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INTENSIFICATION OF THE THERMO-PHYSICAL PROPERTIES OF HYDRATED LIME AND GYPSUM COMPOSITES WITH GRAPHITE MICRO-SCALE PARTICLES

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Abstract. *Thermo-physical properties of composite materials based on hydrated lime and gypsum were acquired in order to evaluate the influence of the addition of graphite on the increase of these properties, in particular of the thermal conductivity. The focus on thermal conductivity is due to the influence of this property on the heat transfer process of solids. Therefore, it was evaluated which is the most appropriate mass fraction of graphite, ranging form 0 to 25 % compared to matrix mass, and what is the amount of water suitable for the production of the materials, in addition to the drying time necessary to guarantee a material that has a high thermal conductivity value. In that way, after the curing process, an increase of approximately 134 % in thermal conductivity was analyzed comparing the sample without the addition of graphite powder (0.22 W/mK) with the sample containing the largest mass of graphite (0.51 W/mK). The same was observed for gypsum composites, with a maximum increase of 106 % being observed in the case of material with a greater amount of graphite powder, with thermal conductivity of 0.76 W/mK, since pure gypsum, with the same amount of water in the production, resulted in thermal conductivity of 0.37 W/mK. A statistical analysis was also performed, and proved that the addition of graphite had a significant influence on the specific mass, thermal diffusivity and mainly on thermal conductivity of the composites. In addition, the amount of water used in the mixture had a negative effect on the values of diffusivity and thermal conductivity.*

Keywords: Thermal conductivity, Thermo-physical properties, Hydrated lime, Gypsum, Graphite

1. INTRODUCTION

Composites with high values of thermal conductivity can enable efficient thermal management in many modern applications, such as electronics, batteries, aerospace devices and LED lighting (Depaifve et al., 2020; Ren et al., 2020; Yetgin et al., 2020). Thermally conductive fillers are one of the most important factors in the thermal conductivity values of composites. Metals, ceramics and carbon materials are the most used thermally conductive charges. Metals and carbon materials filled with thermally conductive polymer composites are used mainly in areas of heat transfer and dissipation where electrical insulation is not necessary, such as heat exchangers (Guo et al., 2020). Expanded graphite (EG) and Graphene Nanoplates (GNP) are the most promising fillers for increasing thermal conductivity in the polymeric composites industry due to their commercial availability, low cost and excellent thermal conductivity (Depaifve et al., 2020).

Gypsum-based composites are widely used in civil construction due to their wide availability, easy manufacture, because they are not harmful to the environment and have good thermal insulation and fire protection qualities (Du et al., 2020). Gypsum and hydrated lime are considered accessible and less complex matrices to produce a stable composite. And to improve the thermo-physical properties of these composites, it is necessary to use an efficient conductive thermal load that is also accessible in most applications. The graphite-based materials are the ones that most fit this profile (Flores Medina et al., 2016). In the works published by Jeong et al. (2017), Barbero-Barrera et al. (2017) and Flores Medina and Barbero-Barrera (2017) the authors observed an increment of 71 %, 97 % and 115 %, respectively, in gypsum composites thermal conductivity with the addition of filling particles, and the last two works used the same graphite mass fraction,

equivalent to 25 %. Due to the lack of data related to the thermo-physical properties of hydrated lime, this work produced composites of hydrated lime with graphite and compare the results with gypsum composite, also with addition of graphite.

Graphite powder is a residual product obtained from crushed isostatic graphite blocks for the production of molds (Flores Medina et al., 2016). European standards place great emphasis on reintroducing waste into the production chain. Analyzes of the use of revalued isostatic graphite powder residues have already been carried out on gypsum-based boards (Barbero-Barrera et al., 2017). Isostatic graphite charge (IG) is a waste by-product that has been performing well with gypsum (Flores Medina and Barbero-Barrera, 2017).

In that sense, the thermo-physical properties of the hydrated lime and gypsum composites were measured in order to evaluate the influence of the addition of graphite on the increase of these properties, in particular of the thermal conductivity. The largest amount of graphite added to the composites was equivalent to 25 % mass fraction compare to matrix mass. The focus on thermal conductivity is due to the influence of this property on the heat transfer process of solids. Experiments were performed in order to assess the effect of graphite and water mass fractions in the production of the materials. Also, the drying time necessary was studied, to guarantee a stable material that has a high thermal conductivity value.

2. MEASUREMENT METHOD

In this item, the details of the production of hydrated lime and gypsum composites will be presented. In addition to the equipment used to obtain the values of the thermo-physical properties of the manufactured materials.

2.1 Production details

Regarding the preparation of solid materials, for the evaluation of properties two samples of the materials are necessary, so that the probe will be positioned between them. A digital scale (GEHAKA, BK500), with maximum resolution of ±0.001 g, was used to weight the mass of each component before and after preparation. The measuring range of the digital scale is from 0.01 to 510 g with an uncertainty of ± 0.002 g. The mass variation with time was also analyzed. The specific mass, ρ, was determined experimentally with the weighted mass and the final dimensions of the sample.

