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THE DEVELOPMENT OF SHORT, HIGH SURFACE AREA ACTIVATED CARBON FIBERS

By
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Biddeford, ME 04005

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TABLE OF CONTENTS

	Page
LIST OF FIGURES	v
LIST OF TABLES	vii
PREFACE	ix
1.0 INTRODUCTION	1
1.1 Goals of the Program	1
1.2 Approach	1
1.3 Program Plan	2
2.0 MATERIALS AND PROCESSING	2
2.1 Materials	2
2.2 Processing	3
2.3 Evaluation	6
3.0 RESULTS AND DISCUSSION	7
4.0 CONCLUSIONS	11
5.0 RECOMMENDATIONS	12
APPENDIX	13

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LIST OF FIGURES

<u>Figure</u>	<u>Page</u>
1 Carbonization Facility	5
2 Activation Facility	5
A-1 Kevlar-Carbonized @ 800°-Not Activated	14
A-2 Run 6-1-Kevlar-Carbonized @ 800°C-Activated @ 950°- with Tumblers-S.A. = 1899 M ² /g	14
A-3 Run 8-5-Kevlar-Carbonized @ 800°C-Activated @ 900°- No Tumblers-S.A. = 2202 M ² /g - High Speed	15
A-4 Run 8-16-Kevlar-Carbonized @ 800°C-Activated @ 900°- No Tumblers-S.A. = 1985 M ² /g - Slow Speed	15
A-5 Run 8-18-Kevlar-Carbonized @ 800°C-Activated @ 900°- No Tumblers-S.A. = 1960 M ² /g - Slow Speed	16
A-6 Run 8-25-Kevlar-Carbonized @ 1000°C-Activated @ 900°- No Tumblers-S.A. = 1688 M ² /g - Slow Speed	16
A-7 Kynol - Precarbonized - Not Activated	17
A-8 Kynol - Carbonized @ 800°C - Not Activated	17
A-9 Run 8-17-Kynol - Precarbonized - Activated @ 900°C - No Tumblers-S.A. = 1036 M ² /g - High Speed	18
A-10 Run 9-1-Kynol - Carbonized @ 800°C - Activated @ 900°C- No Tumblers-S.A. = 1364 M ² /g - Slow Speed	18
A-11 T300 - Precarbonized - Not Activated	19
A-12 Run 8-12 - T300 - Precarbonized - Activated @ 900°C- With Tumblers-S.A. = 662 M ² /g - High Speed	20
A-13 Run 8-11 - T300 - Precarbonized - Activated @ 900°C- No Tumblers-S.A. = 786 M ² /g - High Speed	20
A-14 Run 9-15 -Rayon - Carbonized @ 800°C - Activated @ 900°C- No Tumblers-S.A. = 1364 M ² /g - Slow Speed	21
A-15 Run 9-22 -Kevlar - Carbonized @ 800°C- Activated @ 900°C- No Tumblers - Large Lot - S.A. = 2031 M ² /g - Slow Speed	21

LIST OF TABLES

<u>Table</u>	<u>Page</u>
1 Precursor Fibers	3
2 Carbonization Furnace Cycle	4
3 Activation Furnace Cycles	7
4 Results of Activated Fiber Program	8

PREFACE

This report was prepared by Fiber Materials, Inc. (FMI) under U.S. Army Natick Research, Development and Engineering Center (Natick) Contract No. DAAK60-93-C-0015. The objectives of this SBIR Phase I contract were to develop short, activated carbon fibers possessing BET surface areas over 1500 M²/gram and to deliver 50 grams of the most promising carbon fibers to Natick. The program was conducted during the period of February 1993 to September 1993. Mr. John Herrick was the Program Manager at FMI and Ms. Marie Jean-Pierre was the Natick Project Officer.

THE DEVELOPMENT OF SHORT, HIGH SURFACE AREA, ACTIVATED CARBON FIBERS

1.0 INTRODUCTION

1.1 Goals of the Program

This program was initiated to develop a process for the preparation of short, activated carbon fibers that can be made into a flock fabric. The desired fiber length was 0.03-0.04 inch. The goals of the program were to:

1. Prepare short, activated carbon fibers with BET surface areas of over 1500 M²/gram.
2. Prepare 50 grams of the most promising carbon fibers to be delivered to the U.S. Army Natick RD&E Center.
3. Prepare activated carbon fibers with good mechanical strength.

