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# A High Grade Decolorizing Carbon From Extracted Cotton Seed Hulls

By

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AUBURN, ALABAMA.

1931

A High Grade Doctoring  
Carbon from  
Carbon

The Board of Trustees of the Alabama Polytechnic Institute has made a ruling that a patent should be secured on all worthwhile processes developed through the Engineering Experiment Station. Consequently a patent application on the following process has been filed with the U. S. Patent Office. Those interested in using this method are invited to confer with Dr. Bradford Knapp, President of the Alabama Polytechnic Institute.

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## **A HIGH GRADE DECOLORIZING CARBON FROM EXTRACTED COTTON SEED HULLS**

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### **ABSTRACT**

The object of this investigation was to find a method for the profitable disposal of the large quantity of residual cotton seed hulls resulting from the extraction of xylose from cotton seed hulls.

As a result of this investigation, a process was developed for the manufacture of a high grade decolorizing carbon involving the following steps:

- (a) The residual or extracted hulls after air drying were mixed with lime and heated to 1800°F. The lime reduced the quantity of gases absorbed by the carbon. Air may or may not be added during this step.
- (b) After cooling, the lime was dissolved with hydrochloric acid.
- (c) After washing, the carbon was activated by air. This involved passing air into the carbon at 1600°F. The resulting carbon was of high quality, one gram of the best grade absorbing more than one gram of iodine from solution. This grade is superior to each of eleven commercial carbons collected from this country and Europe.
- (d) The optimum condition for carrying out the various steps were determined.
- (e) The production of the carbon was carried out on a semi-plant scale and a study made of the probable large scale equipment and cost of manufacture.

# A High Grade Decolorizing Carbon From Extracted Cotton Seed Hulls

## INTRODUCTION

The researches by which the cotton seed has been utilized, for the production of oil, are well understood. The success of this work has been responsible for the addition of much wealth to the cotton farmer. The cotton seed hulls collect in large quantities and bring a low price.

In an attempt to find a profitable method for the utilization of the hulls, the Alabama Polytechnic Institute entered into a cooperative project with the Bureau of Standards and the University of Alabama for the production of Xylose, a five carbon sugar, from this material. The work of the Alabama Polytechnic Institute was carried out through the Engineering Experiment Station. The actual experimental work was carried out at Anniston, Alabama, in cooperation with the Federal Phosphorus Co. The method employed can be found in the Industrial and Engineering Chemistry for May, 1930.

The Alabama Polytechnic Institute not only cooperated in the semi-plant production of Xylose at Anniston but it also played a lead part in the utilization of the by-products incident to the production of the Xylose. One of the chief by-products remaining after extraction of the Xylose is the residual cotton seed hulls. After hydrolysis of the Xylans into Xylose and extraction of the latter, the residual hulls remain. This material is dark brown in color and represents about sixty-five percent of the original hulls, after correcting for the increased water content of the extracted hulls.

For every pound of Xylose extracted there are several pounds of residual hulls. Consequently the successful utilization of cotton seed hulls for Xylose depends in large measure upon a profitable method for the disposal of the residue. Further, the utilization of cotton seed hulls for the production of carbon is not necessarily dependent upon the manufacture of Xylose. For example, if desired the residual hulls might be produced from the hulls solely for the purpose of making decolorizing carbon. Under these conditions, the Xylose may or may not be recovered. Also, preliminary results seem to indicate that it is possible that carbon could be made **directly** from the hulls. Only a very limited amount of work was carried out along this line.

## BIBLIOGRAPHICAL REVIEW

Nothing was found in the literature relative to the utilization of **extracted** hulls. However, the methods for the utilization of **fresh** hulls and other forms of cellulose may be conveniently classified as follows:

### Various Uses of Cotton Seed Hulls and Other Forms of Cellulose

O. Sheppard<sup>1</sup> produces a stock feed from cotton seed hulls. The hulls are reduced to bran, mixed with cotton seed meal, and steamed. This mixture is rolled into flakes.

Cotton seed hulls have been employed<sup>2</sup> for the production of wood flour.

E. C. Sherrad and G. W. Blanco<sup>3</sup> have produced reducing sugars by the acid hydrolysis of sugar cane fibre or cotton seed hulls.

Sugars have been produced by S. F. Acree<sup>4</sup> from wood, cotton seed hulls, corn cobs, etc., by the acid hydrolysis of these materials at 70 to 100°C.

P. G. Eckstrom<sup>5</sup> produces fermentable sugar from sawdust by acid hydrolysis.

Sugar is made from cellulose by R. A. Kocher<sup>6</sup> who hydrolyzes this material with hydrochloric acid of at least thirty nine per cent, in an atmosphere of hydrogen chloride.

Plastics<sup>7</sup> have been made from sawdust and formaldehyde. These have a number of uses.

The conversion of cellulose materials into paper, oxalic acid, acetate or nitrate of cellulose, and the use of cellulose material as a fuel, is too well known to deserve special mention here.

#### **Decolorizing Carbon from Wood and Other Forms of Cellulose**

Decolorizing carbons have been produced from wood and other forms of cellulose by a great variety of processes. According to Mantell<sup>8</sup> the various methods may be classified as follows:

- (1) Sawdust, seaweed, peat, and similar materials are mixed with porous substances such as infusorial earth, pumice, or insoluble salts, and the mixture heated strongly, whereupon the carbon in the vegetable matter is deposited throughout the porous base.
- (2) Vegetable materials are mixed with lime, chalk, calcium chloride, phosphoric acid and so forth and after carbonization the inorganic matter is dissolved, leaving the resulting carbon.
- (3) Vegetable materials are carbonized under controlled conditions of temperature and pressure. The carbon is then activated by air, oxides of carbon, chlorine, superheated steam or mixtures of steam and air.

Another method mentioned by Mantell<sup>9</sup> consists of treating porous woods with dehydrating agents such as lime and calcium acetate, carbonizing them at nearly a white heat under a layer of lime, dissolving the lime with acid and recarbonizing at a red heat with exclusion of air.

Special processes that appeared to be of interest and which are representative are as follows:

A. H. Bonnar<sup>10</sup> heats vegetable matter up to 1000°C. with thirty per cent of its weight of chalk, without access of air. The carbon is then washed with hydrochloric acid and dried.

A. Zelniczek<sup>11</sup> heats carboniferous materials with zinc chloride.

According to a German process<sup>12</sup>, vegetable material is impregnated with calcium or magnesium chloride solution, centrifuged, carbonized at about 350°C. with access of air, the salts washed out with acid and the carbon dried.

Muir<sup>13</sup> heated sawdust with various inorganic compounds. Calcium compounds gave the best results. Ten, twenty, fifty and one hundred parts of calcium hydroxide were employed.

An active carbon has been made by L. H. Bonnard<sup>14</sup> by incorporating moist carbonaceous material with calcium hydroxide and heating to 1000°C., until the carbon monoxide ceases. The carbon is washed with hydrochloric acid.

#### **Disadvantages of the Present Processes for Utilization of Cotton Seed Hulls**

As previously stated, apparently no work has been done on *extracted* cotton seed hulls. Most of the possible uses for *fresh* hulls were successfully

applied to *extracted* hulls by the writer. None of these processes looked particularly promising. For example it was found that the extracted hulls could be successfully briquetted without a binder (see Bull. 1, Eng. Exper. Station, Ala. Polytech. Inst. for details). These briquettes were of fair strength and no doubt could be made cheaply but it is doubtful if the process would be highly remunerative in this case. Further, by hydrolysis with concentrated hydrochloric acid, a yield of six to ten per cent of reducing sugars was secured. The cost of the acid is considerable and at best this process could yield only fair profit. Also, cellulose acetate was made from the extracted hulls but the yield was so low that this process did not look promising. Oxalic acid was produced from the extracted hulls. The yield was from fourteen to eighteen per cent. This process did not look very promising due to the limited market for the acid. The production of wood flour from the extracted hulls has the same objection as the production of oxalic acid.

