GEORGIA INSTITUTE OF TECHNOLOGY
Engineering Experiment Station

PROJECT INITIATION

Date: 3/8/72

Project Title: Evaluation of Pyrolysis as a Means of Resource Recovery from Solid Wastes of the Cotton Industry

Project No.: A-1402

Project Director: Dr. James A. Knight

Sponsor: Cotton, Incorporated

Effective March 1, 1972 Estimated to run until: September 30, 1972

Type Agreement: Cooperative Agreement No. 71-576 Amount: $ 21,700

Reports Required: Monthly Progress
Quarterly Report
Final Report

Contact Persons: Administrative
Mr. Arthur B. Bond
Administrative Assistant
Cotton, Incorporated
P. O. Box 18039
3901 Barrett Drive
Raleigh, N. C. 27609

Technical
Mr. R. B. Cleaver, Manager
Textile Products Research
(same address as above)

Assigned to Technology Applications Group Division

COPIES TO:

- ☐ Project Director
- ☐ Director
- ☐ Associate Director
- ☐ Assistant Director(s)
- ☐ Division Chiefs
- ☐ Branch Head
- ☐ General Office Services
- ☐ Engineering Design Services
- ☐ Photographic Laboratory
- ☐ Research Security Officer
- ☐ Accounting
- ☐ Purchasing
- ☐ Report Section
- ☑ Library
- ☐ Rich Electronic Computer Center
- ☐ _____

EA-62(12-66)

A-344-22

GEORGIA INSTITUTE OF TECHNOLOGY
Engineering Experiment Station

PROJECT TERMINATION

Date December 11, 1972

PROJECT TITLE: Evaluation of Pyrolysis as a Means of Resource Recovery from Solid Wastes of the Cotton Industry

PROJECT NO: A-1402

PROJECT DIRECTOR: Dr. J. A. Knight

SPONSOR: Cotton, Incorporated

TERMINATION EFFECTIVE: October 31, 1972

CHARGES SHOULD CLEAR ACCOUNTING BY: All acceptable charges have cleared.

Final report submitted 11/9/72.

TECHNOLOGY APPLICATION Group

COPIES TO:

Project Director
Director
Associate Director
Assistant Directors
Division Chief
Branch Head
Accounting
Engineering Design Services
General Office Services
Photographic Laboratory
Purchasing
Report Section
Library
Security
Rich Electronic Computer Center

A-1402

GEORGIA INSTITUTE OF TECHNOLOGY
EXPERIMENT STATION

Office of the Director

225 North Avenue, Northwest · Atlanta, Georgia 30332

April 6, 1972

Monthly Progress Report I.

Mr. R. B. Cleaver, Manager
Textile Products Research
Cotton, Incorporated
P. O. Box 18039
Raleigh, N. C. 27609

Dear Mr. Cleaver:

The attached report covers the work through March 31 with the gin trash sample from Tifton, Georgia. Pyrolytic experiments are now underway with a representative sample of this material, and the level of activity on the project will increase during April.

We have not received the sample of material from California. It would be highly desirable if you could provide us with two or more samples of other waste material in the very near future. By having the waste materials on hand, we can plan our experimental work program more efficiently and effectively.

If I can supply you with any additional information about the program, please let me know.

Sincerely yours,

James A. Knight, Jr.

JAK:ct

Enclosure

cc: A-1402 file

Project A-1402: "Evaluation of Pyrolysis as a Means of Resource Recovery from Solid Wastes of the Cotton Industry"

Sponsor: Cotton, Inc.

Monthly Report: March 1 to March 31, 1972

Prepared by: J. A. Knight, Project Director
Engineering Experiment Station
Georgia Institute of Technology
Atlanta, Georgia 30332

I. Cotton Gin Trash Sample--Approximately 100 lbs of gin trash were received March 10 from Mr. W. E. Seigler, Tifton, Georgia. Mr. Seigler described this gin trash as follows: "This material did not include the trash from unloading fan or any of the condenser exhausts. It did include all trash that is collected from overhead cleaners, extractor feeders, gin stand, and lint cleaners." The moisture content of a sample of this material as received was 14.7%. The density of this material for a shaken sample was 4.9 lb/cu.ft. and for a hand packed sample, 7.1 lb/cu.ft.

II. Representative Sample--One cubic foot of waste material was removed from each of the four bags of gin trash received. This material was thoroughly mixed and ten pounds removed for grinding. Efforts to grind the original material in a Mikro Bantam Pulverizer were not successful. The sample (ten pounds) was ground in a Mikro Samplmill. This material is suitable for the pyrolysis experiments.

III. Data on the Representative Samples--

1. Moisture*

Sample #1	12.3%
Sample #2	12.3%

*The slight decrease in the moisture content from the original sample could be due to heat generated in the grinding operation.

2. Total Ash

Sample #1	5.70%
Sample #2	5.40%

3. Acid-insoluble Ash

Sample #1	2.65%
Sample #2	2.38%

4. Density

Sample Description	lb/cu.ft.
Loose sample	7.5
Shaken sample	9.4
Hand pressed sample	14.5

IV. Future Work--Pyrolytic experiments of the representative gin trash sample will be carried out beginning in the first week of April.

GEORGIA INSTITUTE OF TECHNOLOGY
EXPERIMENT STATION 225 North Avenue, Northwest · Atlanta, Georgia 30332

May 4, 1972

Mr. R. B. Cleaver, Manager
Textile Products Research
Cotton, Incorporated
P. O. Box 18039
Raleigh, North Carolina 27609

Dear Mr. Cleaver:

The attached report covers the work for April with the representative sample of the cotton gin trash from Tifton, Georgia. During May, we will do some additional pyrolysis experiments and also carry out activation experiments with the char.

If I can supply you with any additional information about the program, please let me know.

Sincerely yours,

James A. Knight, Jr.

JAKjr/edh

Attachment (seven)

MONTHLY PROGRESS REPORT II

Sponsor: Cotton, Incorporated

Project A-1402: "Evaluation of Pyrolysis as a Means of Resource Recovery from Solid Wastes of the Cotton Industry"

Monthly Report II: April 1 to April 30, 1972

I. Several pyrolysis experiments with the representative sample of cotton gin trash were carried out during this report period, and the results are summarized in Table I. Pyrolysis experiment 1 and 3 were carried out at 700°C and 2 and 4, at 800°C.

II. Partial analysis of the gases from pyrolysis experiments 3 and 4 showed the presence of hydrogen, methane, carbon monoxide, nitrogen, and oxygen. The hydrogen, methane, and carbon monoxide are pyrolytic gases, and the nitrogen and oxygen are present because of the air in the system at the beginning of the experiment. A more complete analysis of the pyrolytic gases will be given in the next report.

III. Future Work--During May, additional pyrolysis experiments will be carried out, and activation experiments with the char will be started. The product from the activation experiments will be tested to determine its adsportive characteristics.

TABLE I

PYROLYSIS EXPERIMENTS OF COTTON GIN WASTE

Experiment	Weight of Sample (gram)[a]	Temperature	Percent Pyrolytic Products[b]			
			Char	Water	Condensible Organic Material	Gases
1	100	700°C	32.2	19.4	11.4	37.0
2	190	800°C	31.7	19.4	10.4	38.5
3	200	700°C	32.4	20.5	11.5	35.6
4	200	800°C	31.5	18.2	11.0	39.3

[a] Samples are of the representative material, which contained 12.3 percent mositure.

[b] The pyrolytic products are given in percent by weight on a dry basis of original sample.

A-1402

MONTHLY PROGRESS REPORT III

Sponsor: Cotton, Incorporated

Project A-1402: "Evaluation of Pyrolysis as a Means of Resource Recovery from Solid Wastes of the Cotton Industry"

Period Covered: June 1 to June 30, 1972

Prepared By: James A. Knight

I. Kleen-Seed Sample. Two pyrolysis experiments with the Kleen-Seed sample were carried out during June. An activation experiment with steam at 800°C with the char from the Kleen-Seed sample produced a carbon which had an iodine value of 67.7 and a modified phenol value of 31.4. A water grade activated carbon should have a modified phenol value of 30 or less.

II. Cotton Gin Sample, Tifton, Georgia. Two pyrolyses of the cotton gin sample, Tifton, Georgia were made for the purpose of determining the composition of the gaseous phase, and the results are given below:

Gaseous Phase

Component	Pyrolysis CIT-7	Pyrolysis CIT-8
Air	3.5%	1.4
Carbon Monoxide	20.7	20.5
Methane	14.9	14.2
Carbon Dioxide	29.9	30.9
Hydrogen	26.6	23.2
Not Identified	4.4	9.8

III. Cotton Shearing Waste. Four pyrolyses of the shearing waste sample were carried out during June.

IV. Future Work. During July, activation experiments with the char from the shearing waste sample will be carried out. Experimental work with the cotton gin trash from Lubbock, Texas, will be started.

MONTHLY PROGRESS REPORT IV

Sponsor: Cotton, Incorporated

Project A-1402: "Evaluation of Pyrolysis as a Means of Resource Recovery from Solid Wastes of the Cotton Industry"

Period Covered: July 1 to July 31, 1972

Prepared by: James A. Knight, Jr.

I. Cotton Shearing Waste.

A sample of the char from the cotton shearing waste was activated at 800°C for 30 minutes with steam at the flow rate of 1 g. steam/g. char/hour. The yield of activated char was 89%. The following values were obtained.

Iodine value of char before activation	39.3
Iodine value of char after activation (unground)	90.5
Iodine value of char after activation (ground)	98.0
Modified phenol value after activation (unground)	18.3
Modified phenol value after activation (ground)	18.7

The iodine value is used as an indication of the degree of activation of carbon, and a high value indicates a high degree of activation. The modified phenol value (MPV) is used to characterize activated carbon used in water purification. The MPV for water grade activated carbon is 20 $\pm$ 2, and the values obtained for the activated carbon from the char of the cotton shearing waste show that this material would be an acceptable activated carbon for water purification.

Ash analyses on the char and activated carbon from cotton shearing waste are as follows:

	Total Ash	Acid Insoluble Ash
Pyrolytic Char	2.42%	0.74%
Activated Carbon	3.24%	1.43%

A low ash content is a very favorable characteristic of water grade activated carbon.

II. Cotton Gin Trash from Tifton, Georgia

A lignin analysis (1) of a representative sample of the cotton gin trash from Tifton, Georgia, gave a value of 20.9%.

III. Kleen-Seed Sample

An analysis (2) of a representative sample of the Kleen-Seed sample for oil gave a value of 6.4% oil content.

IV. Cotton Gin Trash Sample from Lubbock, Texas

1. Pyrolysis. Samples (250 grams) of the Lubbock cotton gin waste were pyrolyzed at 800°C for 1 hour, and the results are summarized below.

	Pyrolysis Results		
Experiment	C 4-1	C 4-2	C 4-3
% Char	—[a]	29.2	29.4
% Condensible Organic Material	—[a]	7.6	7.5
% Water	30.2	24.8	27.6
% Gases[b]	—	38.4	35.5

[a] Samples lost due to experimental error.

[b] % Gases obtained by difference.

[1] "Official Methods of the Association of Official Analytical Chemists," 10th Edition, A.O.A.C., Washington, D.C., 1965, pp. 112-113.

[2] Reference cited in 1, p. 331.

2. Gases. Analyses of gases from pyrolysis experiments are given below.

Experiment Component	C 4-1	C 4-3
Air	3.5%	1.9%
Carbon Monoxide	20.0	20.2
Methane	13.0	13.5
Carbon Dioxide	35.1	34.0
Hydrogen	24.6	24.0
Not Identified	3.8	6.4

3. Pyrolysis Char. The following properties were determined on the pyrolysis char.

Total Ash	20.7%
Acid Insoluble Ash	10.2%
Density	12.0 lbs./cu. ft.
Iodine Value	14.1

4. Activation Experiment. A sample of the char was treated with steam (1 gram of steam per hour per gram of char) at 800°C for 30 minutes. The yield was 66.4%. The material after treatment had the following properties.

Total Ash	32.34%
Acid Insoluble Ash	13.58
Density (unground)	15.1 lbs./cu. ft.
Density (ground)	32.9 lbs./cu. ft.
Iodine Value (unground)	61.5
Iodine Value (ground)	85.8
Modified Phenol Value (unground)	52.0
Modified phenol Value (ground)	29.3

V. Future Work

During August, additional pyrolyses and activation experiments under

a variety of conditions will be carried out with the cotton shearing waste. Elemental analyses (carbon, hydrogen, and nitrogen) will be made on the chars, activated carbons and condensible organic material obtained from the four different samples. Oxygen analyses will be made on some representative samples.

A-1402

MONTHLY PROGRESS REPORT V

Sponsor: Cotton, Incorporated

Project A-1402: "Evaluation of Pyrolysis as a Means of Resource Recovery from Solid Wastes of the Cotton Industry"

Period Covered: August 1 to August 31, 1972

Prepared by: James A. Knight, Jr.

Cotton Shearing Waste

The activated carbon, obtained from the cotton shearing waste char from the 800°C pyrolysis experiments, retains the fibrous nature of cotton, and shows a high degree of activation based on a modified phenol value of 18.3. The fibrous nature of this activated carbon is extremely interesting and suggests that is has potential use as a gas adsorbent for polluting vapors. During August, additional experiments were carried out with the objective of determining the optimum laboratory conditions for pyrolysis and activation. This work will continue into September. As the experimental work is incomplete, it would be premature to draw any conclusions from the data at this time.

The experimental results to date from the pyrolysis of the cotton shearing wastes are given in Table I.

