The Production of Activated Carbon from North Dakota Lignite

Robert C. Thelen

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THE PRODUCTION OF ACTIVATED CARBON FROM
NORTH DAKOTA LIGNITE

by
Robert C. Thelen

B.S. in Chemical Engineering, University of North Dakota, 1962

A Thesis
Submitted to the Faculty
of the
Graduate School
of the
University of North Dakota
in partial fulfillment of the requirements
for the Degree of
Master of Science

Grand Forks, North Dakota
June 1963
This thesis submitted by Robert C. Thelen in partial fulfillment of the requirements for the Degree of Master of Science in the University of North Dakota, is hereby approved by the Committee under whom the work has been done.

Chairman

Dean of the Graduate School

306835
This thesis submitted by Robert C. Thelen in partial fulfillment of the requirements for the Degree of Master of Science in the University of North Dakota, is hereby approved by the Committee under whom the work has been done.

(signed) A. M. Cooley
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(signed) C. W. Bullard

(signed) R. C. Ellman

(signed) James L. Elder

(signed) W. R. Kube

(signed) E. D. Wahl

Dean of the Graduate School
ABSTRACT
THE PRODUCTION OF ACTIVATED CARBON FROM NORTH DAKOTA LIGNITE IN A FLUIDIZED BED REACTOR

by

Robert C. Thelen, Master of Science

The feasibility of producing activated carbon from lignite using a fluid bed reactor was investigated. Steam was used as the activating agent and was reacted with carbonized lignite at elevated temperatures. The reaction was performed in a fluidized bed type reactor 1 1/2" in diameter and 2 1/2" in length.

Tests indicate that activated carbon with adsorptive properties equal to or better than commercial Darco activated carbon can be produced from lignite in a fluidized reactor. This conclusion was based on the results from heat of wetting and service time tests which were applied to both Darco activated carbon and the present product. These tests measure the adsorptive powers of an activated carbon. The heat of wetting and service time values for the present product were 1.24° C/gram and 11.61 minutes respectively. For the corresponding Darco activated carbon the values were 0.82° C/gram and 10.37 minutes respectively.

The variables investigated were feed rates, particle size, temperature, and steam flow rate to determine optimum conditions for the activation process. No optima were found for the temperature and particle size variables due to the effect of a changing residence time in each case. Investigation of the feed rate variable from 150 to 500 gram/hr indicated no significant change in the product. For the apparatus used in this investigation, the best product resulted at steam rates below 0.35 SCF*/min, at a temperature of 1750° F, a feed rate of 300 grams/hr, and 40 by 60 mesh particles.

ACKNOWLEDGEMENTS

The author wishes to express his sincere thanks to the U. S. Bureau of Mines Research Laboratory for the fellowship program under which this work was done.

The author also wishes to express his appreciation to Mr. Robert C. Ellman, of the Bureau of Mines Staff, and Professor A. M. Cooley for their invaluable assistance.

Indebtedness is also expressed to Professors C. W. Bullard, W. R. Kube, D. E. Severson and E. D. Wahl for their constructive criticism and suggestions throughout the investigation.

* Standard Cubic Feet
Credit is also given to Messrs. R. Porter, J. Hoeppner, G. Gronhovd, and D. Winge of the Bureau of Mines Staff for their aid and counseling.

The author also wishes to thank Miss Marjorie Anderson for typing this thesis.

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INTRODUCTION

The primary objective of this investigation was to determine the feasibility of producing activated carbon from North Dakota lignite in a fluidized bed reactor. This objective was to be accomplished by reacting carbonized lignite with steam at elevated temperatures. Accomplishment of the objective could result in a new use for lignite.

North Dakota has an abundant supply of lignite and the development of new uses for this material would benefit both the state and the industry as a whole. The possibility of finding new applications for this resource is of interest to the Grand Forks Lignite Research Laboratory and also to the Department of Chemical Engineering in the University of North Dakota. This mutual interest prompted the present investigation on the production of activated carbon from lignite.

Activated carbon is presently being produced from Texas Lignite by the Atlas Chemical Company. Amick and Lavine have prepared activated carbon from North Dakota lignite by activation with steam. They obtained a product which compared favorably with Darco activated carbon. Cooley has also prepared an activated carbon from North Dakota lignite which compared well with the Darco product. He used a rabble-type furnace and steam as an activating agent. Additional work needs to be done before activated carbon can be produced commercially from North Dakota lignite.

In addition to the above procedures, consideration should be given to the fluidized bed method of activation. The advantages of the fluidized bed method of activation are high rates of heat and mass transfer due to the turbulent nature of fluidization. These conditions are necessary for the activation of lignite since the reaction is endothermic. The present investigation deals with the fluid method.

The scope of the present investigation consisted of designing and constructing the necessary equipment for fluidized bed activation and the optimization of certain variables involved with this apparatus. A considerable amount of time was required to obtain an operable and working apparatus. For this reason, a large portion of the paper deals with the specific problems encountered in designing the apparatus.
CHAPTER I
BACKGROUND INFORMATION

The first phase of this investigation involved a literature survey on active carbon and also on fluidization. The surveys included the theories and applications involved with active carbon and fluidization.

Active Carbon

Definitions and history

Active carbon is defined as any material which contains carbon and has adsorptive properties. These adsorptive carbonaceous materials may be produced from bones, coconut, wood, lignite, and many other raw materials (1). The term activated carbon is also used in reference to the reactivity of carbon, that is the rate of reaction of a carbon with \( \text{O}_2 \) or \( \text{CO}_2 \). The present investigation deals only with activated carbon in relation to adsorptive properties. These adsorptive carbons characteristically have a large surface area. Activation is the process by which this surface area is increased, and the hydrogen removed.

The adsorptive powers of carbonaceous materials were first discovered by Scheele in 1773 who observed the adsorption of gases on charcoal (2). Although early uses were known for active carbon, they were not produced on an industrial scale since the activation process resulted in the corrosion of structural materials. Also, the successful production of active carbon requires use of controlled environmental conditions within narrow limits. Such accurate instrumentation is a fairly recent development.

More recently, however, active carbon has become of commercial importance. Eponit was the first active carbon produced commercially in 1909 (3). These early applications were in the areas of sugar purification and later for use in gas masks.

Theory and physical properties

The theory involved in activation is the removal of substituents from the carbon structure and the reaction of this structure with steam to produce a porous material with a large surface area (4). Carbonization is used to remove the substituents, rendering the carbon structure free to react with steam. The steam then reacts with carbon atoms in the carbon structure and increases the surface area of the structure. The final structure has many exposed carbon atoms which attract certain compounds and hold them on the carbon surface.

The temperatures at which this carbonization and reaction with steam occurs is very critical on the quality of the resulting product. Chaney (5) states that the coal should first be carbonized between 400 and 600\(^{\circ}\) C to make it susceptible to activation with
steam. Then, he states, the steam activation should occur at 1750° F to give the best product.

If the carbonization temperature is above 600° C some of the substituents begin to fuse with the carbon structure and form a larger structure. The disadvantage of this large structure is that steam activation will occur only on its surface and the inside of the structure will not be affected (6). On the other hand if the carbonization occurs below 400° C too many substituents will remain on the carbon structure and will interfere with the steam activation. The best structure then, for activation with steam, results when the coal is carbonized between 400 and 600° C.

The reason that activation should occur at 1750° F is that above this temperature the yield decreases and the ash content increases. At temperatures below 1750° F the rate of reaction of steam with the carbon structure declines.

