NRL REPORT 3611

FILTER PAPER STUDIES VII
GENERAL MILL PROCEDURE AND SUMMARY

Harold W. Knudson and Robert D. Parsons

Hollingsworth and Vose Company
East Walpole, Massachusetts

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Approved by:

Dr. E. A. Ramskill, Head, Protective Chemistry Branch
Dr. P. Borgstrom, Superintendent, Chemistry Division

NAVAL RESEARCH LABORATORY

CAPTAIN F. R. FURTH, USN, DIRECTOR
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PREFACE

This Naval Research Laboratory report consists of the following two Research and Development Reports and one Summary Report written by H. W. Knudson and R. D. Parsons of the Hollingsworth and Vose Company, East Walpole, Mass., on Navy Contract N7-onr-430:

"Research and Mill Trial on the Development of a Domestic Substitute for Esparto Fiber in the Navy H-60 Filter Paper," Seventh Quarterly Period of Contract N7-onr-430, referred to as the N-13 Trial.

"Research and Mill Trial on the Development of a Domestic Substitute for Esparto Fiber in the Navy H-60 Filter Paper," Eighth Quarterly Period of Contract N7-onr-430, referred to as the N-14 Trial.

"Summary of Research and Mill Trials on the Development of a Domestic Substitute for Esparto Fiber in the Navy H-60 Filter Paper," report on contract N7-onr-430.

This report concludes the work on the second year of the contract. Filter paper studies are being continued by the Hollingsworth and Vose Company under a renewal of the contract. Additional reports will be published when received.

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N-13 TRIAL

N-14 TRIAL

ABSTRACT

This is an interim report describing in detail two mill runs and the associated laboratory research work on mill procedures for the manufacture of causticized viscose-asbestos filter paper. It is shown that the Hydrapulper can be successfully used in the processing of viscose fibers. A production time schedule is shown to be feasible. A summary of work done under contract N7-onr-430 to date is included. The need for additional studies on asbestos dispersion and distribution in the sheet is indicated.

PROBLEM STATUS

This is an interim report; work on the problem continues.

AUTHORIZATION

NRL Problem C04-28R (NS 181-011)

RESEARCH AND MILL TRIAL ON THE DEVELOPMENT OF A DOMESTIC SUBSTITUTE FOR ESPARTO FIBER IN THE NAVY TYPE H-60 FILTER PAPER

(N-13)

INTRODUCTION

This report is a summary of the laboratory work done under contract N7-onr-430 from January 1, 1949 to March 31, 1949 and the N-13 mill trial. A statement of the general objectives and an outline of previous work accomplished are contained in past reports covering mill trials and laboratory work from April 1946 to December 1948.*

The mill trial reported here is designated as the N-13 trial, H & V Lot No. 887, dated April 21, 1949. As stated previously, the Navy Hydrapulper installation was delayed. Consequently, the mill trials required under the present contract had to be run in close succession.

Considerable progress has been made in the development of viscose rayon as a domestic substitute for esparto in the Navy Type H-60 filter material. Commercially cut viscose rayon flock has been used made from tow furnished by either American Viscose Company or the duPont Company. Although both bright and dull (delustered) types have been used, only the bright viscose rayon (1.5-denier) has been used recently (See N-11 report for reasons).

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*Knudson, H. W., "Filter Paper Studies I. Effect of Replacing Esparto with Yucca Fiber," NRL Report C-3172, September 1947. (Confidential)

Knudson, H. W. and Pasternak, S. J., "Filter Paper Studies II. Effect of Replacing Esparto with Wood Pulp Fiber," NRL Report C-3225, January 1948. (Confidential)

Knudson, H. W. and Pasternak, S. J., "Filter Paper Studies III. Effect of pH and Added Electrolytes," NRL Report C-3226, January 1948. (Confidential)

Knudson, H. W. and Pasternak, S. J., "Filter Paper Studies IV. Effect of Replacing Esparto with Viscose Rayon Fiber," NRL Report C-3299, June 10, 1948. (Confidential)

Knudson, H. W., "Filter Paper Studies V. Effect of Viscose Fiber Processing," NRL Report C-3394, December 7, 1948. (Confidential)

Knudson, H. W. and Parsons, R. D., "Filter Paper Studies VI. Effect of Viscose and Asbestos Types," NRL Report 3610, January 16, 1950. (Confidential)

Due to the fact that it has been possible to produce uniformly good results in the laboratory using this type of furnish and due also to the fact that the Navy Hydrapulper installation was only recently completed, it was decided to concentrate as much work as possible during the time remaining under this contract on problems associated directly with commercial production of this new type of paper.

The use of Blue African asbestos was continued for the reasons outlined in the N-11 report, since the Vortrap units continued to do a good job in removing the foreign material from the asbestos.

From the N-12 trial it has been shown that the most rapid means in causticizing thus far has been through the utilization of the Hydrapulper. This fact is proven again in the Mill Trial N-13.

LABORATORY WORK

Since the first use of the Hydrapulper in Mill Trial N-12, and the data obtained therefrom, correlation between the Waring Blendor and pulper became more predictable. The results from the first Hydrapulper mill trial checked very closely with work done in the laboratory. All the variables now recognized in the causticizing procedure of viscose rayon were kept constant except the time of agitation. Of course, this time of agitation in the Blendor is vastly different for identical results in the Hydrapulper.

