THERMAL CONDUCTIVITY ENHANCEMENT OF LiNO3/GRAPHITE COMPOSITE FOR ENERGY STORAGE

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ABSTRACT

This paper deals with the analysis of a LiNo3/ graphite composite for thermal storage at high temperature. In such a composite, the salt (LiNo3) serves as a latent heat storage material and the graphite has been used to enhance the thermal conductivity of the salts. The elaboration method consists of a cold uni-axial compression of a physical mixing of salts powder and graphite particles.

The thermal conductivity of the LiNo3/ graphite composites with different mass fraction of graphite was measured using transient hot wire technique. Also, the influence of the moisture content on the measurement was studied.

1. INTRODUCTION

The last 20 years were characterized by an essential increase of energy consumption, according to the increasing of the demand for air conditioning and large demands of electric power. The limited reserves of fossil fuels and the increase of the use of energy are the main driving forces behind efforts to search a new and renewable energy. In this scene, Thermal energy storage is a key element for effective thermal management in the sector process heat and power generation, it is indispensable for solar thermal application.

Thermal energy storage at high temperature (>120 °C) is an efficient way for energy saving in industrial processes. It is also an important method for electricity generation based on new conversion techniques and renewable energy [1]. Three major techniques are currently considered for thermal storage: sensible heat storage [2], thermo-chemical heat storage [3] and the latent heat storage.

Among these different methods, the latent thermal energy storage (LTES) method using phase change material (PCM) is the most preferred forms of thermal energy storage, which can provide high storage density and nearly isothermal heat storage/retrieval process [4, 5]. Different phase transition for the charge/discharge process can be considered. In practice, solid-liquid phase change is preferred because of simultaneous weak volume variation and important enthalpy variation [6].

A large number of phase change materials are available for latent heat storage in any required temperature range. Several authors classify the PCM materials into three major categories [7-9]: organic compounds, inorganic compounds and eutectics PCMs. Organic PCMs include paraffin, esters, fatty acids and alcohols. Whereas inorganic PCMs are comprised of salt hydrates, salts and metals. Eutectics are specific alloys of inorganics and/or organics PCMs. Each of these groups has its typical range of melting temperature and melting enthalpy.

The increasing industrial interest in finding heat storage materials for efficient use of thermal energy in corresponding applications simulates the investigation of phase change materials. Salts have been evaluated as suitable thermal storage at high temperature (>120°C). In some applications the thermal energy must be absorbed or released at a very fast rate. However, most salts have sufficient capacity for energy storage, but it has low thermal conductivity, so these drawbacks reduce the rate of heat storage and extraction during the melting and solidification cycles. To increase PCMs thermal conductivity, different methods have been proposed and investigated in the past. The methods include dispersing high conductivity particles within the PCM: metal particles [10] or carbon fibers [11, 12], the methods include impregnated PCM into high thermal conductivity material with porous structure such a carbon or metal foam matrix [13, 14], and micro-encapsulation of the PCM [15].

However, metal fillers add significant weight and cost to the storage systems. In addition, corrosion could appear when using salts. Some other authors have recently proposed conductivity enhancement technique based on graphite additives [16, 17], impregnating a porous graphite matrix with PCM [18-20].

The use of graphite particles has advantages such as low density in contrast to metals and high resistance to corrosion and their thermal conductivity is considerably high.

This paper aimed to elaborate low cost materials with improved thermo-physical properties for energy storage at high temperature. New LiNo3/ graphite composites are prepared by cold uni-axial compression method. In such a composite, the salt (LiNo3) serves as a latent heat material and graphite serve to enhance thermal conductivity of the composite. Different mass fractions of graphite varying from 5% to 20% are used. Then we investigate the effect of Graphite waste addition and the moisture on thermal conductivity of the composites using a transient hot wire technique.

2.THE HOT-WIRE METHOD FOR THE MEASUREMENT OF THERMAL CONDUCTIVITY

The method proposed for the measurement of the thermal conductivity of composites is the transient hot wire (THW) method which is a transient technique based on the measurement of the temperature rise of a linear heat source (hot wire) embedded between two tested materials (Fig.1). This method, originally proposed by Carslaw and Jaeger [21], is based on the idealized one-dimensional radial heat flow model. As an electric current of fixed intensity flows through the wire, the thermal conductivity can be derived from the resulting temperature change over a known time interval.

