Materiales de Construcción
Vol. 71, Issue 344, October-December 2021, e260
ISSN-L: 0465-2746
https://doi.org/10.3989/mc.2021.03621

Evaluation of the influence of the degree of saturation, measuring time and use of a conductive paste on the determination of thermal conductivity of normal and lightweight concrete using the hot-wire method

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Received 22 February 2021
Accepted 21 June 2021
Available on line 24 August 2021

ABSTRACT: The determination of thermal conductivity of cement-based materials is relevant from the perspective of buildings' energy efficiency. The absence of unified tests for its measurement in mortars and concrete results in a heterogeneity of the data available in the literature. This work's purpose is to determine the relevant influence from a a statistical viewpoint that three factors; degree of saturation, measuring time and use of a conductive paste, have in the measurement of the conductivity using the hot-wire needle probe method in two concretes with different thermal behavior: standard-weight concrete and lightweight concrete. The results obtained allow for the establishment of recommendations for future researchers on the minimum information to be included in their reports of thermal conductivity of cement-based materials by the needle probe method, the need to treat outliers, the most favorable saturation conditions and measuring times, as well as the possible benefits of using conductive pastes.

KEYWORDS: Concrete; Physical properties; Characterization; Thermal conductivity.

Citation/Citar como: Revuelta, D.; García-Calvo, J.L.; Carballosa, P.; Pedrosa, F. (2021) Evaluation of the influence of the degree of saturation, measuring time and use of a conductive paste on the determination of thermal conductivity of normal and lightweight concrete using the hot-wire method. Mater. Construcc. 71 [344], e260. https://doi.org/10.3989/mc.2021.03621.

RESUMEN: *Evaluación de la influencia del grado de saturación, el tiempo de medición y el empleo de una pasta conductora en la determinación de la conductividad térmica en hormigones de peso normal y ligeros mediante el método del hilo caliente.* La determinación de la conductividad térmica en materiales cementicios es relevante desde el punto de vista de la eficiencia energética de edificios. La ausencia de ensayos unificados en morteros y hormigones da como resultado una heterogeneidad de los datos disponibles en la literatura. El objetivo del trabajo es determinar la influencia estadísticamente relevante que tres factores, grado de saturación, tiempo de medición y uso de una pasta conductora, tienen en la medición de la conductividad con aguja de hilo caliente en dos hormigones con diferente comportamiento térmico: hormigón de peso convencional y hormigón ligero. Los resultados permiten establecer recomendaciones para futuros investigadores sobre la información mínima a incluir en sus informes de conductividad térmica de materiales cementicios por el método de aguja de hilo caliente, la necesidad de tratar valores atípicos, las condiciones de saturación y tiempos de medición más favorables, así como posibles beneficios de utilizar una pasta conductora.

PALABRAS CLAVE: Hormigón; Propiedades físicas; Caracterización; Conductividad térmica.

1. INTRODUCTION

According to the European Union (1), heating and cooling accounts for half of the EU's energy consumption. Part of the EU's strategy to reduce energy bills fosters the improvement of façade enclosures' insulation capacity in buildings. Thus, thermal properties of cement-based products, as the principal constituent material of many enclosure elements, may play an important role in this strategy. Unfortunately, these properties "are the most overlooked and the least understood by the general concrete engineering and construction industry" (2), since until energy considerations began to be relevant, the thermal behavior of cement products was important only in a reduced number of applications, basically in the construction of large concrete volume elements (3). Due to this lack of need for data, a sizable amount of available databases and scientific literature on thermal properties display a shortfall homogeneity and even certain amounts of incongruity (4). Usually, the experimental conditions are vaguely described, and many values are quoted without any statement as to whether they correspond to single or multiple measurements. Furthermore, the lack of unified standard procedures worldwide leads to the data becoming overly scattered (5).

Among the thermal properties of mortars and concretes, thermal conductivity plays an important role since it is a measure of the material's ability to conduct heat. This is defined as the ratio of the heat flow rate versus the temperature gradient (6). It depends directly on the moisture content of concrete, the mineralogical composition of the constituents (mainly the aggregate part) and density. Although these influences have been qualitatively well-known from more than 50 years (7, 8), the quantification of the conductivity for concrete is especially problematic due to the major bearing of the constituents and the amount of free water in the pores. Traditionally, thermal conductivity has been reported as a function of the material density (9, 10), yet figures for this correlation often tend to be unreliable, particularly for masonry products (11, 12). Another reason for the wide data scattering is related to the large number of available testing methods for the determination of this property in general, and in building materials in particular. Table 1 lists some of the current standards, showing the large number of current methods; there are no specific tests defined for the determination of concrete's thermal conductivity.

The existing test methods can be divided into steady and transient tests. Among the steady tests, the guarded hot plate and the heat flow method are the most common. Both techniques involve placing flat solid samples between plates, one of these being heated. Samples are monitored until a steady state is reached. These methods were the first ones to be used and offer greatest levels of accuracy and comprehension. However, certain disadvantages are visible that hinder application (13-15): usually, to obtain representative results in non-homogeneous materials it is necessary to test sizable specimens that are difficult to handle; conductivity determination may take hours, since the steady state must be reached; as a result of the long duration of the tests, a redistribution of the water inside humid samples may occur, affecting the scattering of the results. Conversely, in order to ensure suitable heat transfer between the specimen and the plates, the flatness tolerance of the specimens is somewhat low for both sides of the slabs, which increases considerably the difficulty in readying the specimens. All these issues make these techniques difficult and expensive to apply for concrete, especially when a large number of tests is required, for instance for the evaluation of different constituents and dosages on the value of the thermal conductivity of the material.

