

Research Paper

Experimental evaluation of porosity, axial and radial thermal conductivity, of an adsorbent material composed by mixture of activated carbon, expanded graphite and lithium chloride

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HIGHLIGHTS

- Porosity, axial-radial thermal conductivity of an adsorbent mixture are obtained.
- Particle size, compaction pressure and composition are the experimental factors.
- Axial thermal conductivity is 10 times greater than radial one, regardless of factors.
- Maximum values for axial and radial thermal conductivity are 76.5 and 13.8 W/m K.

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ABSTRACT

In this study, an adsorbent material made up of a mixture of activated carbon, expanded graphite and lithium chloride, is proposed to evaluate its thermal properties. Ratio effect between mixing components, component particle size, and compaction pressure on porosity and thermal conductivity of composite material was experimentally evaluated. Axial and radial thermal conductivity were evaluated by ASTM C177-13 standard using the hot plate and hot wire method for the respective axial and radial conductivities. Experimental results indicate that the highest porosity reaches 0.78 and is produced with a 70% mixing ratio of activated carbon mass, 10% of LiCl mass, and 20% of expanded graphite mass. With the levels used in experimental design, axial and radial thermal conductivity obtain maximum values of 51.2 W/m K and 11.9 W/m K, respectively. After optimization process based on design of experiments for mixtures, axial and radial conductivity reach their highest values of 76.5 W/m K and 13.8 W/m K, respectively, when mixture is elaborated with a proportion of 30% activated carbon, 40% expanded graphite and 30% Lithium Chloride. This study shows that conductivity results do not vary significantly due to tests temperature.

1. Introduction

Currently, compression refrigeration systems are used in multiple processes from food preservation to design of environments with comfort conditions for human comfort [1–3]. These systems demand a large amount of energy and use refrigerant fluids that can result in negative environmental effects. For this reason, in the past 40 years researches have been studying environmentally friendly cold generation systems; such as sorption cooling systems [4,5]. Typically, these systems use natural refrigerants as water, methanol and ammonia, that having a zero-ozone depletion potential (ODP) and zero-global

warming potential index (GWP), meet the requirements from the Montreal and Kyoto Protocol [6–8]. That is why several investigations have been conducted considering adsorbent-adsorbate pairs allowing to work with low-grade heat. The analyzed adsorbent materials are generally blocks made up from materials that improve physical properties, as porosity and thermal conductivity in the adsorbent bed.

Graphite matrices can be considered as a new heat conductor in fixed-bed reactors for cooling systems by chemical adsorption. The key advantage of graphite structures is that its thermophysical properties are more favorable compared with metal foams made from aluminum, copper or nickel [4].

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Alternatives such as those proposed by Zheng et al. [5] have been studied to improve the physical characteristics of adsorbent materials, where influence of expanded graphite (EG) composite blocks has been analyzed. Authors evaluated surface area and porosity of the composite material by porosimetry and compared results with silica gel blocks. Kiplagat et al. [6] proposed the impregnation of lithium chloride with expanded graphite in mass proportions not exceeding 23%. As a result, the cooling capacity increased by 20%. Oliveira et al. [7] studied a prototype on a laboratory scale with an adsorbent compound (NaBr-expanded graphite). In his work, he obtained 219 kJ/kg of cooling power at 5 °C and 510 kJ/kg of cooling power at 15 °C, with a 65 °C heat source and 30 °C of condensing temperature. With the same heat source and evaporator conditions, the system achieved cooling power between 75 and 79 kW/m³, with a COP ranging from 0.43 to 0.46 when the cooling temperature was set at 15 °C. Mitra et al. [8] studied the influence of the adsorbent particle size on the dynamic adsorption characteristics using a bidimensional unsteady CFD model. taking methanol activated carbon as par. The model showed that smaller particles do not have effect on the adsorption dynamics. Yu et al. [9] analyzed a composite material with a porous activated carbon matrix where surface area and pore size were studied with a gas adsorption analyzer ASAP-2020 from Micromeritics, obtaining a 0.767 cm³/g total pore volume. Demir et al. [10] studied the effect caused by three different porosity values (0.1, 0.2, 0.3) of a silica gel block on the temperature distribution and adsorbate concentration. They observed that the adsorption period increases when the porosity value is higher, affecting pressure distributions and adsorption rate at the beginning of the process for a considerably brief time.