### A NEW DECOLORIZING CARBON FROM A NEW MATERIAL

It has been seen that few of the miscellaneous processes outlined above show much promise of commercial success. Consequently it was thought advisable to study the production of a decolorizing carbon from the extracted hulls. These carbons commands a good price and have a broad market.

Carbon made by heating extracted hulls with inorganic materials such as slag or bauxite had some decolorizing action but was of high ash content. The unsatisfactory nature of this carbon suggested the use of an inorganic material that could be removed with acid. As stated later, lime was selected for this purpose due to its cheapness and great covering power. After removal of the lime with acid, an improvement was noted in the quality of the carbon when the latter was activated, i.e., heated to a red heat in the presence of air, steam, etc. The effectiveness and the cheapness of air suggested its use for this purpose.

Carbons made by the process outlined above were of unexpected quality, and it became apparent that it was possible to produce a carbon of a desirable type from this waste material. The record of this investigation is presented below.

### EXPERIMENTAL WORK

Note: Since all instruments used in this work were calibrated for the English system of units, all temperatures are reported as degrees fahrenheit.

Preliminary work indicated that to remove adsorbed gases during the first heating with lime, it was necessary to heat to approximately 1800°F. This temperature is above the convenient range of gas, but is within the range of the electric furnace. Only two materials seemed to be available for the construction of the retort which would withstand the high temperature employed. These were earthenware or porcelain and iron or steel. The former was not seriously considered because of the difficulty in securing the proper shapes, breakage, and possible reaction of the lime with the clay of the earthenware.

Consequently a retort which would hold a convenient quantity of material was made from a black iron pipe ten inches long and two and one half inches in diameter. The pipe was provided with caps, one of which contained an opening one quarter of an inch in diameter for the escape of the gases.

The retort was heated by a Hoskins crucible furnace with an inside diameter of four inches and six inches deep.

The final heating was found to require a temperature of 1600°F. Consequently a retort was made from a black iron pipe six inches long and one and one-half inches in diameter. The air was carefully metered by a calibrated wet gas meter, and passed into the bottom of the retort through a copper tubing one quarter of an inch in diameter (inside diameter). The tubing was provided with two small holes, made with a small nail, on opposite sides of the tubing and one quarter inch from the end of same. The holes were for the even distribution of the air throughout the charge.

#### Materials and General Procedure.

A large quantity of the extracted hulls was air dried and placed in an air tight can to serve as a sample. Analysis of this material is given in Table I.

**Table I**  
Analysis of Extracted Hulls

Moisture percent by weight	Ash percent by weight	Size percent held	Ultimate analysis percent by weight carbon—dry basis
30.0	0.58	21 held by 20 mesh 92 held by 48 mesh	48.88

The fibre (largely cellulose) amounted to 47.11 per cent, while the fat was 0.58 per cent.

Commercial lime was employed in this work. A suitable amount of this material was secured and placed in an air tight can. Analysis of this product is given in Table II.

**Table II**  
Analysis of Lime

H <sub>2</sub> O + CO <sub>2</sub> -----	25.81 (percent)
Insoluble matter -----	1.32
Fe <sub>2</sub> O <sub>3</sub> & Al <sub>2</sub> O <sub>3</sub> -----	1.45
CaO -----	70.73
Size. All through 48 mesh.	
All through 100 mesh.	
2% held by 200 mesh.	

The course of a run was usually as follows: One hundred and fifty grams of extracted hulls were carefully mixed with the required amount of lime (this was later found to be fifteen grams), and the mixture placed in the retort. This quantity of material filled the retort nearly half full. The retort was then placed in the electric crucible furnace where it was heated to the desired temperature. As the temperature of the furnace increased the gaseous products resulting from the decomposition of the hulls made their appearance. These were followed by considerable quantities of carbon monoxide at higher temperature. After practically all the gaseous products resulting from the decomposition of the hulls were gone (end of first hour) the contents of the retort were stirred with an iron rod at ten minute intervals. In some cases air was passed into the retort during this heating. When the contents of the retort had been heated for the proper length of time at the



desired temperature, the retort was allowed to cool. The small outlet in the top of the retort was closed with asbestos during the cooling.

After the retort had cooled, the contents were transferred to a beaker, an excess of dilute hydrochloric acid (70cc. HCl and 140 cc. H<sub>2</sub>O) added, the mixture stirred, warmed for about fifteen minutes, filtered and the carbon washed until the washings were neutral to litmus.

The carbon was then divided into two equal portions. Half was placed in the small retort where it was heated to a red heat. The required amount of air was then slowly added through the copper pipe. When the air treatment was completed, the opening in the top of the retort was closed with asbestos, and the retort was allowed to cool. After cooling, the contents of the retort were screened and treated with a magnet to remove the iron scalings from the retort, ground in a Braun Type U. A. Pulverizer and the adsorptive value determined by means of an iodine solution (see below). In every case the per cent yield was calculated on the basis of the original hulls rather than on hulls plus lime.

It should be mentioned at this time that the use of iron retorts for this work is not ideal. At the temperature employed, the retorts scaled and corroded appreciably. It was found that if the caps were tightly screwed on the ends of the pipe after the threads had been leaded, the hammering necessary at times to remove them increased the scaling and weakened the retorts. Consequently the cap forming the bottom of the retort was tightened until air tight (tested by water) while the top cap which must be constantly removed was rather loosely tightened.

#### TESTING OF CARBONS AND DETERMINATION OF OPTIMUM CONDITIONS

Before the optimum condition for making the carbon could be determined, it was necessary to establish a method for the evaluation of this material. In the first work done along this line, a ten per cent brown sugar solution was employed. This did not prove to be very satisfactory since it was difficult to determine what proportion of the color was removed from solution by the carbon. Attention was then directed to the use of a standard iodine solution for this purpose. This method has the advantage that the quantity of iodine adsorbed can be very accurately determined. Chaney<sup>15</sup> finds that all carbons of equivalent porosity or apparent density, possess adsorptive capacities for *colloidal particles* directly proportional to their activity (iodine adsorbed). Since the use of decolorizing carbon in the great industries using large quantities of this material, such as sugar, glucose, petroleum, water purification and others depend upon the absorption of *colloidal particles*, it was believed that the iodine adsorption method would give a definite and satisfactory method for the evaluation of the carbon. A curve showing the relation between the amount of iodine absorbed and the decolorizing power of activated carbons for sugar solution, is given in "Industrial Carbon" by C. L. Mantell, page 197.

The method as given by Chaney<sup>16</sup> for the determination of the amount of iodine adsorbed, consists of shaking one gram of dried carbon (200 mesh) with 50 cc of 0.2 Normal iodine-potassium iodide solution for three minutes. The solution is filtered, and the first runnings discarded. The iodine concentration of an aliquot portion of the filtrate is determined by titration.

It should be mentioned at this time that the carbon made from the extracted

hulls adsorbs carbon *after* three minutes. In other words a period of contact of three minutes between the carbon and the iodine solution is not sufficient for the carbon to adsorb all of the iodine which it is capable of adsorbing. This is clearly shown in Fig. 1.

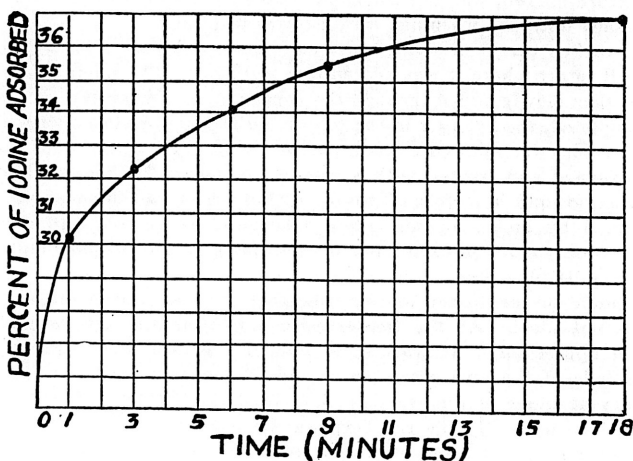


Fig. 1.—Relation Between Iodine Adsorbed and Time

It is seen that the quantity of iodine adsorbed continues to increase up to eighteen minutes. At this point the curve is nearly horizontal.

