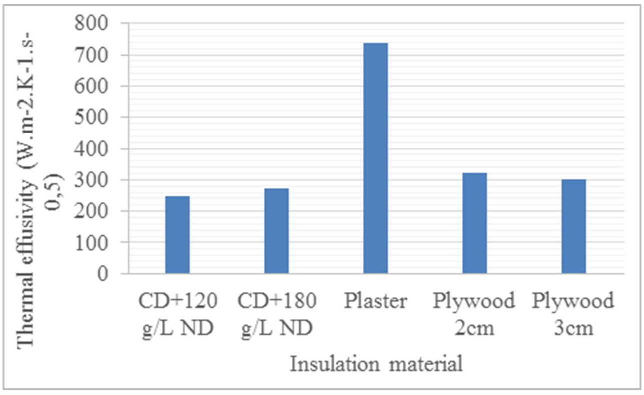

Figure 12. *Eco-insulation based on cow dung + néré pods macerate; plaster and plywood thermal effusivity; (CD = cow dung; ND = néré dops).*

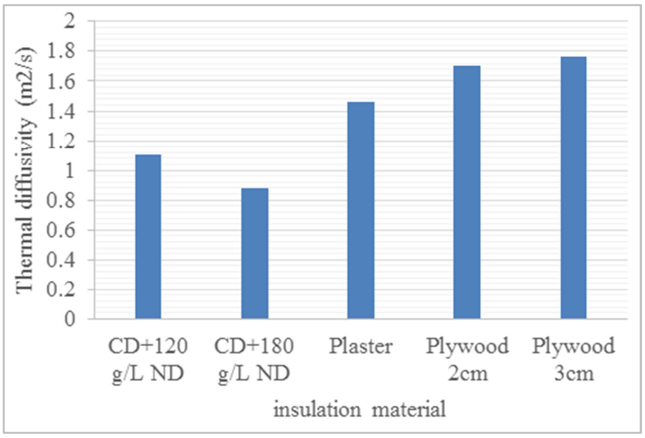

Figure 13. *Eco-insulation based on cow dung + néré pods macerate; plaster and plywood thermal diffusivity; (CD = cow dung; ND = néré dops).*

4.5. Comparative Analysis

Table 2 presents a comparative study of results obtained with those of literature. It is noted that eco-insulating developed and characterized has better thermal performance compared to insulating based on vegetable fibers encountered in literature. Thermophysical properties of all insulating materials characterized with hot strip method used agree well with standardized thermophysical properties of insulating provided by literature [17]. This observation testifies to the quality of experimental device and reliability of results obtained.

Table 2. *Comparative study of results obtained and those of literature [17].*

Materials	Thermal properties	Our study	Literature [17]
Fiberboard NF EN 316 120 g.l^{-1} - 180 g.l^{-1} of néré pods	Dry density (kg.m^{-3})	397.7 - 391.20	350 - 550
	Thermal conductivity (W.m^{-1}.K^{-1})	0.082 - 0.08	0.1 - 0.14
	Thermal effusivity (J.m^{-2}.K^{-1}.s$^{-0,5}$)	247.732 - 270.732	243.926 - 361.801
	Thermal diffusivity (.10^{-7} m^{2}.s^{-1})	1.106 - 0.881	1.681 - 1.497
Plywood NF EN 313-1; NF EN 313-2; EN 12775 Thickness: 2 cm - 3 cm	Dry density (kg.m^{-3})	453.5 - 497.2	450 - 500
	Thermal conductivity (W.m^{-1}.K^{-1})	0.133 - 0.126	0.13 - 0.15
	Thermal effusivity (J.m^{-2}.K^{-1}.s$^{-0,5}$)	320.594 - 299.457	305.94 - 346.41
	Thermal diffusivity (.10^{-7} m^{2}.s^{-1})	1.707 - 1.763	1.805 - 1.875
Plaster	Dry density (kg.m^{-3})	1018.8	700 - 900
	Thermal conductivity (W.m^{-1}.K^{-1})	0.282	0.25
	Thermal effusivity (J.m^{-2}.K^{-1}.s$^{-0,5}$)	736.867	433.013 - 474.341
	Thermal diffusivity (.10^{-7} m^{2}.s^{-1})	1.463	3.33 - 2.477

5. Conclusion

It should be noted from this study that eco-material based on cow dung and néré pods offers greater insulating power than plywood and plaster. The relatively weak thermophysical characteristics obtained are a very important criterion in assessment of wall thermal performance. These parameters give indications for insulating materials judicious choice with a view to minimizing heat input into building. With results obtained, materials characterized in this work will be able to improve building energy efficiency. The development of material from cow dung fairly known in rural areas for its thermal properties and vegetable fibers of nere pods as matrix is a concept that fits perfectly with the challenges to be met in terms of energy efficiency and thermal comfort in habitat sector. From an ecological point of view, material based on cow dung and néré pods can effectively contribute to limiting greenhouse gas emissions and environment protecting compared to characterized plywood and plaster.

Nomenclature

a: Thermal diffusivity, m^2.s^{-1}
E: Thermal effusivity, J.K^{-1}.m^{-2}.s$^{-0,5}$
l: Length, m
m: Mass, kg
R: Electrical Resistance, Ω
S: Surface, m^2
T: Temperature, K
t: Time, s
w: Water content, %
x, y, z: Space variables, m

Greek Letters

ρ: Density, kg.m^{-3}
λ: Thermal conductivity, W.m^{-1}.K^{-1}
θ_s: Laplace transform of probe temperature
φ: Heat flux, W

Indices / Exhibitors

s: Related to probe

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