Seeking to improve the conductivity of materials used in energy storage applications and adsorption systems, expanded graphite was used by Fayazmanesh et al. [11] in an experimental study to evaluate the effect of graphite flakes addition on the thermal conductivity of a CaCl₂-silica gel composite adsorbent. Results showed an increase in thermal conductivity from 0.13 W/m K to 0.57 W/m K. Askalany et al. [12] studied the effect of metallic additives on thermal conductivity of a granular activated carbon. Different mass concentration of iron, copper and aluminum were considered. A theoretical model was developed in order to analyze the behavior of the composite in a cooling cycle. Authors concluded that aluminum addition present the highest effect on raising the thermal conductivity and specific cooling power. Jiang et al. [13] evaluated experimentally a consolidated compound of CaCl₂ with the matrix of expanded natural graphite treated with sulfuric acid (ENG-TSA). The developed composite presented 400 and 22 times higher thermal conductivities compared with natural matrix of expanded graphite and CaCl₂ respectively. Wang et al. [14] measured parallel and perpendicular thermal conductivity of a consolidated expanded natural graphite treated with sulphuric acid (ENG-TSA). It was obtained a strong anisotropic behavior of the compound, which perpendicular thermal conductivity was 50 times higher than the measured in parallel direction of compression. Zheng et al. [5] proposed novel desiccant material elaborated from silica gel with expanded natural graphite treated with sulfuric acid (ENG-TSA). Different silica gel mixture ratios and densities were considered. It was obtained an improvement of 270 times higher thermal conductivity compared with pure silica gel. Tian et al. [15] studied an adsorbent compound elaborated with CaCl₂ and activated carbon using expanded natural graphite composite as host matrix. Thermal conductivity of the composite reached 1.08 W/m K, five times higher compared with granular CaCl₂. Wu et al. [16] developed a novel method to synthesize an expanded graphite (EG)/stearic acid composite with phase change material. Results indicate that EG enhance significantly the thermal conductivity of the compound up to 23.27 W/m K. Xiao et al. [17] evaluated the heat transfer in EG/nitrate compounds. The developed composites presented about 7 times higher thermal conductivity compared with pure sodium nitrate. Cheng et al. [18] developed a Lightweight wall material composed with EG/paraffin composite. Experimental results indicated

Table 1

Thermal conductivity ranges for lithium chloride, activated carbon and expanded graphite.

Mixture component	Thermal conductivity (W/m K)
Lithium Chloride (LiCl)	0.2–0.8 [21]
Activated Carbon (AC)	0.15–0.5 [22]
Expanded Graphite (EG)	50–150 [23]

Table 2

Experimental factors.

Mixture proportion	Low	High	Mixture preparation conditions	Low	High
LiCl Mass (%)	10	30	Pressure (MPa)	8	12
AC Mass (%)	30	70	Mesh (No)	200	80
EG Mass (%)	20	60			

Table 3

Response variables.

Variable	Measurement method	Units
Thermal Conductivity	ASTM C177-13 [24]	W/m K
Porosity	Kiplagat et al. [6]	–
Apparent Density	Kiplagat et al. [6]	kg/m ³

that EG improves the thermal conductivity of the composite without chemical interaction with paraffin. Yang et al. [19] evaluated experimentally a polyethylene glycol/EG composite. It was found that EG improve thermal conductivity of the mixture with remarkable adsorbability, thermal stability and storage capacity. Yuan et al. [20] reported an experimental analysis of an adsorption/desorption composite of strontium chloride (SrCl₂) impregnated into expanded graphite. It was found that the developed composite is suitable for cooling application. Results above indicate that expanded graphite is a good complement to increase thermal conductivity and porosity of adsorbent materials. Nonetheless, this material presents high energy production costs. Therefore, it is necessary to develop materials with better porosity and thermal conductivity at lower cost, hence, analyzing activated carbon as an alternative material to EG is proposed in this study.

Despite the improvements in the properties of composite materials, previous research papers in this matter have been focused on material impregnation with fixed percentages of their components, without considering mixture design and particle sizes of the composite adsorbent. The objective of this study is to analyze the effect of these factors on an adsorbent material made up of activated carbon (AC), expanded graphite, and lithium chloride (LiCl). It is aimed to determine the proportions of mixture favoring the conductivity and porosity of the composite material considering the factors involved in the adsorbent block preparation, (particle size and compaction pressure). For this, preparation of 10 samples was proposed. The preparation factors' influence on physical properties of the new material (porosity, apparent density and thermal conductivity) was evaluated to estimate optimal proportions in mass, particle size and compaction pressure.

2. Experimental procedure

Experimental tests were carried out to study the effect of proportion among mixture components (LiCl, AC, EG), mesh size used in the preparation of mixture and compaction pressure used on the porosity, radial conductivity and axial conductivity of the composite adsorbent material. Thermal conductivity ranges of these individual materials are shown in the Table 1. Design factors and its experimental levels are shown in Table 2, while Table 3 shows the response variables and their measurement methods associated.