1.2 Approach

The approach selected to prepare these activated fibers was based on the technique developed by Wennerberg and O'Grady⁽¹⁾ in which potassium hydroxide (KOH) was used at high temperatures to activate carbon powder. In a previous program at Fiber Materials, Incorporated⁽²⁾, it was found that carbonized rayon fiber activated by carbon dioxide (CO₂) gas at high temperatures resulted in reduced fiber strength. It was proposed that the KOH activation process will yield a highly active carbon fiber without excessive degradation of fiber strength. The retention of good fiber strength is important because the fibers will be used in military uniforms where fabric strength is critical. In the CO₂ process, activation occurs by the consumption of carbon by oxidation to create the porosity. In general, 85-90% of the carbon fibers had to be consumed to result in good adsorption properties, leaving a weak fiber. The KOH process generates microporosity and very high surface areas by a different mechanism. The potassium cation penetrates into the carbon structure during heat treatment to 800-900°C. After cooling and water washing to remove the potassium, the resulting porosity exhibits a cage-like geometry with very small dimensions. BET surface areas of over 3000 M²/gram are

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1. Wennerberg, Arnold, and O'Grady, Thomas, U.S. Patent No. 4,082,694, Active Carbon Process and Composites (1978).
 2. Herrick, J. (1993 October) Activated Carbon Fiber Felt for Improved Chemical Protective Fabric, Natick Technical Report MATICK/TR-93/0201, U.S. Army Natick Research, Development and Engineering Center, Natick, MA 01760.

common with very high adsorption properties. The yield is generally over 50%, so that 50% or less of the fiber is consumed during activation, compared to 90% for the CO₂ process. Consequently, the fiber strength should be much higher for fibers activated with KOH rather than CO₂.

1.3 Program Plan

The program was organized to evaluate two fundamental parameters; the precursor fiber and the KOH activation process. A minimum of four (4) different precursor fibers were to be processed by KOH activation into short, activated carbon fibers. Then, the KOH process was to be varied in efforts to increase BET surface area and reduce fiber damage. The activation process equipment to be used was the same as used to activate carbon powder. It was proposed that the short fibers (e.g., 0.04 inch) would process in the rotating tube furnace in a manner similar to carbon powder.

2.0 MATERIALS AND PROCESSING

2.1 Materials

The precursor fibers used in the program are listed in Table 1. Both carbonized and non-carbonized (organic) fibers were received for evaluation. Several fibers were available in 1 mm lengths, which is very close to the desired fiber length. Three fibers were available only in ¼-inch lengths (about 3 mm) and the carbonized Kynol was available only as ½-inch long fibers. These longer fibers could be cut to shorter lengths but the lengths would vary considerably. It was believed that the longer fibers could be activated by the KOH method. If any of these longer fibers demonstrated outstanding properties, shorter fibers could be obtained by chopping continuous tow to 1 mm length.

The other material used in the process is KOH. A technical grade in flake form was utilized for most activation processes. In one run, the KOH flake was ground to a coarse powder to see if particle size affected the resulting activated fiber properties.

TABLE 1. PRECURSOR FIBERS

NAME	SOURCE	TYPE	CARBONIZED	LENGTH
KEVLAR	DUPONT	ARAMID	NO	1 mm
KYNOL	AMERICAN KYNOL, INC.	PHENOLIC	NO	3 mm
KYNOL	AMERICAN KYNOL, INC.	PHENOLIC	YES	¼ inch
ACRYLIC	FLOCK FIBERS INC.	PAN*	NO	1 mm
RAYON	FLOCK FIBERS INC.	RAYON	NO	1 mm
T-300	AMOCO	PAN*	YES	½ inch
P-25	AMOCO	PITCH	YES	½ inch
PANEX 33-40	ZOLTEK	PAN*	YES	1 mm

* Polyacrylonitrile

2.2 Processing

2.2.1 Equipment

The organic precursor fibers were first carbonized to 800° or 1000° in an inert gas retort furnace sketched in Figure 1. The organic fibers were loaded in a container inside the retort furnace and the lid made gas tight by a sand seal. Nitrogen (N₂) flow was regulated through the retort to exclude oxygen. The temperature was raised to the final temperature by the cycle listed in Table 2. The furnace was cooled to near room temperature before removing the carbonized fibers.

TABLE 2. CARBONIZATION FURNACE CYCLE

Temperature (°C)	Rate (°C/Hour)	Operation
Room	-	Furnace on. N ₂ gas on.
Room - 450	50	Furnace heating.
450	0	Hold for ½ hour.
450-800	50	Furnace heating.
800	0	Hold for 1 hour. Furnace off. Cool under N ₂ .

The carbonized fibers were activated by the equipment sketched in Figure 2. A three-zone Lindberg furnace was used to heat a 4-inch diameter by 31½-inch long Inconel tube. The Inconel tube was tapered at the exit end to facilitate the removal of reaction products. Floor-mounted bearings provided support for the tube. A Rinco rotary valve was installed at the entrance to the tube. The valve allowed N₂ gas and/or water to be added to the tube while still being rotated by the variable speed drive motor. This feature was necessary so that the reaction products could be water quenched under N₂. Without the facility, residual potassium remaining in the tube after processing might present a safety problem. During the activation process, the N₂ was bled into the tube at a constant rate and the water tube closed. After activation and cooling, water was poured into the tube while keeping the N₂ flowing and rotating the tube for mixing. Normally, the tube would warm up a few degrees due to reaction and then cool because of the excess water. The reaction products in water were then poured from the exit end of the tube furnace.

