# THERMAL CONDUCTIVITY OF CARBONACEOUS BRIQUETTES

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## Introduction

The purpose of this work was to determine the effect of temperature and briquetting pressure on the thermal conductivity of briquettes made from bituminous coal, anthracite, and petroleum fluid coke. The briquettes were formed by pressing these materials with suitable binders.

In spite of the promising future for carbonized briquettes of these materials there is a dearth of fundamental and systematic data on their properties, such as thermal conductivity. There is a need particularly for thermal conductivity data which would be used for the proper design of briquette carbonizing retorts and briquette using furnaces. Consequently it was felt that any further study, such as covered by this paper, will go a long way towards upgrading this process from its present stage as an art to one based upon sound technological principles.

### Briquetting

Coals having the properties shown in Table I were dried in an oven at 225°F and ground in a hammer mill through a 3/64" screen. The bituminous coal was then contacted with air at 550°F until it was adequately oxidized to reduce coking power as observed by the ASTM free swelling test. The petroleum coke also shown in Table I was dried and ground in a ball mill for 15 minutes.

The pulverized coals and coke were blended with suitable binders, i.e., coal with coal tar pitch and coke with coker bottoms. The properties of the binders which were chosen on the basis of practical considerations as to general availability and adequacy are shown in Table II.

The mix was then pressed in a hydraulic press to form a cylindrical briquette, 3½" in diameter, and approximately 1" thick. Briquetting pressures of 1000, 3000, 4000 and 5000 psig were used to determine subsequently the effect of pressure on thermal conductivity.

Table I - Properties of Carbonaceous Materials

| | Anthracite | Bituminous | Coke |
|---|---|---|---|
| Fixed C | 80.1% | 73.3% | 86.8% |
| Ash | 15.4% | 6.5% | 0.0% |
| Volatile | 4.1% | 19.5% | 6.1% |
| Sulfur | 0.4% | 0.7% | 6.1% |

Table II - Properties of Binders

| | | Pitch | Coker Bottoms |
|---|---|---|---|
| Conradson C | | 45.8% | 33.0% |
| Ultimate Analysis - | Carbon | 94.0% | 84.0% |
| | Hydrogen | 5.1% | 8.5% |
| | Sulfur | 0.6% | 5.2% |
| | Ash | 0.2% | |

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Carbonization

A liner was made to fit an ordinary laboratory muffle furnace in order to drive off the volatile matter and designed to carbonize the briquettes at various temperatures in an inert atmosphere of nitrogen. The nitrogen was preheated in the outer section of the liner before coming in contact with the briquettes. The furnace was heated at a rate of 3° to 5°F per minute for the petroleum coke and anthracite, while for the bituminous coal this heat-up rate was maintained up to 900°F and reduced to 1°F per minute above this temperature. Experience with these materials had shown that, if these rates were adhered to, cracking due to too rapid vaporization of the volatile matter and shrinkage of the briquettes could be avoided. The purpose of this procedure was to drive off the volatile matter so that it would not subsequently foul the conductivity cell and so that the dimensions of the test specimen could be stabilized and be accurately measured prior to the determination.

Thermal Conductivity

The thermal conductivity cell used in these determinations was based on that described by J. S. Finck (1, 2) modified to enable these determinations to be conducted in an atmosphere of nitrogen in order to prevent combustion of the samples. A cross-section diagram of the cell is shown in Figure I with an enlarged view showing the relative locations of the thermocouples, test heater, guards and sample in Figure II.

The test heater element was imbedded in a 3" diameter alundun plate and delivered 250 watts at 110 V. In the same plate was a guard heater 2¼" wide, which formed a ring around the test heater. The bottom guard heater was separated from the other heaters by 2½" of diatomaceous earth. Atop each of these heating elements was an isothermal plate of 3/4" silicon carbide in which chromel alumel thermocouples were imbedded. The samples, which, after shrinkage during carbonizing, were slightly over 3" in diameter, were placed on the test heater isothermal plate and covered by an additional silicon carbide isothermal plate.