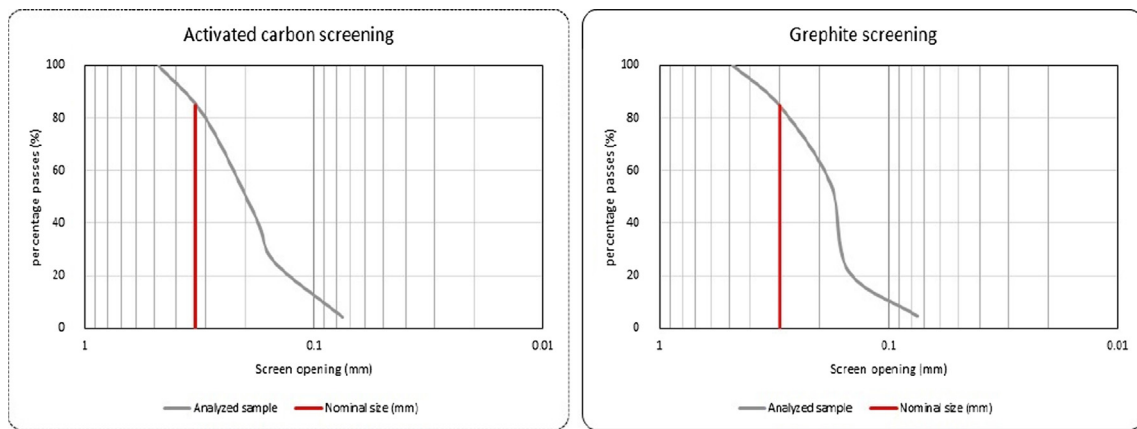


Fig. 1. Granulometric analysis by sieving: activated carbon (left), Graphite (right).



Fig. 2. Block of composite adsorbent material.

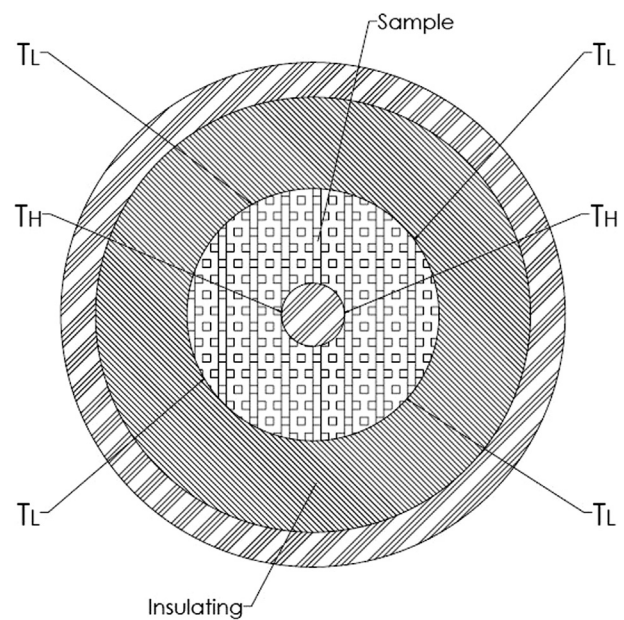


Fig. 4. Hot wire test scheme.

2.1. Mixture preparation

2.1.1. Sieving process

Prior to the sieving process, the characterization of the nominal particle size of activated carbon and graphite was carried by a granulometric analysis according to ASTM C-136 standard [25], these results are presented in Fig. 1. The analysis determined that the nominal size

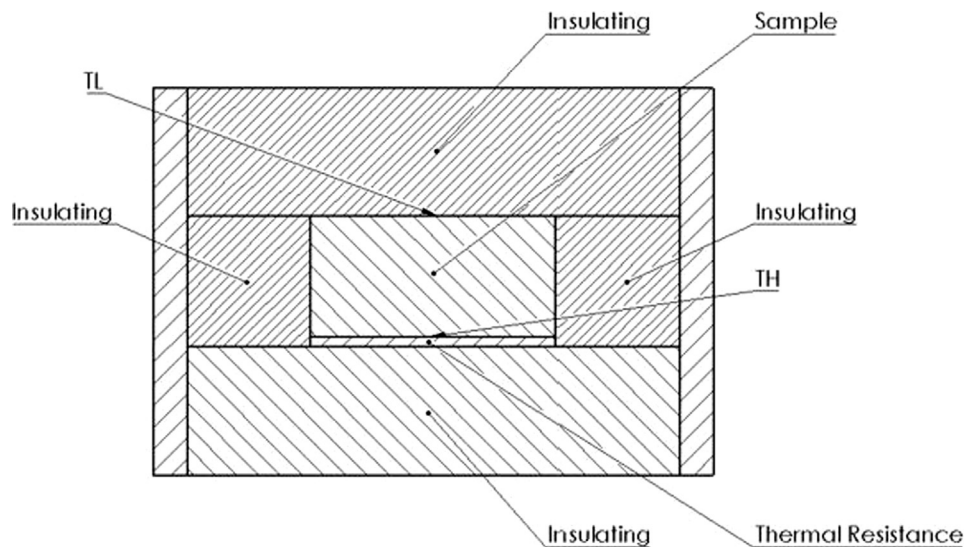


Fig. 3. Hot plate test diagram.

Table 4
Results of calibration tests.

T_{Amb} K	T_{In} K	T_{Out} K	ΔT K	Q W	λ_M (W/m k)
293	353	350	3.0	20	66.1
294	353	350	3.1	20	63.9
294	349	350	3.3	20	60.1
293	349	345	3.2	20	61.9
292	303	302	1.0	5	49.6
292	303	302	0.9	5	55.1
293	307	306	0.9	5	55.1
293	307	306	0.8	5	61.9

Table 5
t-student test for the experimental thermal conductivity.