With a view to overcoming these disadvantages, non-steady methods have been developed. Transient determinations are much swifter than steady-state tests; they do not alter the water profiles as significantly; sample sizes are usually not so large; and the measuring devices tend to be smaller than the steady-state apparatus. On the other hand, they tend to be less accurate (16-19). Among these techniques, the hot-wire method has been successfully used in different materials (16-19). Typically, a hot-wire probe consists of a needle with a heater and a temperature sensor inserted. A current passes through the heater and the system monitors the sensor's temperature over time. Analysis of the sensor temperature determines thermal conductivity. It was first used for liquids (20), but was rapidly adopted for the determination of the thermal conductivity in a wide range of solid materials with medium-low conductivities, including concretes (21, 22).

The magnitude and accuracy of the hot-wire measurements in concrete depend on several factors. The water content of the specimen affects the measurement, as with steady-state methods (7, 8). However, the data scattering should be reduced because of the reduced re-distribution of the water profile inside the specimen. Another expected factor affecting the hot-wire techniques is the measuring time (16), due to the thermal resistance existing between the heated sensor and the material to measure, called contact resistance. If the measuring time is overly short, the value of the thermal conductivity decreases owing to the influence of the contact resistance on the heat exchange. In order to reduce this effect, measuring device manufacturers usually recommend the application of thermal paste to the measuring needle (23).

In spite of all the above-mentioned issues mentioned (absence of a widely accepted standard, influence of several parameters), the authors of this work have been able to verify how, in many of the recent papers studying concrete's thermal properties, there is still a lack of information on the conditions under which the tests are carried out. This may partly explain the widespread dispersion and heterogeneity of results encountered. Demirboğa (24) and Gül (25) researched the influence of different mineral admixtures or lightweight aggregates on concrete. They report that the specimens were oven-dried and the brand of the measuring device, but they do not inform of the measuring time or the use of a conductive paste. The number of measures is not specified. In other papers by the same research group, Uysal, Demirboğa et al. (26) inform that the measuring time is 100 – 120 s. Ünal et al. (27) investigated the properties of low-strength lightweight concrete, yet they solely report the brand of the measuring device, without further information on testing conditions or number of samples measured. Mun (28) appraises the performance of lightweight aggregates, including thermal properties of concrete, though on the measuring device and the fact that concrete specimens were oven-dried are reported. Jerman et al. (29) investigated the thermal properties, among others, of aerated concretes. They measured concretes with different water contents and for different room temperatures, yet they do not report the measuring times, number of measurements or the use of conductive pastes. Collet and Pretot (30) carried out an investigation on the thermal conductivity of hemp concretes. They established a fixed measuring time of 120 s, and change the relative humidity of the pre-conditioning to evaluate its effect (dry point, 33%RH, 50%RH, 81%RH and 90%RH). The given thermal conductivity is the average of five values. They do not mention the use of a conductive medium. Chabannes et al. (31) investigated the use of aggregates from the sunflower stem for the manufacturing of building materials with some insulating abilities. They performed thermal measurements on three oven-dried specimens and at 35±5% RH, yet again they do not inform of the measuring time or the use of conductive pastes. Koçyigit et al. (32) studied mixtures of cement, tragacanth and pumice materials. Their measuring time is 100 – 120 s, the samples are dried at "room temperature (20±2°C)" and the results are the average of nine measures. They do not report the relative humidity at which the samples are stored or the use of a conductive paste. Seng et al. (33) characterized, among others, the thermal properties of hemp concretes, including the measurement of thermal conductivity by both the guarded hot plate and the hot-wire method. They carried out measurements on 18 samples, divided in wet and oven-dried specimens, but no information is provided on measuring times or the use of conductive pastes for the hot-wire method. Shafigh et al. (34) characterised the thermal properties of cement mortar in terms of its thermal conductivity, heat capacity and thermal diffusivity in a wide range of grades. They used the same equipment as in this paper, measuring three oven-dried samples per mix with measuring times of 10 minutes, and using a conductive paste. Anyway, the characterization of the thermal conduc-

Table 1. A selection of existing standards for the determination of different materials' thermal properties.

Standard	Method	Steady/Transient	Materials under the scope	Range ($W \cdot m^{-1} \cdot K^{-1}$)	Testing time
ASTM E1530	Guarded hot plate	Steady	Homogeneous opaque solid specimens of thickness less than 25 mm	0.1 – 30	Hours
ASTM C518	Heat flow	Steady	Flat slab specimens	< 10	Hours
ASTM C177	Guarded hot plate	Steady	Flat, homogeneous specimens	< 16	Hours
ASTM C1044	Guarded hot plate, single sided	Steady	Not specified	< 16	Hours
ISO 8301	Heat flow	Steady	Flat slab specimens		Hours
ISO 8302	Guarded hot plate	Steady	Flat slab specimens	< 10	Hours
EN 12664	Guarded hot plate/ heat flow	Steady	Dry and wet building materials with low and medium thermal resistance	< 10	Hours
EN 12667	Guarded hot plate/ heat flow	Steady	Dry building materials with high and medium thermal resistance	< 2	Hours
ASTM C1113	Hot wire	Transient	Non-carbonaceous, dielectric refractories	< 15	Minutes
ASTM D5334	Hot wire	Transient	Soil and soft rocks		Minutes
ISO 8894-1	Hot wire	Transient	Non-carbonaceous, dielectric refractory products and materials	< 15	Minutes
ISO 22007-2	Transient plane source	Transient	Plastics	0.01 - 500	Minutes

Materiales de Construcción 71 (344), October-December 2021, e260. ISSN-L: 0465-2746. https://doi.org/10.3989/mc.2021.03621.

tivity is also important in other construction materials, such as gypsum (35), masonry materials (36) and even alkali activated materials (37).

The scope of this research is to assess the influence of three important parameters, pre-conditioning of the specimens, measuring time, and the use of a conductive paste, on the measuring of the thermal conductivity with a particular hot-wire transient test method, the thermal needle probe. Thermal conductivity was determined in two different concretes, normal-weight concrete and lightweight concrete, in order to issue recommendations for researchers on the minimum information to include when reporting the results of the determination of this property by using that specific method.