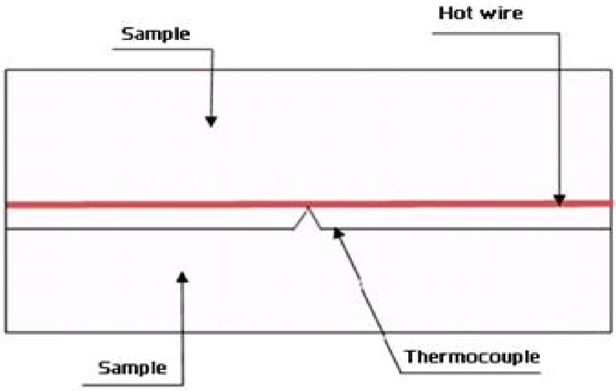

Fig.1: the physical principle of the hot wire

2.1. Theoretical formulation of the THW method

The ideal mathematical model is based on the assumption that the hot wire is an infinitely thin and long line heat source (continuous line source). It produces a thermal pulse for a finite time with constant heating power and generates cylindrical coaxial isotherms in an infinite homogeneous medium initially at equilibrium. The transient temperature, for sufficiently long time from the start of the heat generation can be expressed by the following expression [21]:

$$T(r,t)=\frac{Q}{4\pi\lambda}\left[\ln(\frac{4\alpha.t}{r^2})+\frac{r^2}{4\alpha t}-\frac{1}{4}(\frac{r^2}{4\alpha t})-...-\gamma\right] \quad (1)$$

Where:

- λ is the thermal conductivity (W/m K);
- Q (W/m) is the power supply per unit length of the heating line source;
- α is the thermal diffusivity (m^2/s) of the sample;
- r is the radial position where the temperature is measured;
- $\gamma=0.5772156$ is Euler's constant.

For extended range of time $\left(\frac{r^2}{4.\alpha.t}\right)\prec\prec t$, the second and the third terms of equation (1) can be neglected, thus the temperature can be expressed with good approximation by the simplified form:

$$T(r,t)=\frac{Q}{4\pi\lambda}\left[\ln(\frac{4\alpha.t}{r^2})-\gamma\right] \quad (2)$$

That can also be written as:

$$T(r,t)=\frac{Q}{4\pi\lambda}\left[\ln(t)+\ln(\frac{4\alpha}{r^2})-\gamma\right] \quad (3)$$

Applying a constant electric current throughout the wire, and recording continuously the temperature increase (ΔT) at the measuring point located in the middle of the wire. The thermal conductivity of the sample is calculated according to the following Eq (4):

$$\Delta T=\frac{Q}{4\pi\lambda}(\ln t+A) \quad (4)$$

Where Q is the heat generation rate per unit length of the wire, t the time and A a constant.
If the temperatures are measured at times t_1 and t_2 within the validity rang of time $\left(\frac{r^2}{4.\alpha.t}\right)\prec\prec t$,

The temperature rise of the wire is given by:

$$\Delta T=T(t_2)-T(t_1)=\frac{Q}{4\pi\lambda}(\ln\frac{t_2}{t_1}) \quad (5)$$

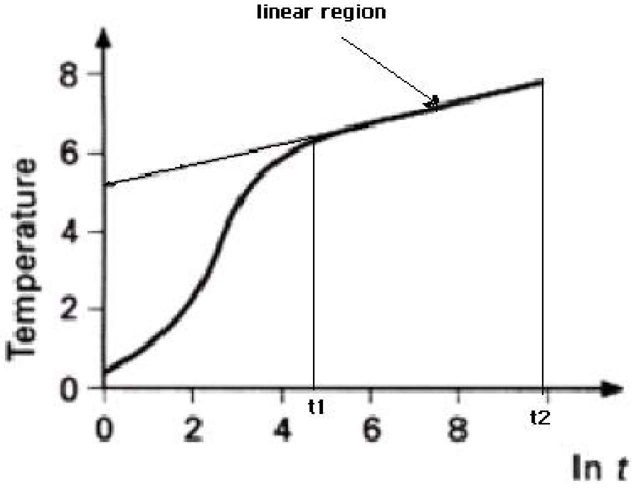

Fig.2: Temperature rise versus the logarithm of the time.