There are two principal reactions which occur in the process activation. Both reactions are highly endothermic and

\[ H_2O + C \rightarrow H_2 + CO \]
\[ 2H_2O + C \rightarrow 2H_2 + CO_2 \]

occur almost instantly (7). These phenomena resulted in a number of problems in the preparation of active carbon.

Methods of producing active carbon

One of the first methods used to prepare active carbon with steam as the activating agent consisted of a vertical tube filled with char, and steam entering at the bottom. This method failed to give a uniform product. Due to the speed of the steam-carbon reaction, the bottom particles in the tube were almost completely reacted before the steam reached the upper portions of the char bed. Also, since the reaction is highly endothermic and carbon is a poor conductor, a large temperature gradient existed both horizontally and vertically in the bed. In an attempt to solve these problems early investigators used very large particles (8). This resulted in a higher steam velocity through the bed and eliminated the temperature differential and also resulted in more uniform reaction in the bed. The problem with using larger particles, however, was that only the outside surface of the particles were activated. This again resulted in a poorly activated carbon upon grinding.

Investigation was continued with different types of apparatus. Wickenden and O'Hill's (9) studies led to the fluidized bed method of activation. In this method steam is again passed through a bed of
carbon but at a velocity sufficient to hold the particles in a suspended and turbulent state. Under these conditions very small particles could be used with uniform reaction and temperature throughout the bed. The particle density decreased as activation proceeded and eventually the particles became entrained and were expelled through the exit gas stream.

Another method employing steam activation involved the use of a Herrsechoff furnace. In this apparatus the steam was passed over rather than through a bed of carbon. Stirring of the bed maintained a fresh supply of particles at the surface.

The above methods were applied to char other than lignite. Other methods have been used to prepare activated carbon from lignite by Amick, Lavine, and Cooley (10). Amick and Lavine reacted Velva lignite char with steam at temperatures above 950° C. Their product gave a benzoic adsorption considerably higher than that of Darco (2% to 21.3 percent respectively) (11). Cooley employed a rabble-type furnace with steam as the activating gas and also obtained a product which compared favorably with Darco for water purification purposes.

In addition to using steam as an activating agent it is also possible to use other oxidizing agents. Certain mineral salts, acids, and bases have been successfully used in activating carbon (12). Among these agents are dolomite, sulfates, phosphoric acid, and caustic. The raw coal and oxidizing agent are thoroughly mixed and carbonized at temperatures between 600 and 800° C. The basic principle in using these oxidizing agents is the uniform oxidation of the coal resulting in the removal of side chains from the carbon atoms throughout its structure. The exposed carbon atoms are then able to adsorb other materials. Chemical treatment has been used successfully for activating lignite, although from an economic standpoint it would appear that the steam activation method is best. Steam is relatively inexpensive as compared to the chemicals which would be required for chemical activation.

Fluidization

In order to understand the advantages, disadvantages, and design factors, a study on the principles and theory involved in fluidization was made. Presently, the literature on fluidization is highly unorganized and at times there are conflicting views regarding theories and design equations. This is due to the very recent development of the principle of fluidization. The first work on fluidization was done by the Standard Oil Company for their catalytic cracking process (13). It was through their studies that the concept of a "fluid bed" was developed. These studies indicated that there was a high degree of slippage between the solids and a moving fluid. The slippage could be employed to obtain a dense build-up of solids which was very turbulent and had definite boundaries.
A high degree of turbulence has definite advantages in areas where a large quantity of heat transfer, good solids circulation, and intimate contact between gas and solid are required. Each of these conditions is a requirement for the activation of coal. The high rate of heat transfer would eliminate the temperature gradient which exists in the fixed bed situation as discussed previously. Also, good contact of coal and steam would eliminate the difference in degree of reaction which occurs between the top and bottom of a fixed bed reactor.

Physical phenomena

There are some variations in the physical appearance of a fluid bed depending on the velocity of the gas stream. In the process of fluidizing a material, four different stages are encountered (14). These are the fixed bed, dense phase fluidization, two-phase fluidization, and pneumatic transport.

**FIGURE I**

**PHYSICAL PHENOMENA OF FLUIDIZATION**

![Diagram of pressure drop through a bed as a function of gas velocity](image)

Figure 1 is a plot of the pressure drop through a bed as a function of gas velocity (15). The line A-B represents the fixed bed situation. Here the pressure continues to increase as the gas velocity increases because of the increased resistance of the bed. The flow is streamlined and the gas percolates slowly through the bed. As the velocity if increased to the B-C and C-D region, there is a rearrangement of the particles as they begin to have some freedom of motion. This rearrangement is one toward less resistance causing the pressure drop to decline slightly. The entire B-E line represents the condition of dense phase fluidization. Here the bed has definite properties such as density, heat capacity, thermal conductivity, and viscosity.
The line E-F in figure 1 symbolizes the situation when the bed is completely supported in the gas stream. In this case the pressure drop is equal to the weight of the bed. This situation is termed two phase fluidization and the smaller particles now began to separate from the larger ones forming two different phases. This situation is very unstable since the smaller particles become entrained and leave the system. If the velocity is increased still more, all the particles become entrained and are expelled.

Of the two types of fluidization discussed, that is dense phase and two phase fluidization, the former is the most common. The dense phase fluidization occurs in three different patterns. These are; cohesive flow, aggregative flow, and slug flow (16). Cohesive flow usually occurs with small particles which agglomerate or lump together. This is not a favorable situation since there is less intimate contact between fluid and particles and some of the particles may not be contacted at all. Aggregative flow is the most favorable of the three types since in this case the particles are free to move about and are fully suspended and separate from one another. Heat and mass transfer are very high in this type of flow since the particles are in continuous motion to and away from the heating surface or container walls. The bed appears to be boiling in this type of flow.

Slug flow is the third type of dense phase fluidization. This type of fluidization occurs with large particles and large bed height to diameter ratios (10). Two layers are formed in this case; one being highly concentrated with particles and the other being almost entirely fluid. The heat and mass transport is lower in this case than in the aggregative flow situation since the particles are confined to a definite area.

Transport phenomena

The most important advantage of a fluidized over a fixed bed is the increased transfer of heat and mass and the uniform temperature distribution throughout a fluidized bed (17). Heat transfer in a fluidized bed occurs between the bed and the heated surface or wall. It has been estimated that the conductivities between points within the bed are 100 times that of silver due to the mixing action of the bed (18). One theory which has been advanced to account for the high rates of heat and mass transfer is that the fluid bed is comparable to a well stirred liquid (19). In this theory the gas acts as the stirring agent and maintains the particles in a turbulent condition. The result is heat and mass transfer properties similar to those of a stirred liquid. Another theory which has been advanced for the high rate of transfer in a fluid bed is that the particles slide down the laminar velocity layer which exists around the inside surface of the reaction tube. The gas velocity in this laminar layer is lower then that in the rest of the tube. When the particles slide down
In this laminar layer they carry heat away from the wall and into the main stream upon re-entry of the particles into this stream (20). The result is an increase in the heat transfer coefficient.