For the first analysis, six samples were produced for the composites based on hydrated lime and other six for gypsum composites, with and without the addition of graphite powder, always maintaining the mass of matrix and water, in approximately 100 and 75 grams, respectively. Only varying the mass fraction of graphite powder, from 0 to 25 % compared to matrix mass. Before removing lime samples from the molds. They were dried in a temperature controlled, oven at 30 °C for about 17 h. In addition, all samples were sanded to ensure the flatness of the parts and, consequently, a uniform contact between the parts and the probe that allows the reading.

2.2 Experimental apparatus

To measure the thermal conductivity, diffusivity and specific heat capacity, from a pre-established specific mass, was used the LINSEIS THB-1 conductivity meter (Figure 1). The measurement parameters used in the conductivity meter were kept constant for all samples. Thus, 3 multiple tests were carried out with 5 measurements in each.

Tests were carried out at room temperature, between 20 and 25 ° C. The probe used was the “B7620”, being necessary a sample with a minimum size of 10 mm x 20 mm x 3 mm. The measurement ranges of the equipment are illustrated in Table 1.

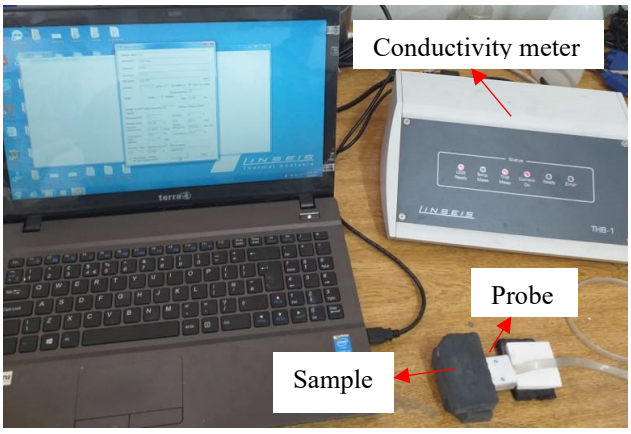

Figure 1. Apparatus for measuring thermo-physical properties.

Table 1. Measurement range and errors of the LINSEIS THB-1 conductivity meter with the "B7620" probe.

Property	Measuring range	Instrument error
Thermal conductivity (k)	0.01–5 [W/mK]	< 2 %
Thermal diffusivity (α)	0.05–10 [mm^2/s]	< 5 %
Specific heat capacity (c)	0.01–1 [J/gK]	< 5 %

3. RESULTS

3.1 Hydrated lime composites

Initially, several samples of hydrated lime and water were produced with the addition of the graphite powder to observe the behavior of the materials according to the variation of the mass fraction of graphite compared to the mass of lime. Figure 2 is a photo of the samples of lime hydrated with water, without the addition of graphite powder. Since Figure 2 (a) was taken shortly after mixing the lime with the water, before drying. While Figure 2 (b) presents the first and the second batch of samples at different curing periods.

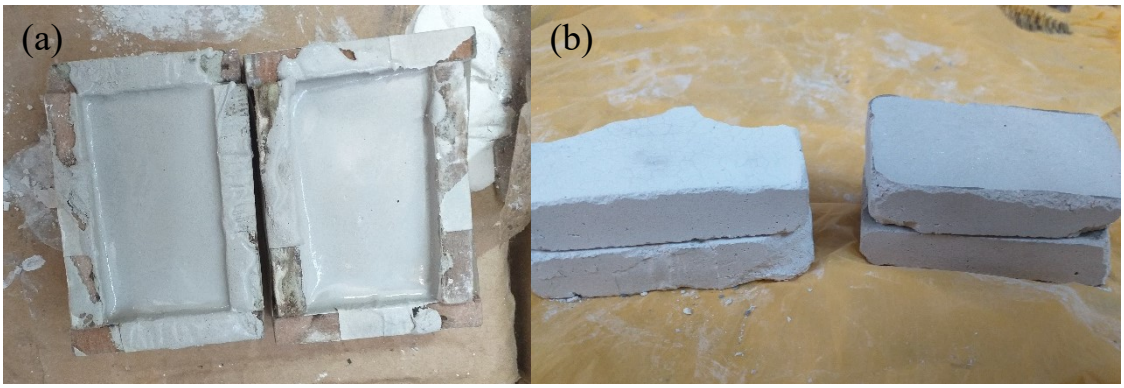

Figure 2. Samples of lime hydrated with water at different time of mixing (pre-drying) and post-drying.

In Figure 2 (b) it is possible to notice visually the difference in humidity between the lime samples, without graphite, since the blocks on the left are lighter, having been the first to be produced and, thus, with the curing process already finished. On the other hand, the blocks to the right of the image are darker due to the fact that they were produced later, so that the sample was still moist at the time of the photo.

Figure 3 shows the production of the samples with a mass fraction of 1/20 (graphite/lime). Figure 3 (a) shows the mixture after preparation. Figure 3 (b) was taken after 17 hours in the oven, at 30 ° C. It is important to highlight that this temperature was used to prevent the structure from cracking and crumbling as much as possible during the drying process. Figure 3 (c) is a photograph of the samples completely dried and sanded.