In practice, the thermal conductivity of the sample is calculated using the slope of the linear portion of the relation between ΔT and ln t from Equation (6):

$$\lambda = \frac{U.I}{4\pi.l\left[T(t_2)-T(t_1)\right]}.\ln(\frac{t_2}{t_1}) \qquad (6)$$

2.2 Measurement setup

The experimental apparatus is schematically shown in Fig.3. It is constituted:

- A stabilized power supply,
- A hot wire,
- A thermocouple,
- A PC ,
- A data acquisition unit (Agilent 34970 A) which allows reading and recording the measured temperatures then transferring data to a computer via an RS-232 interface,
- Two composites samples of the same material and of the same dimensions.

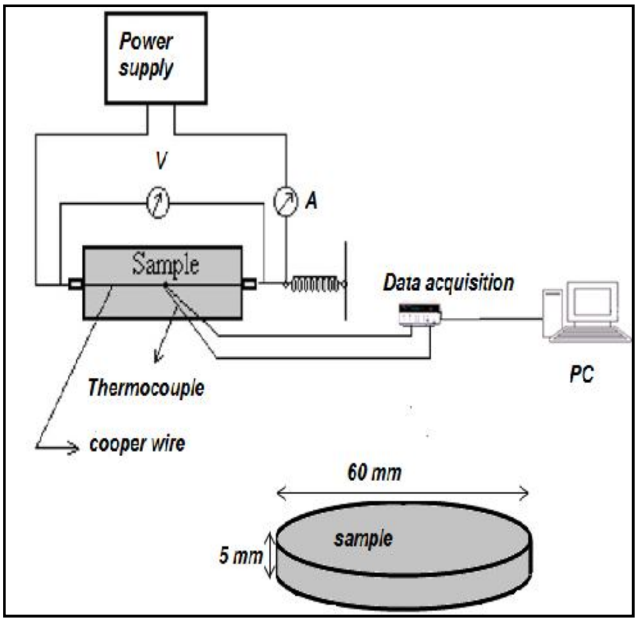

Fig. 3: schematic of the experimental device of hot wire method

The copper wire was 61mm in length and 0.1mm in diameter (the high ratio between length and diameter, R=610, guaranties the validity of the hot wire method utilized to measure the thermal conductivity of solid materials [22]. The wire was sandwiched between the two composite samples having cared to locate the thermocouple junction at the core of the sample. Particular attention was put to ensure an efficient contact among salt/graphite composite and the cooper wire.

After the connection of the thermocouple to the measurement device, the wire is connected to a constant stabilized power supply, which serves to give out a constant voltage of about 0.2 V, was applied for about 2 min which transfer through the sample. And the temperature increasing caused by the current circulating along the cooper wire was registered with a data acquisition unit (Agilent 34970 A) which allows transferring data to a computer via an RS-232 interface.

This procedure was repeated several times in order to decrease the error that could be done on the basis of a single measurement. The increasing of the temperature was reported as a function of natural logarithm of time and the slope of the straight line obtained (see Fig.2) represent the parameter used to calculate the thermal conductivity. In fact according to Eq.(6) the thermal conductivity can be calculated knowing the slope of the linear regression curve temperature vs. time in which time is defined as natural logarithm.

3. SAMPLE PREPARATION

3.1 Raw materials (PCM and Graphite)

Numerous PCMs especially salts are available for the high temperature energy storage purposes, mainly in application concerning electricity generation by solar concentration technologies. In this study the PCM Lithium nitrate (LiNO3) is selected for this experimental investigation (Fig. 4).