TABLE I

PYROLYSIS EXPERIMENTS WITH COTTON SHEARING WASTE

Experiment[1]	Temperature °C	Time (minute)	Percent Pyrolytic Product[2]			
			Char	Water	Condensible Organic Material	Gases[3]
1	800	60	17.4	27.2	11.0	44.4
2	800	60	15.6	30.0	15.8	38.6
3	800	60	15.0	31.4	14.7	38.9
4	800	60	15.3	31.2	10.4	43.1
6	800	30	16.1	27.6	11.7	44.6
7	800	15	15.8	29.0	8.9	46.3
8	700	15	16.0	26.3	12.4	45.3
9	700	15	16.0	26.3	11.7	46.1
10	500	30	19.0	27.4	13.6	40.0
11	400	44	24.1	24.7	9.8	41.5
12	400	40	24.2	30.0	8.2	37.6
13	400	46	23.8	30.0	8.2	37.9
15	400	46	24.4	-	-	-

[1]The charge was 175 grams for each experiment except no. 15 in which 200 grams was used.

[2]Pyrolytic products are in percent by weight on a dry basis of original sample.

[3]Gases obtained by difference.

The results to date from the activation experiments with the char from the cotton shearing waste are given in Table II.

TABLE II

ACTIVATION EXPERIMENTS WITH CHAR FROM COTTON SHEARING WASTE

Experiment[1]	Pyrolysis Temperature	Time (minutes)	I. V. (unground)	M.P.V. (unground)	Yield
5	800°C	30	90.5	18.3	89%
14[2]	400°C	10	(a) 57.9[3] (b) 83.2	-[4]	67%
16	400°C	30	77.0	-[4]	61%

[1]Activation conditions: 800°C; steam flow for experiments 5 and 16, 4g. steam/hour/gram of char.

[2]The steam flow rate for this experiment was 22.5 g. steam/hour/gram of char due to an error in flow meter, and therefore, the experiment was run for 10 minutes instead of 30 minutes.

[3]The iodine values are for samples of the activated char taken from different parts of the plug of material. The differences in the I.V.'s are maybe due to the fact that the char was not uniformly activated.

[4]M.P.V.'s are to be determined on these samples.

Elemental Analysis--The carbon, hydrogen, oxygen, and nitrogen content of a representative samples of the chars and condensible organic material from the different waste materials were determined.

Future Work--During September, the work on the optimum conditions for laboratory pyrolysis and activation of the cotton shearing waste will be completed. Heating values on representative chars and condensible organic material from the different waste materials will be determined.

ENGINEERING EXPERIMENT STATION

Georgia Institute of Technology
Atlanta, Georgia

Project A-1402

EVALUATION OF PYROLYSIS AS A MEANS OF RESOURCE RECOVERY FROM SOLID WASTES OF THE COTTON INDUSTRY

by

J. A. Knight, Jr.

Quarterly Progress Report No. 1
1 March 1972 to 31 May 1972

Performed for

COTTON INCORPORATED
P. O. Box 18039
Raleigh, North Carolina 27609

EVALUATION OF PYROLYSIS AS A MEANS OF RESOURCE RECOVERY FROM SOLID WASTES OF THE COTTON INDUSTRY

I. SUMMARY

Samples of solid wastes of the cotton industry have been received from four different sources for experimental work. The major portion of the work has been with the cotton gin trash sample from Tifton, Georgia. Several pyrolysis experiments have been carried out with this material, and the pyrolysis products are characteristic of lignocellulosic material in that the pyrolytic products can be classified as char, water, condensible organic material, and gases. A sample of the char from the cotton gin trash from Tifton, Georgia was treated with steam, and the iodine values indicate that a reasonable degree of activation was achieved.

Pyrolysis of a sample of the material from the Kleen Seed Delinting Company gave results similar to those for the gin trash sample. Gas analysis of the gaseous material from this pyrolysis experiment showed the presence of hydrogen, air, carbon monoxide, methane, and carbon dioxide.

II. FUTURE WORK

The experimental work with the cotton gin trash from Tifton, Georgia will be continued so as to obtain information on the composition of the gaseous product and additional information on the activation experiments. Additional pyrolysis of the sample from Kleen Seed Delinting Company will be carried out, and activation experiments on the char will be initiated. Experimental work on the shearing waste sample (Cotton, Inc.) and the cotton gin sample from Lubbock, Texas, will be started in the near future. Work progress is generally proceeding in accordance with the overall project schedule as originally contemplated.

III. WASTE SAMPLES RECEIVED

Four samples of waste material have been received for experimental work. These are: (1) cotton gin trash sample from Tifton, Georgia; (2) a sample from the Kleen Seed Delinting Company, Shafter, California; (3) shearing material from Cotton, Incorporated; and (4) cotton gin sample from Plains Cotton Co-op Association, Lubbock, Texas.

IV. EXPERIMENTAL WORK

A. Cotton Gin Trash Sample

1. Sample as Received

Approximately 100 pounds of gin trash were received March 10 from Mr. W. E. Seigler, Tifton, Georgia. Mr. Seigler described this gin trash as follows: "This material did not include the trash from unloading fan or any of the condenser exhausts. It did include all trash that is collected from overhead clearners, extractor feeders, gin stand, and lint clearners."

Data obtained on sample are received:

Moisture	14.7%
Density	4.9 lb/cu.ft. (Shaken)
Density	7.1 lb/cu.ft. (Hand packed)

2. Representative Sample

One cubic foot of waste material was removed from each of the four bags of gin trash received. This material was thoroughly mixed and ten pounds removed for grinding. Efforts to grind the original material in a Mikro Bantam Pulverizer were not successful. The sample (ten pounds) was ground in a Mikro Samplemill. This produced a sample suitable for the pyrolysis experiments.

3. Data on Representative Sample

a. Moisture*

Sample #1	12.3%
Sample #2	12.3%

b. Total Ash

Sample #1	5.70%
Sample #2	5.40%

c. Acid-insoluble Ash

Sample #1	2.65%
Sample #2	2.38%

d. Density

Sample Description	lb/cu.ft.
Loose Sample	7.5
Shaken Sample	9.4
Hand Pressed Sample	14.5

4. Pyrolysis

Several pyrolysis experiments with the representative sample of cotton gin trash were carried out during this report period, and the results are summarized in Table I.

TABLE I

PYROLYSIS EXPERIMENTS OF COTTON GIN WASTE (TIFTON, GEORGIA)

Experiment	Sample (gram)[a]	Temperature	Percent Pyrolytic Products[b] Char	Water	Condensible Organic Material	Gases[c]
1	100	700°C	32.2	19.4	11.4	37.0
2	190	800°C	31.7	19.4	10.4	38.5
3	200	700°C	32.4	20.5	11.5	35.6
4	200	800°C	31.5	18.2	11.0	39.3
5	200	800°C	32.2	20.2	10.9	36.7

[a]Samples are of the representative material, which contained 12.3 percent moisture.

[b]The pyrolytic products are given in percent by weight on a dry basis of original sample.

[c]Weight of gases obtained by difference.

5. Activation Experiment

The char material from pyrolysis experiments 2, 4, and 5 were combined for the activation experimental work. One activation experiment with the combined char was carried out. An 80g. sample was treated with steam at 800°C for 30 minutes at a rate of 4g. water/hr/g. char. The yield of treated char was 58.0%. Data obtained on the combined char and treated char are given below.

	Combined Char (non-treated)	Char (steam treated)
Density	14.9 lb/cu.ft.(non-ground)	32.2 lb/cu.ft.*
Ash	19.15%	31.7%
Acid Insoluble Ash	9.55%	17.7%
Iodine Value	12.4	61.6(non-ground)
Iodine Value	-	76.2*

*Sample ball milled for 30 minutes.

The iodine value is used as a measure of the degree of activation of carbon sample. The I. V. of 76.2 for the steam treated char (ball-milled sample) indicates that a reasonable amount of activation has been achieved.

B. Kleen Seed Delinting Company Sample

1. Sample as Received

The material as received is a short fibrous material with small amounts of seed hulls intermixed.

Data obtained on sample as received:

Weight loss on heating at 110° for 4 hours	6.75%
Total ash	2.47%
Acid insoluble ash	0.49%
Density	16.9 lb/cu.ft. (Shaken)
	26.4 lb/cu.ft. (Hand Packed)

2. Pyrolysis

A sample was pyrolyzed and the data are given in Table II.

TABLE II

PYROLYSIS OF KLEEN SEED DELINTING COMPANY SAMPLE

			Percent Pyrolytic Products[a]			
Experiment	Wt. of Sample	Temperature	Char	Water	Condensible Organic Material	Gases[b]
1	160g.	800°C	26.9	27.5	7.1	38.5

[a]The percent by weight is based on the weight of the original sample.

[b]Weight of gases obtained by difference.

3. Gas Analysis

The gases from the pyrolysis experiment of the Kleen Seed sample were analyzed by gas chromatography and the results are given in Table III.

TABLE III

ANALYSIS OF GASES FROM PYROLYSIS OF KLEEN SEED SAMPLE

Gas	% by Volume
Hydrogen	17.7
Air	14.7
Carbon Monoxide	17.8
Methane	14.7
Carbon Dioxide	24.8
Sum*	89.7

*The 10.3 percent of unaccounted for gas is most likely due to the fact that there are probably some gaseous hydrocarbons, such as ethane, that were not detected by this analysis and to experimental errors involved in the overall method.

V. MAJOR EXPERIMENTAL DIFFICULTY

The major difficulty that has been encountered in the experimental work has been in the analysis of the gaseous products from the pyrolysis experiments. The difficuly was due to the gas chromatographic columns used initially and to the experimental techniques involved in collecting the gaseous product. The problem with the gas chromatographic column has been solved, and the experimental technique for collecting and handling the gaseous products has been improved so that the major sources of error have been reduced to a minimum.

ENGINEERING EXPERIMENT STATION
Georgia Institute of Technology
Atlanta, Georgia

Final Report
Project A-1402

Evaluation of Pyrolysis as a Means of Resource Recovery from Solid Wastes of the Cotton Industry

by

J. A. Knight, Jr.

1 March 1972 to 30 October 1972

Performed for

Cotton, Inc.
Raleigh, N. C.

ENGINEERING EXPERIMENT STATION
Georgia Institute of Technology
Atlanta, Georgia

Final Report
Project A-1402

Evaluation of Pyrolysis as a Means of Resource Recovery from Solid Wastes of the Cotton Industry

by

J. A. Knight, Jr.

1 March 1972 to 30 October 1972

Performed for

Cotton, Inc.
Raleigh, N. C.

ACKNOWLEDGEMENT

The very able assistance and performance of Mr. Lewis W. Elston, Mr. Alton R. Colcord, Mr. David R. Hurst, and Mr. J. K. Hendrix in carrying out the laboratory experimental program on this project is acknowledged. Also, Mr. Lewis W. Elston prepared the experimental section of this report.

TABLE OF CONTENTS

TABLES

FIGURES

EVALUATION OF PYROLYSIS AS A MEANS OF RESOURCE RECOVERY FROM SOLID WASTES OF THE COTTON INDUSTRY

1. Introduction

The purpose of this investigation was to evaluate on a laboratory experimental basis the potential of utilizing pyrolysis for resource recovery from solid wastes of the cotton industry. The solid waste materials investigated were cotton gin waste from Tifton, Georgia; seed lint from the Kleen Seed Delinting Company, Shafter, California; cotton shearing waste, supplied by Cotton, Inc., Raleigh, North Carolina; and cotton gin waste from Lubbock, Texas. Representative samples of these materials were pyrolyzed, and the yields of the pyrolytic products determined. Char samples from each of the waste materials were treated with steam at elevated temperatures for activation and the degree of activation was determined by adsorptive tests.

The activated carbon obtained from the cotton shearing waste represents a unique physical form of activated carbon. This activated carbon has the fibrous nature of the original cotton material, and this physical property suggests its use as a gas adsorbent. Because of the unique nature of the activated carbon from the cotton shearing waste, the cotton shearing waste was investigated to a greater extent than the other waste materials.

2. Summary

The samples of waste materials (two different samples of cotton gin waste, lint from seed cleaning, and a cotton shearing waste) were characterized as received with determinations of moisture content, ash, elemental analysis, and density. Pyrolyses of each waste material were made to determine the yields of char, condensible organic material, water and gas.

The pyrolytic chars from each waste material were characterized with determinations of density, ash, iodine value, elemental analysis, and heating value. The pyrolytic chars were used in activation experiments with steam as the activating agent to determine if an activated carbon could be prepared from them. The adsorptive properties of the steam treated chars from the activation experiments were characterized by the iodine value and the modified phenol value. The steam treated chars were further characterized by density measurements, ash content, elemental analysis, and heating value determinations.

The pyrolytic char from the cotton shearing waste was converted on a laboratory scale to an activated carbon which meets the specifications for water grade activated carbon for water purification. The activated carbon from our laboratory experiments had a density which is higher than the best water grade carbon. This would not preclude its use as a water grade carbon, but it would reduce its value some. It is believed that the density property can be improved by a different grinding or pulverizing technique. This activated carbon also has the fibrous nature of the original cotton shearing waste. The fibrous nature of this activated carbon is unique, and suggests its use as a gas adsorbent for gases and vapors.

The pyrolytic chars from the two different samples of cotton gin waste were converted to carbons which had a resonable degree of activation. The activated carbons from the cotton gin wastes also had a high ash content ($\sim$ 32%). This is a disadvantage as a water grade carbon, as the specifications for water grade carbon call for 7% or less total ash. The high ash content would not necessarily preclude its use, for example, in the

treatment of different types of industrial waste water. The non-activated chars from the pyrolysis of the cotton gin waste should be useful as a material for charcoal briquettes. In addition, the char could possibly be useful as a starting material for the preparation of granular carbon for use in filter beds.