The variables which were kept constant in the laboratory and in the mill were as follows:

a) Temperature - 75°F
b) Caustic concentration - 7.50%
c) Consistency during causticizing - 4.0%
d) Variac reading (laboratory) - 80 volts

In all causticizings performed, a 1.5-denier bright viscose rayon was used (75% of 1/8″ fibers and 25% of 1/4″ fibers). The asbestos was beaten and defibered in a solution of Daxad No. 11 (2% of fiber weight), a dispersing agent. As stated previously, the use of this dispersing agent aids in defibering the asbestos. It is believed that this results in better asbestos distribution in the finished sheet, but the evidence is inconclusive as yet.

A series of handsheets was made from bright viscose rayon that was causticized under the above conditions. Increasing time intervals were chosen in order to determine just what effect time of agitation had on efficiency and tensile strength.

Table I shows the smoke penetration, air resistance, and physical characteristics of paper made from bright viscose rayon at 75° F in 7.5% sodium hydroxide in the Blendor for a varying length of time. A variac setting of 80 was used, as this speed correlated favorably with results obtained from Mill Trial N-12.

TABLE I

Performance and Physical Characteristics vs. Time of Causticizing

Sample	Time of Causticizing (sec)	% BSA	Resistance* (mm H_2O)	Penetration* (%)	Efficiency+ (%)	Caliper (in.)	Tensile (lb/in. width)
1	15	8.0	103	.011	3.86	.045	3.8
2	30	7.0	86	.050	3.89	.041	5.2
3	45	7.0	87	.027	4.07	.040	6.6
4	60	7.0	98	.021	3.82	.043	10.2
5	75	7.0	94	.027	3.82	.038	5.5
6	90	7.0	89	.046	3.76	.039	8.7
7	105	7.0	99	.028	3.69	.045	6.3
8	120	6.0	99	.024	3.74	.044	8.9

*Measured by NRL Smoke Penetration Meter E2, E2R1, or E3. For operating instructions see NRL Instruction Manual A 825A, "Instructions for Canister Tests, Part II, Filters Section A, Smoke Penetration," 13 July 1945.

+Percent efficiency = $\frac{-\log P}{R}$ x 100, where P is the DOP penetration expressed in decimals rather than percent, and R is the resistance across the sample in mm of water under the standard conditions of test.

It can be seen that the efficiency decreases slightly as the time of causticizing increases. The amount of asbestos needed also decreases with time. Each time runs similar to the above were made in the laboratory the filtration efficiency never decreased by a large amount.

From this data it seems that with an increase in time the tensile also increased. Although the results were somewhat erratic on this run, there is every reason to believe that up to a certain point in time of causticizing there will be a uniform increase in strength.

Because the efficiency is gradually lowered as the tensile strength was raised, an optimum causticizing condition had to be decided upon. As already reported from Mill Trial N-12, two causticizings were performed at 2 and 5 minutes respectively. From the information obtained at that trial and also from laboratory studies it was thought that the time could be lengthened to 10 minutes without having a detrimental effect on filtration characteristics while building up the tensile.

It was stated in the N-12 report that, because of too much dilution in the chest, no appreciable amount of white water (asbestos fines) could be returned to the wet end. This noncirculation of white water was believed at that time to decrease the efficiency somewhat. However, since that trial some tests were made in the laboratory on this white water problem. At this time these studies would tend to indicate a plugging up of the sheet with an increasing white water return. Further investigation is needed along this line. It was decided to experiment as much as possible with the white water return in the present trial N-13.

Viscose rayon stock saved from the Mill Trial N-12 was made into handsheets (using laboratory prepared asbestos) in order to investigate the relaxation and flow rate vs. penetration properties further. It was stated in the last report that these tests on the machine-made paper were obtained rather hurriedly. From the results obtained in the laboratory these handsheets showed practically no relaxation on standing and the decrease in penetration with the flow rate was entirely normal.

THE MILL RUN

With the information obtained from these laboratory studies and previous mill trials concerning the causticizing problem of bright viscose rayon in the production of filter paper, it was decided to make the following mill run using the Navy Hydrapulper.

Furnish

The base furnish was to consist of 100% causticized rayon flock, as follows:

75 parts 1.5-denier 1/8″ bright
25 parts 1.5-denier 1/4″ bright

To this would be added approximately 10% Blue South African asbestos on the weight of the base furnish. This asbestos was to be beaten in a Daxad No. 11 solution (2% of fiber weight).

Preparation of Stock for Mill Trial

On the basis of laboratory studies in the blendor it was decided to causticize two 500-pound batches in the Hydrapulper. The first batch of 500 pounds was to be causticized 7 minutes at 7.50% caustic concentration and 75° F. The causticizing procedure for the first batch operation proceeded in the following manner.

A quantity of water just under the total amount required was added to the Hydrapulper, and a predetermined amount of 50% caustic solution was then added. Since the temperature at this point was 69° F, the make-up water was added hot. A steam line then had to be added in order to bring the temperature up to exactly 75° F. After the temperature and concentration had been adjusted 500 pounds of bright viscose (75 parts 1.5-denier 1/8″ and 25 parts 1.5-denier 1/4″) was added to the Hydrapulper. The consistency at this point was 4%. The stock was agitated in the pulper for exactly 7 minutes, at which time a sample was taken. The stock was then dumped to a chest containing several times the pulper volume of water. After this quenching the stock was pumped over the Fourdrinier section of the paper machine and removed as wet lap. This wet lap was transferred to the beater for a final washing. After 1-1/4 hours of washing the stock registered a pH of 7.5. At this time the stock was considered washed, and it was dropped to the beater chest.