2. MATERIALS AND METHODS

2.1. Materials and Mix Proportions

Two different concrete mixes were designed to evaluate the influence of the three parameters mentioned before by means of a hot-wire thermal probe. In order to ensure that the measured data belonged to clearly distinct populations, a normal-weight concrete mix (NW) and a lightweight concrete mix (LW) were used. Both mixes were batched and blended using the same basic components: a CEM I 42.5R (EN 197-1), a siliceous gravel (4/12 mm) and a siliceous sand (0/4 mm). LW concrete was achieved by means of an air-entraining agent. Air entrainment reduces the thermal conductivity (2, 7, 8). No other admixtures were used. Table 2 shows the nominal compositions and the real compositions after considering the air inclusion.

2.2. Test Methods

Two batches were prepared, one of NW and another of LW. Air content was determined in the fresh state (EN 12350-7). From each batch, twelve 150 mm side cubic specimens (EN 12390-1) were fabricated according to (EN 12390-2) and stored under water at 20±2°C for at least 90 days, in order to neglect the influence of continuous hydration in the thermal properties. For control purposes, compressive strength was determined (EN 12390-3) on two specimens for each batch after 90 days' curing.

In order to measure thermal conductivity by means of a needle probe, a series of pilot-pins were inserted into ten of the specimens from each batch in the fresh state, just immediately after the troweling of the specimens (Figure 1). Pins were removed after concrete hardened. The pilot-pins were 10 cm long and 2.4 mm in diameter, equal to the measuring needle, to allow for the introduction of the probe in the hardened state. A total of 25 measuring points were molded for each set of ten concrete cubic specimens, five specimens with two measuring points and five others with three points.

Table 2. Nominal and real composition after air correction of the fabricated concretes (kg/m^3).

	Normal-weight concrete (NW)		Lightweight concrete (LW)	
	Nominal	Real	Nominal	Real
Water	163	160	195	160
CEM I 42.5R	450	444	450	368
Coarse aggregate (4/12)	918	905	863	706
Sand (0/4)	900	888	847	693
Air-entraining agent	--	--	9	7
Air content (%)		1%		22%
Density (g/cm^3), water-saturated		2.40		1.84
90 days compressive strength (MPa)	59.1		10.2	

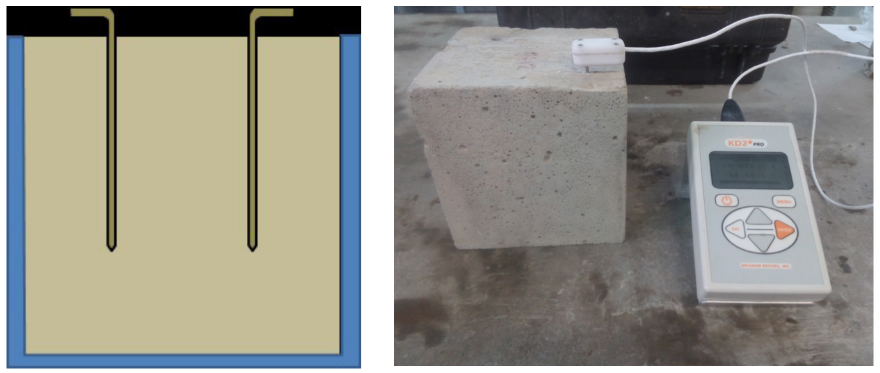

Figure 1. Left: Pilot-pin disposition during casting; Right: Measurement at room conditions.

Thermal conductivity was measured using the hot-wire needle method by means of a Decagon KD2 Pro Thermal Properties Analyzer (23) equipped with a TR-1 probe (2.4 mm x 10 cm long, range 0.1 to 4.0 W/m·K, accuracy ±10% from 0.2 to 4.0 W/m·K, ±0.02 from 0.1 to 0.2 W/m·K). The probe is heated at a constant rate for a predetermined time, while the temperature in the needle is monitored. Heating stops for the same period of time, and the cycle is applied several times depending on the instrument's settings. Thermal conductivity can be computed from the acquired data (38). The longer the measuring time, the larger the data collected and the accuracy of the prediction. The apparatus provides an indication of the goodness of fit of the model to the data (39) by means of the dimensionless value *err*. If the *err* value is quite large (> 0.0100), this can be an indication of issues usually arising from contact resistance between the needle and the material, and rejection of the value must be considered. However, this is only a qualitative indicator that must be carefully interpreted by the operator of the equipment, and rejection of data must be done only after enough indications of misreading are collected.

Thermal conductivity *k* was first measured in three different saturation conditions for each set of ten specimens and concrete type, NW and LW, without the utilization of any thermal paste to prevent contact resistance. Firstly, the specimens were measured in a fully saturated state immediately after removing from the water tank at room temperature. After completion of all the measurements on a specimen, it was left to dry at room temperature (20±2°C, 50±5% RH) up to constant mass (Dm < 0,1%) and *k* was measured again at each point. Finally, the specimens were oven-dried at 70±5°C up to constant mass again, with thermal conductivity being determined once more. For each measuring point and saturation degree, *k* was determined for three different measuring times allowed by the device's setting menu: 2 min, 5 min and 10 min.

Eventually, thermal conductivity was measured again, but this time using two different thermal pastes, a ceramic and a silver compound, whose purpose was to reduce contact resistance between the measuring probe and the material, improving the accuracy and precision of the measurement. The pastes were tested versus three different saturation conditions and three measuring times, meaning the specimens were again submerged in the water tank until constant mass, then dried at room temperature and finally oven-dried. On this occasion, the measurements could not be carried out with a single type of paste in all the measuring holes for each set of ten specimens, since once the hole is filled with thermal grease, the substance cannot be removed and all subsequent measurements on that point are considered to be influenced with that type of thermal paste. Furthermore, since the tolerance between the hole and the measuring probe is so tight, in some cases the needle did not fit again once the paste was introduced. Measuring points were then randomized for each type of paste.