While the carbons adsorb more iodine *after* three minutes, nevertheless the results secured at three minutes are comparative. Consequently three minutes was adopted in all this work.

The method employed for the adsorption of the iodine is essentially the same as that described above. One gram of dried carbon (for the mesh of this carbon see the following section) was shaken in a small Erlenmeyer with 50 cc of 0.2 Normal iodine-potassium iodide solution for three minutes, filtered through a small Buchner funnel, the first twenty-five cubic centimeters discarded, ten cubic centimeters of the remainder accurately measured in a small Erlenmeyer, about fifty cubic centimeters of distilled water added, and the solution titrated with standard sodium thiosulphate solution, using starch as the indicator.

Note: Results secured in this way are probably not strictly comparable to those secured by Chaney, due to a difference in the mesh of the carbon.

#### Degree of Fineness.

Preliminary work indicated that the degree of fineness affected the quantity of iodine absorbed. Consequently before the optimum conditions for making the carbon could be determined, it was necessary to select a definite degree of fineness.

Carbons of several degrees of fineness were made in the Broun pulverizer and the quantity of iodine adsorbed determined by the method outlined above. The results are given in Table III.

**Table III**  
Relation Between Fineness and Adsorption of Iodine.

Sample No.	Screen Analysis				Iodine Added	Percent Iodine Removed
	On 50 mesh percent	On 100 mesh	On 200 mesh	Through 200 mesh		
2	1.6	34.6	34.1	29.7	1.1295 gm.	58.86
3	0.16	0.36	21.7	77.78	1.1295 gm.	66.37
4	31.5	51.7	7.7	9.1	1.1295 gm.	60.42
5	0.3	13.2	31.7	54.8	1.1295 gm.	65.86

It will be noted that the amount of iodine adsorbed reaches the maximum in number three which is also the finest of the samples. It is believed, however, that this carbon is finer than is desired. Undue fineness leads to considerable loss as dust. The difficulties in filtering a carbon of this fineness might be considerable. Number five although considerably coarser than number three shows nearly the same amount of iodine adsorbed. This sample is rather closely comparable to Norite in fineness, but somewhat finer than a number of industrial carbons given by Mantell<sup>17</sup>. Taking everything into consideration it was decided to select the degree of fineness represented by number five for all future work. This degree of fineness can be easily secured by the Braun pulverizer. The filtering action of a carbon of this degree of fineness apparently is satisfactory.

Further work on the degree of fineness showed that the quantity of iodine adsorbed was almost directly proportional to the per cent passed through a 200 mesh screen.

#### Amount of Lime.

The object of the lime is to prevent the adsorption of gases given off during the decomposition of the hulls. Other inorganic compounds have the same effect as the lime, but the latter was selected due to its great covering power, the ease with which the excess can be removed later, and its cheapness.

The proper quantity of lime was the object of considerable investigation. It is evident that as the quantity of lime is increased, the quality of the carbon should steadily increase. This being the case the quantity of lime should be as great as possible. On the other hand, when the quantity of lime exceeds a certain point it appears to have little effect on the quality of the carbon and might be said to merely dilute this material. Further, since it is necessary to remove the excess lime with an acid (preferably hydrochloric acid), the cost becomes prohibitive when the lime exceeds a certain point.

It is clearly advantageous to keep the quantity of lime as low as possible as long as the quality of the carbon can be maintained. In the preliminary work 150 grams of hulls and 20 grams of lime were used. The lime was clearly in excess here since it had a tendency to separate out on standing. Further, the cost of the lime and the necessary acid would be considerable when this quantity is used. With these points in mind a series of carbons were made using 150 grams of hulls and 20, 15, 10 and 5 grams of lime respectively. The preliminary conditions employed in making these carbons

are as follows: Temperature of heating with lime 1700° F, time of heating three and one half hours (one hour at 1700° F), stir every ten minutes after the first hour: rate of heating, two and one half hours required to secure 1700° F. The contents of retort are allowed to cool, the lime dissolved with an excess of dilute hydrochloric acid (210 cc of HCl 1:2), the solution warmed, filtered and the carbon washed until neutral. The carbon was then divided, half was heated to 1800° F. and an excess of air (1.215 cu. ft.) was passed into same over a period of one and one-fourth hour. As stated previously the carbon was then ground in a Braun pulverizer and tested with the iodine solution. As expected the yields secured with various amounts of lime were practically constant. The average yield on the wet basis was approximately fifteen per cent. As stated previously in all this work, yields were calculated on the weight of extracted hulls rather than on the weight of hulls plus lime. It was believed that results secured this way would have more significance than if they were calculated on the total weight of hulls plus lime.

The iodine adsorbed by the various carbons is given in Fig 2.

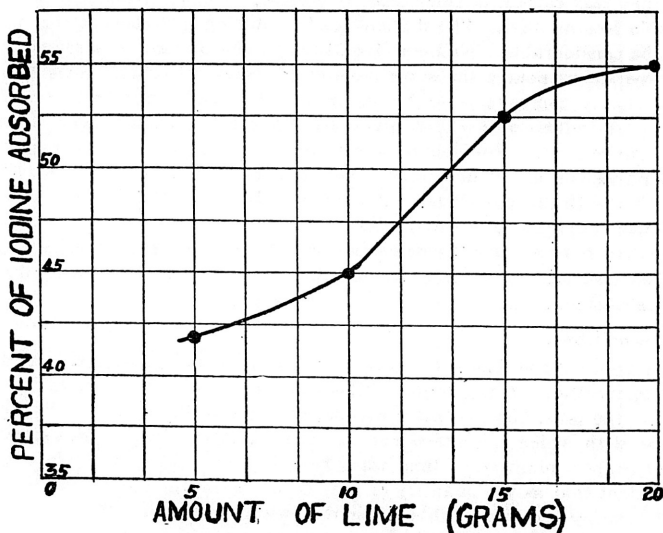


Fig. 2.—Relation Between Iodine Adsorbed and Amount of Lime.

It will be seen that the quality of the carbon steadily improves as the amount of lime is increased until the latter reached 15 grams. At this point the inclination of the curve changes further addition of lime having comparatively little effect. Consequently 15 grams of lime was adopted for all further work. Tests with 25 grams of lime were not considered worth while since the increase from 15 to 20 grams was comparatively small.

#### Temperature of Heating.

It is well known that the gases adsorbed by the carbon are strongly held

Consequently the quality of the carbon should improve as the temperature is increased. The upper limit in this case, is the point where the lime reacts with the carbon to form calcium carbide. A practical limit is the maximum temperature that can be secured by the furnace available and the point where scaling and weakening of the iron retort becomes excessive.

In the preliminary work good results were secured at 1700° F. In order to determine if this was the best temperature a series of runs was made at 1800° F, 1900° F and at 1500° F. These runs were made employing the preliminary conditions discussed on page 11. The results of these runs are summarized in Figure 3.