Statistical	Experimental thermal conductivity	Thermal conductivity literature [33]
Half	59.24	60.50
Variance	30.44	0.00
Observations	8.00	8.00
Grouped variance	15.22	
Hypothetical difference of the means	00.0	
Degrees of freedom	14.00	
Statistic t	-0.65	
P (T <= t) a tail	0.26	
Critical value of t (one tail)	1.76	
P (T <= t) two tails	0.53	
Critical value of t (two tails)	2.14	

for each material was 0.28 mm and 0.3 mm for graphite and activated carbon, respectively. From these results, 80 (0.177 mm) and 200 (0.074 mm) mesh sizes were selected because they showed the highest percentages of population in the granulometric analysis. It is observed that the particle size distribution of the analyzed materials is homogeneous and it is guaranteed that the particle sizes selected are uniform in the experimental range.

2.1.2. Graphite expansion and mixing

In order to ensure a correct graphite expansion, the samples were heated in a muffle furnace at 800 °C during two minutes, this procedure was suggested by Oliveria et al. [26] and it has been widely employed in scientific specialized studies related to compound materials with graphite [15–18,27,28]. After the graphite expansion for the particle sizes, five samples were prepared for sieve No 80 and sieve No 200 varying the percentages by mass of the mixture components (LiCl, GE, CA) and compaction pressure.

Samples mixing was done according to the procedure used by Jiang et al. [13], Yu et al. [29] and El-Sharkawy et al. [30]. The procedure to obtain each sample is described as follows: Firstly, the lithium chloride

Table 6
Experimental results: axial conductivity, radial conductivity and porosity.

Block Sample	m_{LiCl} (%)	m_{AC} (%)	m_{EG} (%)	Mesh (No.)	Pressure (MPa)	λ_a (W/m K)	λ_r (W/m K)	Porosity ϕ	ρ_{Block} (kg/m ³)
E1	10	30	60	200	12	41.9	8.44	0.71	1122.5
E2	10	30	60	80	12	51.1	6.96	0.69	1218.2
E3	10	70	20	80	8	25.6	5.67	0.73	1046.6
E4	10	70	20	80	12	28.7	2.84	0.75	986.75
E5	10	70	20	200	8	32.6	9.45	0.73	1076.2
E6	10	30	60	200	12	21.4	4.50	0.75	987.68
E7	10	70	20	80	12	16.3	4.07	0.78	865.81
E8	30	50	20	200	8	29.5	4.39	0.72	1174.7
E9	10	30	60	200	8	27.6	11.9	0.73	1061.0
E10	30	50	20	80	8	44.3	4.62	0.75	1060.3

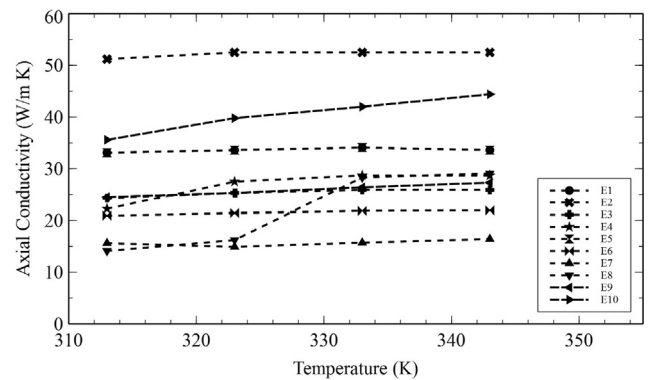


Fig. 5. Behavior of thermal conductivity as a function of Temperature.

was hydrated with distilled water with a 1 ml water per gram LiCl ratio. Secondly, the expanded graphite and activated carbon are included. Finally, the mixture is mixed in an RZR analog rod stirrer 1, HEIDOLPH® at 600 rpm for 10 min to homogenize the composite.

2.1.3. Mixture compaction

The adsorbent composite is mixed with distilled water in 1:1 mass ratio. Subsequently the mixture is heated in a muffle furnace at 110 °C for 24 h to evaporate the water content. Once samples were dehydrated, compaction was carried out using the procedure described by Oliveira et al. [26], where a 10 MPa pressure is applied during 30 s to produce a solid block. Two compacting pressures were used in this work: 8 MPa for the lower level and 12 MPa for the higher level. Samples were made using a cylindrical mold capable to supporting loads applied in the universal machine, obtaining cylindrical composite material blocks as is shown in Fig. 2.

2.2. Measurement of thermal conductivity

2.2.1. Axial conductivity λ_a

Based on the ASTM C177-13 standard [24], axial thermal conductivity was determined using the hot plate method described by Wang et al. [31] and Fayazmanesh et al. [32]. Specimen of the composite was placed in the tests on a silicone thermal resistance of equal dimensions to the sample and that was isolated on the opposite side (see Fig. 3). Then a 5 W heat flow was applied with the resistance and the temperature difference in steady state between the upper and lower surfaces of the sample was measured.