The pyrolytic char from the Kleen Seed lint was activated to yield a material which had a modified phenol value of 23.4. The activated carbon had a slightly higher ash content ($\sim$ 9%) than the 7% maximum specification. Also, the density (60.3 lbs./cu. ft.) of this one sample of activated carbon is very high as compared with the density specification of 15 to 25 lbs./cu. ft. The high ash content of the activated carbon could possibly be improved by separating, if feasible, any extraneous dirt, sand, etc. before pyrolysis. The high density could possibly be reduced to a more acceptable value by a different grinding or pulverizing technique. The oil content of the sample of waste material was found to be 6.4%. This means that approximately 16 tons of the lint waste, as received in our laboratory, would yield one ton of oil. If this oil is essentially cottonseed oil, then the economics of the recovery should be investigated.

The yields of condensible organic material from the pyrolyses were determined, and the percentages of carbon, hydrogen, nitrogen, and oxygen and heating values of the organic material were calculated. The organic material from each waste material is a viscous, dark colored substance with a pungent odor.

The major components of the non-condensible gases from the pyrolysis of each waste material and the percent composition were determined. The major

gases were hydrogen, methane, carbon monoxide, and carbon dioxide. There was an unknown portion of gas from each waste material which is most likely hydrocarbons in the ethane to butane range. From the percent composition, the heating values of the gases were calculated.

Based on the data and results of this investigation, recommendations are made for additional applied research, which is oriented toward the development of marketable products. These recommendations are discussed in detail in Section 6 and are summarized as follows:

(a) Utilization of the cotton shearing waste char for water grade activated carbon and as a gas adsorbent;

(b) Utilization of the cotton gin waste char for charcoal briquettes and granular activated carbon;

(c) Work to determine if the char from the Kleen Seed lint can be improved so that it would be acceptable as a water grade carbon;

(d) Utilization of the char from seed lint for charcoal briquettes and granular activated carbon;

(e) Extraction of oil from seed lint;

(f) An exploratory experimental program with the condensible organic material obtained in the pyrolysis of the waste materials.

3. Experimental Section

3.1. Pyrolysis. The waste material to by pyrolyzed was packed into a two inch ID stainless steel tube. The ends were capped, and the closed unit was placed in a Lindberg tube furnace. After connecting the downstream end

of the tube to a condensation train, the material in the tube was heated to the desired temperature and held for a predetermined time. Spacers inside the pyrolysis tube confined the charge to a uniformly heated zone in the furnace. Internal temperatures were monitored with a chromel-alumel thermocouple attached to a potentiometer or to a recorder. A schematic diagram of a typical pyrolysis apparatus arrangement is shown in Figure 1.

Condensible off gases were collected in the condenser, ambient trap, Berl saddle column, glass wool scrubber, and finally a trap cooled with dry ice and acetone. Non-condensible gases were passed through a wet test meter and collected in a plastic bag for subsequent analysis by gas chromatography. On completion of each pyrolysis the flexible tube between the Berl saddle column and the glass wool scrubber was closed by means of a clamp to prevent reaction between air and the heated char while the system cooled.

After cooling, the system was disassembled and the char was collected, dried, and weighed. The organic material from the pyrolysis tube, ambient trap, condenser, Berl saddle column, and cold trap were collected by extraction and washing with methylene chloride. Both the methylene chloride and water phases were filtered through glass wool into a large separatory funnel; and the heavier methylene chloride layer containing the dissolved organic products was drawn off into a tared beaker. The aqueous portion was extracted with small portions of methylene chloride until the extracting solvent remained colorless. The combined methylene chloride extracts were evaporated on a slightly heated magnetic stirrer under an infra-red lamp until the temperature reached approximately 60°C. The residue was weighed,

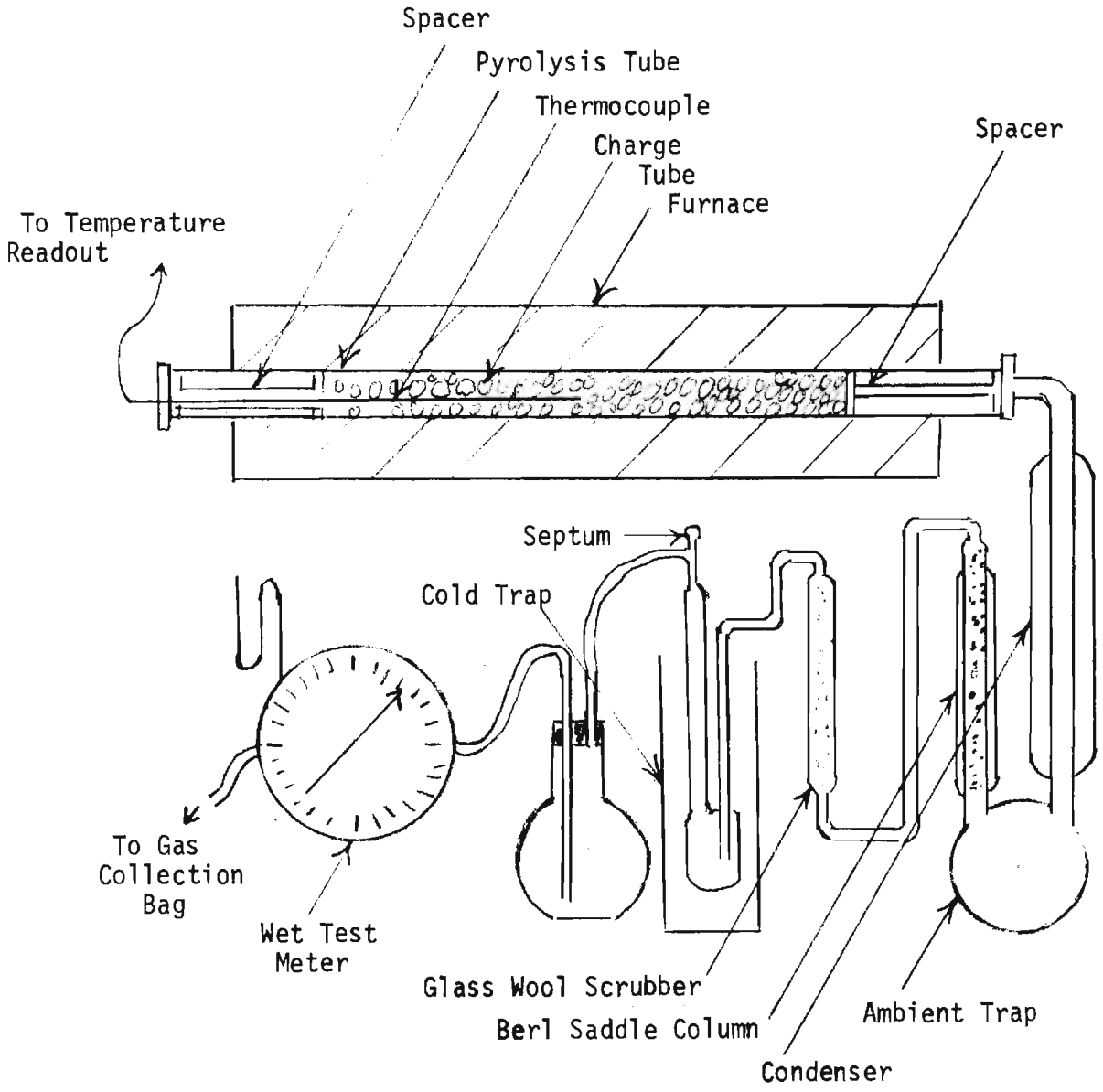

Figure 1. Pyrolysis Apparatus.

and the percent yield of organic material was calculated. The weight of the extracted aqueous phase was used to calculate the percent yield of water from the pyrolysis. Samples of each of the products were reserved for microanalysis and display.

At the end of each pyrolysis the gases remaining in the system were sampled by inserting a syringe through the septum located in the top of the cold trap. The gas collection bag was closed by means of a ball valve in its inlet line.

3.2. Activation. The pyrolysis char to be activated was loaded into a two inch ID Monel tube approximately five and one half feet long. The tube was closed and heated to the desired temperature in a Lindberg tube furnace before exposure to steam. A schematic diagram of the activation apparatus is shown in Figure 2.

The steam generator consisted of a stainless steel tube passed through the upstream end cap of the tube into a stainless steel wool plug, which also served as an upstream spacer. With the charge heated to the desired reaction temperature ($800^{\circ}C$) water was admitted to the inlet tube at a controlled rate for thirty minutes. In early experiments the desired rate of steam per hour per gram of char was controlled by means of needle valves and a flow meter. In later experiments water was transferred from a graduated cylinder by means of a calibrated metering pump.

At the end of each experiment the valves at both ends of the tube were closed to exclude air during cooling. The cooled, activated char was dried and weighed, and the percent yield of activated char was calculated.

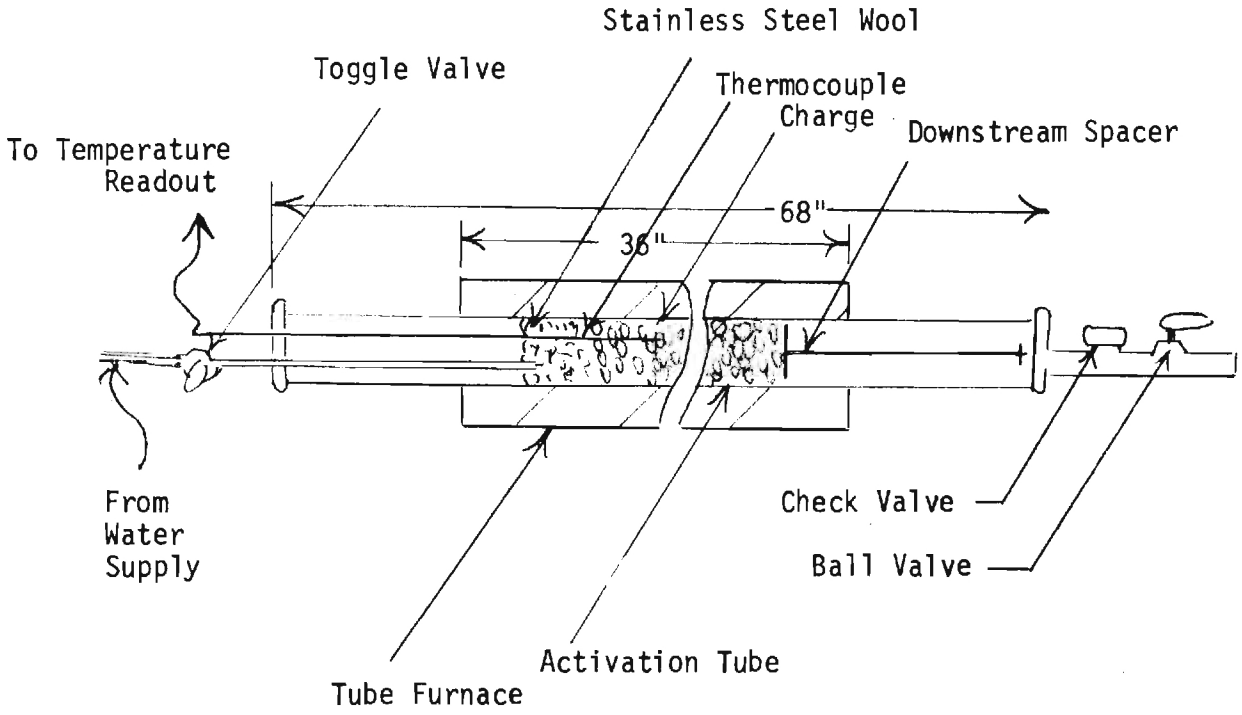

Figure 2. Activation Apparatus

3.3. Gas Chromatography. The gases, collected in a plastic bag during pyrolysis runs and samples drawn from the cold trap at the end of the run, were analyzed by gas chromatography. The composition of the gases evolved could then be calculated from the known volume of the system corrected for furnace temperature plus the volume of gas in the collection bag as shown by the wet test meter, less the known volume of air in the system at the beginning of the run. The principal gases found were air, carbon monoxide, methane, carbon dioxide, and hydrogen. Minor quantities of ethane, ethylene, acetylene, propane, and propylene were detected in the products of an early pyrolysis.

Hydrogen was determined with a Perkin Elmer 820 instrument using a hot wire detector. The 1/8" x 12' molecular sieve column using argon carrier gas at a flow rate of 25 ml/minute was operated at room temperature. The hydrogen volume in each sample injected was determined by comparing the height of the sample hydrogen peak with that of the average height of several standard peaks resulting from injection of 100 microliters of hydrogen.

The remaining major gas components were determined on a 10 foot activated charcoal column using helium carrier gas and operated at 100°C. Roughly half of the analyses were run in a Perkin Elmer Model 154 B Vapor Fractometer using a 1/4" OD column and a flow rate of 100 ml/min. The remaining analyses for major components were performed on a Perkin Elmer Model 990 using 25 ml of helium per minute. Both instruments employed thermal conductivity detectors, thermistor and hot wire, respectively.

The order of gas elution from these instruments was air, carbon monoxide, methane, and carbon dioxide. Determinations of air and carbon monoxide in the

samples were made by comparison of their peak heights with those of peaks generated by 100 microliter samples of room air and cylinder carbon monoxide. The broader methane and carbon dioxide peaks permitted comparison of sample peak areas (measured by planimeter) with those of standard peaks arising from injection of 200 microliters of methane and 300 microliters of carbon dioxide.

The reliability of this method was checked by running known mercury pumped mixtures of hydrogen, air, carbon monoxide, methane and carbon dioxide under the conditions used to analyze the pyrolysis gas.

The minor components were run on a Hewlett Packard Model 776 Autoprep unit using a four foot 1/4" OD column packed with Apiezon grease on Chromosorb P at room temperature with nitrogen carrier gas and a flame ionization detector.

3.4. Elemental Analysis. Nitrogen, carbon, hydrogen, and oxygen were determined by a microanalytical method using a Perkin Elmer Model 240 Elemental Analyzer. Details of the procedure are given in the instruction manual (Perkin Elmer Part No. 990-9572) and in "Instructions, Oxygen Analysis Kit" (Perkin Elmer Part No. 990-9630).