2.2.2 Activation Process

The carbonized fibers were blended in a Hobart mixer with KOH in a ratio of 1 part carbon to 3 parts KOH. A typical charge to the furnace was 50 grams of fiber and 150 grams of KOH. Although flake KOH was used in most runs, the KOH was ground to a powder in one run to evaluate the effect of KOH size on fiber properties. In several mixtures, the KOH flake did not mix well with the fibers and were added to the furnace separately. Several mixtures were made by blending the KOH and fibers by hand to reduce potential fiber damage by the KOH particles.

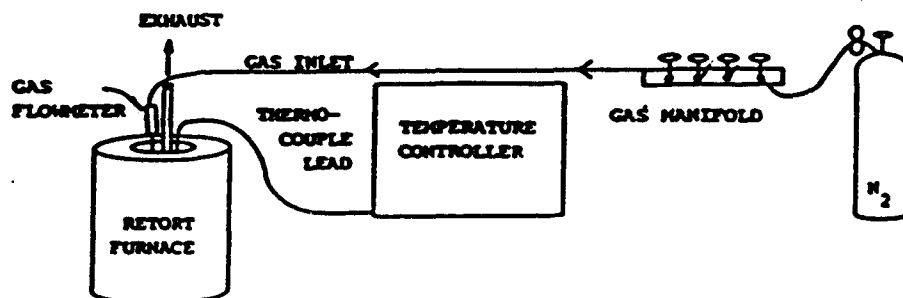


Figure 1. Carbonization Facility

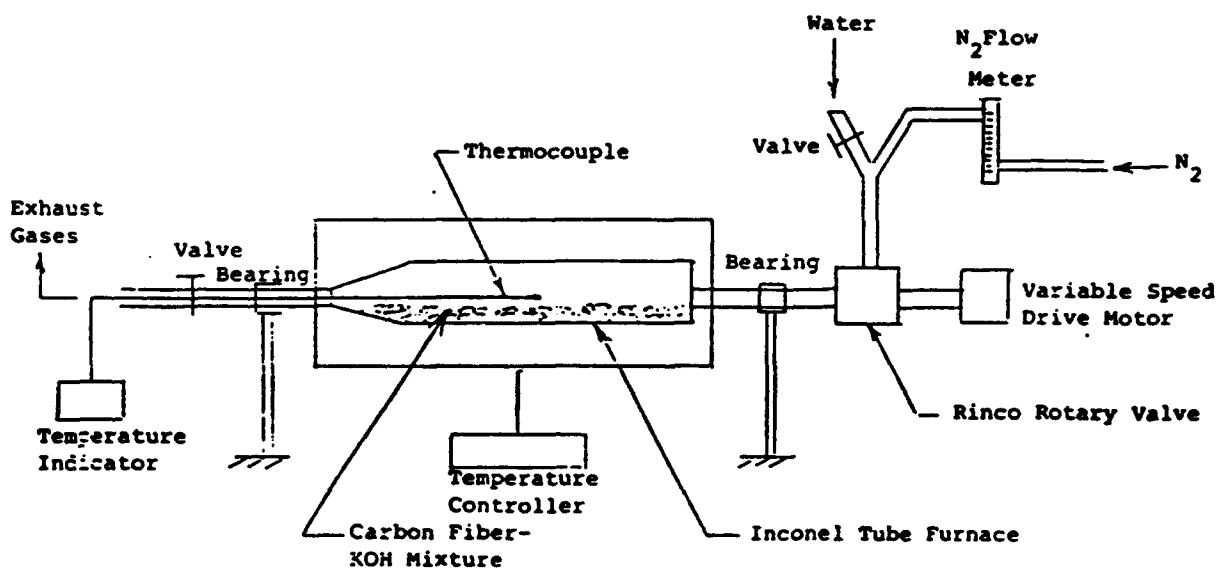


Figure 2. Activation Facility

The KOH-fiber mixture was poured into the entrance tube with the exit valve closed. In some runs, steel balls or short rods (tumblers) were in the furnace to aid in mixing the material while the tube was rotated. However, most activation runs were made without steel tumblers in the furnace to reduce fiber damage.

After placing the tube on the support bearings in the Linberg furnace, the entrance tube was connected to the drive motor through the Rinco valve. The N_2 gas was turned on to a flow of 5 CFM and the tube rotated at about 3.5 or 18 RPM. The water valve was shut off.