Applications and design of fluid bed systems

With the high rates of heat and mass transport, the fluidized bed has been utilized extensively in the chemical industry during the past few years (21). In the past 10 years over 60 applications have been found. Some of these applications were studied to determine the design factors for the present investigation since there is a lack of applicable design equations. It was found during this study that a large range of reactor bed height to diameters have been successfully applied to fluid bed reactors. The Brown Company, for example, uses a fluidized reactor with a ratio of 0.333 for roasting pyrite ores (22). Others are the New England Lime Company using a ratio of 3.5 (23), a fluid bed coal gasifier using a ratio of 13.0 (24), and a fluidized coal carbonizer using a ratio of 17 (25). It is obvious from this that quite a wide range of heights to diameter are applicable.

To provide additional background for the reactor design, a study was also made to determine what equations could be applied with the information available in this investigation. The first equation needed was one relating the gas velocity required for fluidization as a function of particle size. Leva (26) developed an equation for the minimum gas velocity required to fluidize particles, which will be discussed later.

Other equations relating to the bed height and diameter were found during the literature survey but involved a knowledge of pressure drops, void fractions, and bed density which are unknown. Franz (27) recommends that for original design purposes the flow condition be observed in various sized glass tubes to determine the best combinations of height to diameter for a reactor. This investigation employed the glass tube method.

CHAPTER II
EQUIPMENT DESIGN AND REVISIONS

A great deal of time was required to obtain a working apparatus due to the many unexpected problems which were encountered. The original design was a batch apparatus. This was later revised to a continuous feeding apparatus. The original, or batch system design, will be discussed first. Since all the drawings and diagrams illustrate the final design, certain parts will not be included in the discussion of the batch apparatus. The final design will be discussed in a following section.
Batch Feeding

The primary objective in the batch design system was to construct a bench scale apparatus in which lignite char could be reacted with steam at temperatures above 1700° F. The reacted char was to be expelled from the reactor after approximately two hours of reaction time. With these objectives in mind, the necessary equipment was selected.

Figure 2 is a flow diagram of the process and the equipment involved. For the batch system the coal feeder was not included and the char was introduced to the reactor at the top of the furnace. With these corrections, figure 2 is a flow diagram of the batch process.

In the process water is converted to steam in the steam generator, superheated in the primary and secondary superheaters, and then enters the reaction chamber in the furnace. The superheated steam makes contact with the batch sample located in the reactor and reacts for two hours. Gases formed during the reaction period leave the reactor and enter the cyclone separator and condenser. Tars and steam are separated from the exit gases and collected in the catch pot. A stream of inert gas is used to discharge the sample after reaction. The char is separated from the inert gases in the cyclone and collected in a glass jar. Figure 2 indicates that the necessary pieces of equipment are a steam generator, superheater, reaction tube, cyclone separator and a furnace.

Steam Generator

The steam generator used in this investigation was available at the Bureau of Mines Laboratory. The generator and the primary superheater are shown in figure 3. Steam leaves the exit line at approximately 400° F.

Secondary Superheater

In addition to the primary superheater, a secondary superheater was needed to increase the steam temperature more nearly to the reaction temperature. The secondary superheater consists of an electric circular pipe furnace of 700 watt capacity containing a 3/4" diameter copper coil of 1' length and made of 1/4" copper tubing. Temperature in the superheater was controlled with a thermocouple at approximately 1000° F. The exit temperature from the secondary superheater was calculated on the basis of a heater capacity of 600 watts, an entrance temperature of 400° F, and a steam flow rate of 1.65 lb/hr. The steam rate is the maximum which could be used for fluidization as determined from observation of air and coal particles in a glass tube. By equating the heat output from the heater to that gained by the steam an exit steam temperature of 1250° F was calculated. This assumes that
Fig. 2 FLOW DIAGRAM OF STEAM ACTIVATION PROCESS.
Fig. 3 SCHEMATIC DIAGRAM OF STEAM GENERATOR AND SUPERHEATER.
there are no losses through the furnace walls. Due to this high temperature and the corrosive action of the steam, the copper coil in the heater had to be replaced after every thirty hours of operation.

Reaction Tube

The next item of equipment to be considered is the reaction tube. Figure 4 is a drawing of the reactor after it was revised for continuous feeding. The original, or batch reactor, differed from that illustrated in figure 4 in that it did not have a 1/4" pipe concentric in the 3/4" pipe. Instead, only a 3/4" pipe was present and extended to the steam entrance point shown in figure 4. Also, in the batch reactor, there was a screen at the bottom point where the 1 1/2" and 3/4" pipes are joined. This screen served the dual purpose of holding the char from falling into the entrance line while at the same time allowing the steam to pass into the reactor. The rest of the batch reactor is the same as the continuous reactor shown in figure 4.

Temperature was controlled in both reactors by three thermocouples. The thermocouples indicated by dotted lines in figure 4 are located inside the reactor while the one indicated by a solid line is located on the outside wall of the reactor. Control of the heat supplied was accomplished with the inside thermocouples. The wall thermocouple was used to assure that the heating element and tube wall did not exceed maximum allowable design temperatures.

A solid shaft was placed inside the 3/4" pipe above the horizontal exit line of the reactor (refer to figure 4). The extension of the 3/4" pipe above the horizontal line was used for charging the reactor and the shaft prevented the product from entering this section. The shaft also served to position the thermocouple in the center of the reactor. Upon charging the reactor, the shaft, thermocouple, and cap are removed.

Sizing of the reaction tube was based on the observation of fluidizing conditions in a 1 1/2" diameter glass tube and from fluidized reactor designs used in other investigations. Observations of the flow conditions in a glass tube indicated that turbulent dense phase fluidization of a 200 gm sample could be achieved in a 2h" section. Other investigators have successfully employed reactor height to a diameter ratio of 13:1 for the fluidized carbonization of coal (25, 26, 27, 28) which should approximate the conditions of this investigation. This ratio resulted in a tube length of 20" if the diameter is fixed at 1 1/2". Consideration of these two lengths resulted in the design of a reaction tube with a diameter of 1 1/2" and a length of 2h". The tube diameter was fixed because of the availability of a 2" diameter ceramic tube at the Bureau of Mines. The ceramic tube was used in the construction of the heating element and will be discussed later.
Coal and steam

Supporting collar

1½" I.D. stainless steel pipe

Wall T.C. 2

T.C. top

To cyclone

24"

18"

6"

6"

6"

1⅛" I.D.

T.C. bottom

Fig. 4 REACTION TUBE
Cyclone Separator

The exit material from the reactor is composed of a mixture of steam, carbon monoxide, hydrogen, carbon dioxide and the product. Since the product had to be separated from the other components, a cyclone separator was required. The cyclone design was based on the dimensions recommended by Parry (29). Figure 5 is a drawing of the cyclone. To prevent steam condensation in the cyclone, a 2" layer of DFC number 89 insulating cement was placed around the cyclone. There was little difficulty encountered with the cyclone and the efficiency was estimated at 95% for particles in the -100, +150 mesh size range.

Condenser

The exit gas stream from the cyclone enters a condenser where steam and condensable tar are removed. The condenser consists of a copper tube surrounded by a water jacket.

Steam Flow Rate Determination

In order to design the electric furnace required to heat the reactor, it was first necessary to determine the gas flow rate required for fluidization of a 200 gm coal sample. Two methods were used to determine this velocity. One was the glass tube experiment discussed previously, and the other involved the use of equations.