The housing was 22" in diameter by 23" overall height. The cell was sealed by a 14" flange cover in order to make it gas tight. A water cooler, to provide an adequate temperature differential, was attached to the flange cover.

The inside of the cell housing was lined with 5" of 2000°F castable insulation. The spaces between the cell components and the lining were filled with loose diatomaceous earth.

The heaters were controlled by means of powerstats, 45 amp, 110 V for the guard heaters and 7.5 amp, 110 V for the test heater. The power imput to the test heater was measured by a 0-500 ma milliameter and 0-50 V voltmeter at low power levels and 100/200 watt dual range wattmeter at higher levels. This wattmeter was accurate to ±½ watt.

The actual temperature differential across the test piece was measured by stainless steel sheathed chromel alumel thermocouples at the points indicated in Figure II.

The formula for thermal conductivity in a steady state system is as follows:

$$K = \frac{Qs}{At}$$

where K = thermal conductivity
Q = quantity of heat transferred
s = thickness of sample

A = cross sectional area of heat transfer
t = temperature differential across sample

In order for this equation to be valid certain conditions must be met. 1) The system must be in a state of thermal equilibrium. 2) The heat flow must be normal to the cross sectional area through the entire height of the sample. 3) All the heat generated in the test heater and only this heat must flow through the sample. 4) The temperature differential must be reasonably small. The guarded hot plate cell used in this study substantially satisfied the first three conditions, and minimized the ill effects of the fourth.

If the first condition is satisfied by maintaining the cell at constant temperature for a reasonably long period of time and the third condition is satisfied by maintaining the test, ring guard and bottom guard isothermal plates at essentially the same temperature, thus negating any lateral or downward thermal driving forces.

It can be shown by the following analysis that the second condition is also satisfied:

The heat generated in the test heater flows upward into the test sample except for a small amount which flows into the gap between the ring guard and test heaters and then flows upward. (See Figure II) By placing an isothermal plate on top of the test sample there is little tendency for heat migration toward point 3, since the temperature at points 3 and 4 are now essentially equal. With this top plate the heat flow will tend to diverge toward point 3 due to the difference in conductivity in the test sample and the insulating material above the ring guard plate and the resultant temperature difference at points 3 and 4. The same analysis would hold for the heat generated in the ring guard heater. An additional item which effects the direction of the flow of heat from the ring heater is the effect of heat losses. Heat losses from the ring will affect the flow of heat through the test sample if they become excessive. However, with adequate insulation the adverse effect of end losses can be kept within the limits of experimental accuracy.

The fourth condition must be somewhat satisfied because the conductivity is actually a mean value between the conductivity at hot and cold face temperatures. The use of this value assumes that a linear relationship exists between conductivity and temperature.

The values for thermal conductivity determined in this study are tabulated below in Table III and plotted in Figure III. In all cases, except were noted, the briquettes were pre-carbonized at approximately the same temperature as the conductivity determination.

TABLE III

| Material | Briq. Press PSIG | Mean Temp. $^oF$ | K BTU/HR-$^oF$-FT$^2$/IN | Material | Briq. Press PSIG | Mean Temp. $^oF$ | K BTU/HR-$^oF$-FT$^2$/IN |
|---|---|---|---|---|---|---|---|
| Anthracite | 1000 | 295 | 2.69 | Coke | 1000 | 285 | 4.10 |
| | 3000 | 287 | 2.51 | | 2000 | 293 | 3.80 |
| | 5000 | 284 | 2.87 | | 5000 | 309 | 3.92 |
| | 1000 | 890 | 4.09 | | 4000 | 726 | 3.36 |
| | 3000 | 894 | 3.76 | | 1000 | 888 | 2.97 |
| | 5000 | 916 | 3.80 | | 3000 | 928 | 2.82 |
| | 5000 | 1271 | 5.36 | | 3000 | 1277 | 2.56 |

Table III (Cont.)