Sulfur was determined by conventional precipitation as barium sulfate and ignition in tared Grooch crucibles. The samples were prepared by igniting a weighed portion of the material in a Parr oxygen bomb. In practice, the residues from heating value determinations in the oxygen bomb calorimeter were rinsed into beakers, acidified with dilute hydrochloric acid, heated, then carried through the precipitation, digestion, ignition, and weighing steps.

3.5. Calorimetry. Heating values of chars and organic fractions were determined in a Parr Oxygen Bomb Calorimeter, plain type with 1108 double

valve bomb. Details of the procedure are given in the Parr Instrument Company Technical Manual No. 130, pp. 33-36.

3.6. Total Ash and Acid Insoluble Ash. Total ash was determined by igniting a 1.000 gram portion of ground char or a 5.000 gram portion of unburned sample to constant weight in a tared crucible in a muffle furnace at 800°C.

The crucible and total ash were transferred to a beaker and heated with a mixture of 3:1 nitric acid: hydrochloric acid until brown fumes were no longer evolved. The solution was diluted with cold water and passed through a Whatman No. 40 filter paper, taking care to transfer all solids adhering to the crucible and the beaker to the filter paper. After thorough rinsing with water the paper and retained solids were returned to the crucible and ignited to constant weight. The percent acid insoluble ash was calculated from the weight of ash remaining in the crucible.

3.7. Iodine Value. The iodine value represents the amount of iodine adsorbed from a given volume of a standard iodine solution by 0.4 gram of dry activated carbon. Therefore, an increase in the iodine value indicates that the carbon has a greater degree of activation. An I.V. of 100 would mean that the carbon sample had adsorbed all of the iodine.

The iodine test solution was prepared by dissolving 2.70 to 2.75 grams of iodine per liter in an aqueous solution of 4.05 to 4.12 grams per liter of potassium iodide. Immediately before use the solution was standardized with N/100 sodium thiosulfate solution. Titer A is defined as the ml N/100 sodium thiosulfate required to titrate 25 ml iodine test solution acidified with 10 ml 5 percent hydrochloric acid.

Duplicate 0.4000 gram samples of dry char were weighed into 150 ml beakers and wet with 10 ml of 5 percent hydrochloric acid. A 100 ml portion of standardized iodine test solution was added to the beaker, and the solution was transferred twelve times from one beaker to another. The solution was then filtered through a 15 cm. Whatman No. 5 filter paper. A 55 ml portion of the solution was transferred by pipet to a 250 ml flask, and titrated with N/100 sodium thiosulfate solution. The volume of thiosulfate required for this titration is defined at Titer B. The iodine value of the char was calculated by the equation:

$$\text{Iodine Value} = \frac{2 \text{ Titer A} - \text{Titer B}}{2 \text{ Titer A}} \times 100.$$

3.8. Modified Phenol Value. The modified phenol value (MPV) is the amount of activated carbon in ppm required to reduce the phenol content of a standard 200 ppm phenol solution to 20 ppm phenol. Therefore, a low MPV indicates that the carbon has a high degree of activation. A carbon should have a MPV of 30 or less to be considered an activated carbon. Commercial activated carbon which is sold as water grade carbon is specified as having a MPV of 20±2.

Modified phenol values were determined by the Colebaugh method developed by the Westvaco Carbon Company. Residual phenol concentrations were determined by optical absorbance measurements at 270 mm. using a Coleman 111 spectrophotometer and a deuterium light source.

3.9. Screen Analysis. Particle size determinations were made by a wet screen method using one gram samples and three inch screens, which could be

weighed on a standard analytical balance. The screen mesh sizes were U.S. Standard 100, 200, 325, and 400. The samples were weighed into a 250 ml beaker, dispersed in water, and gradually poured through a thoroughly wet stack of tared screens. A continuous stream of wash water was passed through the stack during the operation. Addition of a small quantity of detergent (Fisher Sparkleen) to the dispersion prevented screen blocking and speeded the washing. When no more carbon appeared to be passing through the bottom (400 mesh) screen, the top (100 mesh) screen was raised from the stack and thoroughly washed with water with the washings passing into the 200 mesh screen. The procedure was repeated for each screen in the stack. The washed screens with the retained carbon were dried to constant weight at 105°C, and the percent carbon retained on each screen was calculated.

3.10. Grinding. Only the gin waste samples from Tifton, Georgia, and Lubbock, Texas, and the charcoal sample from Cotton, Inc., required any grinding prior to preliminary (as received) analysis. The cotton gin waste from Tifton, Georgia was ground in a Mikro Sample Mill (Spex Industries, Inc.) with no screen on the grinding chamber. The cotton gin waste from Lubbock, Texas was ground in a Wiley Standard Model No. 4 mill (Arthur H. Thomas Co.) using a 6 mm. screen. This is a cutting rather than a hammer mill and requires roughly one fourth of the time to grind a fibrous sample. A portion of the char sample (Cotton, Inc.) was ground in a Wiley Intermediate Mill using an 80 mesh screen.

The activated chars were ground in a one quart jar mill using 13/16" Burundum cylinders at a charge rate of eight pounds of cylinders per gallon. Approximately one hour of grinding time was required to produce a sample of

which less than ten percent would be retained on a 325 mesh screen in the wet screen analysis described above. The activated chars prepared from the cotton gin wastes, the delinting waste, and the char (Cotton, Inc.) were all ground in this manner. Only one sample of the activated char prepared from shearing waste was ground, as the iodine values and modified phenol values of the ground and unground samples were similar. This unground activated char had a very low bulk density and unique fibrous structure.

3.11. Lignin Analysis. Lignin in the cotton gin waste, Tifton, Georgia, was determined by the direct method described on pp. 112 and 113 of Official Methods of Analysis of the Association of Official Analytical Chemists, 10th edition, AOAC, Washington, D. C., 1965.

3.12. Oil Analysis. The delinting waste sample was analyzed for oil by the direct, anhydrous ether method described on page 112 of the reference in Section 3.11.

4. Results

The data and results obtained for each individual waste material are summarized under separate headings in Section 4.1. Tables giving a comparison of the data for the waste materials are in Section 4.2.

4.1. Data and Results for Each Individual Waste Material Investigated.

4.1.1. Cotton Gin Waste, Tifton, Georgia.

4.1.1.1. Description and Properties. Mr. W. E. Zeigler, Tifton, Georgia, described this gin waste as follows: "This material did not include the trash from unloading fan or any of the condenser exhausts. It did include all trash that is collected from overhead cleaners, extractor feeders, gin stand, and lint cleaners."

Data on sample as received:

Moisture Content	14.7%
Density-Shaken Sample	4.9 lb./cu. ft.
Density-Hand Pressed Sample	7.1 lb./cu. ft.

4.1.1.2. Representative Sample. One cubic foot of waste material was removed from each of the four bags of gin waste received. This material was thoroughly mixed and 10 pounds removed for grinding. Efforts to grind the original material in a Mikro Bantam Pulverizer were not successful. The sample (10 pounds) was ground in a Mikro Samplmill. This material which was suitable for laboratory experiments had the following properties:

Moisture Content*	11.7%
Total Ash	5.55%
Acid Insoluble Ash	2.38%
Elemental Analysis	
Carbon	46.4%
Hydrogen	5.80%
Nitrogen	1.76%
Oxygen	36.45%
Density-Loose Sample	7.5 lb./cu. ft.
Density-Shaken Sample	9.5 lb./cu. ft.
Density-Hand Pressed Sample	14.5 lb./cu. ft.

*(The slight decrease in the moisture content from the original sample could be due to heat generated in the grinding operation.)

4.1.1.3. Pyrolysis Experiments. Several samples of the representative material of the cotton gin waste (Tifton, Georgia) were pyrolyzed, and the results are summarized in Table 1. The pyrolysis experiments were carried out by the procedure given in the experimental section.

4.1.1.4. Analysis of Non-condensible Gases. The non-condensible gases from pyrolysis experiments 1-7 and 1-8 were collected and analyzed as described in the experimental section and the results are given in Table 2.

TABLE 1

PYROLYSIS EXPERIMENTS OF COTTON GIN WASTE (TIFTON, GEORGIA)

Experiment	Sample (grams)[b]	Temperature	Percent Pyrolytic Products[c] Char	Water	Condensible Organic Materials	Gases[d]
1-1	100	700°C	32.2	19.4	11.4	37.0
1-2	190	800°C	31.7	19.4	10.4	38.5
1-3	200	700°C	32.4	20.5	11.5	35.6
1-4	200	800°C	31.5	18.2	11.0	39.3
1-5	200	800°C	32.2	20.2	10.9	36.7
1-7[e]	200	800°C	32.9	--	--	--
1-8[e]	200	800°C	33.4	--	--	--
1-9[f]	300	800°C	31.8	--	--	--

[a] Pyrolysis experiments were carried out for 60 minutes at temperature given except 1-9 which was run for only 30 minutes.

[b] Samples are of the representative material, which contained 11.7 percent moisture.

[c] The pyrolytic products are given in percent by weight on a dry basis of original sample.

[d] Gases obtained by difference.

[e] These pyrolysis experiments were run to obtain gases for analysis.

[f] This pyrolysis experiment was run to obtain char for activation experiments.

TABLE 2

ANALYSIS OF NON-CONDENSIBLE GASES FROM COTTON GIN WASTE (TIFTON, GEORGIA)

Component	Experiment 1-7 % Volume	Experiment 1-7 Weight (grams)	Experiment 1-8 % Volume	Experiment 1-8 Weight (grams)
Hydrogen	26.6	1.1	23.2	1.0
Air	3.5	5.0	1.4	0.8
Carbon Monoxide	20.7	11.7	20.5	12.6
Methane	14.9	4.8	14.2	5.0
Carbon Dioxide	29.9	26.7	30.9	29.9
Not Identified	4.4	--	9.8	--

4.1.1.5. Data on Char Obtained from Pyrolysis Experiments. Representative data on the char obtained from the pyrolysis of cotton gin waste from Tifton, Georgia, are given in Table 3.

TABLE 3

CHAR FROM PYROLYSIS OF COTTON GIN WASTE (TIFTON, GEORGIA)

Density	14.9 lb./cu. ft.
Total Ash	19.2%
Acid Insoluble Ash	9.6%
Iodine Value	12.4
Elemental Analysis[a]	
Carbon	63.5%
Hydrogen	1.10%
Nitrogen	1.28%
Oxygen	6.85%
Sulfur	0.25%
Heating Value, BTU/lb.	10,134

[a] Char from pyrolysis experiment 1-1. See Table 5 for additional elemental data.

4.1.1.6. Activation Experiments with Char from Cotton Gin Waste, Tifton, Georgia. Samples of the char from the pyrolysis experiments were treated with steam for activation tests as described in the experimental section. The conditions and results from these activation experiments are given in Table 4.

TABLE 4

ACTIVATION EXPERIMENTS WITH CHAR FROM COTTON GIN WASTE (TIFTON, GEORGIA)

Conditions for Activation	Experiment 1-6	Experiment 1-10
Wt. of char	80 grams	80 grams
Temperature	800°C	800°C
Time	30 min.	30 min.
Steam Rate	4g/hr./g.char	6g/hr./g.char
Yield	58.0%	53.4%
Properties of Activated Char		
Density (ball-milled)	32.2 lb./cu.ft.[a]	36.0 lb./cu.ft.[b]
Total Ash	31.7%	31.5%
Acid Insoluble Ash	17.7%	12.9%
Iodine Value (non-ground)	61.6	--
Iodine Value (ball-milled)	76.2[a]	72.8[b]
Modified Phenol Value (non-ground)	37.6	--
Modified Phenol Value (ball-milled)	31.0	30.4
Screen Size		
+100	0.98%[a]	0.04[b]
100 x 200	16.76%	0.08
200 x 325	14.22%	0.67
325 x 400	1.85%	0.34
-400	66.19%	98.87
Elemental Analysis		
Carbon	62.0%	--
Hydrogen	0.73%	--
Nitrogen	0.64%	--
Oxygen	5.49%	--

[a] Sample ball-milled for 30 minutes.

[b] Sample ball-milled for 60 minutes.

4.1.1.7. Elemental Analysis. (a) Samples of the char and condensible organic material were analyzed for carbon, hydrogen, nitrogen and oxygen, and the results are given in Table 5.

(b) The char and condensible organic material from pyrolysis experiment number three were analyzed for sulfur, and the sulfur content was found to be 0.25 percent for the char and 0.14 percent for the condensible organic material.

TABLE 5

ELEMENTAL ANALYSIS OF PYROLYTIC PRODUCTS FROM COTTON GIN WASTE (TIFTON, GEORGIA)

Experiment	Sample	Percent Element			
		Carbon	Hydrogen	Nitrogen	Oxygen
1-1 - Pyrolysis	Char	63.5	1.10	1.28	6.85
1-1 - Pyrolysis	Condensible Organic	71.7	8.25	4.06	15.87
1-2 - Pyrolysis	Char	65.4	0.83	1.11	--
1-2 - Pyrolysis	Condensible Organic	71.2	8.17	3.97	--
1-3 - Pyrolysis	Char	65.2	1.06	1.51	6.14
1-3 - Pyrolysis	Condensible Organic	71.4	8.23	3.88	15.99
1-6 - Activation	Char	62.0	0.73	0.64	5.49

4.1.1.8. Heating Values. The heating values of the gases were calculated from the percent composition, Table 2, of the gaseous phase, and the heating values of the char and condensible organic material were determined by the bomb calorimetry technique. The heating values obtained are:

Gases - Experiment 1-7	353 BTU/cu.ft. (air-free)
Gases - Experiment 1-8	416 BTU/cu.ft. (air-free)
Char	10,134 BTU/lb.
Condensible organic material	14,355 BTU/lb.