While the above viscose rayon was being causticized, 50 pounds of Blue African asbestos was beaten hard for 55 minutes in the beater containing a 2% solution of Daxad No. 11. This asbestos was sent to the auxiliary chest for the purpose of bleeding in at the headbox.

Another 25 pounds of Blue African asbestos was beaten hard for 1 hour in a 2% Daxad No. 11 solution and dumped to a beater chest containing the washed stock of the first Hydrapulper charge.

Immediately following the first causticizing another quantity of 7.50% sodium hydroxide at 75° F was made up in the Hydrapulper. The second batch was treated in exactly the same manner as the first except that it was agitated in the Hydrapulper for exactly 10 minutes. This stock was then quenched and pumped to the Fourdrinier wire and removed as wet lap, which was sent to the beater for a final washing. After 1-1/4 hours of washing the pH of the second batch was 7.6 and the causticized viscose was dropped to a beater chest where it was mixed with 25 pounds of asbestos prepared like the first. The average yield of the two batches of causticized viscose was approximately 75%.

Manufacturing Data

The constituents of both batches run in the Hydrapulper were kept separate in the beater chest in spite of the fact that preliminary handsheets indicated only a small difference between the 7 and 10 minute runs. Each beater chest contained the following furnish:

Beater chest Nos. 1 and 2

375 lb	Causticized and washed bright viscose rayon (75% 1/8" cut, 25% 1/4" cut, 1.5-denier)
25 lb	Blue South African asbestos beaten with Daxad No. 11

The stock went through a Jordan under pressure with the plug backed off to insure thorough mixing and was sent through the Vortraps for the additional removal of foreign matter from the asbestos. Two to three percent additional asbestos was added to the stock from the beginning of the run. The white water return was used as much as possible for the first half of the run, and stock was fed to the machine at a higher consistency than in the N-12 run.

The last section of driers on the paper machine was lagged in order to prevent excessive tension where the paper exhibited the greatest shrinkage. One roll on the calendar stack was used to soften up the sheet. At the beginning some difficulty was experienced at the calendar stack and rewinders because of the dampness of the paper. The formation of the stock on the wire was normal.

It was noted that when a relatively large amount of white water was recirculated the water drained through the web more slowly and carried further down the wire. This was thought to be desirable at first because it would conserve asbestos (fines) and would give rise to slightly better formation. However, upon closer examination it was revealed that the slower drainage gave rise to asbestos stratification within the sheet. Upon subsequent testing of this portion of the run, it was noted that the efficiency was low, indicating that the presence of asbestos fines was deleterious. Therefore, during the latter part of the run the extent of the white water recirculation was gradually cut down.

Performance of the Paper

Average values of the physical properties of this paper are as follows:

a) Caliper - 0.039 in.
b) Ream - 165 lb
c) Length Tensile - 2.3 lb
d) Moisture - 12.1%

It can be seen that the high moisture content accounts in part for the slightly high ream weight. The tensile strength was outstanding for this type of filter material. This strength is approximately in the range which would be predicted from handsheet studies.

A summary of the smoke filtration characteristics of the paper is shown in Table II. The efficiencies recorded in Table II are all lower than expected; however, this is not thought to be due to any part of the stock preparation. It will be shown later that handsheets made from this stock did in fact produce sheets of high efficiency.

TABLE II

Performance Characteristics of Paper from Mill Run

Sample	Resistance (mm H_2O)	Penetration (%)	Efficiency (%)	Remarks
First batch				
1	88	.13	3.28	Not enough asbestos
2	108	.075	2.89	Slightly damp
3	131	.016	2.90	Too much asbestos
Start of second batch				
4	110	.065	2.90	Less asbestos
5	110	.050	3.00	Stopped white water return
6	114	.015	3.35	Lighter ream weight
7	137	.003	3.30	Fresh water turned off
8	134	.015	2.85	Very damp, high ream weight
Second batch after air drying several days				
9	118	.017	3.20	
10	120	.022	3.05	
11	118	.016	3.22	
12	122	.014	3.16	
13	110	.040	3.09	
14	113	.033	3.08	

Due to the fact that some of the paper from the second batch contained more moisture than normal, it was felt that samples 4-8 in Table II were not entirely representative of this part of the run. Therefore, one small roll of paper was set aside to air dry several days and was subsequently retested. These data are recorded in Table II opposite samples 9-14.

TABLE III

Effect of DOP Exposure on Performance, N-13 Trial Sample

Time (min)	Resistance (mm H_2O)	Penetration (%)
0	112	.037
2	117	.049
4	120	.058
6	123	.069
8	128	.081
10	130	.088

The rate of "break" indicated above is higher than normal for this type of paper.

TABLE IV

Performance vs. Flow Rate, N-13 Trial Sample

Sample Flow Rate (l/mm)	Resistance (mm H_2O)	Penetration (%)
85	114	.035
42-1/2	57	.038
21-1/4	28-1/2	.034

There is little doubt from the data above that the asbestos distribution in this paper is substandard. Normally, the penetration would be expected to decrease with decreasing flow rate more than the data show. This is still another indication that a more careful study needs to be made of the variables on the wet end of the paper machine.

Effect of Aging

The samples shown in Table V were tested at the time of manufacture and again 10 days later. The purpose of this test is to determine to what extent the paper may relax on standing.