From the observation of flow conditions in a 1 l/2" diameter by two foot length of glass tubing, maximum, minimum and optimum gas velocities were found using air as the fluidizing gas. A maximum velocity of 1.8 ft³/min was found where some of the particles began to leave the two foot section. A minimum velocity of 0.9 ft³/min was found. At this velocity the flow was of a slugging nature. The optimum velocity was 1.65 ft³/min corresponding to dense phase fluidization. Each of the above velocities were determined at standard conditions and the reactor temperature was 1750° F. To obtain the velocities at reactor temperature, the corresponding STP or entrance velocities would be 0.424 ft³/min (maximum), 0.212 (minimum), and 0.388 ft³/min (optimum).

An equation by Leva (30) was also employed to determine a minimum fluidization velocity. The equation is

$$Gmf = (1.4 \times 10^5)D_p^{1.82}(D_f)(D_s - D_f)^{0.94}/\mu^{0.88}$$

Gmf = minimum fluidizing velocity (ft/hr)
Dp = average particle diameter (ft)
Df = fluid density (lb/ft³)
Ds = char density (lb/ft³)
μ = viscosity (lb/ft-hr)
Fig. 5  CYCLONE SEPARATOR
Gmf = 1.4x10^5(9.84x10^-4 ft)^1.82(85 lb/ft^3) -94/(.146 lb/ft-hr)^.88
Gmf = 82.0 ft/hr or .0167 ft^3/min

This calculation was made at 1750° F and the value should compare with the value 0.90 ft^3/min obtained in the previous section. Part of the discrepancy is accounted for by the inaccuracy of the equation since it is only accurate to ± 22%. As a safety factor, the value of 1.8 ft^3/min determined with the glass tube was used in calculating the heat requirements.

Electric Furnace Design

The calculations for the design of the electric furnace involved determining the total heat required, and the length of heating element necessary to supply this heat. Heat requirements were for the steam-carbon reaction, heating the steam from the superheat temperature to the reaction temperature, and for losses through the furnace walls.

The heat required for the steam-carbon reaction will be considered first. The two reactions which occur when steam is contacted with a carbonaceous material are:

\[
\begin{align*}
C + H_2O &\rightarrow H_2 + CO \quad \Delta H = -56,400 \\
C + 2H_2O &\rightarrow CO_2 + 2H_2 \quad \Delta H = -38,750
\end{align*}
\]

The former reaction was the only one considered in determining the heat requirement since the amount reacted by each reaction is unknown. Assuming that no more than 1 lb/hr of char would be activated during the investigation, the heat of reaction was calculated.

\[
Q = \frac{1\ lb/hr}{12 \ lb \ C/1b \ mole} \times \frac{-56,400 \ Btu}{1b \ mole} = 4,700 \ Btu/hr \ or \ 1.38 \ kw
\]

This calculation was made on the basis that all the char would be reacted so the value was in excess of that actually required.

Another heat requirement was for heating the steam from the reactor inlet temperature (1000° F) to the reaction temperature (1800° F) with a steam rate of 1.65 lb/hr and a temperature difference of 800° F. The steam heating requirement was 0.22 kw as the following calculation indicates.

\[
Q = 1.65 \ lb/hr (0.57 \ \frac{Btu}{lb \ \circ F}) / 3413 \ \frac{Btu}{kw-hr} = 0.22 \ kw
\]
Heat losses to the air was the last heat requirement accounted for. It was assumed that the maximum loss through the furnace walls should be no more than 1 kw. The required insulation thickness was calculated from this assumption and from outside and inside wall temperatures of 70° F and 200° F respectively. Firebrick and rock wool were used as insulating materials. Various combinations were calculated until the losses were approximately 1 kw and the temperature between the brick and the rock wool did not exceed the maximum allowable for the rock wool. The final dimensions were 4 1/2" of firebrick and 4" of rock wool. (see figures 6 & 7) The furnace height was based on the length of the reaction tube. In addition to the 24" reactor, 12" of insulation were placed at the top and bottom of the heating element to prevent end losses. This gave a total height of 48". The following is the final calculation for the heat losses from the furnace using the above dimensions. Radial losses were the only ones considered and the insulation thicknesses were expressed as equivalent radii. (See Appendix C)

<table>
<thead>
<tr>
<th>Material</th>
<th>K (Btu/hr ft ° F/in)</th>
<th>Thickness (in)</th>
<th>Equivalent Radius (in)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Firebrick</td>
<td>0.20</td>
<td>4 1/2&quot;</td>
<td>r₁ (center to firebrick)=0.239 ft</td>
</tr>
<tr>
<td>Rock wool</td>
<td>0.048</td>
<td>4&quot;</td>
<td>r₁ (center to rock wool)=0.715 ft</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>r₂ (center to outside wall)=0.917 ft</td>
</tr>
</tbody>
</table>

Equation:

A \[ \Sigma R = \frac{\Delta T(\text{total})}{q} \]
B \[ R(\text{Firebrick}) = \frac{\ln r_1/r_1}{2\pi L K_{F.B.}} \]
C \[ R(\text{Rock Wool}) = \frac{\ln r_2/r_1}{2\pi L K_{R.W.}} \]

Calculations:

\[ R_{F.B.} = \frac{\ln 0.715/2\pi 4(0.21)}{0.239} = 0.209 \quad \text{°F-hr/Btu} \]
\[ R_{R.W.} = \frac{\ln 1.13/715/2\pi 4(0.048)}{0.537} = 0.537 \quad \text{°F-hr/Btu} \]
\[ \Sigma R = 0.746 \]
\[ Q(\text{losses}) = \frac{\Delta T_{\text{total}}}{\Sigma R} = \frac{2000-70}{0.746} = 2,590 \quad \text{Btu/hr or 0.76 kw} \]

The temperature between the rock wool and firebrick follows:
Fig. 6 ELECTRIC FURNACE VERTICAL CROSS SECTION.
Fig. 7 ELECTRIC FURNACE.
\[ \Delta T = Q \frac{R}{R.W.} = (2,590 \frac{Btu}{hr})(0.573^\circ F/hr) = 1,390 \]
\[ T_w = 2000 - 1390 = 610^\circ F \]

The results from these calculations are summarized below.

<table>
<thead>
<tr>
<th>Description</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Firebrick thickness</td>
<td>4 1/2&quot;</td>
</tr>
<tr>
<td>Rock wool thickness</td>
<td>4&quot;</td>
</tr>
<tr>
<td>Furnace height</td>
<td>48&quot;</td>
</tr>
<tr>
<td>Outside wall temperature</td>
<td>70° F</td>
</tr>
<tr>
<td>Firebrick and rockwool junction temperature</td>
<td>610° F</td>
</tr>
<tr>
<td>Inside furnace temperature</td>
<td>2000° F</td>
</tr>
</tbody>
</table>

The furnace in its final form is shown in figures 6 and 7. After the furnace was constructed, a series of tests were run to determine the actual heat losses at various temperatures. Figure 8 is an illustration of the results. These tests were accomplished by calculating the power input to the furnace for a given period of time.

**Heating Element Design**

The heating element of the furnace is also shown in figures 6 and 7. Figure 6 shows two sections of heating element wire, one 220 volts and the other 110 volts. The original design consisted of one 220 volt coil which was 2 1/16" long. The heat output from the coil was calculated from the total length of wire in the coil and the resistance of this wire. This illustrated by calculations A and B below. Kanthol A-1 strip heating wire was used for the element, at

A) Wire length of coil

\[ L = \pi d \frac{16/5 \text{ turns}}{\text{in}} \]

B) Power output from coil

\[ R = 0.180 \frac{\text{ohms}}{\text{ft}} (47.5 \text{ ft}) = 8.55 \text{ ohms} \]

\[ \text{Power} = \frac{(220 \text{ volts})^2}{8.55 \text{ ohms}} = 5.67 \text{ kw} \]

A maximum operating temperature of 2300° F.

