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Research article

An experimental and analytical study of the effect of cold compression

on the thermophysical properties of a granular medium

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Abstract: Based on the previous literature, very few models have described the thermal behavior of granular media or powders as a function of the mechanical stresses to which they are subjected. In recent years, many researchers have been interested in establishing laws that can express the relationship between the apparent thermal conductivity and the mechanical behavior of granular media. The present paper seeks to present a simple model that describes the variation of the apparent thermal conductivity of a granular medium as a function of the mechanical stress. One of the main objectives of this paper is to produce a tool for calculating the thermal conductivity of heterogeneous media, especially that of granular media. For the resolution of the problem, it was decided to use an experimental method recently developed in the laboratory and which is due to be the basis of our calculations. This method is called the hot rod method. It was initially developed to evaluate the damage to a soil subject to superficial heat shock (fires, burns). The results show that for short times and a distance between two measurement points large enough, the 2D transfer can be reduced to a 1D transfer which for long times is hybridized to a hot wire transfer. The modeling of thermal transfers within the environment makes it possible to know the temperature field of soil under the effect of a thermal accident.

Keywords: granular media; mechanical stresses; transmission factor; thermal conductivity

1. Introduction

Very large quantities of granular materials are used daily by man, particularly in the field of geophysics, but also in the industrial sectors of building, civil engineering, pharmaceutical industry, food industry, etc. In all these sectors, the problems of handling granular media, such as the flow of powders through orifices or compression, still arise [1].

The compression of granular media is a subject that has been studied for many years. Several empirical laws related to the topic have been developed and allowed obtaining reliable results. However, their validity areas are not accurate because the parameters used are not or are insufficiently related to the parameters characterizing the granular medium (particle size distribution, mechanical properties of materials, surface condition, etc.) [2]. Most often, these parameters are adjusted based on experimental achievements. Therefore, appropriate and relevant methods must be developed to better determine these compression parameters [3].

The thermal characterization of granular media has been the subject of many studies which, in all cases, were interested in developing accurate measurement probes for determining the thermal properties of granular media, and generally of soils. At this stage, some studies [4] that used probes for measuring soil temperature are cited as examples. The principle of measuring the temperature of many media is based on the implantation of probes at different depths in the medium under study [5]. The realization of these measurements requires the use of supports to minimize the effects of shear stresses on the probe. The supports used are generally made of metal that is very conductive, which induces certain thermal disturbances at different depths of the medium [6]. These last observations prompted us to consider these disturbances in the development of our method.

The hot rod method is advantageous because it allows representing the thermal conductivity as a function of depth. We were inspired by this method to develop a new device for measuring the thermal conductivity in a granular medium subjected to mechanical stress. The peculiarity of measuring the thermal conductivity as a function of depth was kept but was improved by taking into consideration the evolution of thermal conductivity as a function of the mechanical constraints [7–11]. Several researchers have developed theoretical and experimental methods to express or determine the thermophysical properties of material media [12–17]. The main advantage of this new method is that it allows following the evolution of the thermal conductivity as a function of the mechanical constraints and depth [18].

This paper aims to position the problem in a general context, which is described in the first part; in particular, it exposes the semi-numerical modeling of the problem. Its modeling encourages us to treat two cases of heat transfer within the studied medium, namely two-dimensional transfer and onedimensional transfer. An estimation of the thermal conductivity for both cases is investigated later. The study of the sensitivity parameter is also discussed in this section. The conclusions drawn from this last study allow us to simplify the calculation of the estimates of the thermal conductivity of the medium under study. The second part is devoted to the validation of the simulation process. In this sense, this part presents an estimation of the mechanical constraints as a function of depth. This estimate is obtained from simple measurements of the apparent thermal conductivity of the medium under study.

2. Methodology

Thermal transfer modeling within the system is achieved through an extension of the thermal quadrupole method to stratified heterogeneous media (the medium is considered as a set of juxtaposed layers). The idea is to associate a numerical resolution in a certain direction in space (z) to a Laplace transform on the temporal variable. The resulting algebraic system is analytically resolved with respect to the remaining direction, which is assumed to be the depth. Transfer matrices connecting the temperature fields to heat fluxes at different depths are therefore obtained; they are expressed as matrix functions. The model was validated through experiments which were carried out with an experimental device developed in the laboratory.