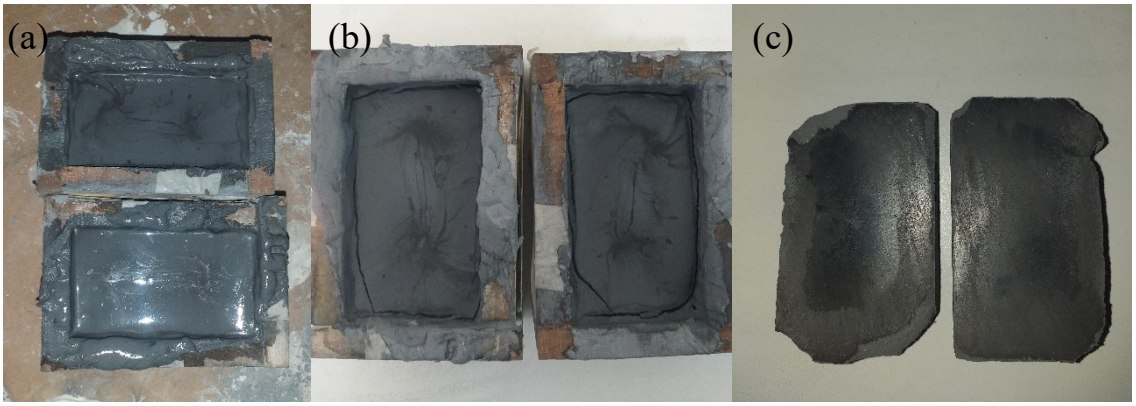

Figure 3. Samples with mass fraction of 1/20 (a) pre-drying; (b) post drying; (c) post sandpaper.

A significant change in the properties of the materials has been observed over the days. Figure 4 illustrates the variation in the thermal conductivity of the samples with different mass fractions of graphite over the days after preparation. Four values of thermal conductivity of the samples were measured on different days randomly. The decay in

thermal conductivity is due to the loss of water and change in the internal structure of the materials during the drying process, while the samples have not yet stabilized. The thermal conductivity of the materials is inversely proportional to the amount of air present in the material and directly proportional to the apparent density. This property still increases due to the moisture content contained in the materials, due to the fact that water replaces part of the gaseous volume contained in the pores and has a higher thermal conductivity value than air (Mendes et al., 2019). The influence of moisture contained in the composite of gypsum was also analyzed by Liu et al. (2017), who observed that the thermal conductivity in the wet curing condition was higher than in the dry curing condition.

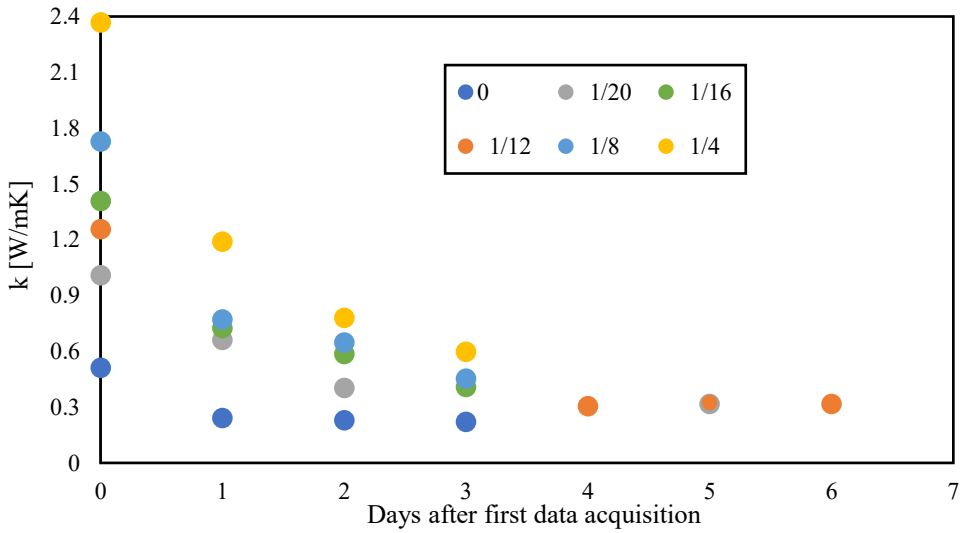

Figure 4. Evolution of thermal conductivity of the samples at different mass fractions over time.

The sample with the highest mass fraction of graphite presented the highest conductivity for every day, while the bare sample, without graphite, showed the lowest thermal conductivity values. This behavior is in accordance with the literature, since the progressive increase in the addition of graphite modifies the properties of the composite, increasing the density and thermal conductivity and reducing its porosity and its water absorption (Flores Medina and Barbero-Barrera, 2017).

The changes in the thermo-physical properties of the composite were evaluated in a period of 5 hours after the sample was removed from the mold (Table 2). The thermal conductivity of the material has dropped significantly. It is worth mentioning that the initial mass of the sample was 55.3 g and the final was 52.8 g, that is, approximately 2.5 g of water evaporated from the material.

Table 2. Variation of the thermo-physical properties of the hydrated lime composite with 1/20 graphite.