A comparison of the calculated heat requirements to that available from the heating element indicates that there is a considerable excess of heat. The heat required was 2.36 kw and the output was 5.67 kw, leaving an excess of 3.31 kw as a contingency factor. At this point the design was completed and the apparatus was ready for operation.
FIG. 8 HEAT LOSS FROM ELECTRIC FURNACE
Operating Procedure

The operational procedure for the batch system involved (1) preheating the entire system, (2) charging the sample and (3) discharging the sample.

To assure that all parts of the system in contact with steam were properly heated, a flow of inert gas was passed through the entire system for one hour while the superheater and furnace were in operation. Steam condensation occurred in the cyclone during the first few runs which made it necessary to perform this preheating procedure.

Considerable difficulty was encountered in charging the reactor. The samples had to be charged while the reactor was heated otherwise the one hour warmup period would result in carbonization of the sample before steam could be applied. At first, the samples were charged with a funnel. This method proved unsuccessful since each time a small portion of the sample was charged and came into contact with the heated reactor, the resulting reaction gases forced the sample back through the funnel. The problem was finally solved by placing a cloth over the funnel and releasing the entire sample into the reactor at once. This procedure allowed the reaction gases to pass through the cloth without loss of the sample.

After charging and preheating the reactor, steam was passed through the sample for two hours. The steam rate had to be adjusted during the run since the density of the particles decreased as the reaction proceeded. The steam rate was controlled so that only a few particles would appear in the collecting jar.

The samples were discharged after a reaction time of two hours. Discharging was accomplished by closing the steam valve and allowing a flow of inert gas to expel the particles into the cyclone separator. The product was removed from the collecting jar and placed in sealed containers to prevent it from picking up moisture from the air. This procedure was used for all the batch runs.

Revisions of the Batch System

The first problem encountered with the batch system was the condensation of steam in the collecting jar. It was not feasible to place insulating materials around the jar since it was used to observe particle emission from the reactor. The problem was solved by jacketing the glass jar with heated mineral oil in a glass beaker.

Another problem encountered was the temperature differential between the top and bottom of the reactor. The data sheet, in table 1 of Appendix B, from one of the runs illustrates that the differential
is 300° F in some cases. This situation is contradictory to the situa-
tion of uniform temperature which should occur in a fluidized bed.
The author believes that the contradiction is due to the highly endo-
thermic nature of the steam-carbon reaction. This reaction occurs
almost instantaneously and most of the reaction takes place at the
instant the steam and char are contacted. As the steam passes through
the tube it becomes less and less concentrated due to reaction at
lower points in the tube. The result is less reaction in the upper
section of the tube and a corresponding higher temperature. In other
words, the bed is not sufficiently turbulent to transfer the required
amount of heat to the bottom of the reactor. It was concluded that
the temperature differential should have no effect on the product.
The element was, however, revised later when a rupture in the reaction
tube made it necessary to replace the element. This is discussed in
a following section.

A final problem with the batch system was the irregularity of the
product. Product formation occurred in two portions, one with a dark
appearance, and the other with a color exactly similar to the raw
material. The two portions were tested for activity, and it was
found that the later portion had not been activated at all. A possible
explanation lies in the charging procedure. As mentioned previously
the samples are charged in portions and approximately fifteen minutes
are required to charge the entire sample. Since the reactor was
preheated to 1750° F, the first portions charged are actually being
carbonized for fifteen minutes at this temperature. This was con-
sidered to be sufficient carbonization to render the first portion
unreactive to steam. The steam was applied immediately after charging
the sample and therefore the last portions charged are immediately
contacted with steam. Therefore, the last portions charged are
activated while the first portions are unaffected by steam and are
inactive.

The only possible way to avoid the effects of this charging
time interval was to contact the steam and char immediately upon
entrance into the reactor. This was accomplished by continuously
feeding the char into the steam entrance line. The result was a
uniform product. The continuous feeding also made it possible to
specify flow and temperature conditions in a run after steady state
conditions were reached. This was not possible in the batch system
as may be seen from table 1 by reading down the steam rate and
temperature columns.

Continuous Feeding

The objective in designing the continuous feeding system was
to contact steam and char continuously in the reactor. The product
was to be removed continuously as its density decreased with activation.
The only change required was the addition of a char feeder to the
system. Figure 2 indicates the equipment and flow lines for the
continuous feeding system.
The coal feeder required for continuous feeding is illustrated in figure 9. It was designed to operate in the range of 0 to 500 gm/hr feed rate. This was accomplished by using a constant speed motor with an adjustable drive wheel which operated the screw of the feeder from 0 to the maximum rpm of the motor. The corresponding feed rates ranged from 0 to 500 gm/hr respectively.

Revision of the system

The first problem encountered with the continuous feeding apparatus was the condensation of steam in the feeder. An attempt was made to solve this problem by placing a pressure balancing line between the top of the feeder and the point where the char is introduced to the steam line. (refer to figure 9) Inert gas at a flow rate of about 3% of the total steam flow was passed through the pressure line. A few runs were made employing this revision without success. The pulsating effect of the fluid bed still forced steam into the feeder unless the inert gas flow was increased to about 10% of the total steam flow. The problem was finally solved by placing a cone directly above the steam entrance line. (refer to figure 9) This cone increased the inert gas velocity into the steam line by a venturi effect and also decreased the size of the opening through which the steam could pass.

A few runs were made with the revised apparatus when a failure in the reaction tube and heating element occurred. The failure in the tube was partly due to the highly corrosive action of the sulfureous materials in the Dickinson char. The corrosion caused failure along the thermocouple groove as may be seen from figure 10. Another contributing factor for the failure was the large temperature gradient between the top and bottom of the reactor. This gradient caused differential stresses vertically in the tube and failure along its weakest point, that is the thermocouple groove.

The tube rupture also caused the entire heating element to collapse. Apparently, the bulge from the tube rupture was sufficient to crack the entire element. The tube and element were repaired with a few revisions being made in the heating element.

Two sections of heating element wire were placed in the revised heating element. The revision was made to eliminate the vertical temperature gradient in the reactor. The top section, where less heat was required was supplied with 110 volts while the bottom was supplied with 220 volts. (see figure 7) Separate recording controllers were used for each section. The revision resulted in a uniform temperature throughout the reactor as may be seen from run 43, table 2, of Appendix B. The table illustrates that the wall temperature was about 100° F higher than the temperature inside the tube, but this is reasonable since the heat must be transferred through the tube wall.
Pressure equalizing line

Inert gas, in

Pressure equalizing line

Auger: \( \frac{3}{8} \)" DIAM. shaft with \( \frac{1}{2} \)" teeth.

\( \frac{3}{8} \)" DIAM. pipe tapering to \( \frac{1}{4} \)" DIAM. cone, connected by 4 unions.

Steam, in

To reactor

Fig. 9 COAL FEEDER
Fig. 10 RUPTURED TUBE
The new element and reactor were assembled and a series of runs was attempted at low steam velocities to increase the reactor residence time. Some difficulty was encountered with this series due to the obstruction of the inlet line with char at low velocities. The initial design contained a 3/4" entry line from the feeder to the reactor. At the low velocities the particles settled at the bottom of this entry line and eventually plugged the entire system. The situation was simulated with a glass tube. Smaller diameter glass tubes were employed until the velocity was sufficiently high to entrain the particles. A 1/4" diameter was found to work well with these low velocities and the entry line was revised to that shown in figure 9. At this point an operable and working apparatus was obtained. Figure 11 is an illustration of the entire apparatus in its final form.