| Material | Briq. Press PSIG | Mean Temp. °F | K BTU/ HR-°F-FT²/ IN |
|---|---|---|---|
| Bituminous | 1000 | 263 | 1.36 |
| | 3000 | 278 | 1.23 |
| | 5000 | 277 | 1.18 |
| | 3000 | 591 | 1.18 |
| | 1000 | 941 | 2.91 |
| | 3000 | 880 | 3.28 |
| | 5000 | 865 | 2.88 |
| | 5000 | 1234 | 5.61 |
| | 1000* | 1466 | 9.98 |
| | 1000* | 475 | 7.10 |
| | 1000* | 1116 | 8.0 |

*These briquettes were carbonized at 1600°F to show effect of temperatures.

A study by Batchelor et al (3) on briquettes formed from 25% Pittsburg seam bituminous coal, 63.5% low temperature char of this material, and 11.5% pitch is substantially in agreement with the results of the present study on bituminous coal briquettes. In both cases the material was pre-carbonized.

Table IV

| Temp. °F | K-BTU/HR-°F-FT²/IN Batchelor et al | K-BTU/HR-°F-FT²/IN Present Study |
|---|---|---|
| 300 | 1.8 | 1.2 |
| 600 | 1.8 | 1.2 |
| 900 | 2.4 | 3.0 |
| 1200 | 4.8 | 5.6 |
| 1500 | 9.6 | 10.0 |

The only other thermal conductivity data found there the results were determined at elevated temperatures is that of Terres (4) on pieces of English coking coals. The values are in reasonable agreement with the present study. The work of Beletzki (5) on pulverized coals is also in good agreement considering that his values were determined at room temperature and to be compared to the current study should include a temperature coefficient. This data together with that of Batchelor et al is shown in Figure IV.

An attempt to show the effect of temperature alone involved the measurement of conductivities on a briquette which had been carbonized at 1600°F. The conductivities of this briquette at lower temperatures were considerably higher than the conductivities of briquettes carbonized at the temperature of the conductivity determination. (See Figure V)

From the above discussion, it will be evident that the values for thermal conductivity determined in this study are values which include the effect of temperature and the effect of carbonization. For design considerations this is the most useful conductivity value.

An attempt was also made to determine the effect of briquetting pressure on the thermal conductivity. Samples briquetted at three different pressures were measured at 300°F and 900°F, and the variations found were well within the accuracy of the determination and therefore could not be attributed to differences in briquetting pressures.

As a result of these tests, it is felt that the conductivity of briquetted coals is dependent on the nature of the base material only slightly modified by the binding agent and independent of the pressure effects of briquetting. Other properties probably enter into the picture, such as particle size, but these were beyond the scope of this study.

It is possible to formulate equations for the conductivities of fluid coke and anthracite, within the range studied, but the equation for the curve for bituminous coal would require a factor related to the coking power of the coal. The equations for fluid coke and anthracite include only a temperature factor since the carbonaceous base in these cases has not undergone any substantial thermal change. If the study could have been continued to higher temperatures, the coefficient of thermal change would probably enter into these equations also.

These conclusions are quite reasonable since the bituminous coal undergoes fusion and loses its particulate nature on carbonizing, while the other materials remain particulate even at much higher temperatures then encountered in this study. They do, however, give up volatile matter consisting mostly of hydrogen indicating a destructive distillation which could probably affect the thermal conductivity.

The equations for the thermal conductivity of the briquetted material based on the curves shown in Figure III are as follows:

<u>Anthracite</u>

300-1200°F -- $K = 2.30 + 1.0 \times 10^{-3}t + 9.0 \times 10^{-7}t^2$

<u>Bituminous Coal</u>

300°- 600°F -- $K = 1.20$
600°- 900°F -- $K = 0.0049t - 1.59$
900°- 1200°F -- $K = 0.0087t - 5.01$
1200°- 1500°F -- $K = 0.0150t - 13.70$

<u>Fluid Coke</u>

300-1200°F -- $K = 4.32 - 1.42 \times 10^{-3}t$
K is the thermal conductivity in BTU/HR-°F - FT²/IN
t is temperature in °F.