In this regard, a total of 774 measurements of the thermal conductivity for different combinations of type of concrete, saturation degree and measuring time and thermal paste were obtained. Once all the

Table 3. Dimension of the mixed linear models used for each type of concrete.

Model Dimension				
		Number of Levels	Covariance Structure	Number of Parameters
Fixed Effects	Intercept	1		1
	Saturation degree	3		2
	Measuring time	3		2
	Thermal grease	3		2
	Saturation * Thermal grease	9		4
	Saturation * Measuring time	9		4
	Thermal grease * Measuring time	9		4
	Saturation * Grease * Time	27		8
Random Effects	Specimen	10	Variance Components	1
Residual				1
Total		74		29

Dependent Variable: thermal conductivity *k* (W/m·K).

measurements were performed, data was analyzed using the statistical analysis software IBM® SPSS® Statistics. In addition to the descriptive statistics for each set of measurements, a linear mixed-effect model (40-42) was used to fit the collected data in order to consider both fixed and random effects. Both types of concretes were analyzed separately, since it can be safely assumed that both populations are statistically independent because of the differences in thermal conductivity between both mixes. The saturation degree, use of thermal grease and measuring time are considered as fixed effects, while the specimen where measurements take place is assumed to be a random effect. Table 3 shows the dimensions of the mixed linear model for each concrete and effect discussed later, including the interactions between the effects.

3. RESULTS AND DISCUSSION

3.1. Compressive Strength

The result of compressive strength after 90 days of curing is shown in Table 2 solely for the purpose of identifying the two concrete types. NW concrete reached more than 59 MPa of strength. As was expected, the inclusion of air in the LW concrete reduces significantly the strength of concrete at the approximate ratio of a 4% drop in strength for each 1% additional content of air, which acts accordingly with the rule-of-thumb usually employed for the effect of air-entraining on strength (6).

3.2. Statistical Tests for Normality

Mixed linear models assume that the response variables follow a Gaussian distribution. This section investigates the normality of the obtained data.

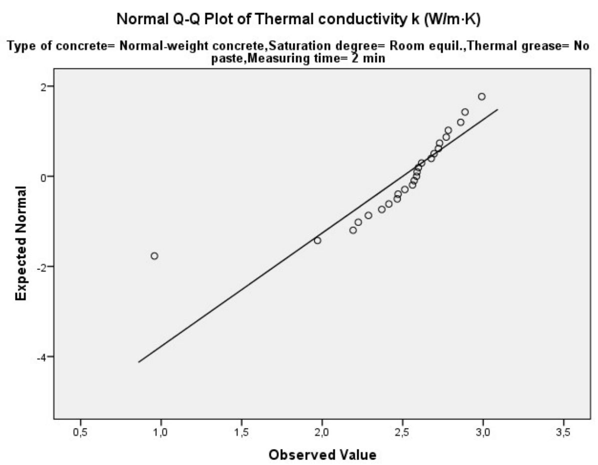

Figure 2. Q-Q plot of the 25 results for normal-weight concrete, concrete at equilibrium with the room conditions (T = 20±2°C, RH = 50±5%), no thermal grease applied within the measuring holes, measuring time = 2 min.

Estimates for the skewness and kurtosis were calculated for each sample and combination of effects, as well as the p-values obtained in the Saphiro-Wilk (S-W) test for normality (43). From the 27 data sets for NW resulting from the combination of effects, 9 of them failed both tests simultaneously, and 1 failed the skewness and kurtosis test, although with a S-W test p-value somewhat close to the limit ($p = 0.051$). For LW, only 3 out of 27 combinations failed both tests simultaneously.

These results might instigate certain doubts regarding the data's normality. However, a more in-depth examination of the data rapidly revealed that the loss of normality is strongly influenced by the presence of possible outliers. First of all, skewness and kurtosis did not show any clear trend that could prove an indication of another distribution, since both parameters displayed quite random occurrences of positive and negative values, somewhat homogeneously distributed around zero.

Q-Q plots (44) were drawn for all the 12 data sets that did not pass the normality tests. Figure 2 shows the Q-Q plot for the 25 thermal conductivity values measured in the NW specimens, when stored at room temperature, without thermal grease and for a measuring time of 2 min. This case is included due to its representative nature. According to the testing results, data distribution is rather removed from normality. The estimated skewness and kurtosis were 5.55 and 10.16 times their respective standard errors, and the p-value of the S-W test is 0.000. Tests for outliers identify only one possible outlier datum out of 25 points. Nonetheless, visual observation of Figure 2 quickly reveals that this single outlier is the reason of the deviation of the data points from the straight line drawn in the Q-Q plot and that indicates normality. If this value identified as the outlier is removed, the estimated skewness of the remaining 24 data points is merely 1.21 times its standard error, the kurtosis is 0.44 times, and the p-value of the S-W test becomes 0.812.

This conclusion was repeated for the rest of cases where doubts regarding normality existed, their analyses not shown herein due to space limitations. Inclusion or rejection of outliers in the subsequent analysis, always a compromised question, is discussed in the next section. In any case the authors estimate that, even before screening possible outliers out of the picture, it can be assumed the hypothesis that the measurement of the thermal conductivity in the two types of concrete follows quite reasonably a normal distribution, which makes possible the subsequent use of mixed linear models, since deviation from normality is mainly due to the presence of outliers, while the rest of the data closely follows a Gaussian distribution.

3.3. Outliers

According to the test proposed by Tukey (45), an observation is considered as outlier if its value is 1.5 times out the interquartile range of its data set, and as far-out value if it is 3 times out. For NW concrete, 18 measurements (4.9%) out of 369 ones were initially labelled as outliers and 7 as far-out points (1.9%). For LW concrete, 8 points (2.0%) were suspected to be outliers and 3 (0.7%) far-out measurements out of 405 points.