The axial thermal conductivity of the samples is obtained from the Fourier Law as shown in Eq. (1)

$$\lambda_a = \frac{\dot{Q}t}{A\Delta T} \tag{1}$$

where \dot{Q} is the heat flow; t , the thickness of the sample; ΔT , the temperature difference (TH-TL); and A , the area of the sample's cross

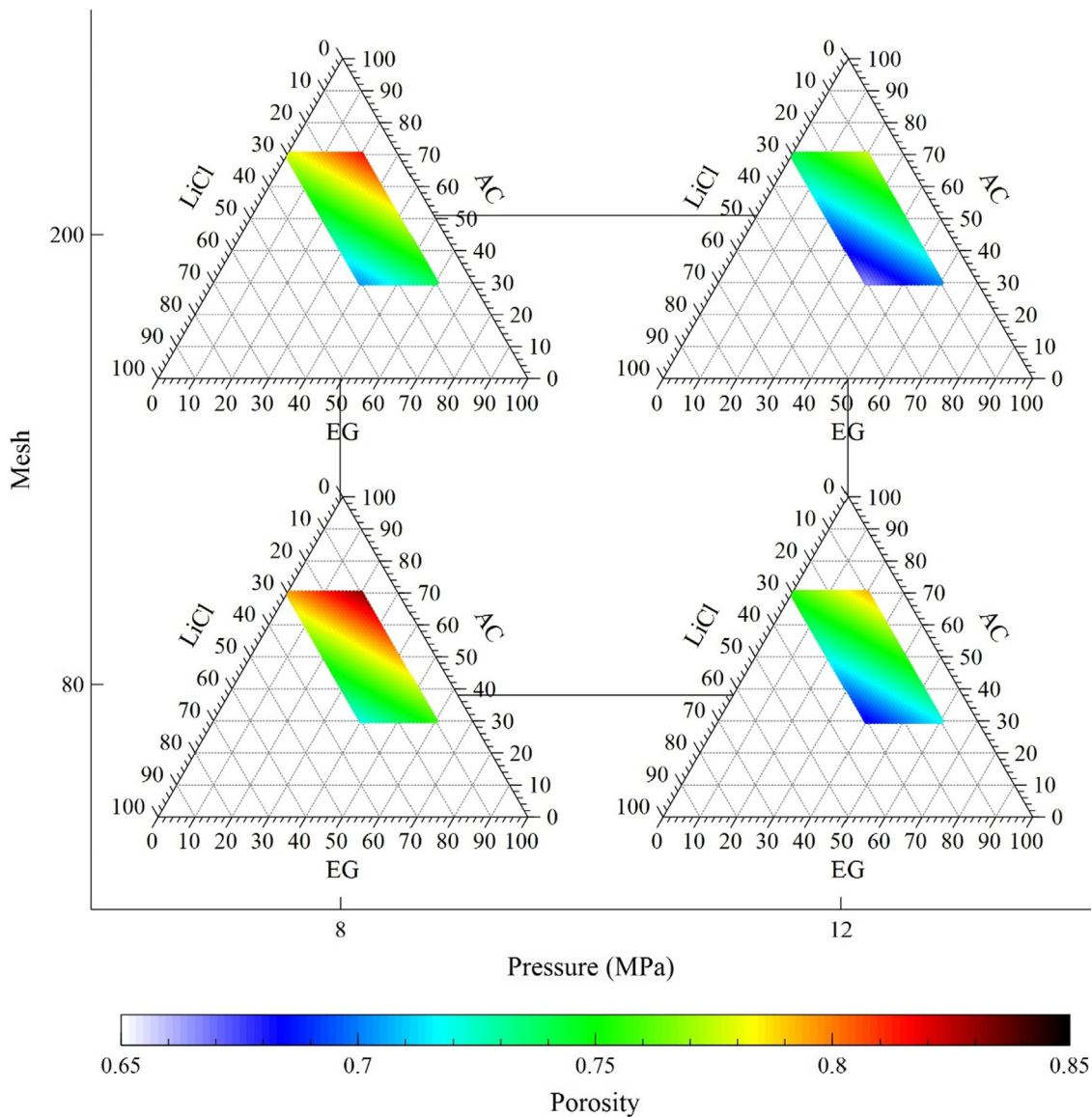


Fig. 6. Porosity according to mixing proportions, mesh size and compaction pressure.

section.

2.2.2. Radial conductivity λ_r

Radial thermal conductivity was determined with the same principle as in the axial conductivity, applying the hot-wire method in a hollow cylindrical specimen with a 20 W thermal resistance in its center. A schematic description of the experimental mock-up is presented in Fig. 4.

The axial thermal conductivity of the samples is obtained from the Fourier Law as shown in Eq. (2)

$$\lambda_r = \frac{\dot{Q} \ln(r_2/r_1)}{2\pi t \Delta T} \quad (2)$$

where \dot{Q} is the heat flow; t is the thickness of the sample; ΔT , the temperature difference ($T_H - T_L$); r_1 and r_2 are the inner outer radius of the sample, respectively.