The average deviation about the mean value for these determinations is 5 to 8% by measurement. The absolute accuracy, as calculated from possible errors in the measurement of temperature and heat input, is in the order of 15 to 20%. This is well within the limits required for engineering work and owing to the very specialized nature of the briquettes and the high degree of variation found in the characteristics of coals, is about as accurate as would be required.

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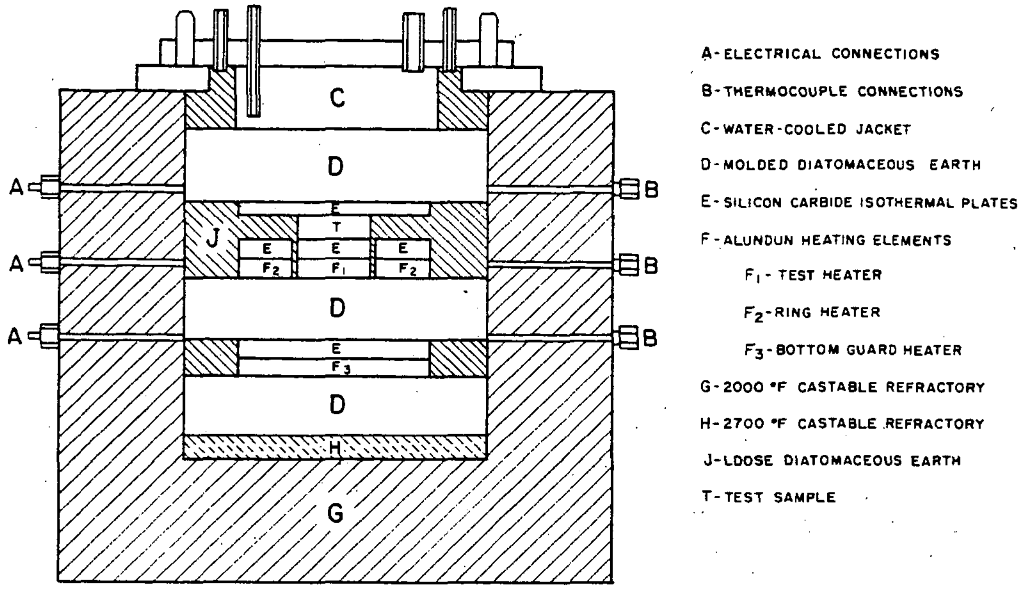
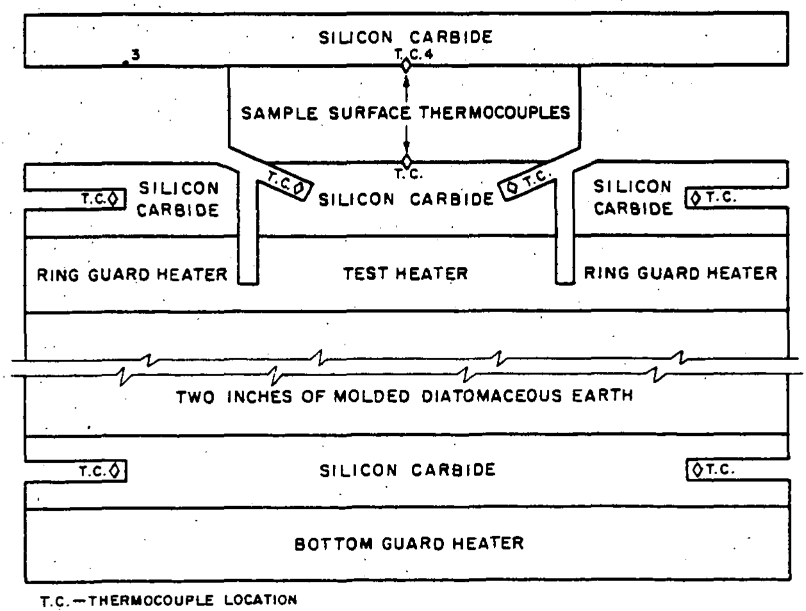



DIAGRAM OF THE THERMAL CONDUCTIVITY CELL

FIGURE I




THERMOCOUPLE ARRANGEMENT IN CONDUCTIVITY CELL

FIGURE II

THERMAL CONDUCTIVITY VS MEAN TEMPERATURE FOR BRIQUETTES MADE FROM ANTHRACITE, BITAMINOUS COAL AND PETROLEUM FLUID COKE.