Mass fraction (graphite/lime)	Hours after dismoulding	k [W/mK]	α [mm^2/s]	ρ [g/cm^3]	c [J/gK]
1/20	0	1.0101	0.7204	1.428	1.0303
	5	0.8303	0.8196	1.362	0.7494

In Table 3 it is possible to observe that all the samples dried completely until stabilizing the result of the thermal conductivity increases with the application of more graphite powder, the sample without graphite being the one with the lowest conductivity. Oppositely, the sample with the highest mass fraction of graphite obtained the highest thermal conductivity. In addition, it was possible to evaluate that with the initial drying in the oven and an additional 7 days of natural curing, it ensures that the material stabilizes its thermo-physical properties.

Table 3. Data obtained for stabilized hydrated lime composite, varying the mass fractions of graphite.

Mass fraction (graphite/lime)	k [W/mK]	α [mm^2/s]	ρ [g/cm^3]	c [J/gK]
0	0.2202	0.5728	1.187	0.3241
1/4	0.5145	1.3607	1.254	0.3016
1/8	0.4010	0.8147	1.204	0.4089
1/12	0.3388	0.3569	1.045	0.9083
1/16	0.2994	0.7177	1.101	0.3811
1/20	0.2984	0.7453	1.083	0.3698

In order to synthesize the values of thermal conductivity in three different curing states. Figure 5 shows the thermal conductivity as a function of the mass fraction of graphite considering the tests on the first day after removing the sample from the oven, on the last day of the first test taking, while the properties still varied, and the tests with the samples stable. It can be concluded that the thermal conductivity reduces with time, until the material stabilizes, due to the loss of water in the structure.

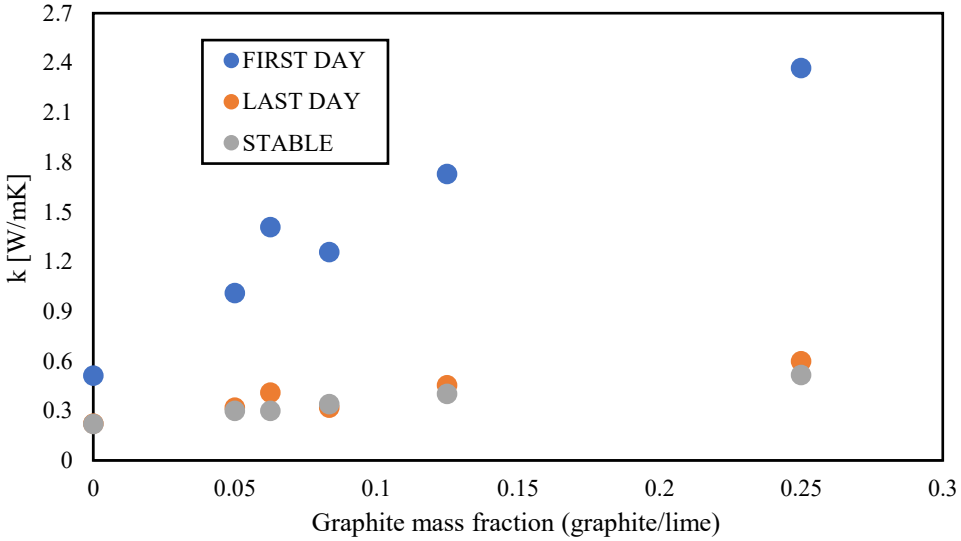

Figure 5. Thermal conductivity as a function of the mass fraction of graphite considering the different drying phases.

3.2 Gypsum composites

Gypsum composites with the addition of graphite powder were also analyzed. Before adding the graphite powder, it was observed the variation of the thermo-physical properties of the samples of pure gypsum with the amount of water used in the manufacture of the material. The results of measurements for pure gypsum are shown in Table 4.

Table 4. Thermo-physical properties of gypsum samples varying the mass fraction of water.

Mass fraction (water/lime)	k [W/mK]	α [mm²/s]	ρ [g/cm³]	c [J/gK]
1/1	0.2858	0.5602	0.975	0.5286
3/4	0.3699	0.5302	1.047	0.6681
2/3	0.3730	0.5361	1.073	0.6484
1/2	0.4286	0.7371	1.100	0.5339

The difference between the thermal conductivity of the gypsum samples was due to the amount of gypsum powder added in the production, since the cured sample that contained more gypsum in proportion to the amount of water had less pores. As a result, the sample with less pores receives less influence from the air - with low thermal conductivity - and has a higher value. It is worth mentioning that this influence of the pores is only seen in the case of completely dry samples. The influence of humidity on the thermal conductivity of the materials was also seen by Gusyachkin et al. (2019) and by Gomes et al. (2017), in these publications the higher humidity of the samples provided an increase in thermal conductivity. In addition, Gomes et al. (2017) analyzed that the thermal conductivity of these porous aggregates was affected not only by moisture and apparent density, but also by porosity, pore size and internal structure. Observing the influence of the place where the samples go through the drying process, Wang et al. (2016) obtained results that indicate that the thermal conductivity of the samples compacted in a dry environment, with 17 % moisture content, slightly decreases with the curing time, while the effect of the time of curing in the samples compacted in a humid environment, with 22 %, was insignificant.

Figure 6 shows the gypsum samples at the same stage of cure with different amounts, by weight, of graphite powder, and with constant gypsum and water masses, equal to 100 g and 75 g, respectively. In the same manner as the samples of hydrated lime, they need to be sanded and flattened to ensure proper sensor contact. Gypsum samples also vary their properties until they lose the water used in their production with drying. However, this process is faster compared to lime samples. For the first samples production, the use of oven for drying was not necessary. Table 5 shows the variation the thermo-physical properties values of the sample with a mass fraction of 1/20 (graphite/gypsum), 5 % of graphite, when comparing the measurements of the second, fifth and tenth days from the sample production.