Since this number of outliers was considered high, actions were taken in order to investigate the possible existence of side effects affecting the compromised measurements. The first action was to repeat the measurements in order to confirm the readings whenever possible. Since some of the holes were already impregnated with a type of thermal grease, it was not possible to restore the conditions for some of the no-paste cases where outliers were detected. In any event, the second set of readings confirmed the first measurements.

The decision to keep or discard the data was then taken with the help of the *err* function given by the Decagon KD2 Pro Thermal Properties Analyzer described in Section 2.2. It was decided that if, for a single measurement detected out of the Tukey's fences, the *err* function was above 0.01, the value would be discarded. In this regard, for NW concrete 3 measurements labelled as outliers and 6 suspected far-out points were rejected, while for LW concrete 2 outliers and 1 far-out point were not considered for further analysis. Table 4 shows the number of valid measurements finally considered for each combination of effects.

It is noteworthy that the number of total measurements outside Tukey's boundaries is larger for NW concrete than for LW concrete, even after removing probable erroneous measurements (16 versus 8). Although there is not statistical evidence to support this assumption, since the investigation submitted in this paper did not contemplate the repetition of batches for each type of concrete which could confirm this observation, a possible explanation is that, for NW concrete, the recorded thermal conductivity is closer to the upper limit of the measuring range of the probe (4.0 W/m·K), which could have a bearing on the appropriacy of the fit.

3.4. Mixed Linear Model

Table 4 shows the main descriptive statistics for the full experimental program: means, standard deviations and number of valid cases for each combination of factors and type of concrete. Table 5 shows the results of the F-tests for the main three fixed effects analyzed in this paper for both NW and LW: saturation degree, use of conductive paste and measuring time, as well as their 2 and 3-ways interactions, when considering 762 thermal conductivity measures after discarding 12 outliers.

According to the model, all three main fixed effects are significant at the 0.05 level for both types of concrete (significance $p < 0.05$); therefore, they are potentially significant predictors of the dependent variable thermal conductivity. When considered individually, ignoring all other variables, degree of saturation (NW: $F = 525.25$; LW: $F = 481.73$) and measuring time (NW: $F = 424.13$; LW: $F = 287.42$) have a much greater effect than the conductive paste (NW: $F = 8.93$; LW: $F = 61.17$) since their F-values are much higher than the F-value of the thermal grease effect.

Figure 3 shows the graphs of the overall means for each main effect. Visual analysis confirms the assumption that both concretes belong to different populations: LW clearly shows lower values of thermal conductivity than NW under the same conditions, as expected.

As reported previously with steady-state methods (7, 8), the amount of water within the concrete increases the value of the measured thermal conductivity for NW (mean k values of 2.683, 2.989 and 3.588 W/m·K for oven-dry, room and saturated conditions, respectively). For LW, there is no significant change between oven-dry conditions (1.404 W/m·K) and room conditions (1.326 W/m·K), although thermal conductivity also increases when the specimens are saturated (1.800 W/m·K), but the rate of change is smaller than the NW. A possible explanation is that, since the entraining of air in LW produces a large number of air pores without connectivity, most of the accessible water is already removed at room temperature, but even the most energetic drying does not remove an appreciable amount of trapped water within the non-connected pores.

Regarding the measuring time, an increase in time significantly increases the value of the thermal conductivity measurements for both concrete types (mean k values of 2.610, 3.201 and 3.462 W/m·K for 2 min, 5 min and 10 min and NW, respectively; and 1.248, 1.549 and 1.728 W/m·K for 2 min, 5 min and 10 min and LW, respectively), as reported in the state-of-the-art (16). This phenomenon is explained by the contact resistance effect, which can be significant for reduced measuring times. Indeed, for both concretes change rate of thermal conductivity measurements is greater between 2 and 5 minutes (23% and 24% for NW and LW concrete, respectively) than between 5 and 10 minutes (8% for NW concrete and 12% for LW concrete).

The use of conductive pastes is recommended by the manufacturer of the equipment in order to reduce the influence of this effect. However, as it has already been mentioned, the influence of this factor considered individually is significantly lower than the degree of saturation or the measuring time. More

TABLE 4. Descriptive statistics for the full experimental program, normal-weight (NW) and lightweight concrete (LW).

			Measuring time					
			2 min		5 min		10 min	
			Thermal conductivity *k* (W/m·K)					
Saturation state	Thermal grease		NW	LW	NW	LW	NW	LW
Oven-dry	No paste	Mean	2.071	1.141	2.688	1.592	3.056	2.078
		Standard Deviation	.294	.100	.173	.110	.273	.092
		Valid N	25	25	25	25	25	25
	Ceramic	Mean	2.464	.979	3.007	1.184	3.373	1.291
		Standard Deviation	.566	.241	.203	.177	.430	.119
		Valid N	10	10	10	10	10	10
	Silver	Mean	2.299	.901	2.962	1.208	3.157	1.364
		Standard Deviation	.144	.192	.600	.149	.552	.150
		Valid N	5	10	5	10	5	10
Room equilibrium (50±5% RH)	No paste	Mean	2.500	1.059	3.360	1.458	3.501	1.497
		Standard Deviation	.398	.303	.132	.137	.182	.177
		Valid N	25	25	25	25	25	25
	Ceramic	Mean	2.193	1.098	2.859	1.329	3.264	1.444
		Standard Deviation	.315	.255	.161	.199	.211	.142
		Valid N	9	10	9	10	9	10
	Silver	Mean	2.347	1.066	2.686	1.358	3.071	1.475
		Standard Deviation	.246	.192	.297	.152	.175	.131
		Valid N	8	10	8	10	8	10
Saturated	No paste	Mean	3.148	1.593	3.617	1.790	3.911	1.929
		Standard Deviation	.287	.206	.129	.156	.226	.120
		Valid N	25	25	25	25	25	25
	Ceramic	Mean	3.117	1.655	3.778	1.850	4.035	1.996
		Standard Deviation	.288	.238	.199	.192	.183	.135
		Valid N	10	10	10	10	10	10
	Silver	Mean	3.244	1.576	3.663	1.874	3.895	2.001
		Standard Deviation	.104	.138	.055	.140	.200	.106
		Valid N	6	10	6	10	6	10

