A RAPID, SIMPLE METHOD FOR THE DETERMINATION OF THE THERMAL CONDUCTIVITY OF SOLIDS

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The rational design of equipment such as shaft coolers, heaters, and rotary kilns for the heating and cooling of solids requires that the thermal properties of the solids be known. Thermal conductivity is one of these properties that to measure necessitates elaborate equipment and time-consuming techniques.

A rapid, simple method has been developed for determining the thermal conductivity of solids. The solids can be either porous or non-porous and of either high or low conductivity. If high-conductivity materials are tested, then both the thermal conductivity and heat capacity can be simultaneously measured by the method.

The procedure involves preparing a cylindrical briquette of the test solid that has a thermocouple located in the center. This briquette is heated to a constant temperature after which it is suspended in an open-end glass tube and cooled by a known flow of nitrogen or any other nonreactive gas. The thermal conductivity is then computed from a digital computer comparison of the cooling curves for the test solid versus a reference solid of known thermal properties and similar size that has undergone the same heating and cooling cycle. The method was validated by using the known thermal properties of lead, aluminum, and silver and computing the theoretical cooling curves. The theoretical curves were in close agreement with the experimentally measured cooling curves for these materials.

Theory

The mathematical basis for determining thermal conductivity by the described method is discussed in a paper by Newman[1] and is summarized as follows. Consider a cylindrical briquette as shown in Figure 1. The differential equation for unsteady state heat transfer by conduction in the x-direction is (see nomenclature for definition of the variables):

$$\frac{\partial t}{\partial \theta} = \alpha\left(\frac{\partial^2 t}{\partial x^2}\right) \tag{1}$$

For a briquette of thickness 2a, the central plane being at $x = 0$ and assuming:

1) uniform temperature at the start of cooling of the initially hot briquette

then $t = t_o$ when $\theta = 0$ (2)

2) the final temperature of the briquette will be the temperature of the surroundings:

therefore $t = t_s$ when $\theta = \infty$ (3)

3) there is no heat flow across the central plane because of symmetry:

consequently $-k \left(\frac{\partial t}{\partial x}\right) = 0$ at $x = 0$ (4)

The heat balance on the briquette surface is made by equating heat transferred to the surface by conduction with heat transferred from the surface by convection. In differential form, the heat balance is:

$$-k \left(\frac{\partial t}{\partial x}\right) = h\ (t - t_s) \text{ at } x = \pm a \quad (5)$$

Newman[1)] showed that the solution to Equations (1) through (5) expressed in terms of a dimensionless temperature ratio Y_x is:

$$Y_x = \frac{t - t_s}{t_o - t_s} = 2\sum_{n=1}^{\infty} A_n e^{-(\beta_n^2 X_a)} \cos\left(\beta_n \frac{x}{a}\right) \quad (6)$$

where $A_n = \dfrac{m_a}{(1+\beta_n^2\ m_a^2 + m_a)\ \cos\beta_n}$ and

β_n are defined as the first, second, third, etc., roots of the transcendental equation:

$$\beta_n \tan \beta_n - 1/m_a = 0 \quad (7)$$

The surface to solid thermal resistance ratio, m_a, is defined as:

$$m_a = k/ha \quad (8)$$

and X_a is defined as: $X_a = \alpha\theta/a^2$ (9)

where the thermal diffusivity is: $\alpha = k/\rho\ C_p$ (10)

Similarly, considering radial heat transfer, the radial briquette heat balance is

$$\frac{\partial t}{\partial \theta} = \alpha\left[\frac{\partial^2 t}{\partial r^2} + \frac{1}{r}\frac{\partial t}{\partial r}\right] \quad (11)$$

The initial condition equation is:

$$t = t_o \quad \text{when } \theta = 0 \quad (12)$$

The final temperature equation is:

$$t = t_s \quad \text{when } \theta = \infty \quad (13)$$

The boundary condition equations are:

$$-k \left(\frac{\partial t}{\partial r}\right) = 0 \quad \text{at } r = 0 \quad (14)$$

and $$-k \left(\frac{\partial t}{\partial r}\right) = h\ (t-t_s) \text{ at } r = R \tag{15}$$