2.2.3. Measurement systems calibration

The measurement system described in previous sections, has the methodology fundament according to the heat transfer theory. The measurement device was developed and constructed for this application. In order to guarantee accuracy on the results, the experimental

procedure to determine the thermal conductivity of the samples was applied in a specimen with widely known properties. In each case, the hot plate and hot wire method were implemented to cylindrical samples made of 1020 steel with 50.8 mm diameter and 20.1 mm thickness. 10 tests were accomplished and its experimental results were compared with literature values. Thermal conductivity was evaluated under two different values of heat flux ($\dot{Q} = 5 \text{ W}$ and $\dot{Q} = 20 \text{ W}$). Results of hot wire calibration tests are shown in Table 4.

Results show that the measurement method is reliable, since the results of a t -student media analysis indicates that there is no significant difference between theoretical value of thermal conductivity [33] and experimental data, results of the t -student test are presented in Table 5.

2.3. Measurement of porosity (ϕ) and apparent density (ρ_{Block})

Porosity and apparent density were determined by Eqs. (3)–(7):
Block Apparent Density:

$$\rho_{Block} = \frac{m_{EG} + m_{AC} + m_{LiCl}}{V_B} \quad (3)$$

Apparent density of expanded graphite plus apparent density of activated carbon:

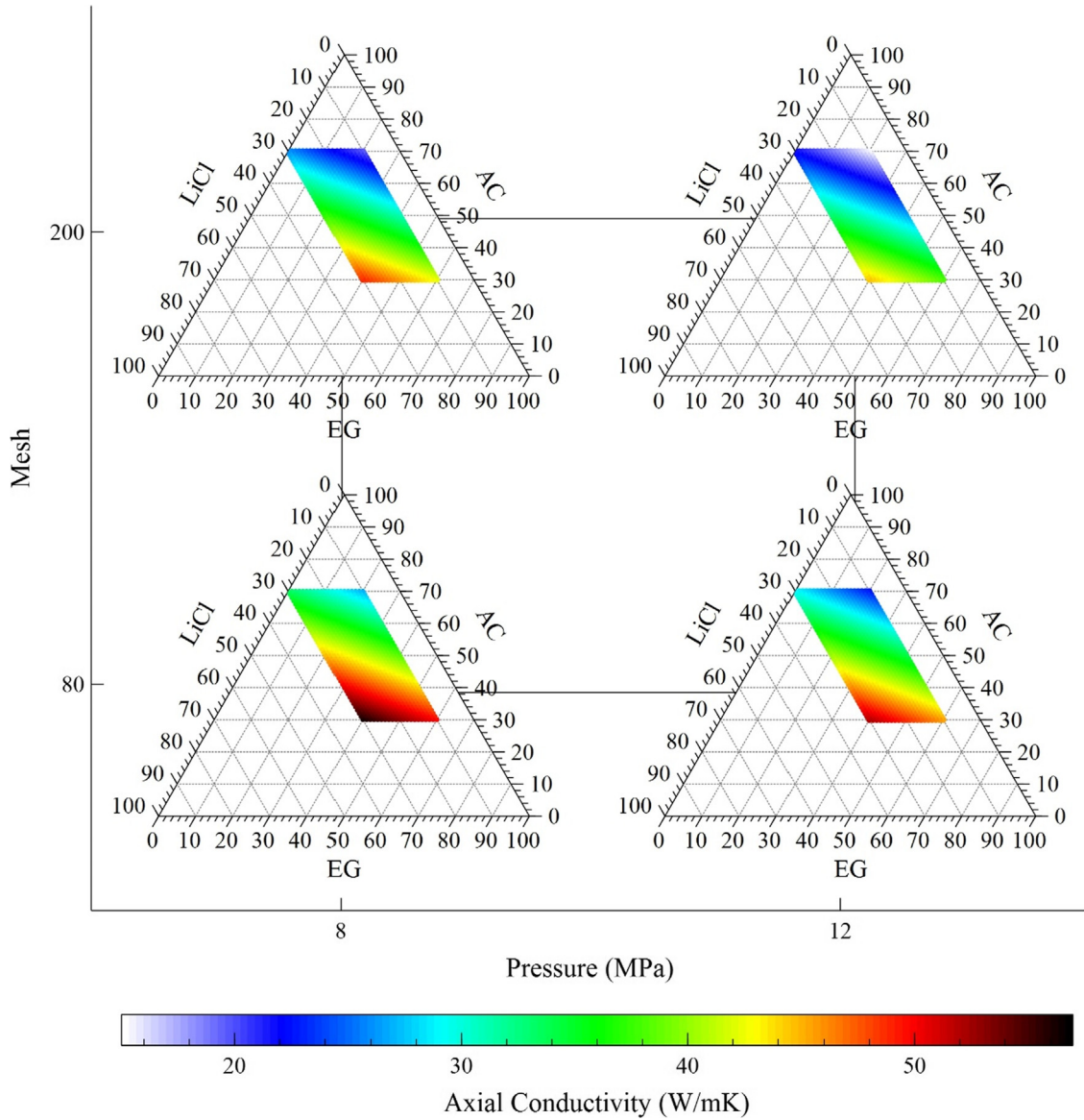


Fig. 7. Axial conductivity according to mixing proportions, mesh size and compaction pressure.