Operating procedure

The operating procedure for the continuous feeding system involves preheating, steam introduction, feed introduction, and sample collecting. The preheating procedure is the same as that discussed in the batch reactor section.

After preheating the reactor, the steam flow rate is adjusted to the desired value. Steam flow continues for about 10 minutes until the temperature becomes stable.

The feeder is then turned on and the system is allowed to reach steady state. Steady state conditions can be ascertained by observing the temperature records. When the temperature became stable sampling was begun.

The first portion of the sample is discarded. The jars are changed by closing the valve at the bottom of the cyclone and allowing the product to build up momentarily in the cyclone. The jar is replaced and the product is collected and placed in a sealed container.

Instrumentation

To clarify the operating procedure the instrumentation involved in the final working apparatus will be explained. The variables which had to be controlled are temperature, steam velocity, inert gas velocity, and char feed rate.

Temperature was controlled with two recording controllers and one recorder. The recording controllers received input temperature signals from the thermocouple located inside the reactor. One recording controller was used for each of the heating element sections.

Steam flow rate was controlled by measuring the water input to the steam generator with a flowmeter. The flowmeter calibration is shown in figure 12.
Fig. 11 FINAL APPARATUS.
FIG. 12 WATER ROTAMETER CALIBRATION
FIG. 13 FEEDER CALIBRATION
Another item which required controlling was the inert gas flow through the coal feeder. This item was controlled with a gas rotameter.

The final variable requiring control was the coal feed rate. This was accomplished by varying the speed of the screw. Figure 13 is a calibration for the feeder.

Having obtained an operable apparatus at this point in the investigation, a series of four tests was begun to carry out the secondary objective of the investigation.

CHAPTER III
METHODS AND RESULTS OF ANALYSES OF THE PRODUCT

This section of the paper deals with the methods used to evaluate the product and the results of these evaluations. Various methods of analyzing activated carbon are discussed. The results are divided into those obtained from batch reactor activation, and continuous feeding reactor activation.

Methods of Analyses

During the literature survey, it was found that there are numerous applications and tests for activated carbon. Each application usually requires a different test. Some examples of this are the phenol adsorption test for water purification, service time test for gas adsorption, and the molasses test for decolorizing. Other tests used to determine activity are acetic acid adsorption, iodine adsorption and the heat of wetting test (31).

In view of the many tests for activated carbon it was decided to limit the testing procedure to two tests which could be used to compare the product from this investigation with Darco activated carbon. The service time and heat of wetting tests were chosen. This choice was based on the reproducibility, and simplicity of the tests. Another reason for employing these tests is that they employed chemicals which did not react with the product. (carbon tetrachloride and benzene).

During the early phase of testing, the acetic acid adsorption test was applied and it was found that the acid reacted with the product. When the acid was added to the char an H$_2$S odor was given off and upon titration of the residual acid, a precipitate was formed. The following are possible reactions which occur. (Other sulfides could cause a similar reaction.)

\[
\begin{align*}
\text{FeS} + 2\text{CH}_3\text{COOH} & \rightarrow \text{H}_2\text{S} \uparrow + 2\text{CH}_3\text{COO}^-\text{Fe}^{++} \\
\text{Fe}^{++} + 2\text{NaOH} & \rightarrow \text{Fe(OH)}_2 + 2\text{Na}^+.
\end{align*}
\]
Since it would be difficult to determine what amount of acid was actually adsorbed the test was abandoned. Other tests involving acidic or basic solutions were also eliminated for this reason and only the heat of wetting and service time tests were used.

Service Time test

The service time testing apparatus is shown in figure 14. In this test a known quantity of air is scrubbed in concentrated sulfuric acid and saturated with CCl₄ at 0° C. The air-carbon tetrachloride mixture then passes through a bed of the active carbon to be tested and the carbon tetrachloride is adsorbed from the air stream. When the first trace of carbon tetrachloride breaks through the bed, the time is recorded and termed the service time of the active carbon. The break through point is determined with a copper coil and bunsen burner. A green color appears when the carbon tetrachloride makes contact with the coil.

Each of the samples was tested at constant conditions. The air flow rate was controlled at 650 ml/min with a glass orifice meter, the calibration of which is shown in figure 15. The volume of active carbon used for each test was also held constant. This was accomplished by measuring 15 cm of the active carbon into a 1.3 cm I.D., U shaped tube. With these conditions constant the test was reproducible within 5% as table 3 in the Appendix indicates.

Heat of Wetting test

This test is a measure of the temperature rise when a weighed sample of activated carbon is added to benzene. The heat of wetting is recorded as degrees temperature rise per gram of active carbon. A high heat of wetting indicates a highly adsorptive carbon since the heat liberated is due to the heat of adsorption of the benzene unto the carbon.

This test was performed on a constant weight basis. Five gram samples were weighed in duplicate and added to 40 cc of benzene. Both the sample and benzene were allowed to reach room temperature before the test was performed. A maximum temperature was reached in 30 seconds for most of the samples. The reproducibility of this test is also shown in table 3 of the Appendix.

Results from analyses of the product

A total of 43 runs were made with the apparatus, 19 of which were made with the batch reactor and the remainder with the continuous feeding apparatus. All the runs were made with Dickinson char as the raw material. Appendix A contains an ultimate and proximate analysis of the Dickinson char. In each case the products obtained were compared to Darco activated carbon. Its analysis is also shown in the appendix.
Fig. 14 SERVICE TIME TESTING APPARATUS.
FIG. 15 GAS ADSORPTION FLOWMETER CALIBRATION

PRESSURE DROP OF OIL AT 1.75 - SPECIFIC GRAVITY

$\Delta P$ (in of oil, Sp. G. = 1.75)

FLOW RATE, $ft^3/min$

0.005 0.01 0.015 0.02 0.025 0.03
Results from batch activation

Initially, the investigation was to be carried out in a batch reactor. The objective of the first series of tests in the batch system was to determine the optimum temperature for activation. For this series, the temperature was varied from 700 to 1900° F while the particle size, sample size, and steam rate were held constant. Figures 16 and 17 depict the evaluation of the resulting products. The steep rise in the curves between 900 and 1100° F indicates the effect of the steam carbon reaction which, according to the literature, becomes significant at 1000° F. Both tests indicate that the product is improved at higher activation temperatures. There was no optimum temperature in the range covered, and the range could not be extended to higher temperatures because of the design limitation on the reactor and heating element.

The results from testing the Darco activated carbon are also indicated on figures 16 and 17. Both tests indicate that the present product compares favorably with Darco grade 2. The top Darco product is, however, considerably better than the present product. As mentioned previously, the product from the batch runs was obtained in two portions, one active and the other inactive. The portions were mixed and tested together since separation of the two was difficult. This resulted in a lower activity than that obtained by activating the entire sample. For this reason, the apparatus was revised and no other variables were investigated in the batch system.