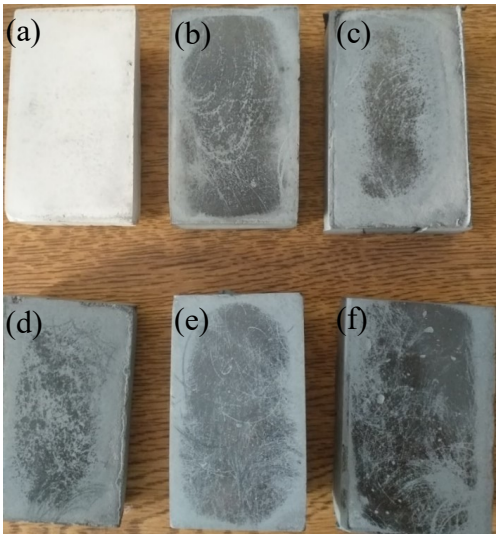

Figure 6. Stable gypsum samples with different mass fractions of graphite: (a) 0; (b) 1/20; (c) 1/16; (d) 1/12; (e) 1/8; (f) 1/4.

Table 5. Thermo-physical properties of gypsum composites with 1/20 graphite, at different drying levels.

Days after production	k [W/mK]	α [mm²/s]	ρ [g/cm³]	c [J/gK]
2	0.6678	0.4618	0.909	1.5908
5	0.4302	0.5394	0.862	0.9337
10	0.4093	0.6186	0.858	0.7712
12	0.4249	0.5565	0.859	0.8891

It was concluded that for the first measurement, the sample had not yet finished the curing process, therefore, the moisture contained within the material influenced the result with greater thermal conductivity, similar to behavior observed for f hydrated lime sample. Proof of this is the reduction in specific mass that was caused due to the decrease in sample mass, from 53.5 g to 50.7 g. The second, third and fourth values were considered stable. It is estimated the curing time is approximately 5 days for the gypsum composite without of any heating equipment.

3.3 Comparison

The results of thermal conductivity of the samples showed a very evident tendency regarding the relation of the increase of the mass fraction of graphite in the increase of the property. Thus, the values obtained for the samples of hydrated lime and gypsum were plotted on the same graph, as shown in Figure 7. As can be observed, there is a significant effect of the matrix material on the graphite composites thermal conductivity.

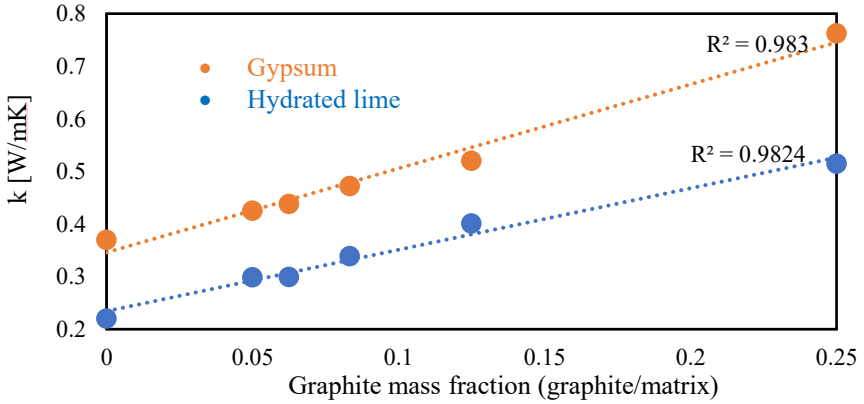

Figure 7. Thermal conductivity as a function of graphite mass fraction, for gypsum and hydrated lime matrices.

Therefore, it is noted that for both materials the graphite powder had an impact on the increase in thermal conductivity. In addition, regard from the figure it is possible to observe a linear relationship between these two parameters. Regarding

the increments in the thermal conductivity values of the samples of stable composites in comparison to the stable samples of the matrix without addition of graphite, an increase of about 134 % in the hydrated lime-based material was observed, reaching approximately 0.52 W/mK, while for the gypsum matrix the increase maximum was 106 %, up to 0.76 W/mK. Similar values were found by Barbero-Barrera et al. (2017) when using the same matrix and filling material, in which the thermal conductivity analyzed by the authors was approximately 0.73 W/mK, with an increase of 97 % in relation to the pure gypsum matrix. Figure 8 presents a comparison between the increments obtained in thermal conductivity for composites with different matrices using graphite as the filling material. It is evident how much graphite had a greater influence on epoxy resin composites compared to the other matrices, and the authors had attributed this effect to the easier formation of heat pathways on the layer structure of graphite in the epoxy matrix (Fu et al., 2014). The increase in the thermal conductivity value was calculated according to Eq. (1). Where "k_c" and "k_m" represents the conductivity of composites and pure matrix, respectively.