$$\rho_{EG} + \rho_{AC} = \frac{m_{EG} + m_{AC}}{V_B} \quad (4)$$

Mass fraction of expanded graphite:

$$f_g = \frac{m_{EG}}{m_s + m_{EG}} \quad (5)$$

Mass fraction of activated carbon:

$$f_c = \frac{m_{AC}}{m_s + m_{AC}} \quad (6)$$

where ρ_B , ρ_{EG} , ρ_{AC} are the apparent densities for the block, the expanded graphite and the activated carbon, respectively; m_{EG} is the mass of expanded graphite; m_{AC} , mass of activated carbon; m_s , mass of salt, V_B the volume of the block, f_g and f_c the fractions of mass of graphite and carbon respectively.

Compound porosity is calculated by Eq. (7).

$$\phi = \frac{\rho_{EG} + \rho_{AC}}{\rho_g + \rho_c} - \left[\frac{(1 - (f_g + f_c))(\rho_{EG} + \rho_{AC})}{(f_g + f_c)} \right] \frac{\bar{v}_s}{MW_s} \quad (7)$$

ρ_g and ρ_c are the real density of the respective graphite and carbon; \bar{v}_s , the molar volume of the salt; and MW_s , is the molar mass of the salt. The

values taken for the real densities of activated carbon and expanded graphite were $\rho_c = 1.8 \text{ kg/m}^3$ and $\rho_g = 2.3 \text{ kg/m}^3$ [34], the volume values and the molar mass were $\bar{v}_s = 0.05$ and $MW_s = 0.042394$ respectively [34].

3. Results

Experimental results of axial conductivity, radial conductivity and porosity are shown in Table 6. Fig. 5 shows the behavior of thermal conductivity as a function of Temperature. This Figure shows that there is no significant effect of the test temperature on the thermal conductivity of the samples. From experimental results, ternary diagrams of Figs. 6–8 were constructed, showing porosity, axial conductivity and radial conductivity according to mesh size used in sieving, compaction pressure and proportion of mixing components.

Compound porosity is higher when mixing proportions close to 70% activated carbon, 20% expanded graphite and 10% lithium chloride are used; as observed in Fig. 8. In addition, porosity of the sample reaches higher values when the mixture is prepared with lower compaction pressure (8 MPa) and thicker mesh sizes during sieving (Mesh # 80).

Axial and radial conductivity results are shown in Figs. 7 and 8.