A thermocouple was inserted inside the reaction tube from the exhaust end to indicate the temperature at the center of the tube. The Lindberg furnace temperature controllers were set over the three zones to achieve uniform temperature over the length of the tube. Several trial runs were made at the start of the program to establish the desired temperature cycle. The temperature cycles used for most runs are listed in Table 3. Maximum temperatures of 900°C or 950°C for 1 hour were selected for all activation runs. The heat was turned off and the tube cooled to room temperature under flowing N_2 . About one quart of water was poured into the rotating furnace under N_2 to quench and terminate the reaction. After carefully removing the thermocouple, the contents of the tube were emptied into a container from the exhaust end and flushed with water until relatively clear.

The reaction products were filtered through a No. 41 filter paper by a Buchner funnel. The fibers were washed with distilled water three times at 90°C for one hour. The fibers were washed in cold, distilled water at least twice and the pH adjusted with dilute hydrochloric acid to near neutral. After the final filtering, the fibers were dried in a vacuum oven for at least four hours.

2.3 Evaluation

The primary evaluation technique was surface area as measured by the Micromeritics Flow Sorb II. In this equipment at FMI, standard BET surface areas in M^2 /gram were obtained by the desorption of "theoretical" monolayers of N_2 from the microporosity in the activated carbon fibers. The BET surface areas are really effective surface areas because true monolayer adsorption does not occur. However, this test is a convenient evaluation of the relative adsorption of activated carbon fibers.

TABLE 3. ACTIVATION FURNACE CYCLES

Temperature (°C)	Rate (°C/Hour)	Operation
Room	-	Furnace on. N ₂ gas on.
Room - 300	550	Furnace heating
300	0	Hold for ¼ hour.
300-400	400	Furnace heating
400	0	Hold for ¼ hour
400-950	360	Furnace heating
950	0	Hold for 1 hour Furnace Off. Cool under N ₂
Alternate Cycle		
Room	-	Furnace On. N ₂ gas on
Room-900	290	Furnace heating
900	0	Hold for 1 hour. Furnace off. Cool under N ₂

The other important fiber property is strength. This property is difficult to obtain for much longer fibers and even more difficult for the short, 1-3 mm fibers. A qualitative evaluation technique was selected in which the activated fibers were inspected under 50X magnification with a metallograph and photomicrographs obtained as a function of processing conditions. Typical photomicrographs of selected fibers are included in the Appendix.

3.0 RESULTS AND DISCUSSION

The results of the program are summarized in Table 4. Identified are the precursor fiber, carbonization, fiber-KOH mixing conditions, presence of steel tumblers, activation temperature and rotation speed of tube furnace in revolutions per minute (RPM). Also listed are the percentage yields of the activation process and the resulting BET surface areas. Photomicrographs of several fibers before and after activation are included in the Appendix.

TABLE 4. RESULTS OF ACTIVATED FIBER PROGRAM

RUN	FIBER	CARB.T	KOH	MIX	TUMBLERS	PROCESS			BET S.A
						RPM	TEMP.	% YIELD	
6-1	Kevlar	800°C	3 x Flake	Robert	Yes	18	950°C	--	1899
6-29	Kevlar	800°C	3 x Flake	Robert	Yes	18	950°C	--	--
7-1	PC33-40	800°C	3 x Flake	Robert	Yes	18	950°C	67	206
7-19	PC33-40	-	3 x Flake	In Furnace	Yes	18	950°C	55	78
7-21	PC33-40	-	3 x Flake	In Furnace	None	18	950°C	64	68
7-22	PC33-40	-	3 x Flake	In Furnace	None	18	900°C	41	86
7-27	PC33-40	-	3 x Flake	In Furnace	Yes	18	900°C	73	84
8-5	Kevlar	800°C	3 x Flake	In Furnace	None	18	900°C	19	2202
8-9	P-25	-	3 x Flake	In Furnace	None	18	900°C	29	26
8-10	P-25	-	3 x Flake	In Furnace	Yes	18	900°C	52	9
8-11	T-300	-	3 x Flake	In Furnace	None	18	900°C	17	786
8-12	T-300	-	3 x Flake	In Furnace	Yes	18	900°C	36	662
8-16	Kevlar	800°C	3 x Flake	By Hand	None	3.5	900°C	8	1985
8-17	Kynol	CF1605	3 x Flake	In Furnace	None	18	900°C	15	1036
8-18	Kevlar	800°C	3 x Ground	By Hand	None	3.5	900°C	12	1960
8-25	Kevlar	1000°C	3 x Flake	By Hand	None	3.5	900°C	21	1688
9-1	Kynol	800°C	3 x Flake	By Hand	None	3.5	900°C	26	1364
9-15	Rayon	800°C	3 x Flake	By Hand	None	3.5	900°C	--	1664
9-22	Kevlar	800°C	3 x Flake	By Hand	None	3.5	900°C	43	2031