TABLE V

Effect of Aging on Performance, N-13 Trial Sample

Sample	Date of Manufacture			10 days later		
	Resistance (mm H_2O)	Penetration (%)	Efficiency (%)	Resistance (mm H_2O)	Penetration (%)	Efficiency (%)
1	122	.032	2.87	119	.042	2.84
2	124	.021	2.97	124	.027	2.88
3	135	.013	2.88	130	.022	2.82
4	117	.046	2.86	111	.060	2.90
5	119	.058	2.72	111	.065	2.90

The data in Table V indicate that this paper shows very little if any relaxation. The small decrease in resistance and slight increase in penetration is common in paper of this type. It is only when these characteristics change over a much wider range that there is cause for concern. Actually, the small variation which is observed here is thought to be due to moisture equilibrium in the paper.

Laboratory Evaluation of Mill Stock

During the mill trial various samples of the stock were saved for laboratory evaluation. Subsequently, handsheets were made from these samples using laboratory prepared asbestos. The following samples were used:

1) Washed Bright viscose rayon causticized for 7 minutes
2) Washed Bright viscose rayon causticized for 10 minutes
3) Rayon wet lap from No. 2 washed after standing 24 hours

The first objective was to determine if the rayon causticized in the Hydrapulper was comparable to that treated in the laboratory Blendor. The second objective was to determine what change if any took place in the fiber if it were to stand in the wet lap form for an extended period of time before being washed and made into paper. The data on these samples are recorded in Table VI.

TABLE VI

Performance and Physical Characteristics of Paper from Mill Stock

Sample	% NaOH	Time of Causticizing (min)	% BSA	Air Resistance (mm H_2O)	Penetration (%)	Efficiency (%)	Caliper (in.)	Tensile (lb/in. width)
1	7.50	7	12.0	99	.014	3.90	.033	2.5
2	7.50	10	13.0	101	.016	3.84	.045	2.5
3	7.50	10	11.0	119	.058	2.75	.042	9.6

Samples 1 and 2, which were washed free of caustic promptly after the causticizing, show very high efficiencies and average tensile strengths. These results are strictly comparable to sheets made from laboratory prepared stock and would indicate that there is good agreement between the laboratory and mill procedures. Sample 3 on the other hand was allowed to stand 24 hours in wet lap form in contact with some residual caustic. Following this, it was washed and mixed with asbestos for handsheet formation. The efficiency is comparatively low and the tensile strength is very high. No satisfactory explanation can be offered for this behavior at this time, but it does indicate the possibility of another variable in the operation which should not be overlooked.

This study also serves to indicate that it should have been possible to make paper of higher filtration efficiency during the mill trial. Apparently there was lack of proper control at the wet end of the paper machine.

DISCUSSION

It has been previously pointed out that the primary objective in this trial was to produce a filter paper of higher tensile strength without causing a disastrous effect on the filtration characteristics. It has also been stated that the over-all objective for the remainder of this contract was to gain experience in the operation of the Hydrapulper during the causticizing procedure. The knowledge obtained from the last two mill trials has been extremely valuable in correlating laboratory and mill runs.

There were no unopened bundles of rayon in the mill-made paper or the laboratory-made sheets indicating that an agitation period of 5 minutes or more is necessary in the Hydrapulper.

Approximately 8.3% asbestos was added to this furnish, somewhat less than in the previous mill trial. The use of less asbestos during this trial may have been due to the fact that the furnish went over the wire at a higher consistency, permitting the use of more white water.

The Hydrapulper stock washed very well in the beater. There did not seem to be any formation of an appreciable amount of gel in this process.

The N-13 trial in general was an improvement leading toward production of this filter paper on a commercial scale. The paper exhibited excellent tensile strength, fair filtration efficiency and very satisfactory folding characteristics. The mechanical strength and scuff resistance are greatly improved over N-11 and N-12 paper. This N-13 paper may not have exhibited as good an asbestos dispersion and formation as some of the former papers, but it is believed that these can be improved with more experience.

RESEARCH AND MILL TRIAL ON THE DEVELOPMENT OF A DOMESTIC SUBSTITUTE FOR ESPARTO FIBER IN THE NAVY TYPE H-60 FILTER PAPER

(N-14)

PREFACE

It is intended that this report serve as the final report under contract N7-onr-430 as well as a detailed report of the last Mill Trial designated as the N-14 Trial. Accordingly the first part will be devoted to the detailed report of the N-14 Mill Trial and associated laboratory work. This will be followed by a summary of all the work done under this contract.

RESEARCH AND MILL TRIAL ON THE DEVELOPMENT OF A DOMESTIC SUBSTITUTE FOR ESPARTO FIBER IN THE NAVY TYPE H-60 FILTER PAPER

(N-14)

INTRODUCTION

This report is a summary of the laboratory work done under contract N7-onr-430 from April 1, 1949 to May 31, 1949 together with the results of the N-14 Mill Trial. At the request of the Office of Naval Research, the work was accelerated to permit the contract to be completed by May 31, 1949 instead of June 30, 1949.

The mill trial reported here is designated as the N-14 Mill Trial, H & V Lot No. 934 dated May 20, 1949. As stated in the reports covering the N-12* and N-13 Mill Trials, it was decided to concentrate as much work as possible during the time remaining under this contract on problems associated directly with the commercial production of this new type of viscose rayon filter material. Blue African asbestos was used similar to that used in other recent trials.