4.1.1.9. Special Analyses. (a) The lignin content of the representative sample of the cotton gin waste (Tifton, Georgia) was determined by the A.O.A.C. official method and was found to be 20.9%. This compares with a lignin content of 33.6% in peanut hulls. Lignin and cellulose are different chemically, and therefore, should give different products when pyrolyzed.

(b) Examination for sand, dirt, etc. A sample of the cotton gin waste (Tifton, Georgia) was examined with a 4x hand magnifier to determine if loosely adhering dirt and sand were present. None was observed. A sample was shaken on the Ro-Tap, and a few small grains of sand were observed in the fines. This indicates that the relatively high ash content is due to inorganic material chemically combined in the original sample, or that extraneous inorganic material is intimately mixed in with the sample and is not easily removed.

(c) Ash in Char. A sample of char was extracted with water for one hour and lost 2.17% ash content due to water soluble material. A second sample was extracted with 5% sulfuric acid for one hour, and then washed free of acid. There was no loss in ash content of these acid washed samples. These experiments indicate that the inorganic material that constitutes the ash is a part of the matrix of the char and is not easily extracted.

4.1.2. Kleen Seed Delinting Company Sample.

4.1.2.1. Description and Properties. The material as received is a short fibrous material with small amounts of seed hulls intermixed. The material has an oily feel.

Data obtained on sample as received are:

Weight Loss on Heating at 110° for 4 hours*	6.75%
Total Ash	2.47%
Acid Insoluble Ash	0.49%
Elemental Analysis	
Carbon	47.4%
Hydrogen	6.65%
Nitrogen	1.61%
Oxygen	43.48%
Density - Shaken Sample	16.9 lb./cu.ft.
Density - Hand-Pressed Sample	26.4 lb./cu.ft.
Oil Content	6.4%

*This weight loss would be due primarily to moisture and any of the oil that volatized at 110°C.

4.1.2.2. Representative Sample. The sample as received was reasonably homogeneous throughout and was used without additional mixing for the experimental work.

4.1.2.3. Pyrolysis Experiments. Four pyrolysis experiments were carried out on the Kleen Seed lint sample by the procedure in the experimental section, and the results are given in Table 6.

TABLE 6

PYROLYSIS EXPERIMENTS OF KLEEN SEED LINT

Experiment[a]	Sample (grams)	Percent Pyrolytic Products[b]			
		Char	Water	Condensible Organic Material	Gases[c]
2-1	160	28.9	22.2	7.6	41.3
2-2	160	31.2	23.4	15.4	30.0
2-3[d]	320	31.2	23.8	9.6	35.4
2-5[d]	350	27.7	--	--	--

[a]All pyrolyses were at 800°C for 60 minutes.
[b]The pyrolytic products are given in percent by weight on a dry basis of original sample.
[c]Gases obtained by difference.
[d]This pyrolysis was carried out for the purpose of obtaining additional char for activation experiments.

4.1.2.4. Analysis of Non-Condensible Gases. The non-condensible gases from two pyrolysis experiments were collected and analyzed, as described in the experimental section, and the results are given in Table 7.

TABLE 7

ANALYSIS OF NON-CONDENSIBLE GASES FROM KLEEN SEED LINT WASTE

Component	Experiment 2-2		Experiment 2-3	
	% Volume	Wt. (grams)	% Volume	Wt. (grams)
Hydrogen	21.3	0.7	25.6	1.5
Air	0	--	0	--
Carbon Monoxide	21.3	10.1	19.3	15.8
Methane	15.3	4.1	17.2	8.1
Carbon Dioxide	30.9	17.6	26.2	33.8
Not Identified	11.9	--	11.7	--

4.1.2.5. Data on Char Obtained from Pyrolysis Experiments. Representative data on the char obtained from the pyrolysis of the lint from the Kleen Seed Delinting Company are listed in Table 8.

TABLE 8

CHAR FROM PYROLYSIS OF LINT FROM KLEEN SEED DELINTING COMPANY

Density	20.4 lb./cu.ft.
Total Ash	7.2%
Acid Insoluble Ash	1.24%
Iodine Value	14.9
Elemental Analysis[a]	
Carbon	84.8%
Hydrogen	1.11%
Nitrogen	2.97%
Oxygen	4.90%
Sulfur	0.22%
Heating Value, BTU/lb.	11,888

[a]Char from pyrolysis experiment No. 2-1 with lint. See Table 10 for additional elemental data.

4.1.2.6. Activation Experiments with Char from Lint from Kleen Seed Delinting Company. The char from the pyrolysis of the Kleen Seed lint was treated with steam as deccribed in the experimental section. The conditions and results from these activation experiments are given in Table 9.

TABLE 9

ACTIVATION EXPERIMENTS WITH CHAR FROM LINT
FROM KLEEN SEED DELINTING COMPANY

Conditions for Activation	Experiment 2-4	Experiment 2-6
Wt. of Char	80 g.	80 g.
Temperature	800°C	800°C
Time	30 min.	30 min.
Steam Rate	4g./hr./g.char	6g./hr./g.char
Yield	85.3%	74%
Properties of Activated Char		
Density (ball-Milled)	41.2 lbs./cu.ft.[a]	60.3 lbs./cu.ft.[b]
Total Ash	8.99	8.68
Acid Insoluble Ash	1.71	1.13
Iodine Value (non-ground)	39.5	92.0
Iodine Value (ball-milled)	67.7	--
Modified Phenol Value (ball-milled)	31.4	23.4
Screen Size		
+100	0.75%[a]	0.18%[b]
100 x 200	6.82%	0.61%
200 x 325	6.21%	2.99%
325 x 400	1.49%	1.94%
-400	84.72%	94.28%
Elemental Analysis		
Carbon	86.4%	--
Hydrogen	0.98%	--
Nitrogen	1.60%	--
Oxygen	2.25%	--

[a] Sample ball-milled for 30 minutes.

[b] Sample ball-milled for 60 minutes.

4.1.2.7. Elemental Analysis. (a) Samples of the char and condensible organic material were analyzed for carbon, hydrogen, nitrogen, and oxygen, and the results are given in Table 10.

TABLE 10

ELEMENTAL ANALYSIS OF PYROLYTIC PRODUCTS FROM KLEEN SEED LINT

Experiment	Sample	Percent Element			
		Carbon	Hydrogen	Nitrogen	Oxygen
2-1 - Pyrolysis	Char	84.8	1.11	2.97	4.90
2-1 - Pyrolysis	Condensible Organic	70.34	8.49	2.05	12.11
2-2 - Pyrolysis	Char	84.5	1.20	2.32	3.53
2-2 - Pyrolysis	Condensible Organic	72.7	9.16	1.93	10.76
2-4 - Activation	Char	86.4	0.98	1.60	2.25

(b) The sulfur content of the char and condensible organic material were determined to be 0.22% and 0.16%, respectively.

4.1.2.8. Heating Values. The heating values of the gases were calculated from the percent composition, Table 7, and the heating values of the char and condensible organic material were determined by the bomb calorimetry techniques. The heating values obtained are:

Gases - Experiment 2-2	439 BTU/cu.ft. (air-free)
Gases - Experiment 2-3	469 BTU/cu.ft. (air-free)
Char	11,888 BTU/lb.
Condensible organic material	14,063 BTU/lb.

4.1.2.9. Special Analysis. The lint from Kleen Seed Delinting Company had an oily feel. A sample was analyzed for oil content and gave a value of 6.4%.

4.1.3. Shearing Waste

4.1.3.1. Description and Properties. The cotton shearing waste was a dyed material and appeared to be free of any extraneous material. Data obtained on the sample as received are given below.

Moisture Content	4.8%
Total Ash	0.29%
Acid Insoluble Ash	None detected
Elemental Analysis	
Carbon	44.4%
Hydrogen	6.30%
Nitrogen	0.38%
Oxygen	47.22%
Density-Shaken Sample	2.6 lb/cu. ft.
Density-Hand Pressed Sample	5.8 lb/cu. ft.

4.1.3.2. Representative Sample. The sample as received was homogeneous throughout and appeared to be free of any extraneous material. It was used as received for the experimental work.

4.1.3.3. Pyrolysis Experiments. Several samples of the shearing waste were pyrolyzed by the general procedure given in the experimental section, and the results are summarized in Table 11.

4.1.3.4. Analysis of Non-Condensible Gases. The non-condensible gases for some of the pyrolysis experiments were collected and analyzed as described in the experimental section, and the results are given in Table 12.

4.1.3.5. Data on Char Obtained from Pyrolysis Experiments. Representative data on the char obtained from the pyrolysis of cotton shearing waste are given in Table 13.

TABLE 11

PYROLYSIS EXPERIMENTS WITH COTTON SHEARING WASTE

Experiment[a]	Temperature °C	Time (minutes)	Percent Pyrolytic Product[b]			
			Char	Water	Condensible Organic Material	Gases[c]
3-1	800	60	17.4	27.2	11.0	44.4
3-2	800	60	15.6	30.0	15.8	38.6
3-3	800	60	15.0	31.4	14.7	38.9
3-4	800	60	15.3	31.2	10.4	43.1
3-6	800	30	16.1	27.6	11.7	44.6
3-7	800	15	15.8	29.0	8.9	46.3
3-8	700	15	16.0	26.3	12.4	45.3
3-9	700	15	16.0	26.3	11.7	46.1
3-10	500	30	19.0	27.4	13.6	40.0
3-11	400	44	24.1	24.7	9.8	41.5
3-12	400	40	24.2	30.0	8.2	37.6
3-13	400	46	23.8	30.0	8.2	37.9
3-15[d]	400	46	25.6	--	--	--
3-17	700	30	15.7	29.1	12.4	42.8
3-18	500	30	19.6	29.4	12.7	38.4
3-20[d]	800	30	16.5	29.4	12.7	38.4

[a]The charge was 175 grams for each experiment except Nos. 3-15, 3-17, and 3-18 in which 200 grams was used, and No. 3-20 in which 250 grams was used.

[b]Pyrolytic products are in percent by weight on a dry basis of original sample.

[c]Gases obtained by difference.

[d]These pyrolyses were run for the purpose of obtaining char for activation experiments.

TABLE 12

PERCENT COMPOSITION OF NON-CONDENSIBLE GASES FROM COTTON SHEARING WASTE

Component	Experiment 3-1 (800°C)	3-2 (800°C)	3-7 (800°C)	3-9 (700°C)	3-10 (500°C)	3-11 (400°C)
Hydrogen	18.0	20.5	25.6	14.8	7.2	0
Air	6.3	7.1	0	7.6	0	0
Carbon Monoxide	29.2	28.8	31.9	29.7	32.9	37.0
Methane	12.3	11.4	10.2	11.9	13.1	4.1
Carbon Dioxide	28.2	27.3	25.6	29.4	41.4	54.1
Not identified	6.0	3.9	6.7	13.6	5.4	4.8

TABLE 13

CHAR FROM PYROLYSIS OF COTTON SHEARING WASTE

Density	4.0 lb/cu. ft.
Total Ash	2.42%
Acid Insoluble Ash	0.74%
Iodine Value	30.3
Elemental Analysis[a]	
Carbon	95.8%
Hydrogen	1.19%
Nitrogen	0.26%
Oxygen	2.24%
Sulfur	0%
Heating Value	
BTU/lb	13,276

[a]Char from pyrolysis experiment No. 3-1. See Table 14 for additional elemental analysis data.

4.1.3.6. Activation Experiments with Char From Cotton Shearing Waste. Samples of the char material from the pyrolysis experiments at various temperatures were treated with steam for activation tests as described in the experimental section. The conditions and results from these activation experiments are given in Table 14.

4.1.3.7. Elemental Analysis. (a). Samples of the char and condensible organic material were analyzed for carbon, hydrogen, nitrogen, and oxygen and the results are given in Table 15.

(b). The sulfur content of the char and condensible organic material were determined to be 0% and 0.21%, respectively.

4.1.3.8. Heating Values. The heating values of the gases were calculated from the percent composition, Table 12, and the heating values of the char and condensible organic material were determined by the bomb calorimetry technique. The heating values obtained are:

Gases	Experiment No. 3-1	370 BTU/cu. ft.
	Experiment No. 3-2	338 BTU/cu. ft.
	Experiment No. 3-11	231 BTU/cu. ft.
Char		13,276 BTU/lb.
Condensible Organic Material		12,595 BTU/lb.

4.1.4. Cotton Gin Waste, Lubbock, Texas.

4.1.4.1. Description and Properties. The material was similar in appearance to the gin waste from Tifton, Georgia. The waste contained stems, cotton fibers, lint, leaves, and similar material.

TABLE 14

ACTIVATION EXPERIMENTS WITH CHAR FROM COTTON SHEARING WASTE

Conditions for Activation	Experiment 3-5	3-16	3-19	3-21
Weight Char	80g.	80g.	80g.	80g.
Pyrolysis Temp. Char Obtained	800°C	400°C	700°C	500°C
Activation Temp.	800°C	800°C	800°C	800°C
Activation Time	30 min.	30 min.	30 min.	30 min.
Steam (g/hr/g char)	4	4	4	4
Yield	89%	61.1	76.7	64.0
Properties of Activated Char				
Density (non-ground) lb/cu. ft.	5.9	----	3.9	3.7
Density (ball-milled) lb/cu. ft.	34.6[a]	----	----	----
Total Ash	3.24%	2.80	2.73	3.04
Acid Insoluble Ash	1.43%	0.94	2.33	2.69
Iodine Value (non-ground)	90.5	77.0	79.3	87.0
Iodine Value (ball-milled)	98.0	----	----	----
Modified Phenol Value (non-ground)	18.7	18.8	22.6	23.6
Modified Phenol Value (ball-milled)	18.3	----	----	----
Screen Size				
+ 100	0.35%[a]	----[b]	----[b]	----[b]
100 x 200	0.69%	----	----	----
200 x 325	0.97%	----	----	----
325 x 400	0.64%	----	----	----
- 400	97.35%	----	----	----
Elemental Analysis				
Carbon	91.9%	----	----	----
Hydrogen	1.17%	----	----	----
Nitrogen	0%	----	----	----
Oxygen	2.10%	----	----	----

[a] Sample ball-milled for 30 minutes

[b] These samples were not ball-milled since this procedure does not effect the modified phenol value.