The yield of the activation process varies considerably according to the precursor fiber, ranging from 8% for a carbonized Kevlar run to 73% for a carbonized PAN (PC33-40) run. The PC33-40 and P-25 (pitch precursor) carbon fibers had the highest yields, but also the lowest surface areas. These fibers were obtained as carbon fibers and the maximum carbonization temperatures were well above 1000°C. The yield of T-300 was much less than PC33-40 and the surface area much higher. The reason for this difference is probably related to carbonization temperature as both fibers are based on PAN fibers. The PC33-40 fibers were carbonized at about 1600°C according to the supplier, while the T-300 fibers were probably carbonized nearer 1200°C. The organic fibers, Kevlar, Kynol and Rayon, that were carbonized at FMI to 800°C or 1000°C, all exhibited much lower yields and higher surface areas. Kevlar yields ranged from 8-43% but possessed the highest BET surface areas, up to 2200 M²/gram. The wide range in yields appears to be affected by the quantity of material processed as the 43% yield resulted from the largest lot of fiber. It can be concluded that decreased carbonization temperature results in decreased activation yield, but increased surface area. The BET surface areas of the activated fibers ranged from a low of 9 M²/gram for pitch fibers (P-25) to a high of 2202 M²/gram for one of the Kevlar runs.

All the Kevlar carbonized at 800°C and activated yielded surface areas over 1900 M²/gram. One lot of Kevlar was carbonized to 1000°C and the resulting surface area after activation was only 1688 M²/gram. This confirms the conclusion stated above that higher carbonization temperatures yield lower surface areas.

Phenolic (Kynol) and rayon fibers were also activated after being carbonized to 800°C and exhibited surface areas of 1364 M²/gram and 1664 M²/gram, respectively. Kynol carbonized by the supplier (CF1605) yielded a lower surface area after activation, 1036 M²/gram. It is probable that CF1605 was carbonized above 800°C. These data indicate that Kevlar yields higher surface areas than phenolic and rayon fibers after equivalent carbonization and activation processes.

There were several other process variations explored during the program which are listed in Table 4. These variables included the type of KOH, fiber-KOH mixing process, presence of steel tumblers, rotational speed of the tube furnace and maximum activation temperature. The last variable, process temperatures of 900°C and 950°C, was included to evaluate the effect of temperature on surface area. The other variables were designed to investigate their effect more on fiber damage rather than surface area.

In one Run, 8-18, the KOH flake was ground to a coarse powder and then mixed with the fiber. This was done to see if ground KOH would yield less damage to the activated fibers. Inspection of the photomicrographs in the Appendix does not indicate that ground KOH damaged the fibers any less than flake KOH. The photomicrographs of Runs 8-5, 8-16 and 8-18 show that the fibers appear to be approximately equal in length.

The procedure for mixing the KOH and carbonized fibers before activation was varied throughout the program. The three variations were: 1) mixing in a Hobart mixer, 2) mixing by hand, and 3) adding the KOH and fibers separately to the reaction furnace. The mixing procedure for each Run is identified in Table 4. Inspection of the activated fiber lengths in the photomicrographs does not reveal any visible effect of mixing. It is probable that adequate mixing of the fibers and KOH occurs during the initial rotation of the reaction furnace. The mixing procedure can be selected more on the basis of convenience rather than its effect on fiber properties.

The effect of the steel tumblers, as $\frac{1}{4}$ -inch diameter balls or $\frac{1}{4}$ -inch diameter x $\frac{1}{4}$ -inch long rods, was more clearly seen on fiber lengths. In Run 6-1, both balls and rods are present and the photomicrograph in the Appendix indicates that significant fiber damage occurred. Run 8-5 was made with the same carbonized Kevlar and same activation process but without any tumblers in the reaction furnace. The photomicrograph of Run 8-5 indicates that reduced fiber damage may have occurred without any reduction in BET surface area. In fact, the surface area increased slightly without tumblers. Some fiber damage still occurred without tumblers although many fibers appeared to retain their original length. A screening procedure should remove most of these shorter fibers.

The reaction tube was operated at two speeds, 18 RPM and 3.5 RPM, to test for the effect of rotational speed on fiber damage. Comparing photomicrographs of fibers made in Run 8-5 to fiber made in Runs 8-16 and 8-18 indicates that slower rotational speed appeared to reduce fiber damage. The surface areas were slightly lower for the low RPM Runs but the values are probably within experimental error.

The last process variation was the maximum temperature of activation, 900°C and 950°C. Although most runs were made at a maximum temperature of 900°C, the limited data at 950°C indicate no effect on the BET surface area of the activated fibers.