LABORATORY WORK

Since the laboratory Waring Blendor most nearly approximates the action of the Hydrapulper, all studies in the laboratory were based on causticized rayon treated in the Blendor. The Blendor is operated at 80 volts by means of a Variac. The causticizing conditions observed both in the laboratory and in mill practice are as follows:

a) Temperature - 75° F
b) Caustic Concentration - 7.5%
c) Consistency - 4.0%

The time of agitation in the Blendor is obviously somewhat shorter than in the Hydrapulper, but good correlation exists between the two under the conditions now observed.

In all causticizings performed, a 1.5-Denier bright viscose rayon flock (75% 1/8″ cut and 25% 1/4″ cut) was used. Unless otherwise indicated, the asbestos was beaten and defibered in a solution of Daxad No. 11 (2% of fiber weight), a dispersing agent.

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*NRL Report 3610

It was thought that the addition of Daxad aided in the defibering of the asbestos, causing a better dispersion of asbestos throughout the finished sheet. However, during the N-13 trial, attention was drawn to the white water from the paper machine because it seemed to contain more than a normal amount of asbestos fines. Since it was not known to what extent the loss or retention of these fines might affect the efficiency of the filter paper, a large sample of white water containing the fines was drawn off for laboratory study. Handsheets were made containing the normal amount of laboratory-beaten asbestos and compared with handsheets in which the regular asbestos was replaced with varying amounts of asbestos fines. In general, the efficiency decreased with increasing amounts of fines. The data in Table I are typical of the several handsheets made with relatively large amounts of fines.

TABLE I

Effect of Fines on Performance

Sample	Regular BSA (%)	Resistance (mm H_2O)	Penetration (%)	Efficiency (%)	Tensile (lb/in. width)
Control	10.0	111	.015	3.51	2.3
White water added	7.5	86	.29	2.95	3.1

It is clear from these studies that large amounts of "fines" should be avoided. In terms of operating the paper machine, this means that the recirculation of white water should be held to a minimum.

This is somewhat contrary to the practice followed in the manufacture of Type H-60 paper and again led to the suspicion that Daxad might be the cause of excessive fines. Although Daxad has continued to show beneficial results in handsheets, it was decided to make another series of handsheets to re-evaluate its effect.

Two asbestos stocks were prepared in the laboratory beater. One was beaten in plain water, and the other in a solution of Daxad No. 11 (2% of the fiber weight). The same causticized rayon stock was used to make the two series of handsheets. Typical results are recorded in Table II.

TABLE II

Effect of Daxad on Performance

Sample	BSA (%)	Resistance (mm H_2O)	Penetration (%)	Efficiency (%)
Daxad dispersed asbestos	9.0	97	.024	3.79
Plain beaten asbestos	9.0	103	.027	3.47

Although the difference is not great, there is an unmistakable improvement in efficiency when the asbestos is beaten with Daxad. This is in accordance with previous laboratory studies, but is contrary to observations when practiced in mill trials. Accordingly it was decided to compromise and use only 1% Daxad (on the weight of asbestos) in the next mill trial. It was hoped that this might aid in the dispersion of the asbestos fibers without giving rise to excessive amounts of fines.

THE MILL TRIAL

Furnish

The N-14 Mill Trial was planned in accordance with these laboratory studies using three Hydrapulper charges of 500 lbs each, a total of 1500 lbs of rayon flock. The base furnish was to consist of 100% rayon as follows:

75 parts 1.5-denier 1/8" bright
25 parts 1.5-denier 1/4" bright

To this would be added in the beater furnish approximately 7.5% Blue African asbestos beaten with 1% (on fiber weight) Daxad No. 11. Additional asbestos (approximately 2.5%) would be bled into the system as required from the auxiliary asbestos tank.

Provisions were made to take samples for subsequent laboratory studies from each of the three Hydrapulper charges of causticized rayon, the mill prepared asbestos, and the final mixed stock as sampled directly from the headbox of the paper machine.

Preparation of the Stock for the Mill Trial

Based on experience in the laboratory as well as on the N-12 and N-13 Mill Trials, it was decided to causticize the rayon in the Hydrapulper under the following conditions:

a) Caustic concentration - 7.5% solution
b) Consistency - 4%
c) Temperature - 75° F
d) Time of agitation - 6 minutes

A quantity of water less than the total amount required was added to the Hydrapulper, and a predetermined amount of 50% commercial caustic solution was then added to the water. The temperature of the solution at this point was 74°F. By means of steam, the temperature was increased to exactly 75° F, following which the concentration was carefully adjusted to 7.5% as indicated by specific gravity readings. The total volume of caustic liquor required to give a 4% consistency based on each 500-pound charge of rayon is approximately 1430 gallons. To this solution in the Hydrapulper was added 500 pounds of rayon flock as rapidly as possible. The charge was agitated in the pulper for exactly 6 minutes, at which time a sample was taken and the agitation stopped. The stock was then dumped to a chest containing approximately 5 times the pulper volume of water. In order to remove as much caustic as possible prior to the beater washing, the quenched stock was de-watered over the Fourdrinier wire and removed as wet lap. The second and third batches were treated in a similar manner.

The wet lap was returned to the beater room where it was divided into two equal beater loads for final washing. Each beater was washed approximately one hour. At the end of this time the first beater indicated a pH of 6.6 while the second indicated 7.5. The fiber yield of the rayon treated in this manner was calculated from consistency determinations in the beater and was found to average approximately 70%. Each beater was dumped to a chest containing 37-1/2 pounds of beaten Blue African asbestos. The final consistency of the stock in each beater chest was held at approximately 1%.