The experimental device, which consists of a rod probe, is shown in Figure 1. For this reason, the method was called the "Hot rod method". This rod is composed of several thermocouples, a resistive wire, a central wooden rod and a resin layer. A regulated power supply provides a voltage step across the resistive heating wire. The regular winding of this wire around the wooden rod allows us to consider that the flow of heat produced is uniform over the entire probe surface. It is important that the distance between two thermocouples is significant. As it is subsequently explained in this chapter, this distance plays an essential role in our experimentation. The thermocouples are of type K, and the junction between two thermocouples makes it possible to record the temperature of each thermocouple. It should be noted that the junction of each thermocouple is wedged between two turns of the wire.





2.1. Two-dimensional heat transfer model

The medium investigated is supposed to be cylindrical and heterogeneous; it is in the form of a stack of layers along the z axis (Figure 2). Thermal modeling is done using the semi-digital quadrupole method. Note that the thermophysical properties are assumed to remain constant during the experiment.



Figure 2. Two-dimensional diagram.

The thermophysical study of the environment can be summarized in two cases:

- Transfer to the hot rod: the transfer takes place in two directions (r, and z). We can say that the transfer in this area is characterized by a 2D conductive transfer.
- Transfer into the resin (index r), the contact resistance and the semi-infinite medium. We assume that the following two assumptions are verified:
- The temperature is continuous in r_r . It is assumed that the resin layer better conducts the heat flux delivered by the heating probe.
- The total flux generated by the resistive wire is the sum of the fluxes entering on both sides in r_t.
 Given these assumptions, we can establish the vector of temperatures with respect to the vector

of heat. This temperature writing is translated into a transformed Laplace space.

• Simplification of the model.

For short times and a large distance between two thermocouples, the transfer along the z axis is negligible. Consequently, the problem of 2D transfer is reduced to a unidirectional radial transfer. For calculation, the transfer in a layer is unidirectional, which does not depend on the neighboring layers along the z axis. Given what we have just mentioned. The application of a simplification of the model consists in solving the general equation of the transfer with only a unidirectional transfer (1D model).

2.2. One-dimensional heat transfer model

In this paragraph, it is assumed that the conditions for simplification of the 2D model in a 1D model are well respected. It was decided to modify the writing of the temperature equation in order to simplify the model. The same equation is repeated but this time without taking into account the heat transfer along the direction z (Figure 3).



Figure 3. One-dimensional diagram.

It should be pointed out that when solving the problem, the initial and boundary conditions remain unchanged. The new expression of the temperature is done in a Laplace transform space. Once the one-dimensional problem is solved, the solution found is compared with that of the two-dimensional problem; this is done for the purpose of evaluating the gain obtained and also to have an idea on the reliability of each method.

2.3. Comparing the two models

To compare the two models, it was decided to proceed as before; a bilayer medium is excited with a step-type flow. After this operation, the response of the medium to thermal excitation is then analyzed. Figure 4 shows a schematic comparison between the Janssen Method and Hot Rod Method.



Figure 4. Comparison of the Janssen Method and Hot Rod Method.

Based on experience, we find that the response of the 2D and 1D model are almost identical, this is true only for fairly deep points of the interface. In addition, for the points close to the interface,

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the transfer between strata is essential; consequently the transfer in the middle is done with a 2D transfer. For short times, the transfer is unidirectional; the latter is done with a 1D transfer. For this time range, the simplification of the model is feasible. In order to be able to simplify the 2D model, and to continue working with a 1D transfer, we assume that the simplification hypotheses are verified. To comply with the simplification conditions, we have agreed to use the following configuration:

- The distance between two measurement points is around 2 cm.
- The time of thermal excitation is very short. For the treatment of the problem, we assume that it is less than 600 s.

We put ourselves in a configuration where the transfer is done only with a 1D transfer (unidirectional transfer). This is to highlight one of the advantages of simplifying the 1D model. In Figure 5, we have reported the results of the response of the 1D model and a response of the hot wire model. Before comparing the results, we would like to point out that we have kept the same geometry of the medium, in this case a cylindrical medium.



Figure 5. Responses of 1D model and the hot wire model.

By comparing the results obtained with a 1D model to those obtained with the hot wire model, we were able to observe differences between the temperatures of these models. It follows that the differences observed in the temperature differences are due to the energy stored by the central rod. It should be noted that the long-time logarithmic slopes are identical for the two responses. We therefore observe on these calculations carried out with a 1D model that the estimation of thermal conductivity can be done with the hot wire method, this being verified only with a regression for long times.

It should be noted that the simplification condition of the 2D hot wire model must meet the following criterion of Eq 1:

$$\frac{r_r^2}{a_i} \ll t \ll \frac{\Delta z^2}{a_i}, and \ r_r \ll \Delta z \tag{1}$$

where t is the duration of the thermal excitation, Δz is height of a stratum, a_i is the thermal diffusivity of a stratum, r_r is the radius of the sensor.