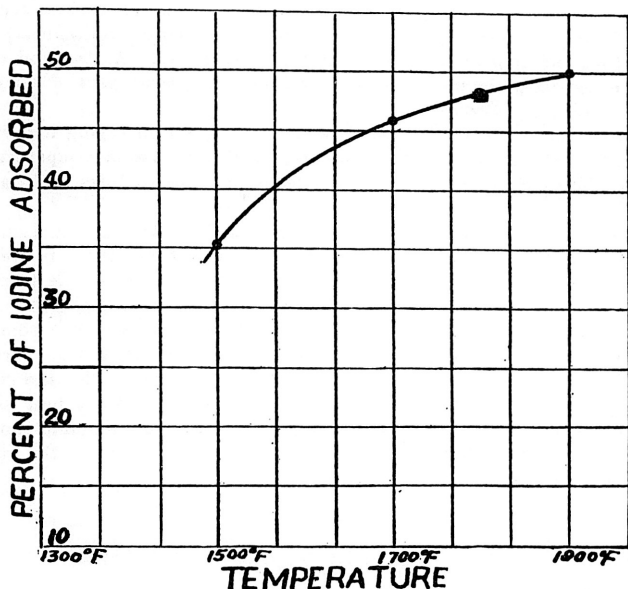


Fig. 3.—Relation Between Iodine Adsorbed and Temperature.

It is noted from Fig. 3 that the results secured at 1700° F are appreciably better than those secured at 1500° F. This advantage is considered sufficient to warrant the selection of this temperature over 1500° F. A further advantage of 1700° F is due to the fact that this temperature is high enough to decompose any calcium carbonate present in the lime, the carbon dioxide escaping. This means that when the lime is dissolved away from the carbon with acid, foaming due to carbon dioxide is reduced to a minimum. The results secured at 1900° F are found to be only moderately better than those secured at 1700° F. This improvement in quality is considered to be more than equivalent to the additional fuel, etc. Consequently 1900° F was preferred. In view of the fact that 1900° F could not be consistently obtained

by the furnace available, this temperature was reduced to 1800° F which was used in all further work.

It should be mentioned here that the yield is slightly less at the higher temperatures. However, in view of the above reasons for employing a higher temperature, the slight decrease in the yield is considered of little importance.

#### Length of Heating.

The period of heating should be sufficiently long to permit removal of all or the greater part of the absorbed gasses. These are chiefly carbon dioxide and carbon monoxide. On the other hand, the shorter the period of heating, the lower the cost, and the greater the output.

In the preliminary work, it was found that oxides of carbon began to come off before reaching the maximum temperature and continued to come off in declining quantities for at least two hours. At the end of one hour after reaching the maximum temperature, the greater part of the oxides of carbon has been removed.

It follows from the above that there is little object in trying a period of heating of less than one hour after reaching the maximum temperature of 1800° F. This corresponds to a *total* period of heating of three and one half hours.

With the above in mind, employing the temperature (1800° F) and quantity of lime (15 grams), the remainder of the conditions being those preliminary conditions discussed on page 11, a series of runs was made, the period of heating at the maximum temperature being one hour (total period of heating three and one-half hours). A series of runs was made also at a somewhat longer period (two hours at the maximum temperature or a total period of four and one half hours). It was not considered necessary under the circumstances to reduce the time below one hour (total three and one half hours). One or two runs which were discontinued as soon as the maximum temperature (1800° F) was attained, gave low results. The results of the runs at one and at two hours are given in Table IV.

**Table IV**  
Comparison of Decolorizing Carbon Made at Different Periods of Heating.

Maintainance of max. temp.	Total period of heating	Iodine added	Iodine adsorbed (Percent)	wet basis 30% water	Yield dry basis (percent)
1. One hour	Three and one-half hours	1.131 grams	45.80	14.1	20.1
2. Two hours	Four and one-half hours	1.131 grams	47.68	13.2	19.0

From the above it is seen that the advantage in favor of the longer period of heating is comparatively small. It is believed that this advantage was not enough to make up for the increased cost of fuel, and deterioration of the retort at the longer period. Consequently the shorter period of heating (one hour at 1800° F, total period of three and one half hours) was adopted for all future work.

### Rate of Heating.

In the preliminary work, a rate of heating was employed which was as rapid as could be secured conveniently and consistently with the furnace available. This same rate was used in most of this work.

In order to determine if the rate makes any appreciable difference a series of runs was made at an appreciably slower rate.

In making all of the above carbons, the quantity of lime was fifteen grams, the temperature was 1800° F, the length of heating was one hour at the maximum temperature (total time of heating three and one half hours). For other conditions see page 11, where the preliminary conditions are discussed. The two rates are summarized in Fig. 4 while the quality of the carbon at the two rates, is given in Table V.

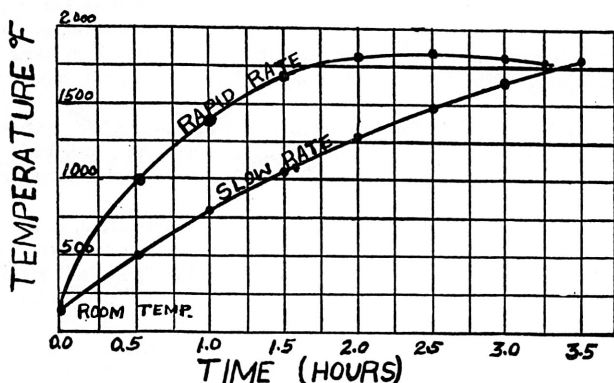


Fig. 4.—Comparative Rate of Heating.

Table V  
Relation Between Quality of Carbon and Rate of Heating

Rate	Amount of iodine added	Amount of iodine adsorbed (Percent)	Yield	
			wet basis Percent	dry basis Percent
1 Rapid	1.131	47.61	14.14	20.20
2 Slow	1.131	45.33	15.8	22.50

As seen from the above table there is little to choose between the rates. The quality of the carbon made at the slower rate is not quite as good as that made at the more rapid rate. On the other hand, the yield at the slower rate is slightly more than that at the rapid rate. No clear cut advantages having developed in favor of the slower rate, the more rapid rate was adopted for all further work.

### SOLUTION OF LIME WITH ACID

After the hulls and lime have been heated according to the method already described, the retort was allowed to cool, and the contents of same transferred

to a beaker. An excess of dilute hydrochloric acid solution (one part by volume of concentrated hydrochloric acid and two parts of water), was added slowly with stirring, the contents of the beaker heated to boiling for about fifteen minutes, the solution filtered, and the carbon washed until neutral.

It should be noted at this time, that in place of the hydrochloric acid, probably any acid other than sulphuric could be used. On a large scale the calcium chloride in the filtrate could be recovered in all probability. This might be treated with sulphuric acid which is much cheaper than hydrochloric, and much of the hydrochloric recovered.

### CONDITION FOR ACTIVATION OF THE CARBON

After removal of the acid, the washed carbon is read for activation. According to Mantell<sup>18</sup> the various methods for activation includes the treatment of the heated carbon with air, steam, a mixture of air and steam, or chlorine. All of these reagents are believed to cause a preferential oxidation, the inactive carbon associated with the active carbon, oxidizing somewhat more rapidly than the active carbon, leaving the latter.

Of the various methods for activation mentioned above, air was selected because of its cheapness and since its use brings about an exothermic rather than an endothermic reaction in the retort. This reduces the necessary fuel required to a minimum.

Before establishing the optimum conditions for the activation, it was necessary to secure a considerable amount of carbon which was uniform. Consequently using the optimum conditions already determined for the first heating with lime, an appreciable amount of this carbon was collected. This sample was used in all this work.

In the various tests carried out along this line, fifteen grams of the dry carbon (this corresponds closely to half the yield up to this point from the 150 grams of hulls) were placed in a small black iron retort. It will be remembered that these retorts were made from black iron pipe one and one-half inches in diameter. Special precautions were taken to make these retorts air tight. The threads on the ends of the retorts were treated with red lead and the caps were carefully tightened.

#### Temperature of Activation.

The temperature used in the preliminary work for the activation was 1800° F. This gave good results. In order to determine if just as satisfactory results could be secured at lower temperatures, a series of runs was made at 1600° F and 1400° F. With the equipment available, we were unable to go above 1800° F. The results of the various runs are given in Table VI.