The organic fibers investigated in this program as precursors for activated carbon fibers were Kevlar, PAN, pitch, phenolic (Kynol) and rayon. The Kevlar fibers have been discussed above. The PAN and pitch based fibers did not result in activated carbon fibers with high BET surface areas. Some PAN flock fibers were carbonized but fused together, preventing the activation process. The Kynol and rayon precursor fibers yielded activated carbon

fibers with BET surface areas near 1500 M²/gram, the minimum value acceptable by the contract. The Kynol surface area was 1364 M²/gram (Run 9-1) and the rayon yielded 1664 M²/gram (Run 9-15). The non-carbonized Kynol fibers were longer than all the other fibers ($\frac{1}{2}$ inch). After carbonization at 800°C and activation at 900°C, this fiber retained good length with almost no visible damage as seen in the photomicrograph for Run 9-1. These fibers appear to be somewhat larger in diameter than the other fibers. It is possible that Kynol may yield a good combination of high surface area and fiber strength if processed by optimized conditions. The other precursor fiber, rayon, yielded higher surface area but appeared to suffer more damage during processing as indicated by the photomicrographs.

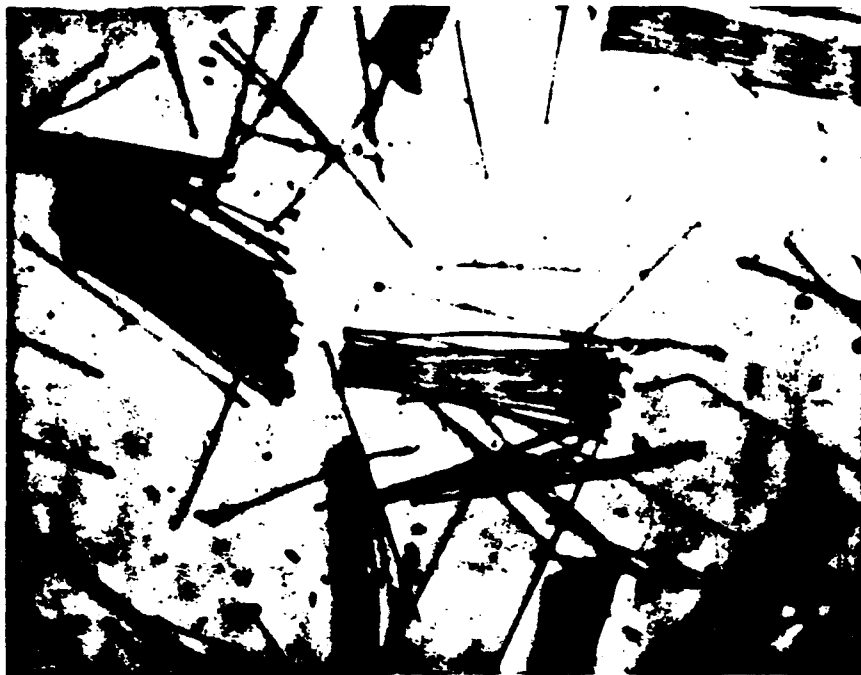
4.0 CONCLUSIONS

1. Short, activated carbon fibers can be made with BET surface areas to at least 2000 M²/gram.
2. For maximum surface area, the temperature of activation must be higher than the temperature of carbonization.
3. The type of precursor fiber affected the surface area of the activated carbon fibers. In order of decreasing surface area, the fibers are Kevlar, rayon, phenolic, PAN (precarbonized) and pitch (precarbonized).
4. Variations in processing did not appear to affect the surface area.
5. The fiber yield of activation ranged from 9-73%. In general, fibers with lower surface areas had higher yields although the quantity of fiber processed affected the yield. The large lot of Kevlar, 57 grams, had a yield of 43% while smaller lots had yields of 10-20%.
6. The presence of steel tumblers in the activation furnace damaged the fibers.
7. Activating fibers at slow speed and without tumblers in the furnace produced the best fibers with minimum damage.
8. The fiber that yielded the best combinations of high BET surface area and fiber integrity after activation was Kevlar carbonized at 800°C. The second best fiber was phenolic (Kynol). The other fibers had lower surface areas and exhibited more fiber damage.

5.0 RECOMMENDATIONS

This Phase I program demonstrated that short, activated carbon fibers can be produced that possess high BET surface area and good fiber strength. Fiber Materials, Incorporated recommends that a Phase II program be initiated to complete the development of these fibers for use in chemical protective uniforms. Kevlar and phenolic precursor fibers should be the primary fibers for continued development. A scaled-up activation facility should be designed and built to prepare larger quantities of fiber. The carbonization and activation processes should be optimized to yield fibers with maximum adsorptive and strength characteristics. Special attention must be given to obtaining short, activated fibers with uniform length. Finally, the processes developed must be amenable to low-cost production so that the activated fibers will be affordable.