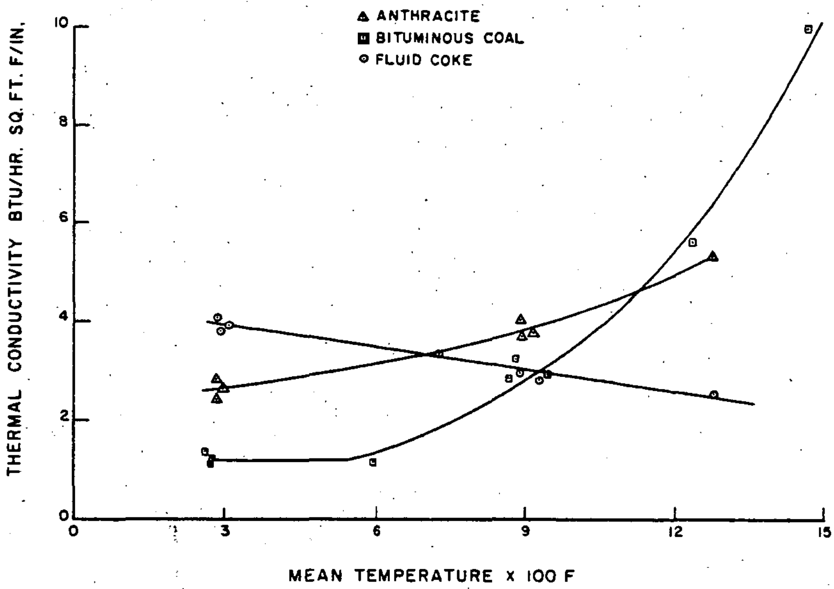



FIGURE III

COMPARISON OF THE THERMAL CONDUCTIVITIES FOR BITUMINOUS COAL FROM LITERATURE

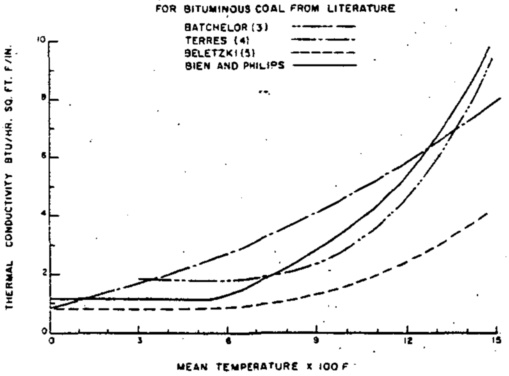



FIGURE IV

COMPARISON OF THERMAL CONDUCTIVITY FOR BITUMINOUS COAL BRIQUETTES BY NORMAL PROCEDURE AND CARBONIZING TO ELEVATED TEMPERATURE AND MEASURING CONDUCTIVITY AT LOWER TEMPERATURE.

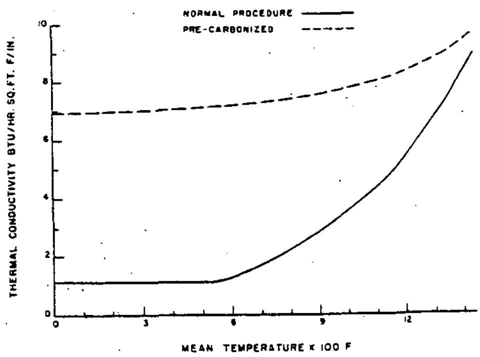



FIGURE V