Table VI  
Activation at Different Temperatures

No.	Temperature	Amount of iodine added	Iodine adsorbed Percent	Yield	
				Wet basis percent	Dry basis percent
1	1800°F	1.131 grams	43.68	12.10	17.30
2	1600°F	1.131 grams	46.10	12.26	17.50
3	1400°F	1.131 grams	38.51	14.26	20.37



On examination of Table VI it is evident that the results secured at 1600° F are fully as good as those secured at 1800° F. As a matter of fact the activity at this temperature due to some unknown reason, is actually better than at 1800° F. While the yield secured at 1400° F is somewhat better than at 1600° F, there is a marked decrease in the activity at this temperature. Consequently 1600° F was selected for all future work.

#### Amount of Air.

Since compressed air is an item of some expense, it is important to use as little as possible. In the preliminary work an improvement was noted in the quality of the carbon up to 0.729 cu. ft. of air for 15 grams of carbon. Beyond this point the improvement appeared to be slight.

With the above work in mind, a series of runs was carried out using 0.729 cu. ft. The air was then varied on both sides of this point. The lower point selected was two-thirds of the above amount or 0.486 cu. ft. The upper point selected was appreciably greater than 0.729 cu. ft. so that any improvement, however slight, might be detected. The quantity of air selected for this point was 1.215 cu. ft. The quantity of air used in the various tests was then in the proportion 2, 3 and 5. The results of the work carried out along this line are given in Table VII.

**TABLE VII**  
Relation of Amount of Air to Activity

No.	Amount of carbon	Amount of air (uncorrected)	Iodine added	Iodine adsorbed (percent)	Yield	
					Wet basis (percent)	Dry basis (percent)
1.	15	0.486 cu. ft.	1.131 grm.	44.78	15.96	22.80
2.	15	0.729 cu. ft.	1.131 grm.	46.65	14.54	20.77
3.	15	1.219 cu. ft.	1.131 grm.	48.68	12.84	18.34

Examination of Table VII indicates that the comparatively small increase in the amount of air from 0.486 cu. ft. to 0.729 cu. ft. improves the quality of the carbon about two per cent. There is a decrease in the yield of about one and one half per cent. In general, however, up to a certain point at least, the improvement in quality is considered more important than a moderate decline in yield. Consequently 0.729 cu. ft. of air was preferred to 0.486 cu. ft. When the large increase of air from 0.729 cu. ft. to 1.219 cu. ft. is made there is an increase in activity of about two per cent, the yield decreasing from 14.54 per cent (wet basis) to 12.84 per cent. Under these conditions the advantage of 1.219 cu. ft. of air over 0.729 cu. ft. appears to be doubtful. Consequently the smaller quantity (0.729 cu. ft.) was adopted for all future work.

#### Rate of Passage of Air.

The rate should be slow enough so that good contact is secured between the air and the bed of the carbon. Further, the rate should not be rapid enough so that carbon in considerable quantity is blown out the top of the retort.

Preliminary work showed that if the selected quantity of air (0.729 cu. ft.) was passed into the retort in less than 45 minutes, there was danger of not

securing the necessary contact. Further, some carbon was blown from the top of the retort.

With the above points in mind, 0.729 cu. ft. in 45 minutes was selected as the maximum rate. Incidentally this was the rate used in all the work carried out up to this point. In order to determine if the rate makes any appreciable difference, a series of runs was carried out at an appreciably slower rate, i.e., 0.729 cu. ft. in 75 minutes. The results of this work are given in Table VIII.

**Table VIII**  
Rate of Passage of Air

No.	Time required for 0.729 cu. ft. of air	Iodine added	Iodine adsorbed percent	Yield	
				Wet basis percent	Dry basis percent
1.	45 minutes	1.131 grm.	46.65	14.54	20.77
2.	75 minutes	1.131 grm.	50.86	14.62	20.90

Examination of Table VIII shows that the activity at the slower rate is several per cent greater than at the more rapid rate. There was little difference in the yields at the two rates. The increase in activity was considered to more than balance the increase in the time required. Consequently the slower rate was adopted. A slower rate than this was not tried since it was believed that such a rate would be abnormally slow and would not be practical.

In summing up the above results, it may be stated that the most favorable conditions for the production of the decolorizing carbon have been shown to be: (1) degree of fineness 54.8 per cent through 200 mesh, 31.7 per cent on 200 mesh, 13.2 per cent on 100 mesh; (2) quantity of lime, 15 grams for 150 grams of hulls; (3) temperature of heating with lime 1800° F; (4) length of heating at maximum temperature of 1800° F, one hour (total period of heating three and one half hours); (5) rate of heating, rapid (see Fig. 3). The best conditions for the activation of half the carbon from 150 grams of hulls, i.e., for the carbon from 75 grams of hulls, have been shown to be: (1) temperature, 1600° F; (2) amount of air, 0.729 cu. ft. uncorrected; (3) rate of passage of air, 0.729 cu. ft. in 75 minutes.

### IMPORTANCE OF TYPE OF RETORT

As has been previously mentioned, in the first heating it is highly important that the gases adsorbed by the carbon be as little as possible. It will be remembered that lime is added for the purpose of reducing the adsorbed gases. One of the factors which probably determine the amount of gases adsorbed in the quantity of gases liberated.

In connection with the above, a series of experiments carried out in the laboratory should be mentioned. The black iron retort used for most of this work burned out and it was necessary to replace it. Two retorts slightly smaller (two inches rather than two and one-half inches in diameter) were secured. With a sample of the same weight the increase in the thickness of the bed of hulls and lime in the small retort was nearly fifty per cent. Carbon made in these small retorts was of inferior quality. The per cent iodine adsorbed in the various runs was from thirty-five to forty per cent. The lower results secured in the small retorts are probably accounted for by the

fact that the increased thickness of the bed of carbonaceous material, gave an increased opportunity for the carbon dioxide formed during the decomposition of the hulls to react with the carbon thereby forming carbon monoxide. Now, one molecule of carbon dioxide reacts with carbon to form **two** molecules of carbon monoxide. The result was that the volume of gas was appreciably greater, which in turn probably increased the volume of gas absorbed. In this connection, what appeared to be abnormal quantities of a combustible gas believed to be carbon monoxide passed off through the opening at the top of the retort. In view of the above, it would appear that on the large scale the retort should be designed so as to reduce the formation of carbon monoxide to a minimum. One of the most effective methods of doing this is to use a shallow bed of material.

The results secured by employing the usual process but employing retorts of different sizes, are given in Table IX.

**Table IX**  
Quality of Carbon Made in Retorts of Different Diameters

No.	Diam. of retort	Charge (grams)	Iodine added	Iodine adsorbed percent	Yield	
					Wet basis percent	Dry basis percent
1.	2 inches	15 lime 150 hulls	1.131 grams	38.7	17.14	24.50
2.	2.5 inches	15 lime 150 hulls	1.131 grams	45.8	14.14	20.20
3.	4.0 inches	15 lime 150 hulls	1.131 grams	67.86	8.66	12.37

On examination of the quantity of iodine adsorbed, it was observed that as the diameter of the retort increased or as the thickness of the bed decreased, the quality of the carbon increased. An air leak was observed in the bottom of the largest retort and consequently results secured in this retort were not strictly comparable to other results. However, the high quality of the carbon made in this retort (the quantity of iodine adsorbed by the carbon was much higher than any commercial sample which had been encountered up to this time) suggested passing air into the retort during the **first** heating with lime. The very interesting results secured in this way are given in the following section.