Solving Equations (11) through (15) gives:

$$\frac{t - t_s}{t_o - t_s} = 2\sum_{n=1}^{\infty} A_n\ e^{-(\beta_n^2\ X_r)}\ J_o(\beta_n \frac{r}{R}) = Y_r \tag{16}$$

where

$$A_n = \frac{m_r}{(1+\beta_n^2 m_r^2)\ [\ J_o(\beta_n)]} \tag{17}$$

and β_n are the first, second, third, etc., roots of the equation:

$$\beta_n J_1\ (\beta_n) - 1/m_r J_o(\beta_n) = 0 \tag{18}$$

The surface to solid thermal resistance ratio, m_r is

$$m_r = k/hR \tag{19}$$

and $$X_r = \alpha\theta/R^2 \tag{20}$$

The complete differential equation for the case shown in Fig. 1 is:

$$\frac{\partial t}{\partial \theta} = \alpha \left(\frac{\partial^2 t}{\partial r^2} + \frac{1}{r}\frac{\partial t}{\partial r} + \frac{\partial^2 t}{\partial x^2}\right) \tag{21}$$

and the solution to Equation (21) is:

$$Y = \frac{t-t_s}{t_o-t_s} = Y_r\ .\ Y_x \tag{22}$$

If the center temperature defined at $r = 0$, $x = 0$ is t_c, then Equation (22) becomes:

$$Y_c = \frac{t_c-t_s}{t_o-t_s} = Y_r\ .\ Y_x \tag{23}$$

where Y_r and Y_x are evaluated at $r = 0$ and $x = 0$.

The preceding mathematical analysis shows that the rate of cooling, or change in center temperature for a cylindrical briquette is a function of time (θ), density (ρ), thermal conductivity (k), the surface heat transfer coefficient (h), specific heat (C_p) and the briquette dimensions as expressed by Equation (23).

The experimental technique can now be described in terms of the previous discussion. If the change in center temperature with time is measured experimentally for a material of known thermal and physical properties (standard briquette), the surface heat transfer coefficient can be calculated from Equation (23), since it is the only unknown.

The surface heat transfer coefficient (h) is a function of the flow rate of the cooling gas and the geometry and size of the briquette. It is independent of all other physical, thermal, or chemical properties of the briquette. Therefore, any other briquette having similar dimensions and cooled at the same flow rate will have the same value for (h).

Once (h) has been determined using the standard briquette, the thermal conductivity of any test material can be determined from Equation (23) since all other variables are known.

A computer program has been written which through an iterative process determines the best value of (h) which makes the calculated values for the dimensionless temperature ratio equal to the experimental values obtained when the standard briquette is cooled.

With (h) determined, another computor program is run for the test specimen. Thermal conductivity is now the unknown variable and through another iterative scheme, the best value for (k) that makes the calculated and experimental values for the temperature ratios equal is found.

The input data for both programs consist of density, specific heat, time, briquette dimensions, and several experimental values for the temperature ratio. The output from the first program (standard) is the best value for (h). Using this value for (h), the second program used to determine the k value for any test material. If a highly conductive material is tested, then it is possible to determine its heat capacity since the solid thermal resistance will be small compared to the surface thermal resistance. A transient heat balance can be written for the test solid cooling in a stream of coolant gas.

$$V\rho C_p \frac{dt}{d\theta} = hA\ (t - t_s) \tag{24}$$

In the above equation, $t = t_c$ since the thermal gradient in the solid is neglected. Integrating Equation (24) and using the dimensionless temperature ratio, Y_c gives:

$$Y_c = \exp -(hA/\rho C_p V)\,\theta \tag{25}$$

Thus, if the internal solid thermal resistance is negligible, a plot of the experimental Y_c versus θ data on semilog paper should be linear as shown by Equation (25). The heat capacity, C_p, can be calculated from the slope of the line for Y_c versus θ since (h) is the same as for the standard briquette and the density, ρ, and total surface area, A, for the test material are also known.

Materials and Experimental Work

A primary advantage of the transient technique for determining thermal conductivities is the ease and swiftness with which the experiment can be conducted.