$$k_{increment}[\%] = [(k_c - k_m)/k_m] * 100 \quad (1)$$

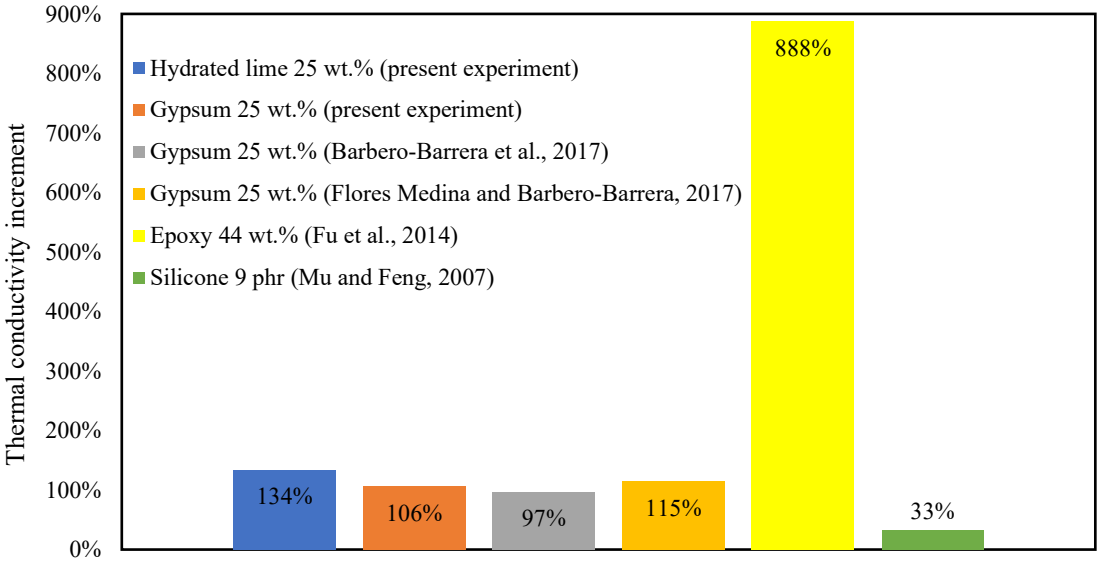

Figure 8. Increase in thermal conductivity for composites with different matrices using graphite as the filling material.

In order to evaluate the variability of the measurements, more samples were replicated with 5 % graphite (1/20) for lime and gypsum matrices. The variation of the results obtained can be applied to all other samples produced with different amounts of graphite. Therefore, Figure 9 indicates the thermal conductivity obtained for the composites of both matrices with a mass fraction of 5 % graphite compared to the matrix mass. For each matrix, the analysis of uncertainties between materials manufactured with the same mass fractions was performed on a sample with 5 composite materials and for a 95 % confidence interval. Measurement uncertainties were similar for materials with both matrices. Being 0.012 W/mK for the hydrated lime matrix and 0.016 W/mK for gypsum. The thermal conductivity range for the hydrated lime and gypsum composites with 5 % graphite, in mass, was (0.275; 0.299) and (0.430; 0.462), respectively.

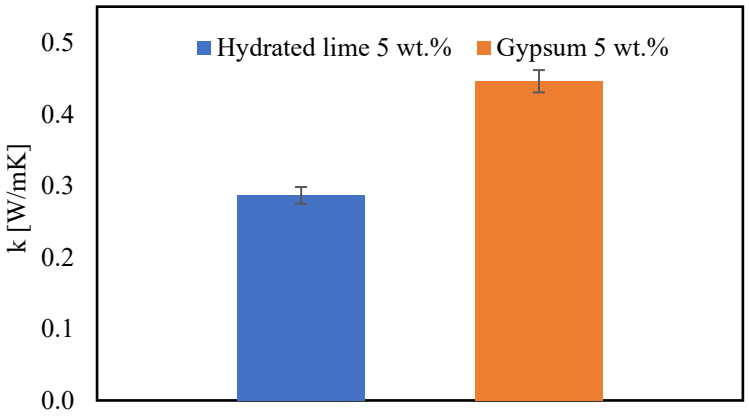

Figure 9. Thermal conductivity for stable samples of hydrated lime and gypsum with graphite mass fraction of 5 %.

4. STATISTIC ANALYSIS

Five samples were produced with different mass fractions of hydrated lime, water and graphite and applied to the statistical methodology of planning triangular surfaces, used in dependent ternary mixtures (Montgomery, 2012). So that three samples were produced from the central point, totaling seven samples. Thus, it was possible to observe the influence of each component on the thermo-physical properties of the final material. The sum of the mass of all components results in approximately 200 g and the sum of the mass fractions must equal one. The samples underwent a seven-day drying process in an oven at 30 °C to ensure complete curing of the materials. Table 6 shows the values obtained experimentally for the thermo-physical properties for each sample manufactured after the entire curing process. Based on these results, a statistical analysis was performed to estimate the influence of each component of the mixture thermo-physical properties.

Table 6. Averages obtained for the thermal physical properties of hydrated lime composites.