TABLE 15

ELEMENTAL ANALYSIS OF PYROLYTIC PRODUCTS
FROM COTTON SHEARING WASTE

Experiment Number	Temp °C	Time-Minutes	Sample	Percent Element			
				Carbon	Hydrogen	Nitrogen	Oxygen
3-1-Pyrolysis	800	60	Char	95.8	1.19	0.26	2.24
3-1-Pyrolysis			Condensible Organic	68.7	8.49	0.22	16.78
3-3-Pyrolysis	800	60	Char	98.2	1.16	0.02	2.74
3-3-Pyrolysis			Condensible Organic	69.3	9.27	0.12	12.3
3-6-Pyrolysis	800	30	Char	93.0	1.85	0	-
3-6-Pyrolysis			Condensible Organic	56.9	6.72	0.28	-
3-7-Pyrolysis	800	15	Char	91.7	1.21	0.07	-
3-7-Pyrolysis			Condensible Organic	58.8	1.73	0	-
3-8-Pyrolysis	700	15	Char	91.8	1.68	0.01	-
3-8-Pyrolysis			Condensible Organic	69.6	9.28	0.26	-
3-9-Pyrolysis	700	15	Char	91.2	1.86	0.15	5.42
3-9-Pyrolysis			Condensible Organic	67.8	8.34	0.34	18.50
3-10-Pyrolysis	500	30	Char	86.2	3.36	0.13	7.49
3-10-Pyrolysis			Condensible Organic	67.8	7.05	0	11.77
3-11-Pyrolysis	400	44	Char	76.2	4.00	0.54	16.35
3-11-Pyrolysis			Condensible Organic	58.3	6.87	0.43	27.52
3-5-Activation	800	30	Carbon	91.9	1.17	0	2.10

4.1.4.2. Representative Sample. A sample of the waste as received was removed from each of the four boxes of gin waste from Lubbock, Texas. This material was ground using a Wiley mill. After grinding, the material was thoroughly mixed and had the following properties:

Moisture	10.01%
Total Ash	6.20%
Acid Insoluble Ash	2.56%
Elemental Analysis	
Carbon	44.2%
Hydrogen	5.40%
Nitrogen	1.36%
Oxygen	38.29%
Density-Shaken Sample	9.3 lb/cu. ft.
Density-Hand Pressed Sample	14.5 lb/cu. ft.

4.1.4.3. Pyrolysis Experiments. Samples of the representative material were pyrolyzed by the procedure in the experimental section, and the results are given in Table 16.

4.1.4.4. Analysis of Non-Condensible Gases. The non-condensible gases from pyrolysis experiments were collected and analyzed as described in the experimental section, and the results are given in Table 17.

4.1.4.5. Data on Char Obtained from Pyrolysis Experiments. Representative data on the char obtained from the pyrolysis of cotton gin waste, Lubbock, Texas, are given in Table 18.

4.1.4.6. Activation Experiments With Char From Cotton Gin Waste, Lubbock, Texas. Samples of the char from the pyrolysis experiments were treated with steam for activation tests as described in the experimental section. The conditions and results from these activation experiments are given in Table 19.

TABLE 16

PYROLYSIS EXPERIMENTS OF COTTON GIN WASTE (LUBBOCK, TEXAS)

Experiment[a]	Sample[b] (grams)	Percent Pyrolytic Products[c]			
		Char	Water	Condensible Organic Material	Gases[d]
4-1	250	--[e]	20	--[e]	--[e]
4-2	250	32.5	16.4	8.4	42.7
4-3	250	32.6	19.5	8.3	39.6
4-5	355	30.8	--	--	--

[a]Pyrolysis carried out at 800° C for 60 minutes.

[b]Samples are of representative material, which contained 10.1 percent moisture.

[c]The pyrolytic products are given in percent by weight on a dry basis of original sample.

[d]Gases obtained by difference.

[e]Sample lost due to experimental error.

TABLE 17

ANALYSIS OF NON-CONDENSIBLE GASES FROM
COTTON GIN WASTE (LUBBOCK, TEXAS)

	Experiment 4-1		Experiment 4-2	
Component	% Composition	wt.(grams)	% Composition	wt.(grams)
Hydrogen	24.6	1.3	24.0	1.3
Air	3.5	2.6	1.9	1.5
Carbon Monoxide	20.0	15.1	20.2	15.7
Methane	13.0	5.6	13.5	6.0
Carbon Dioxide	35.1	41.8	34.0	41.2
Not Identified	3.8	-	6.4	-

TABLE 18

CHAR FROM PYROLYSIS OF COTTON GIN WASTE,
LUBBOCK, TEXAS

Density	12.0 lb/cu. ft.
Total Ash	20.73
Acid Insoluble Ash	10.23
Iodine Value	14.1
Elemental Analysis[a]	
Carbon	68.3%
Hydrogen	0.86%
Nitrogen	1.40%
Oxygen	7.85%
Heating Value	
BTU/lb.	10,041

[a]Char from pyrolysis experiment No. 2. See Table 20 for additional elemental analysis data.

TABLE 19

ACTIVATION EXPERIMENTS WITH CHAR FROM COTTON GIN WASTE, LUBBOCK, TEXAS

Conditions	Experiment 4-4	Experiment 4-6
Wt. of Char	80	80
Temperature	800°C	800°C
Time	30 min.	30 min.
Steam rate	4g./hr./g.char	6g./hr./g.char
Yield	66.4%	40.0
Properties of Activated Char		
Density (non-ground)	15.1 lb/cu. ft.	--
Density (ball-milled)	32.9 lb/cu. ft.[a]	39.0 lb/cu. ft.[b]
Total Ash	32.4%	40.2%
Acid Insoluble Ash	13.6%	12.0%
Iodine Value (non-ground)	61.5	--
Iodine Value (ball-milled)	85.8	78.2
Modified Phenol Value (non-ground)	52.0	--
Modified Phenol Value (ball-milled)	29.3	32.2
Screen Size		
+ 100	0.88%	0.29[b]
100 x 200	2.27%	0.33
200 x 325	9.80%	2.44
325 x 400	2.29%	0.78
- 400	84.76%	96.16
Elemental Analysis		
Carbon	54.9%	--
Hydrogen	0.75%	--
Nitrogen	0.64%	--
Oxygen	8.46%	--

[a] Sample ball-milled for 30 minutes.
[b] Sample ball-milled for 60 minutes.

4.1.4.7. Elemental Analysis. (a) Samples of the char and condensible organic material were analyzed for carbon, hydrogen, nitrogen, and oxygen, and the results are given in Table 20.

(b) The sulfur content of the char and condensible organic material were determined to be 0.31% and 0.18%, respectively.

TABLE 20

ELEMENTAL ANALYSIS OF PYROLYTIC PRODUCTS
FROM COTTON GIN WASTE (LUBBOCK, TEXAS)

		Percent Element			
Experiment	Sample	Carbon	Hydrogen	Nitrogen	Oxygen
4-2-Pyrolysis	Char	68.3	0.86	1.40	7.49
4-2-Pyrolysis	Condensible Organic	64.8	7.10	3.50	11.77
4-3-Pyrolysis	Char	69.0	0.75	1.28	8.36
4-3-Pyrolysis	Condensible Organic	71.3	7.69	4.08	13.75
4-4-Activation	Carbon	54.9	0.75	0.64	8.46

4.1.4.8. Heating Values. The heating values of the gases were calculated from the percent composition, Table XII, and the heating values of the char and condensible organic material were determined by the bomb calorimetry technique. The heating values obtained are:

Gases	Experiment No. 4-1	318 BTU/cu. ft.
	Experiment No. 4-2	358 BTU/cu. ft.
Char		10,041 BTU/lb.
Condensible Organic Material		14,015 BTU/lb.

4.1.5. Char from Cotton, Inc. A sample of char from Cotton, Inc., was examined and treated with steam in one activation experiment. The data and results are given in Table 21.

TABLE 21

Data and Results - Char from Cotton, Inc.

Sample as Received

Moisture	6.0%
Total Ash	19.2%
Acid Insoluble Ash	4.4%
Iodine Value	33.8

Activation Experiment

Conditions	Experiment 5-1
Weight of Char	60 g.
Temperature	800°C
Time	30 minutes
Steam Rate	4 g./hr./g. char
Yield	51.3%
Iodine Value (ground)	68.2
Modified Phenol Value (ground)	43.8

4.2 Comparison of Data from the Waste Materials. In the tables 22-28, data on the representative samples of the waste materials, pyrolysis experiments, gas composition, non-activated char, condensible organic material, and activated char are given for comparison purposes.

TABLE 22

DATA ON REPRESENTATIVE SAMPLES OF THE FOUR WASTE MATERIALS

Property	Cotton Gin Waste[a], Tifton, Ga.	Kleen Seed Lint	Cotton Shearing Waste	Cotton Gin Waste, Lubbock, Texas
Moisture Content%	11.9	6.75[b]	4.8	10.1
Total Ash%	5.55	2.47	0.29	6.20
Acid Insoluble Ash%	2.38	0.49	None Detected	2.56
Elemental Analysis%				
Carbon	46.4	47.4	44.4	44.2
Hydrogen	5.80	6.65	6.30	5.40
Nitrogen	1.76	1.61	0.38	1.36
Oxygen	36.45	43.48	47.22	38.29
Density (lb/cu. ft.)				
Shaken	9.5	16.9	2.6	9.3
Hand Packed	14.5	26.4	5.8	14.5

[a] Lignin content, 20.9%.

[b] The lint contained 6.4% oil, and some of the loss in weight for moisture determination could be due to volatile organic material.

TABLE 23

PYROLYTIC DATA FROM THE FOUR WASTE MATERIALS

Waste Material	Experiment number[a]	Percent Pyrolytic Product[b]			
		Char	Water	Condensible Organic Material	Gas[c]
Cotton Gin Waste, Tifton, Ga.	2	31.7	19.4	10.4	38.5
	4	31.5	18.2	11.0	39.3
	5	32.2	20.2	10.9	36.9
Kleen Seed Lint	1	28.9	22.2	7.6	41.2
	2	31.2	23.4	15.4	30.0
	3	31.2	23.8	9.6	35.4
Cotton Shearing Waste	2	15.6	30.0	15.8	38.6
	3	15.0	31.4	14.7	38.9
	4	15.3	31.2	10.4	43.1
Cotton Gin Waste, Lubbock, Texas	2	32.5	16.4	8.4	42.7
	3	32.6	19.5	8.3	39.6

[a]Each of these pyrolysis experiment was carried out at 800°C for 60 minutes.

[b]Pyrolytic products are in percent by weight on a day basis of original sample.

[c]Gases obtained by difference.

TABLE 24

GAS COMPOSITION OF NON-CONDENSIBLE GAS PHASE FROM THE FOUR WASTE MATERIALS

Waste Material	Experiment Number[a]	Gas Component - % Volume					
		Air	Carbon Monoxide	Methane	Carbon Dioxide	Hydrogen	Unidentified
Cotton Gin Waste, Tifton, Ga.	1-7	3.5	20.7	14.9	29.9	26.6	4.4
	1-8	1.4	20.5	14.2	30.9	23.2	9.8
Kleen Seed Lint	2-2	0	21.3	15.3	30.9	21.3	11.9
	2-3	0	19.3	17.2	26.2	25.6	11.7
Cotton Shearing Waste	3-1	6.3	29.2	12.3	28.2	18.0	6.0
	3-2	7.1	28.8	11.4	27.3	20.5	3.9
Cotton Gin Waste, Lubbock, Texas	3-1	3.5	20.0	13.0	35.1	24.6	3.8
	3-3	1.9	20.2	13.5	34.0	24.0	6.4

[a]Each of these pyrolysis experiments were carried out at 800°C for 60 minutes.

TABLE 25

DATE ON CHAR FROM PYROLYSIS OF THE FOUR WASTE MATERIALS

Property	WASTE MATERIAL			
	Cotton Gin Waste Tifton, Ga.	Kleen Seed Lint	Cotton Shearing Waste	Cotton Gin Waste Lubbock, Texas
Density (lb/cu. ft.)	14.9	20.4	4.0	12.0
Total Ash %	19.2	7.2	2.42	20.73
Acid Insoluble Ash %	9.6	1.24	0.74	10.23
Iodine Value	12.4	14.9	39.3	14.1
Elemental Analysis				
Carbon %	63.5[a]	84.8[b]	95.8[c]	68.3[d]
Hydrogen %	1.10	1.11	1.19	0.86
Nitrogen %	1.28	2.97	0.24	1.40
Oxygen %	6.85	4.90	2.24	7.85
Sulfur %	0.25	0.22	0	0.31
Heating Value BTU/lb.	10,134	11,888	13,276	10,041

[a]Data from pyrolysis experiment No. 1-1 of cotton gin waste, Tifton, Georgia.

[b]Data from pyrolysis experiment No. 2-1 of Kleen Seed lint.

[c]Data from pyrolysis experiment No. 3-1 of cotton shearing waste.

[d]Data from pyrolysis experiment No. 4-2 of cotton gin waste, Lubbock, Texas.