In so far as sample preparation is concerned, any solid that can be briquetted, cast, or fabricated around a centrally located rigid.

thermocouple ($x = 0$; $r = 0$) may be tested. Finished test sample cylinders should be approximately one inch in diameter, and one-half inch in height; however, other dimensions can be used.

Experimental Apparatus

The experimental apparatus (see Figure 2) consists simply of a 3-inch diameter glass tube approximately 3 feet in length. One end of the tube is completely stoppered except for a one-half inch circular opening through which the coolant gas flows. The other end of the tube is open to the atmosphere. A small electric furnace is used to heat the briquette, and an automatic single point temperature recorder connected to the embedded thermocouple is used to measure the center temperature of the briquette.

Experimental Procedure

The experimental procedure is the same for both the standard and test briquettes. Either the standard (aluminum was chosen since its thermal properties are well established), or the test briquette is connected to the temperature recorder by way of the thermocouple leads. The briquette is heated until the center temperature has reached a constant, predetermined value. The briquette is then quickly removed from the furnace and suspended in the cooling tube with the cooling gas flowing at a constant rate. The briquette is usually cooled to the temperature of the cooling gas within 20 minutes.

Data Processing

For the standard briquette, the experimental dimensionless temperature ratio versus time data points for the standard briquette along with the known thermal properties are used to calculate the surface coefficient, h, in the following manner. A digital computer program is written to compute Y_C from Equations (6) through (23). By iteration and assuming various values of (h), the computed values of Y_c can be made to converge on each of selected experimental Y_C versus θ data points. Thus, for a selected data point, the best experimental (h) is that which when used in Equations (8) and (19) results in equal values for the computed and experimental Y_C values.

For low conductivity test materials, the same method is used to determine the best experimental value of k by using the h determined for the standard and the other properties of the test material. If the test material is a good conductor as discussed in the theory section, then experience has shown that h should be computed from the experimental cooling curve and then this value is used to compute k by the same method as for low conductivity test materials.

Discussion and Results

Three briquettes of aluminum, lead and silver were made to test the validity of the experimental technique since their thermal --ope-ties were available from the literature as shown in Table I.

Sintered, dense hematite (Fe_2O_3) and a briquette of porous carbon made from a partially devolatized coal were used as test materials. For these materials, all properties except the thermal conductivities shown in Table I were previously measured. Cooling curves for each briquette were measured for a nitrogen flow rate of 0.9 scfm. Surface heat transfer coefficients for lead, silver and aluminum were calculated by the method discussed in the data processing section. For these materials, the literature conductivity values were used to calculate the surface coefficient. Table I shows that the calculated or experimental h values for each metal are nearly identical. This result is consistent with the theoretical basis of the experiment and may be considered as establishing the validity of the method. Also as additional evidence, aluminum was chosen as the standard and k values for lead and silver were calculated using the h value for aluminum. Table I shows that the calculated or experimental k values were within 0.5 percent of the literature values. The conductivities for hematite and porous carbon were calculated using aluminum as the standard. Figure 3 shows the experimental data points with the solid lines calculated from the theory. Note that the line for the carbon is curved whereas those for the metals and hematite are linear. As discussed previously, a linear cooling curve is obtained if the surface to solid thermal resistance ratios are relatively large. Note that for the metals, lead which has the lowest conductivity and thermal diffusivity cooled the fastest. This result is explained by examination of eqn. (25) which shows that for similar gas flows and briquette dimensions, the rate of cooling for different materials is determined by the heat content, ρC_p. It can be seen in Table I that the heat content for lead is the lowest of all metals tested.

Summary

A rapid, simple method for determining thermal conductivity for a solid has been developed. The solid can be either porous or nonporous and of either high or low conductivity. If high conductivity materials are tested, then both conductivity and heat capacity can be simultaneously measured from one cooling experiment. The method was validated by using the known thermal properties of lead, aluminum, and silver and the experimental cooling curves in a comparison with the computed results.