It should be noted that while the rayon was being causticized and made into wet lap, the asbestos was beaten in accordance with the discussion in Section II. Two beater charges were required. The first containing 75 pounds of asbestos and 3/4 pound of Daxad No. 11 was beaten with a hard roll for about 2-1/2 hours and pumped to the auxiliary chest for bleeding into the headbox as required. The second also contained 75 pounds of asbestos and 3/4 pound of Daxad No. 11 and was beaten in a similar manner. One-half of the latter was dumped to each of two beater chests to be mixed with the rayon as indicated above. Samples were reserved for later laboratory study.

Manufacturing Data

Each of the two beater chests contained the following furnish:

Beater Chest No. 1 and No. 2

418 lb	Causticized and washed bright viscose rayon flock (75% 1/8″ cut and 25% 1/4″ cut - 1.5-denier)
37-1/2 lb	Blue South African asbestos beaten with 1% (on fiber weight) Daxad No. 11

The stocks in the two beater chests were combined and pumped to the machine chest. On its way to the paper machine, the stock was passed through a Jordan under pressure with the plug backed off to insure thorough mixing of the fibers. Following this the additional required asbestos was bled into the system and the stock passed through the Vortraps for removal of any residual foreign matter still present in the asbestos. After passing through the conventional screens, the stock discharged into the headbox and flowed on the Fourdrinier wire. The recirculation of white water (containing asbestos fines) was held to a minimum throughout the entire run. Approximately 3% additional asbestos was required from the auxiliary asbestos tank.

At the beginning of the run, the last section of dryers was immediately lagged slower in order to prevent excessive tension where the paper exhibited the greatest shrinkage. The paper was passed through one nip in the calender stack in order to soften up the sheet. However, three nips were used near the end of the run in the hope of improving the filtering characteristics. The formation on the wire appeared to be normal.

Performance of the Paper

Average values of the physical properties of this paper are as follows:

a) Caliper - 0.030 in.
b) Ream Weight - 148 lb
c) Length Tensile - 2.5 lb
d) Moisture - 7.0%

The tensile strength is that which would be predicted for stock causticized under the conditions of this run. The slightly low caliper can be explained by the somewhat heavier calendering given this paper.

A summary of the smoke filtration characteristics of this paper is recorded in Table III. These values were read on samples taken direct from the paper machine during manufacture.

TABLE III

Performance Characteristics of Paper from Mill Run

Sample	Resistance (mm H_2O)	Penetration (%)	Efficiency (%)	Remarks
1	127	.38	1.90	Damp paper
2	128	.16	2.19	Adding asbestos
3	116	.10	2.58	Enough asbestos
4	113	.096	2.67	
5	118	.095	2.56	More stack
6	133	.035	2.59	
7	125	.044	2.68	Suction increased before dandy roll
8	125	.065	2.55	Light weight

The efficiencies as calculated from the data in Table III are all much lower than those obtained on any previous paper made with this type of furnish. No entirely satisfactory explanation can be offered at this time for this behavior. However, there is abundant evidence to indicate that the asbestos distribution in the finished sheet was far less homogeneous than required. To what extent this may be related to the asbestos preparation and/or formation of the paper web on the Fourdrinier wire can only be determined by further study.

TABLE IV

Effect of DOP Exposure on Performance, N-14 Trial Sample

Time (min)	Resistance (mm H_2O)	Penetration (%)
0	110	.11
2	116	.14
4	118	.16
6	121	.18
8	123	.20
10	125	.22

The rate of "break" shown above is higher than normal for this type of paper. Reference to the N-11 and N-12 reports will show only a negligible increase in penetration after 10 minutes exposure.

TABLE V

Performance vs. Flow Rate, N-14 Trial Sample

Sample Flow Rate (l/min)	Resistance (mm H_2O)	Penetration (%)
85	114	.11
42-1/2	57	.17
21-1/4	28	.17

It can be seen in Table V that the smoke penetration increased by almost 50% when the flow rate is decreased by half. This inversion has always been interpreted in the past in terms of poor asbestos distribution. These data would thus lend increased support to the previous statement that the asbestos distribution in this paper is very substandard.

Effect of Aging

The following table compares the penetration and resistance characteristics of the N-14 paper at the time of manufacture with those obtained seven days later.

TABLE VI

Effect of Aging on Performance, N-14 Trial Sample

Sample	Date of Manufacture			7 Days Later		
	Resistance (mm H_2O)	Penetration (%)	Efficiency (%)	Resistance (mm H_2O)	Penetration (%)	Efficiency (%)
1	130	.055	2.51	127	.10	2.36
2	134	.054	2.44	128	.11	2.31
3	130	.050	2.54	124	.11	2.39
4	116	.089	2.63	115	.14	2.48
5	122	.083	2.52	110	.11	2.47
6	135	.040	2.52	127	.073	2.47
7	117	.088	2.60	112	.16	2.50

This information indicates that the paper relaxes on standing. There is no reason at present to believe that this behavior is related to poor asbestos distribution. Rather, it is thought to be due to over-calendering or calendering at a moisture content inconsistent with the stack pressure used.

Laboratory Evaluation of Mill Stock

Samples of various mill-prepared stocks were collected for subsequent laboratory study. For purposes of identification, these samples are listed below.