Samples	Mass fraction (water:grafite:lime)	k [W/mK]	α [mm^2/s]	ρ [g/cm^3]	c [J/gK]
S1	0.4000:0.0000:0.6000	0.1806	0.9362	1.076	0.1798
S2	0.4500:0.0000:0.5500	0.1576	0.7609	0.966	0.2145
S3	0.4000:0.1000:0.5000	0.4286	0.9251	1.113	0.4166
S4	0.4500:0.0500:0.5000	0.2803	0.6358	0.962	0.4591
S5	0.4250:0.0375:0.5375	0.2274	0.8215	0.882	0.2960
S6	0.4250:0.0375:0.5375	0.2197	0.6947	0.946	0.3460
S7	0.4250:0.0375:0.5375	0.2298	0.6515	0.947	0.3767

The “Statistica” software (StatSoft, 2011) was used to perform the statistical analysis. A linear model of dependent ternary mixtures was applied. Figure 10 presents the region of the mass fractions analyzed. This region was delimited according to the mass fractions of components that made it possible to manufacture a stable solid sample that would guarantee repeatability in the readings in the measurement system. On the other hand, Figure 11 presents the response surfaces generated from the statistical analysis, in which it is possible to perceive which are the components that have more influence in each thermal physical property of the hydrated lime composites with graphite. The triangular surfaces formed by the factors of each component are normalized, so that they present only the region between the points indicated in Figure 10. In addition, the equations composed of the correction values that made it possible to generate each of the triangular surfaces are also presented.

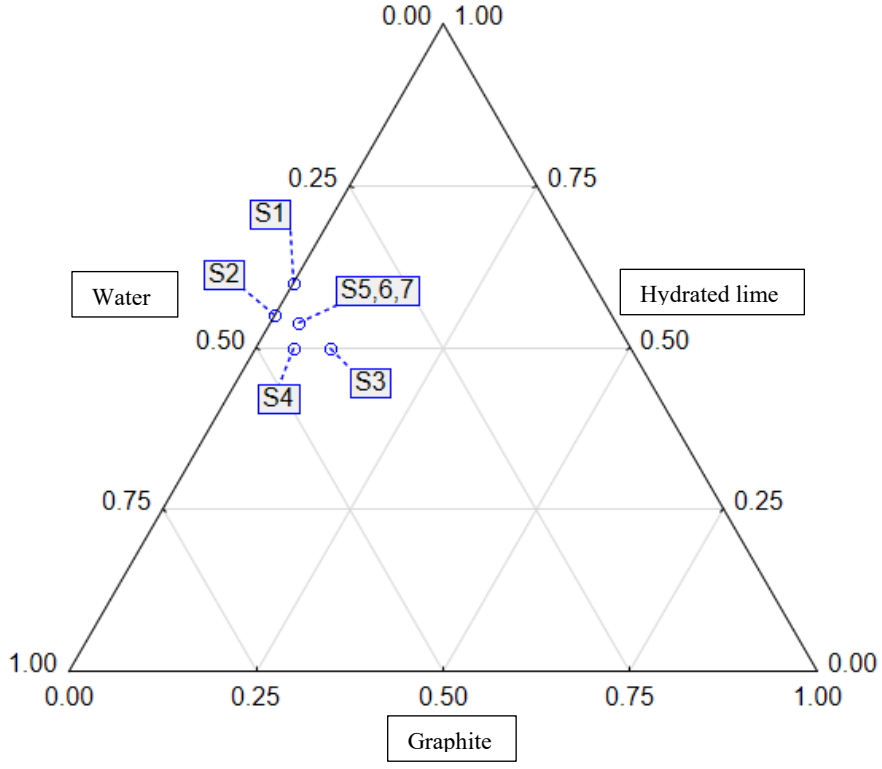

Figure 10. Mass fractions analyzed to generate triangular surfaces.