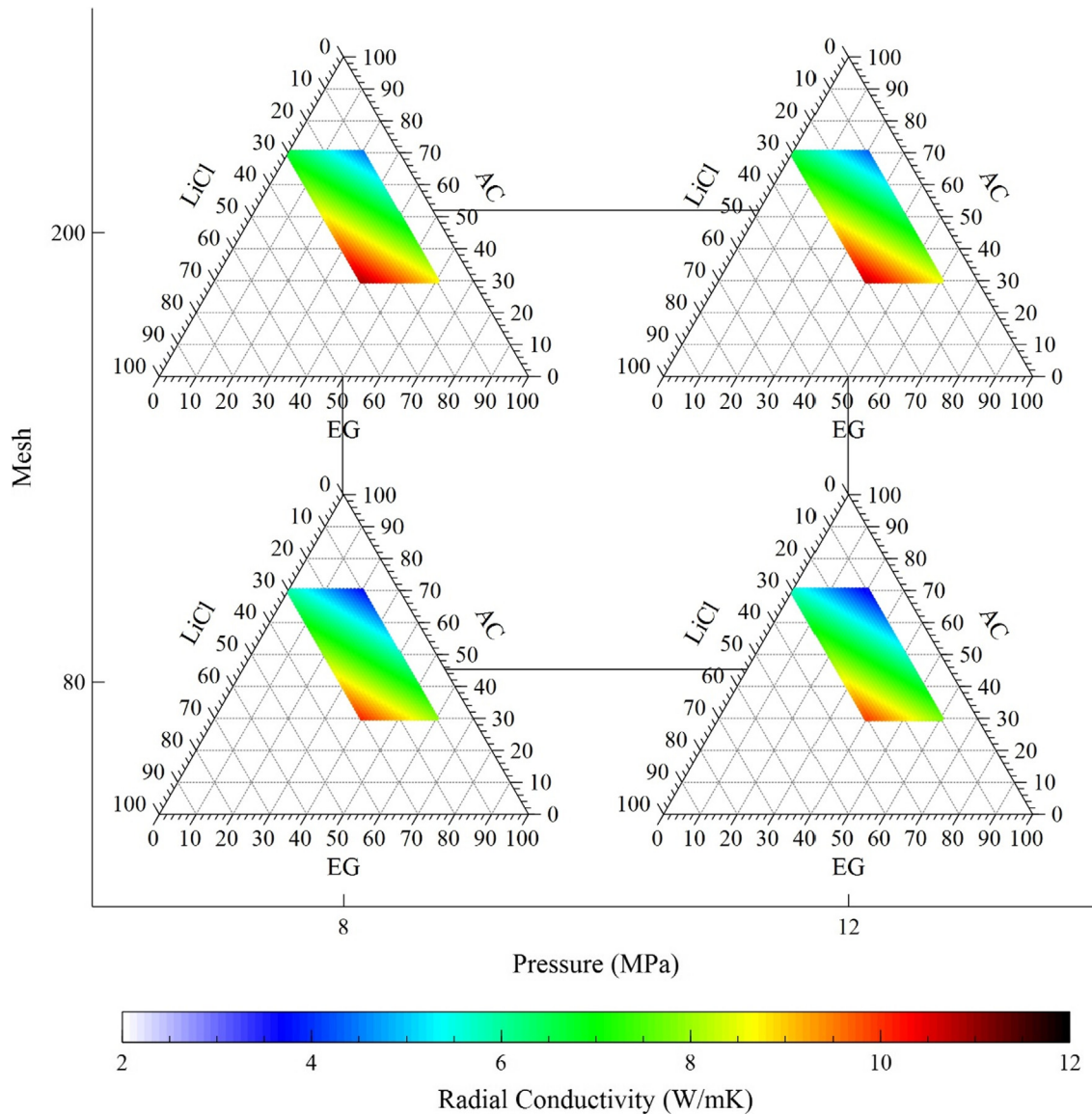


Fig. 8. Radial conductivity according to mixing proportions, mesh size and compaction pressure.

Table 7
Optimal mix experimental results: physical properties.

Block Sample	m_{LiCl} (%)	m_{AC} (%)	m_{EG} (%)	Mesh (N_0)	Pressure (MPa)	λ_a (W/m K)	λ_r (W/m K)	Porosity ϕ	ρ_{AC} (kg/m)
M1	30	30	40	200	12	76.5	13.8	0.72	1164.32
M2	30	30	40	80	8	69.7	5.90	0.70	1192.01

Contrary to the observed with porosity, the axial and radial conductivity values are higher when mixing proportions close to 30% activated carbon, 40% expanded graphite and 30% lithium chloride. As for the preparation conditions of the adsorbent compound, it is observed that the 8 MPa compaction pressure improves the radial and axial conductivity with respect to that of 12 MPa. However, mesh size used during sieving does not have the same effect on both properties. While thicker mesh sizes favor axial conductivity, using a finer mesh favors radial conductivity.

Since the optimal mixture proportion was 30% of activated carbon, 40% of expanded graphite and 30% of lithium chloride, two samples were prepared and experimentally analyzed in order to validate the results. The properties obtained for both samples are shown in Table 7.

It was obtained that both axial and radial conductivity increased considerably and they adjust to the values shown in the previous Figs. 6–8.

It is possible to observe that the addition of EG in the compound increased the overall thermal conductivity of the composite, compared with the adsorbent components of the mixture separately. While thermal conductivity of LiCl and AC presented maximum values of 0.8 W/m K and 0.5 W/m K, respectively, the adsorbent composite enhanced with EG reached 76.4 W/m K. Besides, both samples presented high porosity values. These results indicate that the developed composite has promising potential in several applications, as the sorption cooling systems, where the COP can be improved by using an adsorbent component with high porosity and thermal conductivity, since the porosity increases the sorption capacity due to the increase of

adherence area, while the thermal conductivity improves the heat transfer on the adsorbent bed.

4. Conclusions

Experimental results showed that both porosity and thermal conductivity are influenced by mass ratio of expanded graphite within the mixture, as well as its particle size and compaction pressure. Samples of composite material designed with a higher proportion of activated carbon made with mesh #80 and compacted with an 8 MPa pressure, have the highest porosities. Likewise, as observed in ternary surfaces, composite material blocks with the lowest level in the activated carbon proportion have values of greater axial and radial thermal conductivity, with mesh #80 particle sizes. This suggests that to enhance the adsorbent composite material properties should be made with larger particles.

Due to the polytropic nature of these composite adsorbent materials, it was found that average axial thermal conductivity is 10 times greater than radial thermal conductivity; regardless of particle size or mass proportions of the mixture components. It is noteworthy that composite material samples made with lower compaction pressure had greater thermal conductivity, both axial and radial. Nonetheless, axial thermal conductivity is greater when particle size is mesh #80 (larger particles), and the thermal conductivity radial increases with 200 mesh (smaller particles).

Results indicated that the addition of EG is an appropriate strategy to enhance the thermal conductivity of the adsorbent composite, without significant negative affectation on its porosity. Thermal conductivity also improves when apparent density of composite material blocks increases. This density is affected by the rise in mass proportion of activated carbon due to its porous nature. In addition, this new composite adsorbent material has minor variation in its thermal conductivity at different measurement temperatures; thus, it can have a constant desorption rate in solar energy applications where changes induced by external variables are frequent.

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