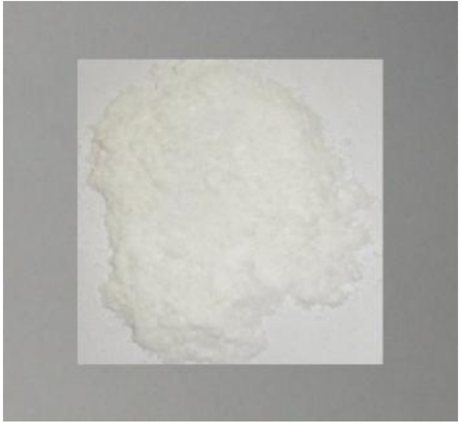

Fig 4. Lithium nitrate (LiNO3) powder

The LiNo3 PCM has many advantages such a : commercial availability at low cost, a little or no supercooling, non-flammable, nontoxic, no phase segregation and chemical stability providing demanded life time of the heat storage system.

Property	salt (LiNo3)
Density, g/cm3	2380
Thermal conductivity (W/m.K),	0.51
Melting temperature, °C	253 [23]
Latent heat, J/g	373 [23]

Table 1: Thermo-physical properties of the salt (LiNo3)

In the present work, the graphite used for the thermal conductivity enhancement of salt, is obtained from damaged Tubular graphite block Heat Exchangers (Fig. 5a and Fig. 5b).

Figure 5: (a) Tubular graphite Heat Exchangers, (b) graphite waste powder.

This type of graphite is a form of carbon with crystalline structure; it has good thermal and mass transfer characteristics.

Property	Graphite
Density, g/cm3	1936
Radial thermal conductivity (W/m.K)	5
Axial thermal conductivity (W/m.K)	160

Table 2: Thermo-physical properties of graphite waste.

3.2 Salt/graphite composite elaboration

The elaboration route that we used in this work is the **cold uni-axial compression**. This method is based on physical mixing of salts ($LiNo_3$) powders and graphite waste particles. Then the obtained mixture ($LiNo_3$+ graphite) can be poured into a highly alloyed steel mold and then compacted in a mechanical press (see Fig .6) with a predetermined pressure (50bar) at ambient temperature.

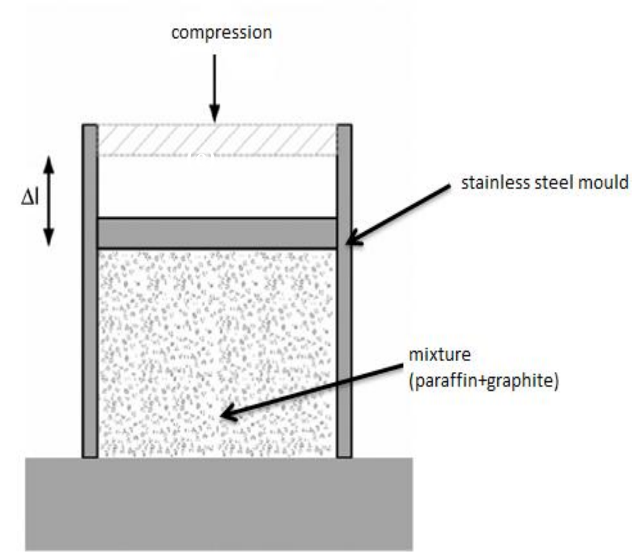

Figure 6 : Cold uni-axial compression technique

This technique leads to cohesive matrix made of graphite whose porosity is partially occupied by salts grains. Cylindrical and anisotropic structure composites containing 5%, 10%, 15% and 20% in weight of graphite were prepared by the cold uni-axial compression method.

The thickness and the diameter of all this specimens were 5mm and 60mm respectively. For an accurate measurement of thermal conductivity a very smooth surface of composites samples is necessary to allow an efficient contact with the hot wire located between salt/graphite samples.

Fig.7: Sample: salt (LiNo3),15%graphite

This technique is characterized by a lower energy cost, because the PCM does not require to be melted before use, and can be applied to PCM with variable size [24].

Then, composites samples are placed in an oven at approximately 60°C and weigh at 2 hours. Samples are dry when the weight is constant for two consecutive reading. Once a constant weight value is obtained, the samples are dried and ready for analysis.

4. RESULTS AND DISCUSSION

4.1 Density and porosity measurement:

The determination of the composites density and the porosity is important for checking the quality of the samples.