#### PASSAGE OF AIR INTO THE HEATED MIXTURE OF LIME AND CARBON

The mixture of hulls and lime was heated in the usual way. When the temperature of the furnace reached 1400° F, air was passed into the retort by means of the copper tubing previously mentioned. The rate of passage of the air was cut down to one cubic foot per hour in order to prevent as far as possible the escape of incandescent particles of carbon. When four or more cubic feet of air were employed, the period of heating was necessarily greater than the period selected in the determination of the optimum conditions previously discussed. With the exception of the period of heating and the passage of air into the retort during the heating of the hulls and the lime, the *optimum conditions previously determined were employed in making all of these carbons.* The results of this work are given in Table X.

**Table X**  
Effect of Air on the Quality of the Carbon During the First Heating

No.	Amount of air	Amount of iodine added	Iodine adsorbed (percent)	Yield		Index number Percent yield x Percent Iodine adsorbed
				Wet basis percent	Dry basis percent	
1.	None	1.131 gms.	50.86	15.10	21.60	767.
2.	2 cu. ft. (uncorrected)	1.131 grms.	58.88	14.0	20.0	824.3
3.	4 cu. ft. (uncorrected)	1.131 gms.	70.77	10.0	14.3	707.
4.	6 cu. ft.	1.131 gms.	87.43	3.74	5.34	326.9

The following points are obvious from this table.

1. The addition of air during the first heating greatly improves the quality of the carbon. The quality of the carbon increases with the amount of air.

2. Carbon of very high quality can be made from the extracted hulls by this method. The highest quality carbon (91 per cent iodine adsorbed) made in this way, is superior to all of ten well known commercial carbons. See page 21 for further details.

3. By adjusting the amount of air it is possible to make carbon of several different grades. If one wishes to make a carbon of a quality comparable to some on the market, and of a comparatively high yield, no air whatever is necessary in this stage of the process. On the other hand, if a carbon of higher quality but of lower yield is desired, this can be secured by regulating the amount of air. In this connection, it will be noted that the index number, secured by multiplying the per cent yield by the per cent iodine adsorbed, is slightly greater when 2 cu. ft. of air are used than when no air is used. It is not known whether this has any significance. Beyond 2 cu. ft. of air, the index number decreases, consequently the most economical carbon to make would seem to be that one where no air or 2 cu. ft. of air is employed. The quality of this carbon, however, would be only fair.

#### GENERAL PROPERTIES OF THE DECOLORIZING CARBON

The ash content, reaction to indicators and other properties of typical carbons are given in the following table.

Ash Percent	Carbon Percent	Reaction to Indicators	pH value	Wt. per cu. ft. loosely packed (lbs.)
4.35	86.8	Neutral to alkaline	7.0 to 8.2	24
This is representative of a carbon where no air was added during heating with lime.				

#### QUALITY OR ACTIVITY OF THE CARBONS

In order to determine the quality of the carbon as compared to other carbons, eleven samples of activated carbons actually on the market were collected and the iodine test applied in the usual way. The iodine adsorbed by each

of these carbons and by several samples of the new carbon made from the extracted hulls is given in the following table.

**Table XI**  
Activity of Various Carbons

No.	Sample	Iodine added (grams)	Iodine adsorbed (Percent)	Iodine index (gms. of iodine adsorbed per gram of carbon.)
A	Best grade carbon made from extracted hulls	1.125	91.0	1.020
B		1.125	81.71	0.910
C		1.125	70.28	0.79
D		1.125	65.42	0.736
E		1.125	64.85	0.729
F		1.125	64.56	0.727
G	Blood charcoal	1.125	62.28	0.700
H		1.125	59.13	0.664
I	Medium grade carbon made from extracted hulls	1.125	58.88	0.663
J		1.125	50.85	0.571
K		1.125	23.13	0.260
L		1.125	7.38	0.083
M	Animal charcoal	1.125	7.11	0.080

From the above it is obvious that employing iodine adsorbed as a measure of quality, the best grade carbon made from the hulls is superior to all eleven typical carbons on the market. Since the adsorption of carbons is *selective*, the iodine adsorption does not necessarily imply that the hull carbon is superior to all the other carbons for the decolorization of sugar, glucose, phosphate liquor and for other purposes. It will be remembered, however, that Chaney<sup>8</sup> finds a direct relation between the iodine adsorbed and the sugar solution purified. One is probably correct in the assumption that a carbon with a high iodine index would be generally satisfactory for most purposes. Direct comparison of the better grade hull carbon with several good grade commercial carbons on brown sugar solutions and di-sodium phosphate liquor, shows that the hull carbon is superior to many and is comparable to the others.

#### REVIVIFICATION OF EXHAUSTED CARBON

Examination of the carbon showed that it could be revived. The carbon was exhausted by treating it with an excess of ten per cent brown sugar solution. The sugar solution was warmed with the carbon for fifteen minutes, the solution filtered, and the carbon washed. The carbon was then removed to a small iron retort where it was heated to approximately 1200° F with exclusion of air. After cooling, fresh carbon and carbon which was revived as explained above were both warmed with a ten per cent brown sugar solution

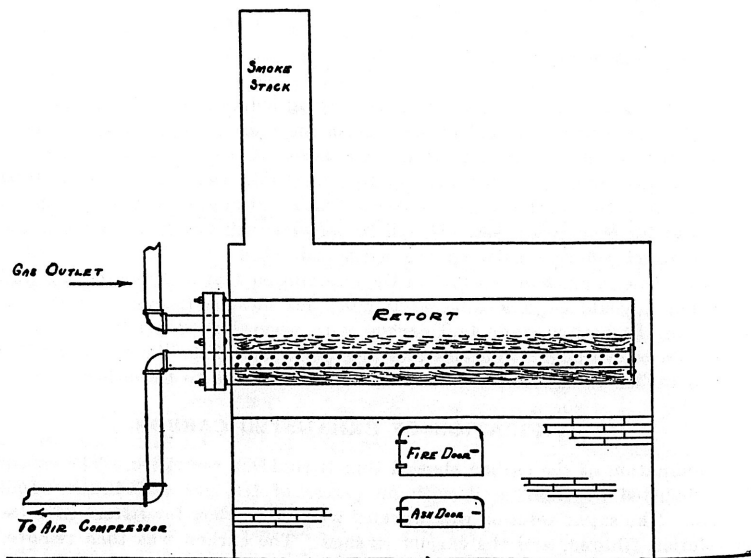
for fifteen minutes and the solutions filtered. Examination showed that the decolorizing action of the revived carbon was practically as good as the fresh carbon. The adsorptive capacity as determined by the iodine solution in a typical run, was 47.71 per cent for the fresh carbon and 45.13 per cent for the revived carbon.

### SEMI-PLANT SCALE

To secure the necessary conditions for carrying out this work, a stationary retort provided with a stirrer, or a rotating retort appeared to be necessary. Since the latter would involve complications as well as considerable expense, the former type was selected for the semi-plant work. Consequently, a wrought iron retort five feet long and fifteen inches in diameter (inside) was secured. One end of the retort was provided with a flange to facilitate charging and discharging. The flange contained the necessary openings for the pyrometer and for the escape of the products of combustion. The flange also contained two one inch openings for two perforated three quarter inch pipes which supplied the necessary air. These pipes were in a parallel position and ran the entire length of the retort about two inches from the bottom of same. When air under pressure was passed through the pipes, the air could be evenly distributed throughout the charge.

The retort was supported in a horizontal position in a brick furnace. Gas not being available in large quantities, the retort was heated by coal. The furnace contained two arches in such a position that the flames passed clear around the retort. Drawings of the retort are shown in Fig. 5 and Fig. 6.

The hulls and lime were mixed by hand and placed in the retort. When



Scale:  $\frac{1}{2}$  in. = 1 foot.

Fig. 5.—Semi-Plant Production of Decolorizing Carbon—Front View.

no air was admitted during the heating with lime, the charge was stirred with a long rod. When air was employed, no stirring was necessary.