Results from continuous activation

The objective of the tests in the continuous feeding apparatus was to determine the optimum temperature, feed rate, particle size, and residence time for activation. Due to the limitation in time for the determination of these optimums, the number of tests made had to be limited. Four series of four runs each were made using constant conditions for all variables except the one investigated in each series. The constant conditions around with the tests were made were 1750° F, -40, +60 mesh particles, 0.578 ft³/min steam rate, and 300 gm/hr feed rate. These conditions appeared to be optimum from the literature and from the experiments performed in a glass tube. All four series were run and then the resulting products were tested for activity. The results from testing the temperature, particle size, and feed rate series indicated that any effect from these variables on the product was cancelled by the effect of residence time variations.

The temperature series best illustrates this conclusion. The results from testing this series are shown in figures 18 and 19. Both tests indicate a decline in activity as temperature increases. A possible explanation for this situation is that the increasing temperature increased the gas velocity through the reactor. The
FIG. 16 SERVICE TIME VS ACTIVATION TEMPERATURE
FIG. 17  BATCH SYSTEM, HEAT OF WETTING VS ACTIVATION TEMPERATURE
FIG. 18 SERIES 1, SERVICE TIME VS ACTIVATION TEMPERATURE*

(*Service time apparatus revised at this point)
FIG. 19 SERIES 1, HEAT OF WETTING VS ACTIVATION TEMPERATURE

Darco: Grade 1 - 0.82 °C/gm
     Grade 2 - 0.67 °C/gm
result is less reaction time and lower activity for the product. In order to determine an optimum temperature, the steam rate through the reactor would have to be corrected so that the residence time would be the same at each temperature.

The second series of tests was performed to determine the effect of particle size variation on the product. Figures 20 and 21 are the evaluation of the series. Both curves show a higher activity as the particle size is increased. A smaller particle size presents more surface area for reaction and therefore should have a higher activity than a corresponding large particle size. The contradiction is again explained in terms of residence time. For the same flow rate through the reaction, large particles require a longer residence time as compared to small particles. Apparently, the effect of the increased residence time of large particles surpassed the effect of a high surface area for small particles. To obtain an optimum particle size, the steam flow rate would again require a correction factor to eliminate the variations in residence time.

Figures 22 and 23 represent the results from the feed rate variation series. The graphs indicate that the variation in feed rate had little effect on the activity of the product. The percent yield for each feed rate was constant, which verifies that the amount of reaction changed insignificantly. Since there was no effect on the product with a variation in feed rate, it was concluded that there was a sufficient amount of steam flow to react with the char. Another conclusion which can be made is that the fluidizing properties were unchanged in the range of feed rates covered. Any change in these properties would have been reflected in a change in the activities and yields obtained.

The final series of runs was made with a variable steam flow rate, and the results are illustrated in figures 24 and 25. The figures indicate a substantial improvement in the product at the lower velocities. This improvement is attributed to the higher particle residence time at the low velocities. Apparently, the product would continue to improve with increasing residence time with the only limitation being on the yield of the product. Table 3 of Appendix B indicates the yield and residence time for this series of runs. Theoretically, the residence time could be increased until the entire sample was reacted providing that there is sufficient steam flow for reaction. The rate of 0.275 ft³/hr was the lowest which could be run with the apparatus used in this investigation. Lower velocities resulted in obstruction of the entrance line with char.

This series of runs points out the fact that the residence time was not sufficient in the temperature, particle size, and feed rate series to obtain a product comparable to Darco's activated carbon. Reference to figures 18 through 23 indicate that none of the runs in the later series of tests compared to Darco's product. These runs were all performed at a steam rate of 0.578 ft³/min compared to rates
FIG. 20 SERIES 2, PARTICLE SIZE VS SERVICE TIME
FIG. 21 SERIES 2, PARTICLE SIZE VS HEAT OF WETTING
FIG. 22 SERIES 3, FEED RATE VS SERVICE TIME

Darco: Grade 1 = 10.37 min
Grade 2 = 8.86 min
FIG. 23 SERIES 3, FEED RATE VS HEAT OF WETTING

**Darco:**
- Grade 1 - 0.82 °C/gm
- Grade 2 - 0.672 °C/gm
FIG. 24 SERIES 4, STEAM RATE VS SERVICE TIME
FIG. 25 SERIES 4, STEAM RATE VS HEAT OF WETTING
of 0.25 ft³/min and 0.35 ft³/min for the last two runs of the steam rate series (figures 24 and 25). These later two runs resulted in a product which had better properties than Darco's activated carbon.

As discussed previously, the rate of 0.578 ft³/min was chosen on the basis of experiments in a glass tube. This velocity gave fluidized conditions in the glass tube with very few particles being lost in the exit stream. There are two reasons why this velocity was too low to hold the particles in the reactor under actual reaction conditions. The first is due to the reaction gases contributing to the total gas flow rate through the reactor. The inlet gas flow would then have to be lower than 0.578 ft³/min to account for these additional gases. The second reason for the discrepancy between flow conditions in a glass tube and those under reaction conditions is due to the substantial change in density as the particles begin to react (see figures 26 and 27). A lower velocity would then be required to maintain the particles in the reactor.

The conclusions from the data and results are summarized below.

1. The particle size, temperature, and feed rate series were performed at velocities too high to give a sufficient residence time for activation. These series of runs did not compare to Darco activated carbon as determined by the heat of wetting and service time tests. In each series, no conclusion could be made regarding an optimum due to the predominance of the effect of change in the residence time with a change in the variable.

2. The steam rate variation series verifies the above conclusion. Low steam rates (high residence time) resulted in a product better than Darco activated carbon.

Time was not available to reinvestigate the temperature, particle size and feed rate series at the lower velocities of the steam rate series. In future tests the steam velocity should be corrected in each series so that the residence time will not vary with a change in the variables being investigated.

CONCLUSIONS AND RECOMMENDATIONS

The objectives of this investigation were to determine the feasibility of producing activated carbon from lignite by activation with steam in a fluidized reactor, to design an operable reactor in which tests could be performed to determine this feasibility, and to determine and examine the variables involved with the apparatus. The degree of accomplishment of these objectives is reflected in the following conclusions.
FIG. 26  SERIES 4, BULK DENSITY VS SERVICE TIME
FIG 27 SERIES 4, STEAM RATE VS BULK DENSITY
1. An operable apparatus has been designed and constructed.

2. The variables involved with this apparatus are temperature, feed rate, particle size, and steam rate. Examination of the first two variables was prohibitive due to a variation in residence time while investigating these variables. Examination of the feed rate variable indicated no effect of varying the feed rate on activity. Examination of the steam rate indicated a better product than Darco at steam velocities below 0.35 ft³/min.

3. It is feasible to produce activated carbon from lignite by the fluidized steam activation method. The resulting product is as good or better than Darco activated carbon.

   The conditions at which active carbon may be produced from lignite are at a temperature of 1750°F, with a particle size of -40, +60 mesh, a steam rate lower than 0.35 ft³/min, a feed rate of 300 gm/hr.

Along with these conclusions, the following recommendations are made.

1. The temperature and particle size series should be reinvestigated at steam rates below 0.35 ft³/min to determine the best conditions for activation.

2. Tests other than the heat of wetting and service time should be applied to the products to determine whether the present product is applicable in more specialized areas.

3. The effect of variation in lignite source on the activity of the product should be investigated.
APPENDIX A

The following analyses are of the raw Dickinson char, the activated carbon produced in this investigation, and the Darco activated carbon, respectively.