The theoretical density of the prepared salt/graphite composite was calculated according to the rule of mixture. It is clear that if the composite samples are well processed i.e. good homogeneity is reached without air bubbles in the sample; the specific density of the composites should have a linear dependence upon the volume fraction of graphite.
The apparent density can be calculated using density values of salt ($LiNo_3$) (ρ_{LiNo_3} = 2380 $kg.m^{-3}$) and graphite (ρ_{gw} = 1936 $kg.m^{-3}$) and according to the following classical rule of mixtures:

$$\rho_{app} = \rho_{gw}\,\phi_{gw} + \rho_{LiNo_3}\,\phi_{LiNo_3} \qquad (7)$$

Where ϕ_{gw} And ϕ_{LiNo_3} are the mass fractions of graphite and $LiNO_3$ respectively.

During the preparation, it was ineluctable to involve a few air bubbles in the blocks of the composite PCMs, which decreased the density of the composite PCMs.
Then, for the porosity measurement, we consider a material sample of volume V and mass m including three phases: salt(s), graphite (g) and air (a). Let Vx and mx (with x= s, g, a) be, respectively, the volume occupied by each one of the phases and their corresponding mass:

$$V = V_s + V_g + V_a \qquad (8)$$

The porosity of the composite sample can be approximated from the following expression:

$$\varepsilon = \frac{V_a}{V} = \frac{V_{real} - V_{theory}}{V_{real}} \qquad (9)$$

The real volumes were achieved using a cylindrical sample, the sample sizes were measured using a caliper squar. So, the value of the real volumes of the composites can be obtained using the following expression:

$$V_{real} = \pi . r^2 . e \qquad (10)$$

The densities and porosities of graphite/salt composites elaborated by uni-axial cold-compression are summarized in Table 3:

samples	Masse (10^{-3}.kg)	V_{air} $.10^{-6}m^3$	V_{real} $.10^{-5}$	Apparent density ρ_{app}(kg.m-3)	porosity
Salt (LiNo3)	33.89	5,782	1,9919	2380	29.03%
5%Gr+95% salt	33.07	5,785	1,9922	2357	29.04%
10%Gr+90% salt	30.46	4,239	1,8376	2335.6	23.07%
15%Gr+85% salt	32.99	5,751	1,9889	2313.4	28.92%
20%Gr+80%salt	29.81	5,690	1,9827	2291.2	28.7%

Table 3: physical properties of PCMs composites.

We remark that the total porosity is about 23-29% and the apparent density range from 2.291 kg/m^3 to 2.38 kg/m^3.

4.2 Thermal conductivity measurement

The thermal conductivity of salts/graphite composite was measured at ambient temperature by using the transient hot wire apparatus. Measurements were performed at constant voltage value of about 0.3 V with an electric power of 0.27 W, which is adequate for measurements at low thermal gradient as required by Carslaws and Jaegers theory [21].
Before to proceed with the thermal conductivity measurement of PCM samples, the measurement accuracy of the system was evaluated using a plexiglass standard sample. According to the literature, the plexiglass thermal conductivity should vary in the range of 0.20–0.244 W/ m.K. The measurement was preformed at ambient temperature and values of 0.2–0.21 W/mK were obtained which are in good agreement with the literature.
In all measurements, the temperature rise of the copper wire was about 0.5-2.5 K and the measurement time was 10-20 second after the initiation of heating by adjusting the current through the wire. Measurements on each sample were repeated more than three times in order to test repeatability of the results.
The thermal conductivity of PCM composites and their uncertainties are summarized in Table 4.

Samples	λ_{eff} (W:m.K)	$\frac{\lambda_{composite} - \lambda_{salt}}{\lambda_{salt}}$
Salt (LiNo3)	0.51±0.02	0
95%(Salt)+5%(graphite)	0.870±0.036	70%
90%(Salt)+10%(graphite)	1.190±0.052	133%
85%(Salt)+15%(graphite)	1.520±0.068	198%
80%(Salt)+20%(graphite)	1.860±0.086	264%

Table 4: measured thermal conductivity of PCM composites

The thermal conductivity of salt (LiNo3)/graphite composite PCMs experimentally measured, indicating that thermal conductivity of composites PCMs is greatly influenced by the graphite waste addition and increased with increasing mass fraction of graphite.