2.4. Experimental device

The experimental device used is shown in Figure 6. It consists of a metal cylinder about 30 cm high and 11 cm in diameter, made of stainless steel, and equipped with a stainless steel piston. A probe (hot rod) is placed in the center of the cylinder and fixed to its walls. The dimensions of the cylinder make it possible to suppose that the medium is semi-infinite. The compression testing was carried out by means of an Instron electromechanical universal testing machine that allows performing a series of compressions of the powder. A regulated power supply is used to provide a voltage step across the hot rod.



Figure 6. Powder production process.

To comply with the safety conditions, it was sometimes necessary to reduce the height of the probe for the purpose of limiting the number of thermocouples used for the measurement of the thermal conductivity.

2.5. Calculation of the coefficient of friction with the walls

The walls, about 1 cm thick, are made of steel and are considered as non-deformable under the effect of mechanical stresses. The shear cell helps to calculate the coefficient of friction with the walls. For this, it was decided to use the Jenik écell (Figure 7).



Figure 7. Experimental device (Jenik écell).

3. Results and discussion

In order to validate the method, it was decided to consider three different granular media. The reliability of the method can be verified by making a quantitative comparison between the measurements from the Hot Disk technique and those obtained with the hot rod method.

3.1. Experimental data

To adapt the hot rod method to granular media, it was decided to choose well known granular media. The following table gives some properties of the media used. It should be noted that the thermal conductivity measurements were performed with the Hot Disk method and using a CT-meter. Only the thermal conductivity values obtained for well-defined initial relative densities of the media are presented here.

Microcrystalline steel powder:

Galvanized steel–Thermal conductivity, $K = 0.095 \pm 0.004 \text{ W/(m K)}$.

Type A2 steel–Thermal conductivity, $K = 0.216 \pm 0.003 \text{ W/(m K)}$.

ASTMA A36 steel–Thermal conductivity, $K = 0.231 \pm 0.011 \text{ W/(m K)}$.

In fact, the measurement error on the apparent thermal conductivity by the Hot Disk method can reach 4%. This error is significantly dependent on the controlled boundary conditions previously described. In the context of the present study, granular media with different mechanical behavior were carefully selected. Therefore, only measurements of the thermal conductivity of a medium at rest were considered. It was found that the relative density (porosity) was not constant when the depth changed. This was done for the purpose of simplifying the thermophysical characterization process of media at rest.

Comparison of the experimental results and the actual value of thermal conductivity of the gel made it possible to determine the power factor of each thermocouple.

The results found are in good agreement with the data obtained by the Hot Disk method. The hot rod method used was validated through experiments (Tables 1–3). The difference between the measurements found with this method and those given by the hot disk method can be reduced if the probe is properly calibrated.

One can clearly see that the results found are consistent with the values obtained with the Hot Disk method, but with an error estimated at 2%.

The results obtained for type A2 steel (Table 2) are in agreement with the values measured by the hot disk method. However, it was found that the difference between the measured values and the actual value increased for the thermocouples 4, 5 and 6. This is certainly due to the fact that these thermocouples are located at the bottom of the cylinder. Therefore, it can be concluded that the relative density of each layer is certainly influenced by the stratification effect. Indeed, the apparent thermal conductivities of the deep layers are strongly modified due to the stress applied by the upper strata. The problems due to the stratification effects arise in granular media having a high bulk density. It should be noted that for the case of galvanized steel, the problem of higher thermal conductivity in deep layers does not occur since the bulk density is low.

Thermogram	Thermal conductivity (W/(m K))	Deviation%
1	0.093	2%
2	0.094	1%
3	0.094	1%
4	0.096	2%
5	0.097	1%
6	0.095	0%

Table 1. Experimental results (galvanized steel).

Thermal conductivity (W/(m K))	Deviation
0.211	1.0/

Table 2. Experimental results (type A2 steel).

Strata	Thermal conductivity (W/(m K))	Deviation%	
1	0.211	1%	
2	0.215	2%	
3	0.211	2%	
4	0.201	4.3%	
5	0.204	3.6%	
6	0.201	4.3%	

Table 3. Experimental results (ASTMA A36 Steel).

Strata	Thermal conductivity (W/(m K))	Deviation%	
1	0.252	5%	
2	0.242	1%	
3	0.241	1%	
4	0.236	1.6%	
5	0.249	3.75%	
6	0.2410	1%	

To better interpret these results, we have plotted them on Figure 8 which traces the variation in mechanical stresses, but this time in 2D.