TABLE 26

DATA ON CONDENSIBLE ORGANIC MATERIAL FROM PYROLYSIS OF FOUR WASTE MATERIALS

Property	WASTE MATERIAL			
	Cotton Gin Waste Tifton, Ga.	Kleen Seed Lint	Cotton Shearing Waste	Cotton Gin Waste Lubbock, Texas
	Expt. 1-1	Expt. 2-1	Expt. 3-1	Expt. 4-2
Elemental Analysis				
Carbon %	71.7	70.34	68.7	64.8
Hydrogen %	8.25	8.49	8.49	7.10
Nitrogen %	4.06	2.05	0.22	3.50
Oxygen %	15.87	12.11	16.78	11.77
Sulfur %	0.14	0.16	0.21	0.18
Heating Value BTU/lb.	Expt. 1-3	Expt. 2-2	Expt. 3-2	Expt. 4-3
	14,355	14,063	12,595	14,015

TABLE 27

DATA FROM ACTIVATION EXPERIMENTS WITH CHARS
FROM FOUR WASTE MATERIALS

Property	WASTE MATERIAL			
	Cotton Gin Waste Tifton, Ga.[a]	Kleen Seed Lint[b]	Cotton Shearing Waste[c]	Cotton Gin Waste Lubbock, Texas[d]
Yield %	58.0	85.3	89.0	66.4
Density (Ball-milled)	32.2	41.2	34.6	32.9
Total Ash %	31.7	8.99	3.24	32.4
Acid Insoluble Ash %	17.7	1.71	1.43	13.6
I.V. (Non-ground)	61.6	39.5	90.5	61.5
I.V. (Ball-milled)	76.2	67.7	98.0	85.8
M.P.V. (Non-ground)	37.6	--	18.7	52.0
M.P.V. (Ball-milled)	31.0	31.4	18.3	29.3
Screen Analysis %				
+ 100	0.98	0.75	0.35	0.88
100 x 200	16.76	6.82	0.69	2.27
200 x 325	14.24	6.21	0.97	9.80
325 x 400	1.85	1.49	0.64	2.29
- 400	66.19	84.72	97.35	84.76
Elemental Analysis				
Carbon %	62.0	86.4	91.9	54.9
Hydrogen %	0.73	0.98	1.17	0.75
Nitrogen %	0.64	1.60	0	0.64
Oxygen %	5.49	2.25	2.10	8.46

[a] Data from experiment 1-6 with cotton gin waste, Tifton, Ga.
[b] Data from experiment 2-4 with Kleen Seed lint.
[c] Data from experiment 3-5 with cotton shearing waste.
[d] Data from experiment 4-4 with cotton gin waste, Lubbock, Texas.

TABLE 28

HEATING VALUES FOR NON-CONDENSIBLE GASES
FROM FOUR WASTE MATERIALS

Sources of Gases	Heating Value, BTU/cu. ft.[a]
Cotton Gin Waste, Tifton, Georgia	
Expt. 1-7	353
Expt. 1-8	416
Kleen Seed Lint	
Expt. 2-2	439
Expt. 2-3	469
Cotton Shearing Waste	
Expt. 3-1	370
Expt. 3-2	338
Cotton Gin Waste, Lubbock, Texas	
Expt. 4-1	318
Expt. 4-3	358

[a] BTU/cu. ft. at 30 in. mercury and 60 F. saturated with water vapor.

5. Discussion

5.1. Purpose of Investigation and Pyrolysis. This investigation of certain waste materials from the cotton industry was directed toward resource recovery utilizing pyrolysis of the solid wastes. Pyrolysis is the degradation of organic material with heat. In the pyrolysis of lignocellulosic material, such as the cotton gin waste, and of cellulosic material, such as the cotton shearing waste, the pyrolytic products obtained are char, condensible organic material, water, and a gaseous phase. The char is mainly carbon and also contains the ash, which is inorganic material. The condensible organic material is usually a viscous oily material with a dark color and a pungent odor. The water usually contains some of the low molecular weight organic material which is water soluble. The non-condensible gases are usually a mixture of hydrogen, methane, carbon monoxide, and carbon dioxide, with a small amount of hydrocarbon gases such as ethane and ethylene. The investigation was directed specifically toward the preparation of activated carbons from the chars and the determination of some of the adsorptive characteristics of the carbons from the activation experiments.

5.2. Cotton Gin Waste, Tifton, Georgia. The cotton gin waste from Tifton, Georgia, is described in section 4.1.1.1, and the properties of the representative sample are given in section 4.1.1.2. This material has a relatively high total ash content as received. A visual examination of this cotton gin waste did not reveal any loosely adhering sand or dirt. After shaking a sample on the Ro-Tap, a few small grains of sand were observed. This would account for some of the ash, but it does not seem

that it would account for the relative high value. It is not possible from our examination of the material and our results to determine if the relative high ash value is due to chemically combined material or extraneous material that is intimately mixed in with the waste.

Several pyrolysis experiments were carried out with this material and the results are given in Table 1, section 4.1.1.3. The yield of the char was approximately 32%; water, 19.5%; condensible organic material, 11%; and gases, 37.4%. The non-condensible gases were analyzed by gas chromatography to determine the major components, and the results for two experiments are given in Table 2, section 4.1.1.4. The mixture has value as a gaseous fuel since the hydrogen, carbon monoxide, and methane are all combustible. In one experiment, the gaseous mixture was analyzed for the minor components. The results from this analysis showed that several compounds were present which were most likely the low molecular weight hydrocarbon gases, such as ethane, propane, and the butanes. The heating values of the gaseous mixtures were calculated from the percent composition and the heating values of the individual components. It was assumed for these calculations that the unidentified portion was ethane. The heating values calculated for the gaseous mixtures from experiments 1-7 and 1-8, were found to be 353 and 416 BTU/Cu.ft. respectively.

Representative data on the char from the pyrolysis experiments are given in Table 3, section 4.1.1.5. As expected, this char has a relative high ash content. The low iodine value shows that it is non-activated. The char retained the physical appearance and form of the original samples. Elemental analysis showed that it has a small amount of sulfur present.

Samples of the char were treated with steam in activation experiments under the conditions given in Table 4, 4.1.1.6. In the activation experment 1-6, the char had a modified phenol value of 31.0. For water grade carbon for use in water treatment plants, the carbon should have a MPV of 30 or less. In the activation experiment, 1-10, a larger flow rate of steam was used, and there was a slight increase in the MPV of the ball-milled char. The high ash content of the char from the activation experiments in not compatible with specifications of 7% or less for the ash content of water grade carbon. A sample of the char was extracted with water for one hour and lost 2.17% ash content due to water soluble material. A sample of the char, extracted with 5% sulfuric acid, showed no loss in ash content. These preliminary experiments indicate that the ash is a part of the matrix of the char and is not easily extracted from the carbon. From the activation results with the char from the cotton gin waste, Tifton, Georgia, the char would not meet the current specifications for an activated char for potable water supply treatment purposes. This does not preclude, however, its utilization as an adsorbant for treatment of industrial waste waters and in the tertiary stage for sewage treatment.

5.3 Lint from Kleen Seed Delinting Company. The lint as received was a short fibrous material with small amounts of seed hulls intermixed and had an oily feel. The weight loss on heating at 110°C for four hours could be due to moisture or a mixture of moisture and some volatile organic material due to the oil present. An oil analysis gave a value of 6.4% oil content.

Samples of the lint waste were pyrolyzed and the results are given in Table 6, 4.1.2.3. The yield of the char was approximately 30%; water, 23%; condensible organic material, 11%; and gases, 36%. The char had some material of a fibrous nauure, resembling the short fibers in the lint, some hard lumpy material, and fine carbon dust. The ash content in the char increased as expected. The iodine value of 14.9 showed that the char was non-activated. The elemental analysis showed that a small amount of sulfur is present in the char. Representative data on the char are given in Table 8, 4.1.2.5.

The non-condensible gases from two of the pyrolysis experiments were analyzed for the major components, and the results are given in Table 7, 4.1.2.4. The major components were hydrogen, carbon monoxide, methane, and carbon dioxide with about 12% of unidentified gas. The unidentified gas is most likely hydrocarbons in the ethane to butane range. The heating values of the gaseous mixtures from pyrolysis experiments 2-2 and 2-3 were cacluated and found to be 439 and 469 BTU/cu.ft. respectively.

Samples of the char were treated with steam in the activation experiments and the results are given in Table 9, 4.1.2.6. The modified phenol value of 31.4 for the char from experiment 2-4 is a little high for the char to be classified as an activated carbon for use in water treatment plants. Increasing the steam flow rate to 6 g. steam/hr./g. char produced a char with a MPV of 23.4, which meets the requirement for water grade carbon. The high density and the total ash content would reduce the value of the activated carbon from the lint waste for use in water treatment plants.

5.4. Cotton Shearing Waste. The cotton shearing waste was a dyed fibrous material and appeared to be free of any extraneous material. The moisture content was low (4.8%) and the total ash of 0.29% was very low. This material was used as received for the experimental work. A number of pyrolysis experiments were carried out at different temperatures and for different lengths of time, and the results are given in Table 11, 4.1.3.3. The yield of char at 800°C is only about 50% of the yield of char from the cotton gin waste and lint waste. The yield of water is greater than the yields from the cotton gin waste and lint waste. This is apparently due to the fact that the cotton shearing waste is essentially pure cellulose. The yield of char increased with decreasing temperature whereas the yield of condensible organic material decreased with decreasing temperature.

The gases from a number of the pyrolysis experiments at different temperatures were analysed and the results are given in Table 12, 4.1.3.4. The hydrogen decreased to 7.2% for the 500°C experiment and to 0% for the 400°C experiment. The carbon monoxide and carbon dioxide both showed an increased yield at the lower temperatures. The methane yield showed a decrease for the 400°C pyrolysis. The heating values for the gases from pryolysis experiments 4-1, 4-2, and 4-11 were calculated from the composition and found to be 370, 231, and 231 BTU/cu. ft., respectively.

The char from the cotton shearing waste retained the fibrous nature of the original material, with some carbon dust. The fibrous char has a low density. The iodine value of 39.3 showed that the char was not activated, but indicated that it had some adsorbent characteristics. The elemental analysis gave a high carbon content and no sulfur. The total ash was a little higher than expected, and acid insoluble ash, although

low, was not expected since none was found in the original material. This ash analysis was run on some material which was ball-milled for 30 minutes, and it is possible that the char could have picked up some silica (acid insoluble material) from the jar and balls used in this operation.

A number of activation experiments were carried out with the char from the cotton shearing waste in an effort to establish optimum conditions for producing an activated carbon, and the results are given in Table 14, 4.1.3.6. The modified phenol values from each of the four experiments show that an activated carbon can be prepared by steam activation of the char from the cotton shearing waste. The activated carbon from the carbon shearing waste meets the specifications for a water grade carbon for use in water purification plants with one exception which is the density of 34.6 lb./cu.ft. for the ball-milled material. It is felt, however, that this is not an insurmountable problem as a different method of grinding or pulverizing the char could yield a less dense material. The Westvaco specifications give an apparent density of 15-25 lbs./cu.ft. for activated carbon for water purification. Activated carbons with higher densities are used in the industry, but these carbons do not bring the premium prices. The fibrous nature of this activated char is an interesting property and suggests its use as a gas adsorbent. Its capability and utility for adsorbing gases would have to be determined experimentally.

From our experimental results, the overall yields of activated char is as follows: 13.8% for 800°C char, experiment 3-5; 14.7% for 400°C char, experiment 3-16; 12.2% for 700°C char, experiment 3-19; and 12.2% for 500°C char, experiment 3-21. These results indicate that the yields are not effected greatly by the original pyrolysis temperature. Additional experimental work

would be needed to establish the optimum conditions for pyrolysis and activation. Other activation methods, such as activation with air, need to be investigated.

5.5. Cotton Gin Waste, Lubbock, Texas. The cotton gin waste from Lubbock, Texas, was very similar in appearance to the cotton gin waste from Tifton, Georgia. Data on the representative sample are given in section 4.1.1.2. The sample had a relative high ash content with a total ash of 6.2% as compared with a total ash of 5.55% for the cotton gin waste from Tifton, Georgia. Four pyrolyses of this waste material were carried out, and the percent yields of the pyrolytic products were in the same range as found for the cotton gin waste from Tifton, Georgia.

The analysis of the non-condensible gases showed that the percent composition was about the same as found for the gases from the Tifton cotton gin waste except that the carbon dioxide was about 5% higher from the Lubbock waste. The heating values calculated for the gaseous mixtures from experiments 4-1 and 4-2 were found to be 318 and 358 BTU/lb., respectively.

Representative data on the char from the pyrolysis experiments are given in Table 18, section 4.1.4.5. A comparison of this data with the data on the char from the Tifton waste shows that the two chars are very similar. Both have a relatively high ash content.

Samples of the char were treated with steam in the activation experiments under the conditions given in Table 18, 4.1.4.6. In experiment 4-4, the ball-milled treated char had a MPV of 29.3, which indicates a fair amount of activation. In experiment 4-6, a higher flow rate of steam was used to determine if a greater degree of activation could be obtained. The MPV of 32.2 on this material shows that approximately the same degree of activation was

obtained as in experiment 4-4. Essentially, the same results were obtained with cotton gin char from the Tifton waste.

5.6. Char from Cotton, Inc. This char had similar characteristics of the pyrolytic chars from the samples of cotton gin waste from Tifton, Georgia, and Lubbock, Texas, except that the acid insoluble ash was lower. The char from the activation experiment had a modified phenol value of 43.8, which indicates that some activation was achieved in this one experiment. This MPV is greater than the values, approximately 30, obtained for the chars from the two cotton gin waste samples. As all of the char was used in this activation experiment, no additional experimental work could be performed with the material.

5.7. Data Comparison and Correlation of the Waste Material. Data on some properties of the representative samples of the four waste materials are given in Table 22, 4.2. An examination of these data shows that the two samples of cotton gin waste are very similar. The Kleen Seed lint contained pieces of hulls from cotton seed, and also contained 6.4% oil. The cotton shearing waste had the least amount of ash and had the least density of the four materials. The lignin content of the cotton gin waste, Tifton, Georgia, was found to be 20.9%, which compares with a value of 33.6% for peanut hulls. The cotton shearing waste is evidently cellulosic material.