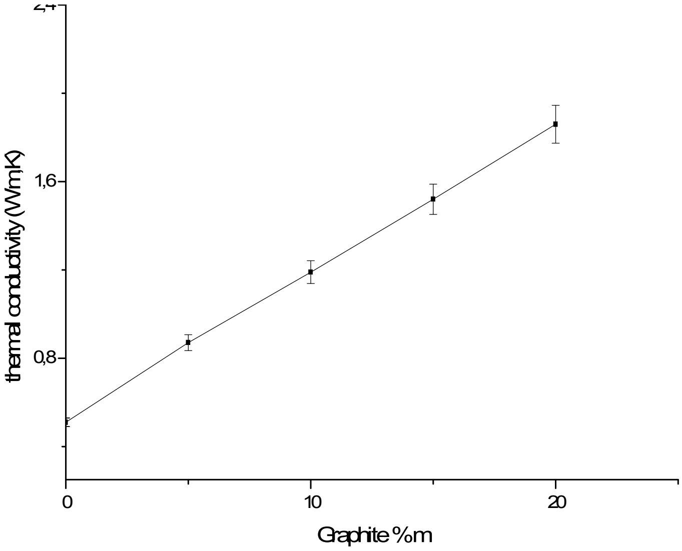

Fig. 8: Thermal conductivities of salt/EG composite PCMs with varying mass fraction of Graphite.

The figure 8 displays the relationship between the thermal conductivity of salt (LiNo3)/graphite composite and the graphite mass fraction. A linear increase of λ_c with increasing graphite masse fraction was observed. This increase of λ_c is foreseeable, because the graphite has a significantly higher thermal conductivity than the salt ($LiNo_3$). And a relationship between the thermal conductivity and mass fraction of EG can be easily drawn out from (Fig. 8) as follows:

$$y = 0.067x + 0.5193 \qquad (11)$$

The thermal conductivity intensification I_λ obtained by the graphite addition was calculated through Eq (12):

$$I_\lambda = \frac{\lambda_{composite} - \lambda_{PCM}}{\lambda_{PCM}} \qquad (12)$$

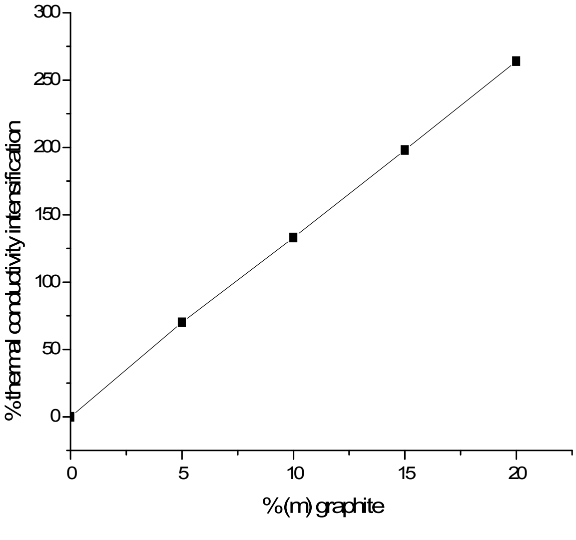

Fig. 9: thermal conductivity intensification vs % (m) graphite

Figure 9 displays the relationship between the thermal conductivity intensification and mass fraction of paraffin/graphite waste composites. It can be observed that the thermal conductivity intensification increases linearly with increase of %graphite waste. Thermal conductivities of the composite PCMs with mass fraction of 5%, 10%, 15%, and 20% graphite indicated that the thermal conductivity of salt(LiNo3) (0.51 W/m K) increased as 70%, 133.6%, 198.1%, and 264.5%, respectively. This was attributed to high thermal conductivity of the Graphite.

4. 3 Influence of moisture content on the thermal conductivity

The porous system of the PCMs composite on natural base enables the transport and the accumulation of moisture.

The value of the thermal conductivity coefficient of the PCMs composite is closely connected with the moisture content. The air in porous systems of this type of materials is substituted during humidification by water which has multiple higher values of thermal conductivity and this means a significant increase of the thermal conductivity coefficient.