Sample	Stock
1	Causticized and washed rayon flock from 1st Hydrapulper charge
2	Causticized and washed rayon flock from 2nd Hydrapulper charge
3	Causticized and washed rayon flock from 3rd Hydrapulper charge
4	Blue African asbestos defibered in mill beater with Daxad
5	Blue African asbestos defibered in lab beater with Daxad
6	Combined stock direct from headbox of paper machine

Handsheets made from various combinations of these samples using 12% Blue African asbestos were made and tested. The data are recorded in Table VII.

TABLE VII

Performance and Characteristics of Paper from Mill Stock

Samples	Resistance (mm H_2O)	Penetration (%)	Efficiency (%)	Tensile (lb/in. width)
1 and 4	85	.052	3.86	2.0
2 and 4	88	.051	3.76	2.4
3 and 4	94	.038	3.65	3.2
1 and 5	86	.034	4.04	1.8
2 and 5	96	.021	3.83	2.3
3 and 5	97	.018	3.89	2.5

It will be noted that the first three handsheets above were made with mill asbestos while the last three were made with laboratory asbestos. Comparison of the average efficiency of the first set with that of the second set will reveal very little difference. This would indicate that the asbestos beaten in the mill beater compares favorably with that beaten in the laboratory. The low efficiency of the N-14 paper as compared with the handsheets in Table VIII would thus have to be explained on the basis of differences in sheet formation.

Handsheets were also made from Sample 6 taken from the headbox. In spite of the fact that extra heavy sheets were made, the resistance of these handsheets were too low to compare directly with the N-14 paper. However, this information might lead to a possible explanation of the difference between paper formed in a handsheet mould and that formed on the paper machine. Since it was noted that the resistance of handsheets formed from Sample 6 was very much lower than the resistance of the N-14 paper formed on the paper machine from the same stock, it can only be assumed that less asbestos was retained in the handsheets than on the machine. It seems only reasonable to assume that the asbestos lost during handsheet formation would be of the nature of asbestos fines pulled through the sheet under the suction used with the handsheet mould. If this be the case, all the handsheets might be expected to contain far less asbestos "fines" than the machine-made N-14 paper. Since it has already been demonstrated earlier in this report that excessive asbestos "fines" is harmful to the efficiency, one might conclude that the N-14 paper contained more than a normal amount of asbestos fines. To what extent Daxad, beating time, and roll pressure might have contributed to this situation is not known. Further study on this asbestos problem should prove extremely valuable.

DISCUSSION

The N-14 Mill Trial was judged to be highly successful in all respects except that involving the proper treatment of asbestos or its distribution throughout the paper web. Each operation in the entire process of this semicommercial run followed a predetermined schedule which was planned around a time cycle adjusted to demonstrate the feasibility of producing this new type of filter material on a much larger commercial scale. The fact that it was possible to follow this schedule gave added assurance that no major oversights had been committed.

The reader is cautioned against drawing the erroneous conclusion that faulty preparation of asbestos or poor distribution is in any way common to the rayon type of paper only. On the contrary, this problem has been associated with asbestos-bearing filter papers from the earliest beginnings. Even now during the manufacture of Navy Type H-60 or Army Type 6 filter material this problem reappears at unexplained intervals. Only skill in reducing stock preparation to a well-standardized routine has made possible the manufacture of these grades.

Thus while it would appear that a satisfactory (if not superior) substitute has been developed for esparto in the H-60 paper, further work needs to be done on the over-all asbestos problem.

In concluding the report on the N-14 Mill Trial, it can be stated that a great deal of valuable information was obtained. Except for further study on the exact nature of the asbestos desired in this sheet, the production of a rayon-asbestos filter material of high efficiency seems assured.

SUMMARY OF RESEARCH AND MILL TRIALS ON THE DEVELOPMENT OF A DOMESTIC SUBSTITUTE FOR ESPARTO FIBER IN THE NAVY TYPE H-60 FILTER PAPER

The general objective of Contract N7-onr-430 was to develop a domestic fiber to replace foreign-grown esparto fiber previously used in the furnish for the Navy Type H-60 Gas Mask Filter Material. The work was also to be carried out with a view toward improving the over-all smoke filtering characteristics of any new type filter material.

Work under previous Contracts, N6-ori-34 and N6-ori-209, revealed the possibility of using either causticized Kraft fibers or causticized yucca fibers as possible substitutes for causticized esparto in H-60 filter material. The use of either of these domestic fibers still remains a possibility. However, the filtration performance of papers made from these materials was only average and the mechanical strength was low.

The initial work under Contract N7-onr-430 led to the investigation of other fibers. Work at this laboratory as well as at Arthur D. Little, Inc. (Army Chemical Corp Contract) revealed the possibility of using causticized viscose rayon flock. Very high smoke filtration efficiencies and good mechanical strength were realized in laboratory handsheet studies. These early successes led to an enthusiastic program centered around the use of this fiber. Accordingly most of the work under the present contract was aimed at the problem of developing a commercial papermaking process for producing a rayon-asbestos filter material suitable for use in the Navy gas mask filter.

The work covered a period of two years, July 1, 1947, to June 30, 1949, and included extensive laboratory investigations as well as eight semicommercial mill runs. Eight quarterly reports have been issued (including the present report) each covering one mill trial and the associated laboratory work. The experimental trials conducted under these various Navy research contracts have all been assigned an N-series number. The trials conducted under this contract together with some of the pertinent data are tabulated below.