Data on the yields of the pyrolytic products from the four waste materials are given in Table 23, 4.2. The two cotton gin waste materials produce about the same percentages of pyrolytic products. The Kleen Seed lint yields about the same amount of char as the cotton gin waste and slightly more water. The cotton shearing waste yields about half as much char as the other waste materials, but it yields more condensible organic material and water on the average than the other waste materials.

The gas compositions of the non-condensible gases from the four waste materials are given in Table 24, 4.2. The major components of the gaseous mixtures from each of the four waste materials are hydrogen, methane, carbon monoxide, and carbon dioxide. The yields of the identified gases are in the same general range for the four waste materials. The cotton shearing waste gave the highest yield of carbon monoxide and slightly lower yields of methane and hydrogen than the other materials. This is probably due to the fact that the cotton shearing waste is essentially all cellulosic material. For each of the waste materials, there was a fraction of the volume that

was unidentified. In one experiment, gas chromatographic analysis showed the presence of ethane, propane, and butanes, which were estimated to be about 4% of the total volumes of gases for that experiment. It seems reasonable therefore, to assume that the unidentified portion of the pyrolytic gases is mainly low molecular weight gaseous hydrocarbons such as ethane and propane.

Representative data on the chars obtained from pyrolysis of the four waste materials are given in Table 25, 4.2. The data from the two cotton gin waste materials are very similar. The cotton shearing waste had the least ash and the greatest carbon content, and these properties enhance its potential for conversion to an activated carbon. The iodine values of the pyrolytic chars show that the cotton shearing waste char has a high degree of adsorptivity relative to the other chars. The iodine value of 39.3 for the cotton shearing waste pyrolytic char does not mean it is activated, but indicates that it has potential to be activated. The heating values obtained for the chars vary from a low of 10,041 BTU/lb. to a high of 13,270 BTU/lb. for the cotton shearing waste char which is approximately 96% carbon. These heating values are in the same range as reported for a large number of bituminous coals, which vary from a low of 10,240 to 14,650 BTU/lb. (1)

Some representative data from the activation experiments on the steam treated chars are given in Table 27, 4.2. These data show that the steam treated char from the cotton shearing waste can be classified as an activated carbon if a MPV of 30 or less is used as the definition of an activated carbon for water purification plants. The other chars are on the borderline of being

---

(1) Lange's "Handbook of Chemistry", (1956 ed.) p. 817.

classified as activated carbons. The treated carbon from the cotton shearing also has a low total ash and a high carbon content, which are specifications for water grade carbon. The ash content of the chars from the two cotton gin waste materials are too high for water grade carbon.

The heating values on an air-free basis of the non-condensible gases from the four waste materials were calculated from the percent composition, and these values are given in Table 28., 4.2. In calculating these values, the assumption was made that the unidentified portion of the gases was ethane. This assumption is believed to be a valid one as, in one experiment, additional gas chromotographic analysis showed the presence of ehtane, propane, and the butanes. This assumption has a marked effect on the heating values since the heating values in BTU/cu.ft. of the hydrocarbon gases increase with increasing carbon content. For example, methane (CH_4) has a heating value of 895 BTU/cu.ft. compared with a value of 1580 BTU/cu.ft. for ethane (C_2H_6). Therefore, these heating values should be regarded as indicative of the range of values one could expect from the non-condensible gases. For comparison, natural gas which is predominantly methane has a heating value of 900-1100 BTU/cu.ft. (2).

---

(2) Perry, et al, Chemical Engineers' Handbook, (4th ed., 1963) p. 9-8.

5.8. Potential for Utilization of the Waste Materials. In the pyrolysis approach to the utilization of cellulosic and lignocellulosic materials, the pyrolytic products are non-condensible gases, a condensible organic material, water, and a char which is mainly carbon and which also contains the inorganic ash. The non-condensible gases are combustible and can be burned as a fuel. The organic material is a viscous dark colored material and usually complex in its chemical nature. The char can frequently be activated to a material of high adsorptive capacity.

5.8.1. Activated Carbon. Generally speaking, activated carbon refers to a material of a high carbon content in the powder or granular form which has a high degree of adsorptivity for gases and liquids. It should be pointed out that an activated carbon does not show necessarily a high degree of adsorptivity for all substances. An activated carbon, for example, may be an excellent water grade carbon for municipal water purification use, but it may not be a good adsorbent for use in the sugar refining industry. For this reason, activated carbons must be tested by specific methods for the specific end use. A few of the uses of activated carbons are as decolorizers for sugars, vegetable oils, and organic solutions, for the removal of odors and tastes, and as gas adsorbents for air purification, odor removal, and solvent recovery.

From the activation experiments with the chars, the carbon from the cotton shearing waste would meet the specifications as a water grade carbon for municipal water purification. Some of the most important specifications for water grade are a modified phenol value of 20 $\pm$ 2 ppm, a minimum carbon content on a dry basis of 93%, and sieve size which is controlled by the grinding operations. The specification of 93% minimum carbon means that the ash content on a dry basis should not be greater than 7%. Carbons with greater

ash content are not considered as high quality activated carbons. The apparent density of water grade carbon of high quality varies from 15 to 25 lbs/cu.ft. The density of the ball-milled activated carbon from the cotton shearing waste was 34.6 lbs/cu.ft. which is high. This property could most likely be controlled and improved by a different grinding or pulverizing technique. The activated carbon from the cotton shearing waste has a fibrous structure with some carbon dust. This fibrous structure is unique, and if the carbon shows adsorptive characteristics for gases and vapors in gas streams, it should find utilization as a gas adsorbent. There are other possible areas of utilization for an activated carbon with a fibrous structure.

The char from the Kleen Seed lint was activated to yield a material with a modified phenol value of 23.4. This material had an ash content of 8.68%, which is slightly above the specification for high quality water grade activated carbon. From experiment 2-6 in which this activated carbon was obtained, the material had a density of 60 lbs/cu.ft. after ball-milling for one hour. This density is quite high for use as a water grade carbon. It should be possible to improve the density by a different grinding or pulverizing operation. This activated carbon has some fibrous material with some dense hard particles which were formed from the pieces of hulls in the original waste.

The chars from the two different cotton gin wastes yielded products with modified phenol values of approximately 30. In the industry, a carbon must have a modified phenol value of 30 or less to be considered as an activated carbon. An increase in the steam rate in the activation experiments for these materials did not yield products with any improvements in the modified phenol values. Also, the total ash content of the steam treated chars is quite high, approximately 32%, for use as a water grade carbon. The high ash content,

however, would not necessarily preclude its utilization in other areas, such as the possible treatment of industrial waste waters.

5.8.2. Charcoal Briquettes. One important outlet for char in our economy today is in charcoal briquettes. The char from the two cotton gin wastes could be converted possibly to briquettes for the consumer market. For this utilization the ash content would not be a disadvantage. For conversion to briquettes, it would be necessary to mix the ground char with a binder and compress it in a mold of the desired shape. It is quite possible that the yield of the char for this purpose could be increased by pyrolyzing at a lower temperature so that much of the volatile organic material is retained in the char.

5.8.3. Fuel Value of the Waste Materials. In those situations where process heat is needed and a combustible solid waste material is available, the waste material can serve as a fuel. If the solid waste material is burned directly, air pollution frequently results. An example of this is the practice of paper mills in burning pine bark in the bark boilders without adequate facilities for preventing air pollution. With this possible use of the waste materials in mind, total heating values on a dry basis for each of the waste materials were calculated from the yields of the gases, condensible organic material, and char. From these values, the equivalent amount of fuel oil was calculated and an equivalent dollar value based on current fuel oil prices. The values of these materials on this basis as fuels are: cotton gin waste, Tifton, Georgia, $9 to $12/ton; Kleen Seed lint, $9 to $12/ton; cotton shearing waste, $7 to $9/ton; and cotton gin waste, Lubbock, Texas, $7 to $9/ton. These calculations are based on pyrolysis of the cellulosic and lignocellulosic materials to yield a mixture of "off-gases" which is made up of the non-condensible gases and the

organic material and which can be burned directly in a boiler. The char from the pyrolysis steps can be converted to a gaseous mixture of hydrogen and carbon monoxide by the water-gas reaction, and this mixture can be burned directly. The main advantage of using the pyrolysis and water gas route to utilize a lignocellulosic waste material as a fuel is that the gaseous fuel from the process can be burned cleanly, and thus air pollution is avoided. The ash from this process can be disposed of in a landfill.

6. Recommendations for Additional Research. Based on the data and results from this investigation of the waste materials, several recommendations are made for additional applied research, which is oriented toward the development of marketable products.

Two areas in which additional work can be done with the cotton shearing waste char are: utilization of the activated carbon as a water grade carbon for water purification and as a gas adsorbent. For utilization as a water grade activated carbon, a preliminary economic assessment of the feasibility of the cotton shearing waste as raw material for this purpose should be made. This preliminary assessment should include the availability, location, and quantity of cotton shearing waste; capital outlay for a large scale pyrolysis and activation unit; cost of operating a large scale unit; and market value of the activated carbon. If the results of this economic assessment are favorable then additional bench scale experimental work should be carried out directed toward determining the optimum conditions for converting the cotton shearing waste to activated carbon. An excellent start has been made on determining these conditions. Some additional bench scale experiments should be carried out with our larger tube furnace. In addition to pyrolysis and activation experiments with steam, some considerations should be given to activation by other techniques such as the use of air. Also, methods for grinding and pulverizing other than ball milling should be investigated. Based on these results, an experimental pilot plant program would be outlined. This program would be directed toward obtaining the necessary information for design of a commercial prototype. The other area of utilization for the activated carbon from the cotton shearing waste is as a gas adsorbent. For utilization as a gas and vapor adsorbent, an experimental program would be necessary to determine the

adsorptivity of the activated char for a number of representative gases and vapors. This would involve a quantitative determination of the amounts of gas adsorbed. If the results of the gas adsorption work are favorable, then additional bench scale work directed toward establishing optimum conditions for pyrolysis and activation should be carried out as outlined above. The bench scale work would be followed by a pilot plant program.

One of two areas in which additional applied research can be done with the chars from the cotton gin wastes is the utilization of the non-activated char for the manufacture of charcoal briquettes. This char should be a suitable material for briquettes as the heating value of the char is slightly above 10,000 BTU/lb. If the char is utilized for this purpose, then some bench scale experiments should be carried out directed toward obtaining the maximum yield of char, suitable for briquettes, and the optimum conditions for pyrolysis. The program would include the formulation and laboratory testing of the briquettes. The bench scale laboratory work would be followed by a pilot plant program which would be directed toward obtaining the necessary data and information for the design of a commercial prototype unit. The other area for utilization of the char from the cotton gin wastes would be the manufacture of granular activated carbon from the char. Granular activated carbon is used in filter beds for purification purposes. Although the cotton gin waste char has a high ash content, this should not be a disadvantage in some types of waste water treatments. It may be that the inorganic material in the char will be of a beneficial nature in some specific uses. The laboratory experimental program would include an analysis of the ash to determine the elements present and bench scale work directed toward the preparation of granular activated carbon. The preparation of granular activated carbon ususally involves the blending of the char with a suitable binder, extruding the mass into a suitable

size and shape, and drying of the extruded material. The bench scale experimental program would include testing of the granular carbon for adsorptive characteristics with some samples of industrial waste water. If the results with the bench scale work for making granular carbon from the cotton gin waste char and with the adsorptive tests, are favorable, then a pilot plant program should be carried directed toward obtaining the necessary data for design of a commercial prototype unit.

The char from the Kleen Seed lint, after the activation experiments, had an ash content which was slightly above the specifications for water grade carbon. If the ash content is due to some extraneous material which could be removed prior to the pyrolysis, then it is possible the ash content could be reduced. The activated char had a density which was higher than the density specified for water grade carbon, and it is possible that the density can be reduced by a different grinding or pulverizing procedure. Additional bench scale experimental work would be necessary to determine if the ash content and density of the activated char can be reduced to acceptable values. Other areas in which bench scale experimental work can be carried out with the char from the lint waste are to investigate converting the char to charcoal briquettes and to granular carbon. This experimental program would be essentially the same as discussed above for the cotton gin waste char. One additional research area which is unique to the Kleen Seed lint waste would be to determine if it is economically feasible to recover the oil from the lint. Our determination of the oil content by a standard mentod (see section 2.12) gave a value of 6.4% oil. The experimental program for oil recovery from the lint would involve some extraction experiments and a chemical characterization of the oil. A preliminary economic assessment of the oil recovery could be made from the data obtained. Based on this assessment, then a decision could be made as to

whether or not a pilot plant study should be carried out. The bench scale experimental program should also include some pyrolysis and activation experiments with the extracted lint.

In the recommendations above which involve the pyrolysis of the waste material, two other pyrolytic products are produced in addition to the char. These are the gases and the condensible organic material. The gases are combustible and could be used as a fuel for process heat in a commercial pyrolysis-activation process. The organic material can also be used as a fuel. There are possibly other areas of utilization for this organic material from the different wastes, and an exploratory experimental program should be undertaken with the objective of determining possible uses for this material.

In summary, the recommendations for additional research are:

(a) Utilization of the cotton shearing waste char for water grade activated carbon and as a gas adsorbent;

(b) Utilization of the cotton gin waste char for charcoal briquettes and granular activated carbon;

(c) Work to determine if the char from the Kleen Seed lint can be improved so that it would be acceptable as a water grade carbon;

(d) Utilization of the char from seed lint for charcoal briquettes and granular activated carbon;

(e) Extraction of oil from seed lint;

(f) An exploratory experimental program with the condensible organic material obtained in the pyrolysis of the waste materials.