After the run was completed, the retort was allowed to cool, the charge transferred to a stoneware vessel, where the lime was dissolved with dilute hydrochloric acid. The carbon was then washed with water in a barrel with a perforated bottom until the washings were neutral. The washed carbon was then transferred to the same retort used for the first heating where it was activated with air. The necessary air was supplied by a small compressor attached to a paint spraying machine. The air entered the perforated pipes previously described where it was distributed throughout the charge.

Data from the various runs carried out is given below:

#### First Heating

Run No.	H <sub>2</sub> O in hulls (percent)	Charge (lbs.)	Amount of air (cu. ft.)	Pressure of air (lbs. sq. in.)	Rate of air (cu. ft. per hr.)	Temp. of heating O°F.
1.	15.36	35 ext. hulls 3.5 lime	None	—	—	1650-1700
2.	15.36	35 ext. hulls 3.5 lime	None	—	—	1650-1700
*3.	32.21	70 ext. hulls 7.0 lime	422.8	30	156	1700
*4.	26.6	70 ext. hulls 7.0 lime	422.8	30	156	1700
5.	27.18	70 ext. hulls 7.0 lime	None	—	—	1700
*6.	54.6	75 ext. hulls 7.0 lime	845.6	30	156	1700

No air was added in runs one and two. Runs number three and four correspond to 2 cu. ft. on a small scale, while run six corresponds to 4 cu. ft.\*

With the furnace available, it was practically impossible to exceed 1700° F. This accounts for the lower temperature used in this work. During the heating of the extracted hulls with lime, a flame more than two feet long burned from the retort. This, no doubt would furnish much of the necessary heat for maintaining the temperature of the retort.

\*NOTE: In runs 3, 4 and 6, the amount of air is calculated on the assumption that the hulls contain 30 per cent water. No correction was made for the variation from this figure.

#### Solution of Lime

No.	Amount of acid (conc.)
1.	1.18 gal. (4500 cc.)
2.	1.18 gal.
3.	2.6 gal. (10,000 cc.)
4.	2.6 gal.
5.	2.6 gal.
6.	2.6 gal.

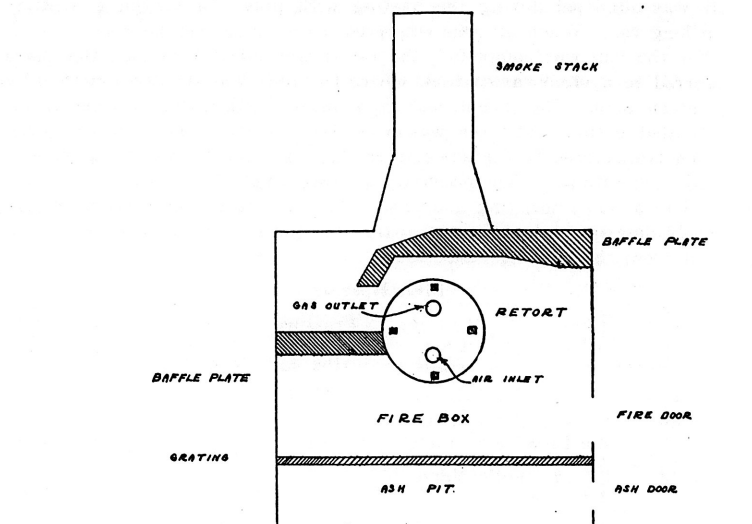


Fig. 6.—End View of Retort for Semi-Plant Production of Decolorizing Carbon.

It was found that the quantity of acid could be reduced appreciably below that used on the laboratory scale.

#### Activation

(All of carbon from the first heating was activated in each case)

No.	Amount of air (cu. ft.)	Rate of air (cu. ft. per hr.)	Temp. (°F)	Iodine added (grams)	Iodine adsorbed (percent)	Yield	
						Wet basis (percent)	Dry basis (percent)
1. & 2.	510.8	156	1600	1.131	47.5	15.7	18.57
3.	428	156	1600	1.131	52.56	18.0	26.4
4.	320.7	156	1600	1.131	56.1	18.5	25.3
5.	320.7	156	1600	1.131	59.06	15.7	21.6
6.	214.0	156	1600	1.131	74.28	8.7	18.0

In carrying out the activation, as in the heating with lime, as far as possible the same conditions were used as in small scale work. The amount of air was calculated directly from the small scale data, while the temperature employed was the same as in the small scale work. The variation in the quantity of the air and in the yield was due to variation in the moisture of the hulls employed in the first heating. Because of the small amount available from each run, runs one and two were combined.

In conclusion, it is of interest that no particular difficulty arose in the production of the carbon on a semi-plant scale. The yield and activity is



somewhat superior to that secured on the small scale. This is particularly noticeable in run 6.

### PROBABLE LARGE SCALE APPARATUS AND COST ESTIMATE

The heating of the residual hulls requires the heating of a mixture of two solids to a temperature of 1800° F.

It is preferable, if not absolutely necessary, that thin section or layers of material be exposed to the heat, so that the gasses may escape freely.

If air is admitted during the heating of these materials the layer of materials could be deeper. In this case it is highly important that the air is evenly and uniformly distributed.

As previously stated a wrought iron or steel retort is only fairly satisfactory because it scales considerably at the temperature employed. It may be that a steel retort lined with fire brick and heated internally with flue gases would be advantageous. Pipes could be introduced for the admission of the necessary air.

For making gas adsorbent charcoal for the United States Army<sup>19</sup>, horizontal fire clay retorts were arranged in a bank of two rows of four retorts each, one above the other. Every retort had a door at each end opening to the full size of the cross section of the retort. The retorts were heated to about 900° C. by hot gases from coke fires situated at each end of the bank and passing through flues around the retorts. Charging was accomplished by buckets on the traveling boom of a charging machine, one at each end of the retorts. The charge was pushed out by a semi-elliptical plate a little smaller than the cross section of the retort.

Probably the above idea could be used to advantage in this work provided a series of perforated pipes were introduced for addition of the necessary air. If this were done both the first and the second heatings could be carried out in retorts of the same type. Also a design of this type would permit the utilization of the waste heat given off during the passage of the air into the retort, for the heating of the retort itself. The hot combustible gases given off from the retort might be burned and the hot products of combustion passed around the retort. The amount of waste heat available during the passage of air into the retort during the first heating is very considerable.

A brick lined rotary retort, heated internally by flue gases might be considered. The good contact between the solids and the air should not offer a problem here, since, in all probability it would be necessary to merely admit a measured quantity of air to the interior of the retort. It is possible to make a retort of this type continuous in operation. The hot gases given off during the addition of this air might be passed through flues into the interior of the retort, thereby utilizing the waste heat.

Assuming that the air dried residual hulls and lime are in separate hoppers, they might be fed by gravity to a rotary mixer. The raw mixed materials might be stored in a suitable hopper. From the hopper, the raw materials might be fed by screw conveyor to the retort. From the retort the carbon might be discharged into a cooler (air being excluded). From the cooler the material might be discharged by a screw conveyor into a storage hopper. From the storage hopper the carbon might be discharged on a conveyor which would deposit it in a steam heated solution tank, where it might be mixed with the required amount of dilute hydrochloric acid. The carbon might be filtered and washed in a centrifugal or filter press and then conveyed to a

storage hopper. From the storage hopper, a screw conveyor might be employed for the transportation of a definite weight of carbon to the activation furnace where it is heated and treated with air. The finished carbon might be discharged into a cooler and then transferred by a conveyor to the storage hopper. A flow sheet of the proposed plant is shown in Fig. 7.

#### Cost of Manufacture of the Carbon.

The capacity of the plant for the manufacture of a product determines in large measure, the cost of the product. The quantity of extracted hulls available for conversion into carbon depends upon the quantity of xylose produced. Not knowing the quantity of xylose production, no attempt is made to estimate labor and depreciation costs of the carbon. However, the cost of the necessary reagents, fuel, and water are discussed below. It is believed that these would represent the greater part of the total cost. In this estimate, no charge is made for the extracted hulls. The cost is estimated on a yield (wet basis) of 16 per cent.