detailed observation of the lower graph in Figure 3 shows that the rate of change of the measurements when using no paste, with a ceramic conductive paste and with a silver paste is small (mean *k* values of 3.110, 3.112 and 2.991 W/m·K for NW, respectively, and 1.579, 1.425 and 1.425 W/m·K for LW), not taking into account its interaction with the other factors. Table 6 shows the estimates of the mixed model for the fixed main effect thermal paste and the results of the t-tests when inter-comparing the three levels of the effect. While the overall F-value for the use of a thermal paste was statistically significant, the t-tests between levels are statistically non-significant ($p > 0.05$) with these confirming the visual observation for both types of concrete which states that the use of a thermal paste in overall terms does not entail a significant change to the *k* value .

Regarding double interactions, for NW concrete, the double interaction saturation degree*thermal grease ($F = 35.375$) and grease*measuring time ($F = 4.124$) are significant at the 0.05 level, while saturation*time ($F = 1.600$) is not statistically relevant, meaning no interaction. For LW concrete, the three double interactions (saturation*grease $F = 59.471$; saturation*time $F = 7.077$; grease*time $F = 7.770$) are potentially significant.

For both concretes, the interaction factor between the saturation degree and the use of a thermal grease has more bearing in the model (F-value is larger). Figure 4 shows the graph of this final interaction between the saturation degree and the use of a thermal paste (upper) for the two concretes studied. The interaction is complex, as it is difficult to comprehend the effect of each principle on the combination of

TABLE 5. Type III tests of fixed effects (46), normal-weight concrete (NW) and lightweight concrete (LW).

Source	Numerator df	Denominator df		F-test value		Significance	
		NW	LW	NW	LW	NW	LW
Intercept	1	9.834	9.183	11305.053	3102.607	.000	.000
Saturation	2	324.886	365.951	525.250	481.733	.000	.000
Thermal grease	2	326.493	366.180	8.930	61.165	.000	.000
Measuring time	2	323.901	365.950	424.132	287.421	.000	.000
Saturation*Grease	4	324.560	365.951	35.375	59.471	.000	.000
Saturation*Time	4	323.888	365.951	1.600	7.077	.174	.000
Grease*Time	4	323.931	365.951	4.124	7.770	.003	.000
Saturation*Grease*Time	8	323.909	365.951	2.005	8.499	.045	.000

Dependent Variable: Thermal conductivity *k* (W/m·K)

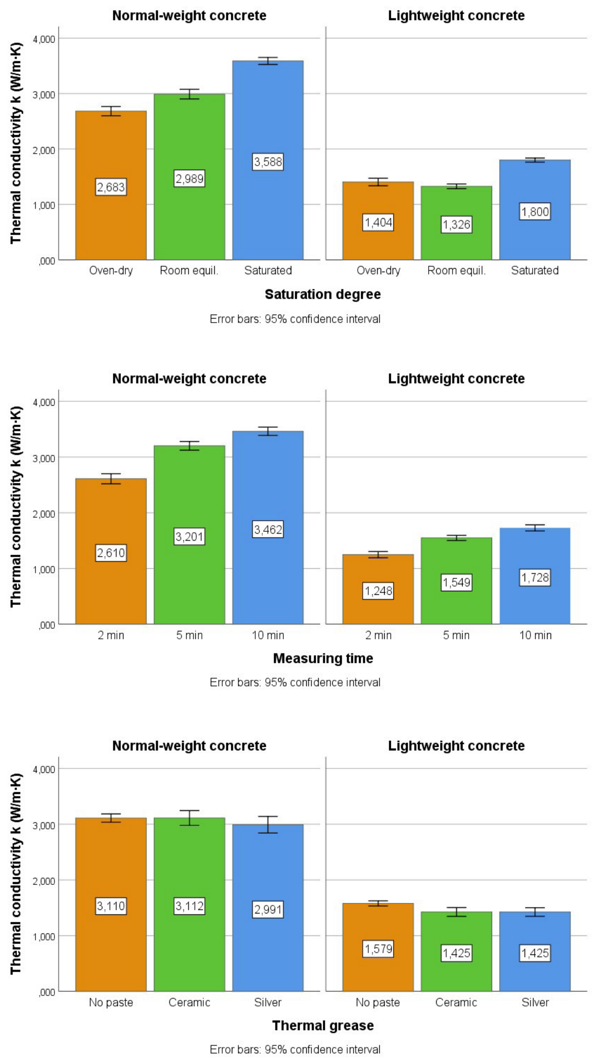

FIGURE 3. Overall means for the three main effects: saturation degree (upper), measuring time (middle) and thermal grease (lower), for normal-weight concrete and lightweight concrete.

both of them. For NW concrete, the no-paste case shows coherent results regarding its saturation state: when increasing the water content, thermal conductivity increases as expected. The use of conductive pastes, contrary to what might be expected, increases the variability of the measurements when considering their interaction with the saturation degree (the 95% confidence interval of the means is wider). Although the use of pastes does not seem to have an effect on the NW concrete when saturated (the green line is approximately flat), the use of conductive pastes results in thermal conductivity values on dry specimens similar to those values of concrete at room equilibrium (RH = 50%).