### RAW DICKINSON LIGNITE CHAR

<table>
<thead>
<tr>
<th></th>
<th>As received</th>
<th>Moisture free</th>
<th>Moisture and ash free</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture</td>
<td>5.84</td>
<td>----</td>
<td>----</td>
</tr>
<tr>
<td>Volatile matter</td>
<td>13.71</td>
<td>16.68</td>
<td>20.54</td>
</tr>
<tr>
<td>Fixed carbon</td>
<td>60.76</td>
<td>64.53</td>
<td>79.46</td>
</tr>
<tr>
<td>Ash</td>
<td>17.69</td>
<td>18.79</td>
<td>----</td>
</tr>
<tr>
<td>British thermal units</td>
<td>10,960</td>
<td>11,640</td>
<td>14,340</td>
</tr>
</tbody>
</table>

### ACTIVATED CARBON PRODUCED DURING THIS INVESTIGATION

<table>
<thead>
<tr>
<th></th>
<th>As received</th>
<th>Moisture free</th>
<th>Moisture and ash free</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture</td>
<td>0.90</td>
<td>----</td>
<td>----</td>
</tr>
<tr>
<td>Volatile matter</td>
<td>5.08</td>
<td>5.13</td>
<td>6.65</td>
</tr>
<tr>
<td>Fixed carbon</td>
<td>71.93</td>
<td>72.54</td>
<td>93.30</td>
</tr>
<tr>
<td>Ash</td>
<td>22.09</td>
<td>22.33</td>
<td>----</td>
</tr>
<tr>
<td>British thermal units</td>
<td>11,930</td>
<td>----</td>
<td>----</td>
</tr>
</tbody>
</table>

### DARCO ACTIVATED CARBON

<table>
<thead>
<tr>
<th></th>
<th>As received</th>
<th>Moisture free</th>
<th>Moisture and ash free</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture</td>
<td>0.80</td>
<td>----</td>
<td>----</td>
</tr>
<tr>
<td>Volatile matter</td>
<td>4.20</td>
<td>4.23</td>
<td>4.92</td>
</tr>
<tr>
<td>Fixed carbon</td>
<td>81.25</td>
<td>81.91</td>
<td>95.08</td>
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<tr>
<td>Ash</td>
<td>13.75</td>
<td>13.86</td>
<td>----</td>
</tr>
<tr>
<td>British thermal units</td>
<td>12,040</td>
<td>12,140</td>
<td>14,090</td>
</tr>
</tbody>
</table>

50
APPENDIX B

TABLE I

REACTOR TEMPERATURE DIFFERENTIAL BEFORE REVISIONS

Run 20

<table>
<thead>
<tr>
<th>Time</th>
<th>(T_T)</th>
<th>(T_2)</th>
<th>(T_B)</th>
<th>Steam flow Scf/min.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1200</td>
<td>1725</td>
<td>1800</td>
<td>1650</td>
<td>0.2</td>
</tr>
<tr>
<td>15</td>
<td>1905</td>
<td>1875</td>
<td>1645</td>
<td>0.3</td>
</tr>
<tr>
<td>30</td>
<td>2020</td>
<td>2020</td>
<td>1650</td>
<td>0.3</td>
</tr>
<tr>
<td>45</td>
<td>1920</td>
<td>1920</td>
<td>1655</td>
<td>0.4</td>
</tr>
<tr>
<td>100</td>
<td>1960</td>
<td>1870</td>
<td>1640</td>
<td>0.4</td>
</tr>
</tbody>
</table>

\(T_T\) = Top thermocouple

\(T_2\) = Wall thermocouple

\(T_B\) = Bottom thermocouple

TABLE II

REACTOR TEMPERATURE DIFFERENTIAL AFTER REVISIONS

Run 38

<table>
<thead>
<tr>
<th>Time</th>
<th>(T_T)</th>
<th>(T_2)</th>
<th>(T_B)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1200</td>
<td>1850</td>
<td>1928</td>
<td>1840</td>
</tr>
<tr>
<td>15</td>
<td>1850</td>
<td>1930</td>
<td>1845</td>
</tr>
<tr>
<td>30</td>
<td>1850</td>
<td>1942</td>
<td>1860</td>
</tr>
<tr>
<td>45</td>
<td>1850</td>
<td>1930</td>
<td>1850</td>
</tr>
<tr>
<td>100</td>
<td>1850</td>
<td>1950</td>
<td>1855</td>
</tr>
</tbody>
</table>

\(T_T\) = Top thermocouple

\(T_2\) = Wall thermocouple

\(T_B\) = Bottom thermocouple

TABLE III

SERIES 4: STEAM RATE VARIATION

<table>
<thead>
<tr>
<th>Steam rate (\text{Scf/min.})</th>
<th>Percent yield of product</th>
<th>Residence time (\text{min})</th>
<th>Heat of Wetting, ° C/gm</th>
<th>Percent difference</th>
<th>Service time, min</th>
</tr>
</thead>
<tbody>
<tr>
<td>154</td>
<td>44.7</td>
<td>12.3</td>
<td>11.33</td>
<td>1.65</td>
<td>6.20</td>
</tr>
<tr>
<td>147</td>
<td>52.2</td>
<td>7.5</td>
<td>10.38</td>
<td>0.96</td>
<td>3.40</td>
</tr>
<tr>
<td>163</td>
<td>56.0</td>
<td>---</td>
<td>7.25</td>
<td>2.40</td>
<td>2.35</td>
</tr>
<tr>
<td>178</td>
<td>57.1</td>
<td>3.8</td>
<td>4.83</td>
<td>1.25</td>
<td>1.90</td>
</tr>
</tbody>
</table>

Percent difference

<table>
<thead>
<tr>
<th>Percent difference</th>
<th>A</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td>6.20</td>
<td>3.40</td>
<td>3.35</td>
</tr>
<tr>
<td>3.25</td>
<td>2.37</td>
<td>0.85</td>
</tr>
<tr>
<td>1.90</td>
<td>1.85</td>
<td>2.70</td>
</tr>
</tbody>
</table>
APPENDIX C

This method of calculation is only approximate and results in a larger value for heat losses than what actually occurs. The equation does not account for the additional insulation provided by the corners of the furnace. For a more accurate determination of heat losses the relaxation method by Eckert should be applied (32). This method would, however, be complicated since the conductivities change through the furnace walls. The Eckert method was not applied in this investigation because an accurate value for the heat losses was not required.

Another clarification in the calculation is that the calculations are based on 4 1/2" of rock wool while the furnace was constructed with 2 1/2" of rock wool and 2" of expanded vermiculite. The supply of rock wool was limited and had to be supplemented with vermiculite. Since the conductivities of the two are about equal, the calculations were based entirely on rock wool.

REFERENCES


(2) Ibid., p. 4

(3) Ibid., p. 5


(5) Ibid., p. 52

(6) HASSLER, op. cit. p. 15

(7) Ibid., p. 27

(8) Ibid., p. 16

(9) Ibid., p. 16

(10) U. S. Bureau of Mines. Technology of Lignitic Coals. p. 110

(11) Ibid., p. 110


(14) Ibid., p. 164


(16) FRANZ, Part I, op. cit. p. 164

(17) OTHMER, op. cit. p. 20


(19) Ibid., p. 90

(20) Ibid., p. 90

(21) OTHMER, op. cit. p. 212

(22) Ibid., p. 213

(23) Ibid., p. 216


(27) FRANZ, op. cit. Part I, p. 174


(30) LEVA, op. cit.

(31) Hassler, op. cit., pp. 312-15