References

1. Newman, A. B., Industrial and Engineering Chemistry, Vol. 28, 1936, pp. 545-548.

Nomenclature

a = Half height of briquette; ft

A = Area; ft^2

A_n = Coefficient in infinite series solution for temperature distribution in briquette

C_p = Specific heat; BTU/lb °F

h = Surface heat transfer coefficient; BTU/hr ft^2 °F

k = Thermal conductivity; BTU/hr ft^2 °F/ft

m_a = Axial surface resistance; dimensionless

m_r = Radial surface resistance; dimensionless

R = Maximum radius of briquette; ft

r = Radius of briquette; ft

t = Temperature; °F

t_c = Temperature at center of briquette; °F

t_o = Initial temperature of briquette; °F

t_s = Temperature of cooling gas; °F

x = Distance of direction; ft

X_a = $\frac{\alpha\theta}{a^2}$ Dimensionless time parameter for axial component

X_r = $\frac{\alpha\theta}{R^2}$ Dimensionless time parameter for radial component

Y_x = Symbol for temperature ratio, axial component; dimensionless

Y_r = Symbol for temperature ratio, radial component; dimensionless

α = $(k/\rho C_p)$ Thermal diffusivity; ft^2/hr

θ = Time; minutes or hours

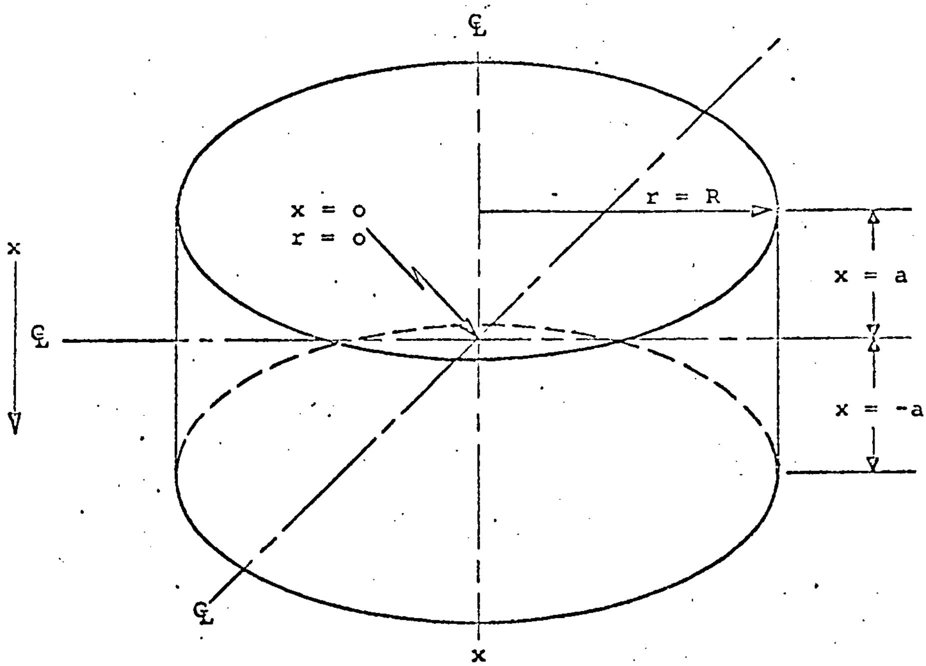
ρ = Density; lb/ft^3

Table I

THERMAL AND PHYSICAL PROPERTIES FOR THE BRIQUETTES

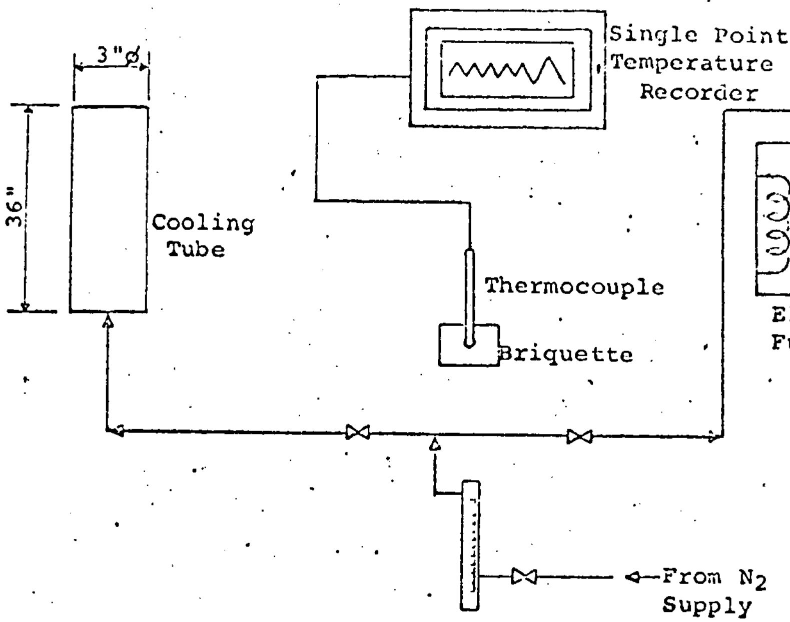
	Aluminum	Silver	Lead	Hematite	Porous Carbon
a	.01842	.02059	.01842	.01842	.01958
R	.04208	.04210	.04117	.04208	.04121
ρ	168.50	655.20	707.43	306.00	75.0
C_p	.2273	.0578	.0306	.2090	.2360
ρC_p	38.30	39.31	21.65	63.95	17.70
h (experimental)	5.58	5.70	5.60	5.58	5.58
k (experimental)	Not Measured	240.3	18.99	12.10	.0307
k (literature)	121.7	240.0	19.00	none	none
α (experimental)	3.178*	6.113	.8770	.1892	.00173

*Average of literature sources



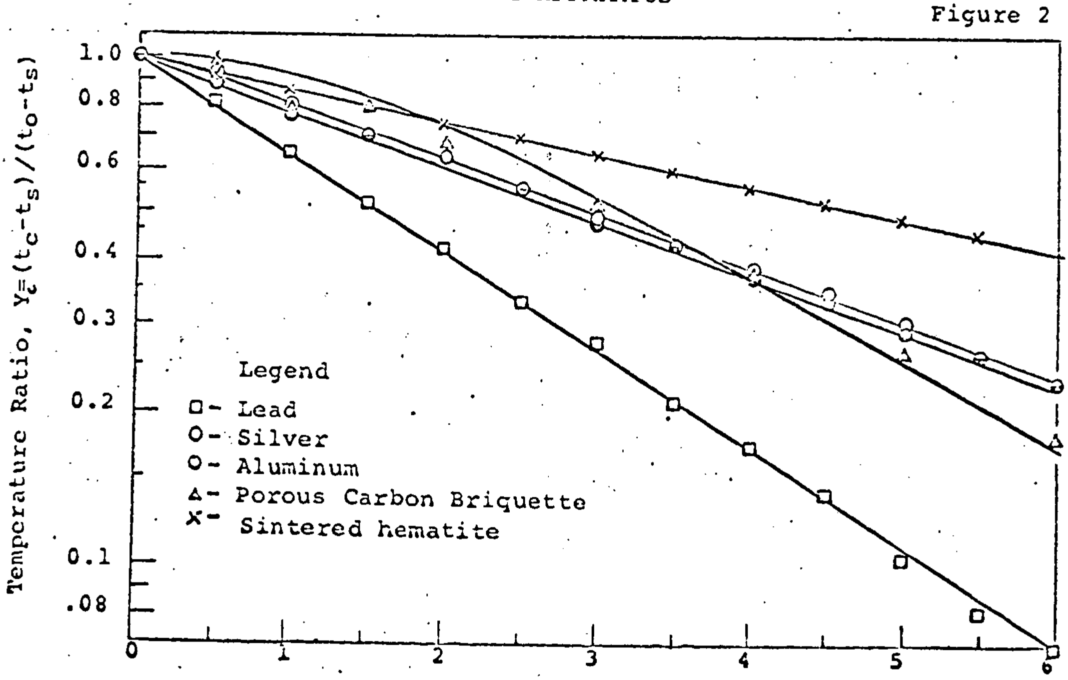
STANDARD CYLINDRICAL BRIQUETTE

Figure 1



EXPERIMENTAL APPARATUS

Figure 2



.. Theoretical Cooling Curves and Experimental Points for five Materials

Figure 3