With regard to LW concrete, the variability of the measurements using a thermal paste also increases when considering its interaction with the degree of saturation, as in the case of NW concrete, albeit to a lesser extent. Similarly, the use of paste has no effect when the concrete is saturated (green flat line). However, when the decision is made not to use conductive pastes, the conductivity results of LW concrete are not as would be expected, since although the test specimens with higher water content do show a higher conductivity, the oven-dried specimens show a significantly higher conductivity than the specimens in equilibrium with the laboratory, with values close to those of the saturated test specimens, which is not consistent. These apparently false readings for LW concrete are corrected with the use of conductive pastes, in which the conductivity increases as the degree of saturation increases. Furthermore, as observed when analyzing the fixed factors individually, the conductivity values for oven-dry concrete and concrete in equilibrium with the laboratory are similar. Both conductive pastes, ceramic and silver, behave similarly without significant differences between them.

Figure 4 also shows the interaction graph between the saturation degree and the measuring time (middle) for the two concretes studied. As it was observed when considering the effect of the measuring time individually, an increase in the measurement time produces an increase in the measured value of thermal conductivity for all saturation states in both concretes. Increasing the measuring time does not significantly affect results' variability (error bars are approximately the same width). The graph confirms the absence of interaction for the NW concrete detected in the F-test (the three lines are quite parallel). In the case of LW concrete, there is no interaction from 2 minutes to 5 minutes between the measurement time and the degree of saturation, nor does it appear that there is interaction between the measuring time in saturated specimens and in specimens at room equilibrium. The interaction detected by the model is due to the results obtained with 10 minutes of measurement on oven-dried specimens that, contrary to what should be expected, present thermal conductivity values higher than those of the specimens in equilibrium with the laboratory, which should have a higher water content. This result is probably due to the interaction between the conductive pastes and the degree of saturation previously analyzed, which, as seen before, has a greater influence than the interaction between time and degree of saturation and may be influencing the means and confusing the graphs. This remark is supported by observation of the results for each combination of factors in Table 4. The ratio between the mean thermal conductivity of the oven-dry/no-paste/10-minutes readings and the room-equilibrium/no-paste/10-minutes case is 1.38, a strong deviation from the expected relationship, while the ratio between the oven-dry/ceramic-paste/10-minutes and the room-equilibrium/ceramic-paste/10-minutes measurements is 0.89, the foregoing being consistent with what should be expected.

Figure 4 also displays the interaction graph between the measuring time and the use of a thermal conductive paste (lower) for the two concretes studied. Again, the use of conductive pastes increases the variability of the measurements when considering their interaction with the measuring time for both types of concrete. The interaction is complex, but for both concretes, there seems to be no double effect between the use of conductive pastes and 5 and 10 minutes measuring times (green and red lines are parallel). The interaction in the conductivity results is caused by the 2 minutes measuring time. In addition, the results at 2 minutes are clearly inferior to those of 5 and 10 minutes, which is consistent with the greater measurement error expected for short times.

Regarding the 3-ways interaction, it is on the verge of significance for NW concrete, while for LW concrete it could be of relevance. Due to the complexity of physical interpretation of the 3-ways interaction and the relatively low weight in the model (F-values of 2.005 and 8.499), it is considered that this interaction is of no practical interest.

Finally, Table 7 shows the estimates of the covariance parameters between the thermal conductivity measurements and the specimens. Since the variance for both types of concrete is close to zero, it can be assumed that there was no linear relationship between the test specimens and the measurements of thermal conductivity.

Table 6. Estimates of fixed effect thermal paste.

Parameter	Estimate	Std. Error	df	t-test value	Significance	95% Confidence Interval Lower Bound	95% Confidence Interval Upper Bound
Normal-weight concrete (NW)							
Intercept	3.885701	0.081404	249.362	47.734	0.000	3.725375	4.046027
No paste to silver paste	-0.008129	0.086022	324.527	-0.094	0.925	-0.177360	0.161103
Ceramic paste to silver paste	0.149299	0.097338	324.594	1.534	0.126	-0.042194	0.340792
Lightweight concrete (LW)							
Intercept	2.000600	0.050938	105.786	39.275	0.000	1.899608	2.101592
No paste to silver paste	-0.065544	0.052264	365.986	-1.254	0.211	-0.168320	0.037232
Ceramic paste to silver paste	-0.005000	0.062447	365.948	-0.080	0.936	-0.127800	0.117800

Dependent Variable: Thermal conductivity *k* (W/m·K).

Table 7. Estimates of covariance parameters.

Parameter		Estimate NW	Estimate LW	Std. Error NW	Std. Error LW
Residual		0.035285	0.019498	0.002773	0.001441
Specimen	Variance	0.006804	0.006449	0.003733	0.003281

Dependent Variable: Thermal conductivity *k* (W/m·K)

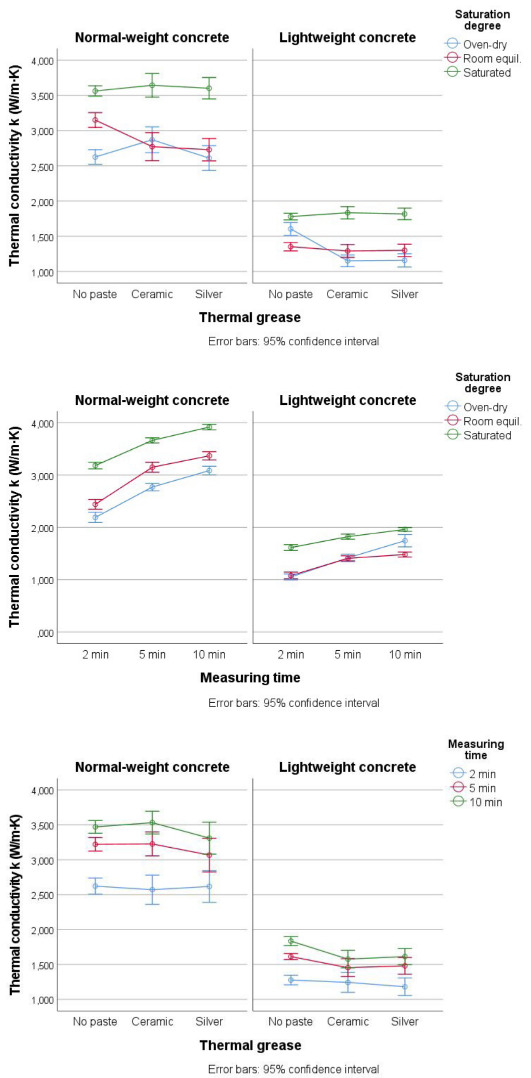

Figure 4. Graphs of the saturation degree*thermal grease interaction (upper), saturation degree*measuring time interaction (middle) and measuring time*thermal grease interaction (lower) for normal-weight concrete and lightweight concrete.