TABLE VIII

Summary of Mill Trials Conducted Under Contract N7-onr-430

Trial	Date of Mill Trial	Approximate Efficiency (%)	Tensile Strength (lb)	Asbestos Treatment	Equipment used for Causticizing
N-7	10/13/47	3.7	2	Plain	Rotary Boiler
N-8	12/18/47	3.7	2	Plain	Beater
N-9	2/10/48	3.6	3	Plain	Beater
N-10	7/8/48	3.1	1	Plain	Beater (50% causticized rope fibers used)
N-11	11/15/48	4.3	1	Plain	Beater
N-12	3/25/49	3.9	2.3	Daxad in part only	Hydrapulper
N-13	4/21/49	3.1	4.8	Daxad	Hydrapulper
N-14	5/20/49	2.6	2.5	Daxad	Hydrapulper

For detailed information concerning each of these trials, the reader is referred to the earlier reports. However, several general statements can be made under this summary.

1. Filter papers of very high efficiency similar to the Navy Type H-60 filter material have been realized in both laboratory work and mill trials using causticized (mercerized) viscose rayon flock as the base furnish in the paper web. It is necessary to carry out the mercerizing treatment under carefully controlled conditions. The exact nature of these conditions depends upon the type of processing equipment available.

2. The causticizing of rayon fiber has been carried out in at least three standard pieces of paper mill equipment, a rotary digester, a paper beater, and a Hydrapulper. This experience has led to the selection of the Hydrapulper as the most suitable equipment for carrying out the treatment under the conditions now practiced.

3. The mechanical strength of the finished paper can be increased over a range of severalfold (at some sacrifice in efficiency) by simply increasing the time of agitation in the Hydrapulper. Increased caustic concentration or decreased temperature may accomplish the same purpose, but time of agitation is a simpler commercial control for adjusting strength.

4. Very abnormal shrinkage is encountered on the paper machine dryers in the manufacture of this type of filter material. In spite of the fact that special adjustments have been made on the paper machine to compensate for this high shrinkage, it would be desirable to be able to incorporate a small amount of other suitable fibers in the furnish to bring the shrinkage within the range ordinarily experienced in papermaking.

5. This investigation has very largely been confined to the use of one or two types of rayon generally reported to be available in large quantities. Only a limited range of fiber diameters and fiber lengths have been studied. Other types of rayon and/or sizes may be more suitable, but it was not possible under this contract to investigate the full range of rayon fibers. Laboratory handsheets indicated that mechanical strength increases somewhat with increasing Denier, other variables constant, while the efficiency of smoke removal decreases. This led to the selection of 1.5-denier rayon as the optimum diameter that was readily available in large quantities. The length of the cut flock was chosen as long as possible consistent with good paper formation. Combinations of 1/8-inch and 1/4-inch lengths in the ratio of about 3:1 were found to be quite suitable. The rayon used for all these studies was purchased as a cut flock made from 100% new rayon. There exists the possibility of utilizing a much cheaper source of this material such as carefully selected thread waste cut with less precision than required for the flocking industry. At the present price of flock cut from new rayon, the furnish cost of this new type filter material is considerably more expensive than the furnish cost of the Type H-60 filter material. The selling price of the new paper would thus be correspondingly higher than the H-60 paper.

6. Both Blue Bolivian and Blue African asbestos have been used. Blue African asbestos is perhaps preferable, but the choice of one over the other is based on small differences in performance. Availability will perhaps determine which should be used at any given time. It should be noted that some preliminary work has indicated the possibility of using certain domestic and Canadian asbestoses in combination with AA Fiberglass and causticized rayon. Overlooking the very high cost of AA Fiberglass this might represent a solution for the manufacture of gas mask filter material from 100% domestic fibers.

7. In spite of the fact that the processing of the rayon flock is now regarded as being under control, results from the last four Mill Trials show that the efficiencies of the four papers vary over very wide limits. These differences are thought to originate from one or both of two sources.

a) Differences in the exact nature of the defibered asbestos prepared at different times.

b) Lack of uniform dispersion of asbestos throughout the filter sheet. This may be related to the many complicated variables encountered during the actual sheet formation on the wet end of the paper machine.

At present there is some reason to believe that Daxad 11 (dispersing agent) used to aid the dispersion of the asbestos during beating might be responsible in part for the low efficiencies observed in N-13 and N-14 papers. Repeated laboratory studies on the effect of Daxad in handsheets will not support this suggestion, however. On the contrary, handsheets made from Daxad-dispersed asbestos show a slight but definite improvement in efficiency over plain beaten asbestos. Nevertheless, reference to Table VIII will show that the highest efficiency papers made on the paper machine were made without the aid of Daxad. In all probability there is something more fundamentally wrong with the preparation of asbestos or its distribution throughout the paper web which is aggravated by the presence of Daxad. Reference is made to the discussion of asbestos fines in the report covering the N-14 Mill Trial.

In conclusion it should be stated that viscose rayon flock causticized or mercerized in the manner described throughout the various quarterly reports holds great promise of not only serving as a domestic substitute for esparto fibers in the Navy Gas Mask Filter Material, but also promises to lead to an improved filter material. Semicommercial quantities have been produced under conditions which are thought to represent commercial methods of manufacture using standard paper mill equipment. Variations in efficiency of smoke removal are thought to be due to variations in asbestos preparation and/or sheet formation on the wet end of the Fourdrinier paper machine. These difficulties are not common to the rayon-type filter material alone, since similar variations have been noted in the past during the manufacture of H-60 filter material. Recommendations for further work should include a study of the characteristics and behavior of asbestos as related to this problem.

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NAVY-DPPO PRNC. WASH., D.C.