#### Cost of Chemicals.

The estimated cost of the lime is as follows:

24 lbs. of carbon require 15 lbs. of lime.

Then 1 lb. of carbon requires 0.623 lbs. of lime at \$20 per ton -----0.623c

Hydrochloric acid is the most expensive reagent used in this process. It will be remembered that the dissolved lime exists in the filtrate, from the carbon, in the form of calcium chloride. Since this salt is already in solution, the hydrochloric acid might be readily recovered from this salt by adding the required amount of sulphuric acid and heating. The hydrochloric acid gas driven off on heating is readily dissolved in water when it is ready to use again. On this basis, using only 45 cc. of hydrochloric acid for 15 grams of lime (this has been found to be sufficient), the cost of the reagents necessary to regenerate the hydrochloric acid is estimated to be as follows:

1 lb. carbon required 0.825 lb. dry HCl which required 1.53 lb. 72%  $H_2SO_4$   
or

1 lb. carbon required 1.53 lb.  $H_2SO_4$  at \$11.00 per ton, or 0.84c.

Assuming that the hydrochloric acid lost on each regeneration is equivalent to one fourth of the total acid, we have:

1 lb. carbon requires  $0.84 + 0.84/4$  or 1.05c.

#### Fuel.

On the semi-plant scale, one pound of carbon requires several pounds of fuel. It is obvious that the small furnace used in this work is not efficient from the standpoint of fuel utilization. Consequently it appears much better to depend upon general data with regard to the efficiency of large, well designed furnaces. It is believed that with furnaces of this type heated to temperature of 1800°F. to 2000°F., an efficiency of at least fifty percent can be expected. On this basis, the cost of the fuel (calculated as coal at \$4.00 per ton) is given by the following calculation:

\*24 lbs. of carbon requires the following heat units:

(a) 45 lbs. (30%) of water heated from 60°F. to 212°F. -- 6.840 B.t.u.

(b) 45 lbs. water converted to steam, i.e.,  $45 \times 970.4$  ----43.668 B.t.u.

(c) 120 lbs. hulls and lime heated from 60°F. to 1800°F.

\* 24 pounds is an average value for the carbon made from 150 pounds of extracted hulls.

(27)

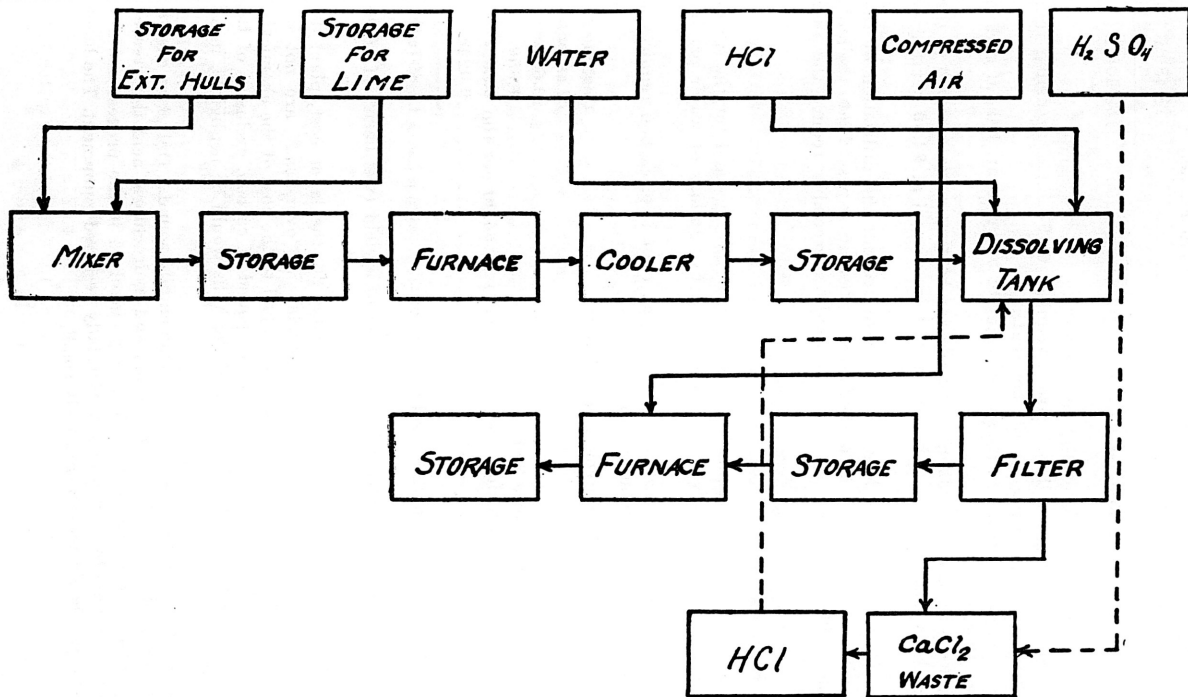


Fig. 7.—Process Diagram.

(specific heat of hulls and lime assumed to be 0.4)	
i.e., $120 \times 1740 \times 0.4$ -----	83.520 B.t.u
(d) 30 lbs. carbon heated from 60°F. to 1600°F., i.e.,	
$30 \times 1540 \times 0.4$ -----	18.480 B.t.u.

Total for 24 pounds of carbon -----152.508 B.t.u.

Note: 24 lbs. is 16% of 150 gms. of hulls.

On the above basis, 1 lb. of carbon requires 6,354 B.t.u. assuming that the efficiency of the furnace is 100 per cent.

Then, with an estimated furnace efficiency of 50 per cent, 1 lb. of carbon requires 12.708 B.t.u.

12.708 B.t.u. might be taken as the average heating value of much of the coal in this vicinity., i. e. 1 lb. carbon = 1 lb. of coal at \$4.00 per ton or 0.20c.

The above calculation does not take into account the utilization of the combustible gases given off from the furnace when air is passed into the hulls and lime. The utilization of this heat would appreciably reduce the fuel consumed.

#### Water Required.

Most of the water is required for solution of the lime and for the washing of the carbon. Experience has shown that five gallons of water per lb. of carbon is sufficient.

Assuming that water costs 5c per 1000 gal. then 1 lb. carbon requires 5 gal. of water at 5c per 1000 gal. or .024c.

The total cost of these items is as follows:

	Cost per lb. of carbon
1 Cost of lime -----	0.623c
2 Cost of hydrochloric acid -----	1.050c
3 Cost of fuel (assuming coal is employed) at \$4.00 per ton	0.200c
4 Cost of water -----	0.024c
	<hr/>
Total cost of chemicals, fuel and water -----	1.897c

Note: The above is calculated on the basis of a yield (wet basis) of 16.00 per cent.

Because of the reasons mentioned on page 26, the labor cost, plant depreciation and other costs commonly included in "over-head" are not included in this estimate. If these items are equal to the cost of the chemicals, fuel and water, the total cost is 3.794c per pound of carbon, exclusive of the cost of the extracted hulls. The cost of the hulls would be negligible, in all probability.

The above process appears to be fundamentally sound. Only two chemicals are used—lime which is very cheap, and hydrochloric acid the greater part of which can be recovered. The steps involved in the process are not unusual nor should they require any especially designed equipment. The labor requirements should be comparatively small.

## SUMMARY

A process for the manufacture of a high grade decolorizing carbon from extracted cotton seed hulls has been outlined and studied in detail. The effect of variation in the process has been studied and the optimum conditions for the process determined. The carbon has been made on both a laboratory and a semi-plant scale.

The probable large scale installation has been outlined and the cost of the manufacture of the carbon estimated.

The results of the experimental work indicate that by the proposed process a good grade decolorizing carbon can be produced from extracted hulls at a moderate cost. This carbon with its high absorptive qualities and neutral reaction could be used in the manufacture of sugar and in many other industries.

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