In order to study the influence of moisture content on thermal conductivity of $LiNo_3$/graphite composites, we let all the samples at ambient humidity for 24 hours. The moisture content on the wet composites refers to the quantity of water per unit weight or volume of the wet material.

The water content can be calculated using the following Equation:

$$\theta = \frac{m_w}{m_{composites}} = \frac{m_{wet} - m_{dry}}{m_{dry}} \qquad (13)$$

Where: m_w and $m_{composites}$ are the weight of water and composites respectively.

The values of weight of the dry and the wet composites and the water content are summarized in Table (5).

samples	Mass dry (g)	mass (g) (after 24 hours)	Water content
Salt	33.33	34.7	4.20%
5%gr+95%salt	33.07	34.13	3.20%
10%gr+90%salt	31.828	32.375	2.80%
15%gr+85%salt	32.87	33.71	2.55%
20% gr+80%salt	29.838	30.59	2.52%

Table 5: comparison between the weight of wet and dry composites

We can note from this table, that the mass of all composite increased after 24 hours

Then, the thermal conductivity of all wet composites samples was measured using the previously mentioned method. The results obtained are depicted in Fig. 10.

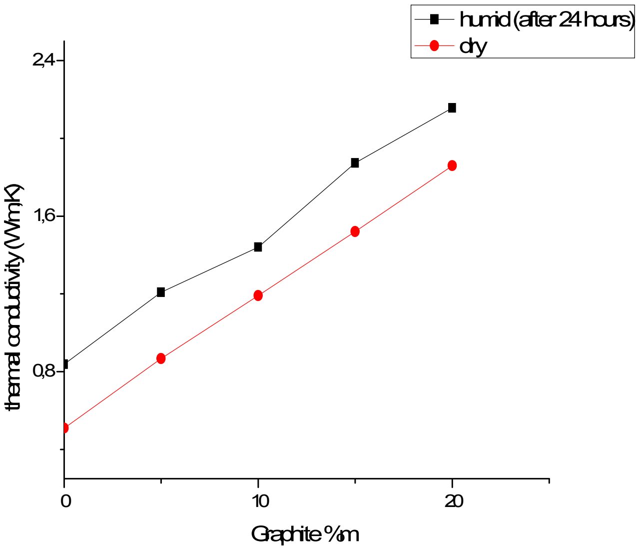

Fig10. Influence of moisture on the thermal conductivity of composites

Fig. 10 presents a comparison between thermal conductivity of dry $LiNo_3$/graphite composites and the values of humid samples. We note, That the thermal conductivity coefficient grows with the increasing moisture content.

It seems reasonable to assume that the influence of a moisture content on the coefficient of thermal conductivity results from the fact that in some of the pores the air, which is a good insulator, is replaced by water which is not as good insulator.

5. CONCLUSION

Salt is one of the most preferred heat storage materials at high temperature, however, the low thermal conductivity limits its utilization.

In this study, graphite waste was used as an effective heat transfer promoter. New Salt (LiNo3)/graphite waste composite PCMs with mass fraction of graphite waste varying from 5 to 20% were elaborated and characterized.

The LiNo3/graphite composites were prepared by using a cold uni-axial compression technique and its effective thermal conductivity were measured by the hot wire method.

Researching results shows that the effective thermal conductivity of salt/graphite composites PCMs is greatly influenced by the graphite waste addition and it increased with increasing mass fraction of graphite.

We can conclude that the utilization of the salt/graphite waste composite PCMs improves significantly the heat storage/retrieval rate of the Latent thermal energy storage.

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Nomenclature :

Q	Power supply
r	Radius (m), Radial position
t	Time (s)
m	measured mass (kg)
l	Wire length (m)
U	Voltage (V)
I	thermal conductivity intensification, current intensity (A)
a	thermal diffusivity (m^2s^{-1})
d	Density (Kg/m^3)
V	volume of composite (m^3)
e	thickness
T	Temperature (°C)

Greek symbols

λ	thermal conductivity ($W.m^{-1}K^{-1}$)
ϕ	graphite mass fraction
ρ	density (Kg/m^3)
ε	Porosity
θ	Water content

Subscripts

eff	effective (composite)
s	salt
g	graphite
w	water
a	air