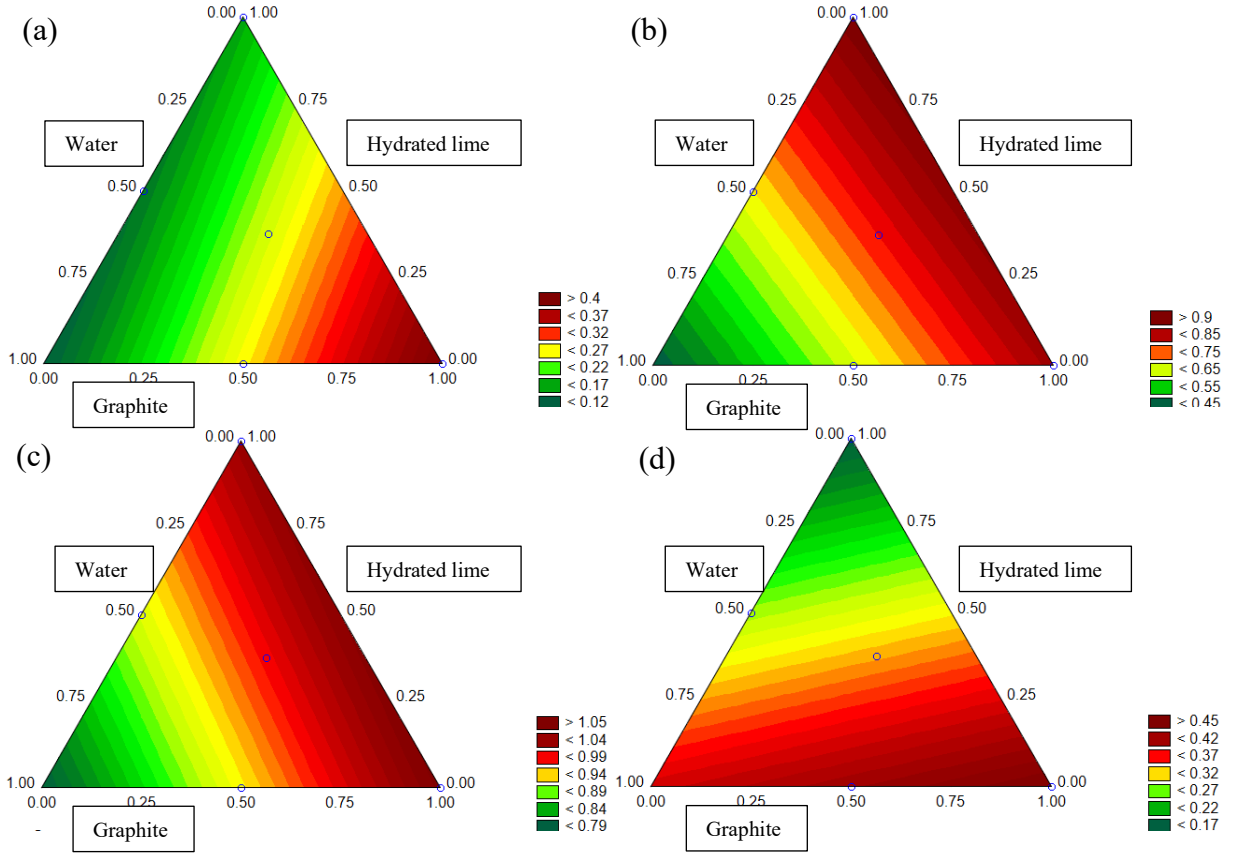

Figure 11. Triangular surface generated for thermal conductivity (a), thermal diffusivity (b), specific mass (c) and specific heat capacity (d).

$$k = 0.1178 * m_{water} + 0.4129 * m_{graphite} + 0.1654 * m_{lime} \quad (2)$$

$$\alpha = 0.4267 * m_{water} + 0.8618 * m_{graphite} + 0.9207 * m_{lime} \quad (3)$$

$$\rho = 0.7888 * m_{water} + 1.0638 * m_{graphite} + 1.0358 * m_{lime} \quad (4)$$

$$c = 0.3849 * m_{water} + 0.4513 * m_{graphite} + 0.1640 * m_{lime} \quad (5)$$

Equations (2), (3), (4) and (5) are used to calculate the value of thermal conductivity and diffusivity, in addition to specific mass and heat capacity, respectively, from the normalized mass fraction of each component, represented by "m_{water}", "$m_{graphite}$" and "m_{lime}" for water, graphite and lime, respectively. The mean squared error of each linear regression is 0.0006, 0.0090, 0.0054 and 0.0026, respectively. As can be seen from the Figure 11, it is important to highlight that the greater the amount of water used in the production of the samples, greater the number of voids in the solid material after the drying process. Thus, we can conclude that with more voids, the thermal conductivity of the material is reduced, acquiring characteristics of an insulating material, due to the influence of the air, as it is possible to observe in Figure 11 (a). It is also noted that the component that had the greatest impact in increasing thermal conductivity was graphite powder. This can also be seen in the coefficients that accompanies the graphite in Eq. (2). While the increase in the amount of matrix, in the analyzed range, will result in a decrease in conductivity. Analyzing the other thermo-physical properties, it was possible to observe that the thermal diffusivity and the specific mass tends to increase with the addition of graphite. Also, the greater the mass fraction of water in the production of the composite, the lower the values of these properties will be. With regard to specific heat capacity, the higher mass fraction of water and graphite provide higher values, while the amount of hydrated lime has a negative effect on heat capacity.

5. CONCLUSIONS

With the tests carried out it was possible to observe that the graphite powder increased the thermal conductivity of the materials based on hydrated lime. It was evident that for the proportions analyzed, the higher the mass fraction of

graphite, the greater the conductivity of the sample. Thus, considering the samples stable containing the largest mass of graphite (0.515 W/mK), an increase of approximately 134% in thermal conductivity was observed compared to the sample without the addition of graphite powder (0.220 W/mK). The largest mass fraction of graphite was 1/4 of the matrix mass, equivalent to 25 %. The same trend was seen for gypsum composites, with a maximum increase of 106 % being observed in the case of material with a greater amount of graphite powder, with thermal conductivity of 0.763 W/mK, since pure gypsum, with the same amount of water in the production resulted in a thermal conductivity of 0.370 W/mK. It was also possible to observe the influence of water on the thermal conductivity of the manufactured materials, since, as the material dries and loses water, the conductivity decreases, and the newly produced samples and still containing a significant amount of water used in the mixture, presented thermal conductivity significantly higher than dry samples. Finally, through the response surfaces, it is possible to conclude that the addition of graphite has a significant influence on the specific mass, on the thermal diffusivity and mainly on the thermal conductivity of the composites. In addition, the amount of water used in the mixture has a negative effect on the values of diffusivity and thermal conductivity.

6. ACKNOWLEDGEMENTS

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7. REFERENCES

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