4. CONCLUSIONS

Thermal conductivity data available in the state-of-the-art unveiled a lack of information on the conditions under which the hot-wire tests are carried out. This paper has studied the statistically relevant influence that three factors, the degree of water content, the measuring time and the use of conductive pastes to reduce the contact resistance effect, have on the determination of thermal conductivity by a particular hot-wire transient test method, the thermal needle probe, on two types of concrete with differentiated thermal characteristics: a normal-weight concrete (NW) and a lightweight concrete (LW). Thermal conductivity *k* was measured in three different saturation conditions (oven-dry, equilibrium at room conditions (50±5% RH) and saturated), three measuring times (2, 5 and 10 minutes), using two different conductive pastes (ceramic and silver) and with no paste.

Normality tests allows to conclude that the thermal conductivity measurements by the thermal needle probe method follow a normal distribution in most cases. In those cases where the response moves away from normality, a more exhaustive analysis shows that the presence of outliers is the cause of this non-normality. The appearance of a considerable number of outliers beyond 1.5 times the interquartile range (6.8% for NW and 2.7% for LW, when the probability that a result lies outside the range 1.5 x IQR is 1%) is precisely one of the relevant conclusions of this study. The presence of an abnormally large number of outlier values is largely due to the lack of fit of the heating-cooling curves during the measurement associated to contact resistance problems. Therefore, initial recommendation the authors would make to researchers who use the hot-wire needle probe method for the determination of the thermal conductivity of concrete and mortars is that, prior to the analysis of the results, a study on the number of outliers should be carried out, discarding those results in which it is suspected that the effect of contact resistance may exist. The number of discarded values should be reported in the paper.

The three main effects, when analyzed individually, are statistically significant for both types of concrete with a significance level of 95%, although the degree of saturation and the measuring time have a significantly much greater effect than the conductive paste. Therefore, it is recommended that researchers' work who report on thermal conductivity values for mortars and concretes using the needle probe method, include in their papers at least the degree of saturation of the specimens on which the measurement is performed and the measuring time used for each measurement.

The amount of water within the material significantly increases the value of the measured thermal conductivity. Saturated specimens showed a much more consistent behavior both considered individually or in combination with the rest of the effects. While for NW concrete clear differences between conductivity values on oven-dry, room equilibrium and saturated specimens were observed, for LW concrete only saturated specimens showed significant differences to oven-dry and specimens at room equilibrium. In addition, the use of a conductive paste has no effect on the values measured on saturated specimens. Thus, for the sake of comparison between different mixtures or between cement-based materials with marked differences in their thermal properties, it is recommended that thermal conductivity measures should be made with saturated materials.

Increasing the measuring time significantly boosts the measured thermal conductivity, regardless of the saturation state of the concrete for both NW and LW, with the exception of LW oven-dry specimens, although this difference is rather attributed to the interaction with the presence or absence of conductive paste. The difference is more marked in short measuring times (2 minutes), while less noticeable for longer times (between 5 or 10 minutes), thus meaning it is recommended that the minimum measuring time should be at least 5 minutes.

Regarding the use of conductive pastes to reduce the contact resistance effect, as recommended by the manufacturer of the measuring equipment, the two pastes tested in this study, ceramic and silver, showed similar behavioral trends. Contrary to what would be expected, the use of the paste increases the variability of the results. For NW concrete (i.e. for cement based materials with high thermal conductivity) it was observed that the use of a conductive paste does not entail any appreciable benefit. Nor does the use of paste also display significant influence for high measurement times. However, the use of a conductive paste would seem advisable if researchers wish to measure thermal conductivities on oven-dried concretes or mortars with low conductivity such as LW concrete, since not using paste resulted in non-coherent measures.

Finally, the analysis confirmed the non-dependence of the thermal conductivity result with the specimen on which the measurement is taken.

In summary, from the analysis of the statistical significance of the influence of three factors, the degree of saturation, the measurement time and the use of a conductive paste, on the results of thermal conductivity through the use of the hot-wire needle probe method on two concretes of high and low conductivity, the following recommendations can be established for researchers who employ said method:

- Due to the effects attributable to the measurement method, one would expect to obtain a number of outliers higher than statistically accepted. Therefore, it is advisable to perform background searches into these values and analyze their inclusion or discard in the subsequent analysis, reporting this fact.

- The results report should include, at least, the saturation degree of the specimens, as well as the measuring times used.

- The use of saturated specimens is recommended, as they provide more consistent measures of the thermal conductivity.

- The use of a minimum measuring time of 5 minutes is recommended.

- The use of conductive pastes does not improve the procedure, except for dry cement-based materials with low thermal conductivity.

ACKNOWLEDGEMENTS

Authors gratefully acknowledge A. Fernández-Escandón and J.C. Porras for their help in the experimental work. They are also grateful to SIKA, S.A., for supplying some of the raw materials used in this study.

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Materiales de Construcción 71 (344), October-December 2021, e260. ISSN-L: 0465-2746. https://doi.org/10.3989/mc.2021.03621.