APPENDIX



A-1 Kevlar-Carbonized @ 800° - Not Activated



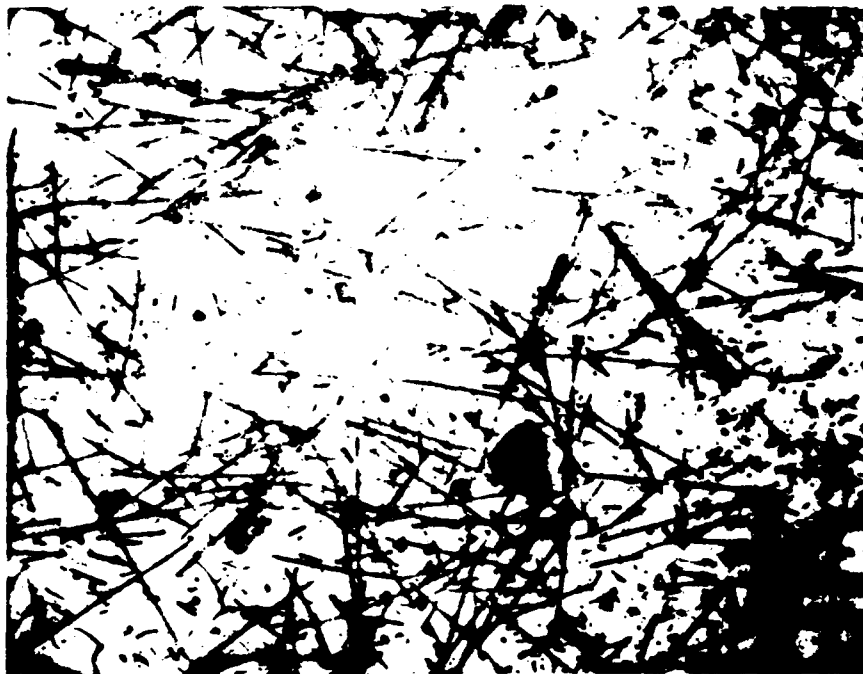
A-2 Run 6-1 Kevlar-Carbonized @ 800°C-Activated @ 950°-
with Tumblers-S.A.= 1899 M²-g



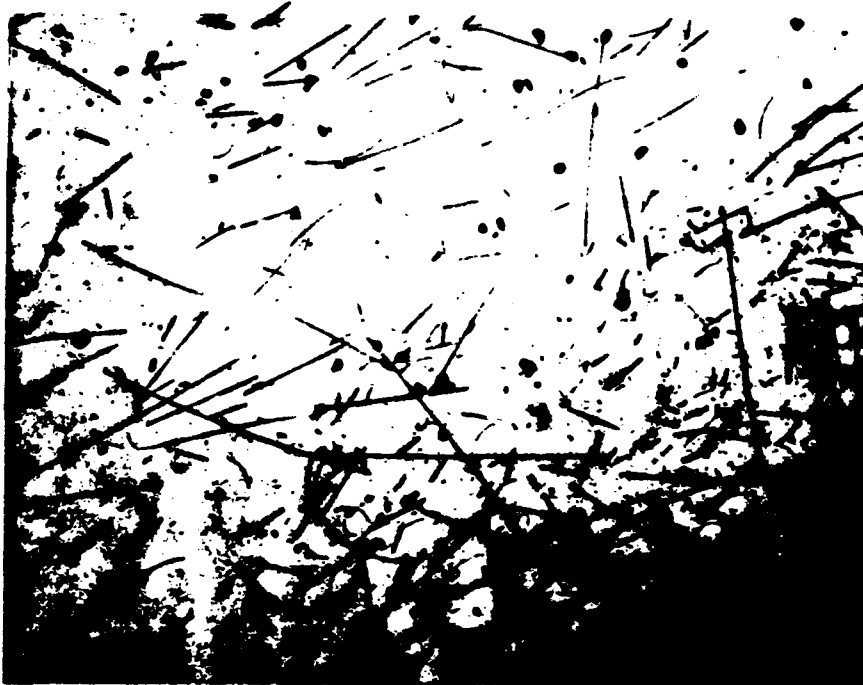
A-3 Run 8-5 Kevlar-Carbonized @ 800°C-Activated @ 900° -
No Tumblers-S.A. = 2202 M²/g High Speed



A-4 Run 8-16 Kevlar-Carbonized @ 800°C-Activated @ 900° -
No Tumblers-S.A. = 1985 M²/g Slow Speed



A-5 Run 8-18 Kevlar-Carbonized @ 800°C-Activated @ 900°-
No Tumblers-S.A. = 1960 M²/g Slow Speed



A-6 Run 8-25 Kevlar-Carbonized @ 1000°C-Activated @ 900°-
No Tumblers-S.A. = 1688 M²/g Slow Speed



A-7 Kynol - Precarbonized - Not Activated



A-8 Kynol - Carbonized @ 800°C - Not Activated



A-9 Run 8-17-Kynol - Precarbonized - Activated @ 900°C -
No Tumblers-S.A. = 1036 M²/g - High Speed