Figure 8. Mechanical stresses as a function of depth.

One can clearly see that the profile of the mechanical stresses decreases regardless of the value of the transmission factor k (Figure 8). It is also noted that the reduction in the amplitude of the mechanical stresses depends on the value of this factor.

For low values of k, the axial stresses are larger than the radial stresses. Therefore, it can be stated that there is a good transmission of forces within the medium along the z axis. However, for significant stresses, the radial forces are larger than those normal to the walls of the cylinder. The primary role of walls is to curb the mechanical stresses from the center.

In this part, the mechanical stresses applied to the medium are changed (Figure 9). The main objective is to examine the effect of walls on the variation of the mechanical stresses at each point of the medium. The height of the medium is the same in all the cases studied.



Figure 9. Mechanical stresses as a function of depth for different values of parameter k.

The most important remark that can be made about these comparisons is that for some values of k there is a critical depth from which the mechanical stresses are constant. This depth is the same for all mechanical stresses applied at the top of the cylinder (Figure 10).



Figure 10. Mechanical stresses as a function of depth.

From the current analysis, it becomes clear that the transmission factor plays an important and fundamental role in the modeling of the final density of the medium subjected to mechanical stresses. Consequently, the mechanical stresses cannot be modeled without the determination of this transmission parameter k.

• Calculation model for thermal conductivity as a function of depth, and mechanical stresses.

For the validation of the model, we kept the same powder, that is to say, the Avicel 102. We apply a mechanical stress of order of 2.5 MPa on the granular medium with a compression speed of order of 10 mm/min. Using the Instron machine, we calculate the displacement of the piston in the middle.

At the end of compression, the medium is exposed to a heat flux with a power of the order of 1.4 W and for duration of 600 s. The variation in temperature between the initial and final state for each thermocouple is shown in Figure 11.



Figure 11. Variation of temperature as a function of time.

Before each test, it is ensured that the temperature of the medium is homogeneous and stable. In addition, it is noted that the variation of the temperature is not homogeneous for all the thermocouples (Figure 11). This remark proves that the thermo physical behavior of each stratum is

different. In the end, it is important to recall that the value of thermal conductivity of each stratum is measured by applying the hot wire technique for each response of a thermocouple.

One may clearly see that the transmission parameter k lies within the range extending from 0.9 to 1.4. The value of k, found by an indirect estimate, indicates that the transmission factor is greater than unity, which implies that the forces are always directed towards the walls (Figure 12). This is demonstrated by the fact that the measurements of the current thermal conductivity are close to those of the initial thermal conductivity. In practice, this remark is justified by the fact that powder in deep layers is still in its granular state. With regard to the thermomechanical characterization of a granular medium, it is highly recommended to use the method that gives results that are as close as possible to reality, namely the modified hot rod technique.



Figure 12. Estimation of the transmission parameter k.

4. Conclusion

The thermophysical characterization of granular media can also be done by a new method, called the hot rod method, which was previously developed. Indeed, this study first focused on the definition and realization of different elements that make up the experimental setup used in this method. The technique developed in this chapter is easy to implement and is intended to measure the apparent thermal conductivity of heterogeneous media.

A semi-digital resolution of the physical problem was performed. The results found were analyzed. Furthermore, the study of sensitivity helped, on the one hand, to highlight the essential role of the distance between two measurement points as well as the warm-up time, and on the other hand, to define the conditions necessary to simplify the resolution of the problem studied. In addition, the study of the influence of various parameters made it possible to restrict the field of study to two interesting cases, namely the 2D heat transfer and 1D heat transfer. Indeed, the thermal transfer in a medium is carried out according to the two main directions (2D thermal transfer). However, for short times and a distance between two measurement points large enough, the 2D transfer can be reduced to a 1D transfer which for long times is hybridized to a hot wire transfer.

Tests related to the reliability of thermal conductivity measurements by this method were carried out. Our method could be validated, among other things, thanks to the good calibration of the sensor. It turned out that the thermal conductivity measurements recorded during a measurement campaign by the hot rod method gave very good results and in a more direct way.

On the other hand, another experimental device, which this time takes into account the evolution of thermal conductivity as a function of the mechanical stresses, was developed. Using the developed model and the experimental results obtained, it was possible to model the evolution of thermal conductivity as a function of depth. It turned out that this method can restrict the scope of use of this parameter.

Conflict of interests

All authors declare no conflicts of interest in this paper.

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