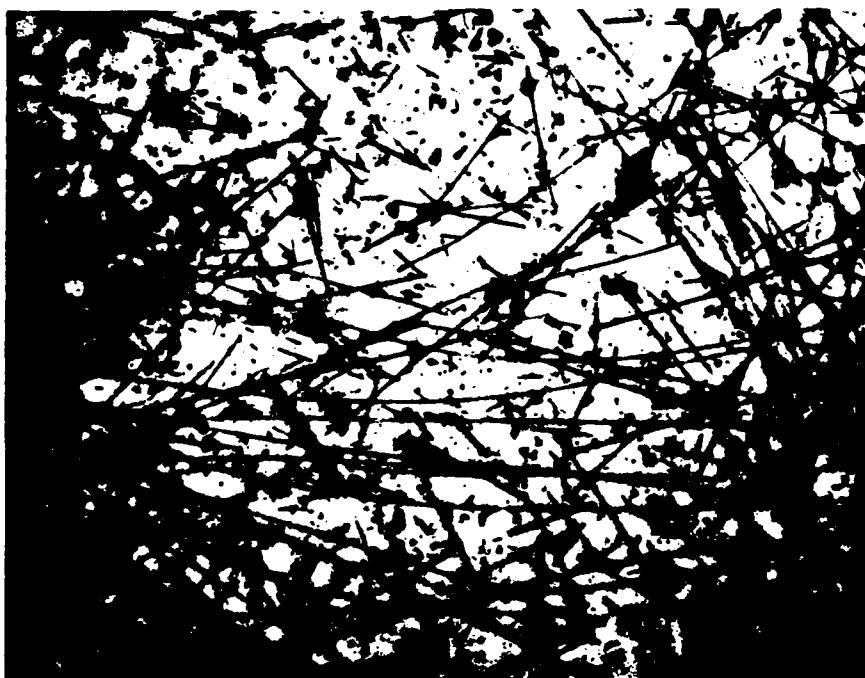
A-10 Run 9-1-Kynol - Carbonized @ 800°C - Activated @ 900°C -
No Tumblers-S.A. = 1364 M²/g - Slow Speed



A-11 T300 - Precarbonized - Not Activated



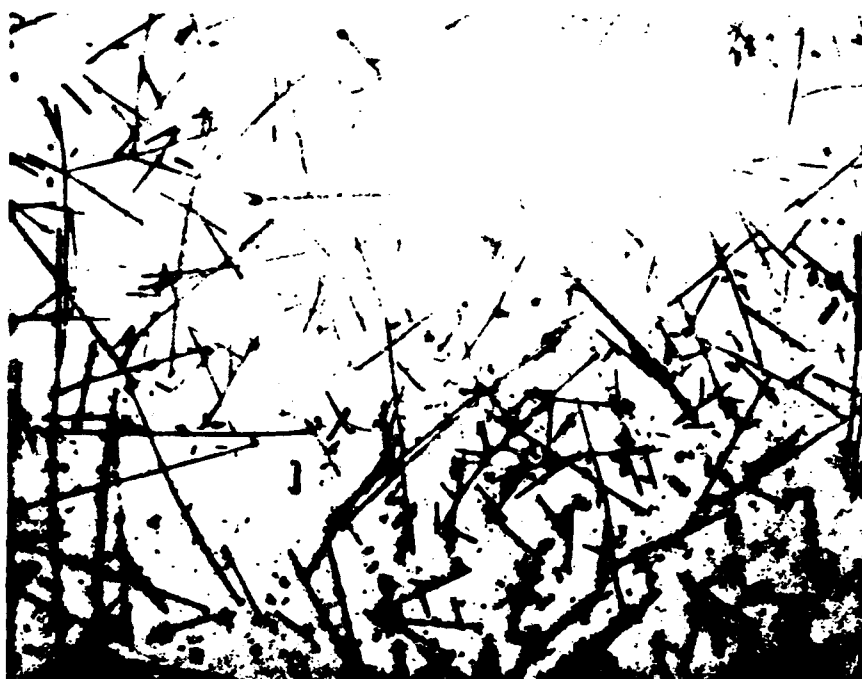
A-12 Run 8-12 - T300 - Precarbonized - Activated @ 900°C-
With Tumblers-S.A. = 662 M²/g - High Speed



A-13 Run 8-11 - T300 - Precarbonized - Activated @ 900°C-
No Tumblers-S.A. = 786 M²/g - High Speed



A-14 Run 9-15 -Rayon - Carbonized @ 800°C - Activated @ 900°C-
No Tumblers-S.A. = 1664 M²/g - Slow Speed



A-15 Run 9-22 -Kevlar - Carbonized @ 800°C- Activated @ 900°C-
No Tumblers - Large Lot - S.A. = 2031 M²/g - Slow Speed