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FINAL REPORT

PILOT PLANT SCALE INVESTIGATION OF PHORESIN III

CONTRACT DA-19-129-QM141

By

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Victor Chemical Works
Chicago Heights, Ill.

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SUMMARY AND CONCLUSIONS

Ten lots of Phoresin III flameproofing solution were prepared at Victor Chemical Works on a pilot plant scale. Each lot contained ten gallons of solution of 22% to 38% solid Phoresin III partial polymer concentration.

These solutions were tested by laboratory procedures and all imparted adequate flame resistance except in certain plasticiser studies.

They were applied either singly or as multiple lots under plant conditions at the Fairforest Finishing Company in Spartanburg, S.C. Under plant conditions only these variables were studied, namely: plasticizers, duration of drying and curing, method of drying and procedures to soften the finish.

It can be concluded from these tests that any degree of flame resistance can be imparted to cotton with Phoresin III. However, the degree of flame resistanse increases at the expense of such qualities as hand and water repellency. A commercially possible process for the preparation of Phoresin III has been developed, and this commercial product will impart permanent flame resistance to cotton cloth.

Aside from a compromise with the desired degree of flame resistance, water repellency and hand, there remains little that has not been attempted to minimize the effect on these qualities.

The basic problem appears to be one of penetration of the fiber. This application problem was partially solved by incorporation of Penetron S to increase the wetability of the cloth with Phoresin III solution. The problem however has only been partially solved. The degree of permanence toward laundering obtained in the laboratory has never been approached on plant scale. This, it is assumed, is due to lack of penetration since the soaking procedure used at Victor and the laboratory padding techniques of the Fairforest Finishing Company and QMC have all led to excellent laundry resistant finishes. The more a finish tends to be a coating, the more susceptible it is to the abrasive and flexile action of laundering.

Another factor which could contribute to lack of penetration has been pointed out by Mr. DeMarco of the QMC and involves the tension under which the cloth is treated, dried and cured under plant conditions. The tightening of the yarn could lead to poor fiber penetration and hence a semicoating treatment.

As a matter of comparison, the data obtained with the brominated triallyl phosphate on a pilot plant scale and the best data obtained with laboratory prepared solution of Phoresin III were included in Table III. Table III indicates that Phoresin III is by far the best flameproofing compound. Even lot No. 9, E4602, which was not considered sufficiently flame resistant by the QMC is at least equal to brominated triallyl phosphate at a considerably lower add-on.

INTRODUCTION

This report fulfills the final requirement under a contract which actually began in 1951. During the investigation several potential flame resistant finishes for cotton were studied on a laboratory scale. Two of these finishes were advanced to the pilot plant scale. These were, brominated triallyl phosphate and beta dialloxyphosphonopropionitrile (Phoresin III).

The investigation of brominated triallyl phosphate was successful in the pilot plant to the same degree achieved in the laboratory. Economic considerations prevented further development of this process at that time.

Phoresin III gave promise of being the most desirable of all compounds tested in the laboratory. It was effective at 15% add-on insofar as it would pass the Vertical Bunsen Test after fifteen launderings and was capable of being softened to a desirable hand. The scale-up to pilot plant quantities was much more difficult than experienced with brominated triallyl phosphate. In fact, only one pilot plant investigation was successful, as to preparation and application. At 20% add-on this commercially representative sample passed all of the flame resistant requirements, both by Vertical Bunsen and 1/4 inch Strip Match Test methods. Even in this case there were problems of application which were not solved. These problems of application are concerned with the inter-relation of the degree of flame resistance desired, the hand of the cloth and ability to incorporate water repellents. These textile problems could only be solved by further textile application studies. The primary object of this contract was accomplished: namely, the preparation of a uniform flame retardant in pilot plant quantities.

HISTORY OF PILOT PLANT INVESTIGATION

The contract was again renewed in 1954 for the purpose of advancing one of the promising Victor compounds to the stage of drum lot production. This was accomplished in stages by preparing a six gallon batch first and applying this solution (E1389) to 9 ounce sateen at Fairforest Finishing Company in Spartanburg, S. C. Within five laundry cycles in the Wash Wheel the finish lost its flame resistance. The failure in light of previous laboratory successes was attributed to the method of precipitation. Toluene had been substituted for the ethyl acetate-hexane system as the precipitation agent.

Another four gallons of solution from a toluene precipitation procedure was reprocessed with ethyl acetate and hexane. This reprocessed solution was tested as F6332 at the Fairforest Company.

The finish had more laundry resistance than obtained with E1369 solution. The flame resistance persisted through only five launderings. The Phoresin III which was extracted from the solid prepolymer during reprocessing with ethyl acetate-hexane could not be converted to good quality "flameproofing" solution in the laboratory.

The final drum of solution (E1455) was the result of concentration of two drums of dilute solution. Laboratory tests of the solution indicated that the ultimate testing of several hundred yards of cloth would be impractical because of lack of permanence to laundering. A small 1 1/2 gallon portion of the drum had been retained and was reprocessed with ethyl acetate and hexane. Laboratory tests of this reprocessed E1455-1 were very encouraging. The drum was returned from Philadelphia QMC Depot and reprocessed as E2211. The solution had evidently degenerated on storage because it was extremely viscous and gave off noxious fumes. Some 800 yards of various grades of cotton cloth were treated with the drum of solution at the Fairforest Company. This application was not promising since only the finish on the denim could withstand more than two laundry cycles in the Wash Wheel.

Prior to any further pilot plant studies it was necessary to determine, if possible, the causes for the inability to duplicate laboratory successes on the pilot plant scale. The quality of monomer prepared on the pilot plant scale was found to be equal to the best laboratory monomer. The monomer process was therefore excluded from further consideration.

There were two factors in which pilot plant polymerization differed from laboratory procedure. A smaller percentage of benzoyl peroxide was employed for safety considerations and consequently a longer period of heating was required to accomplish the desired degree of polymerization. These differences were considered very probable causes of the failures.

Another departure from laboratory procedure concerned solvents used to precipitate the solid prepolymer. The pilot plant procedure, which used toluene as the precipitant, was much more desirable than the two solvent, highly flammable laboratory system of ethyl acetate-hexane. For this reason production personnel were insistent that the necessity for use of ethyl acetate-hexane be shown.

The final process consideration lies in the efficacy of recyclization of unprecipitated Phoresin III by incorporation in further polymerization batches. Experience in the laboratory indicated that it was much better to start with a master charge of recovered unprecipitated Phoresin III and add fresh monomer to increase the Phoresin III content to 50% than to use fresh monomer entirely. This, from the production standpoint, is very desirable.

Of all of the potential causes for the difficulties in scale-up, the change from ethyl acetate-hexane to toluene for precipitation of the solid, partially polymerized Phoresin III was considered critical. Therefore, four solutions were prepared in the laboratory by starting with either recovered unprecipitated Phoresin III from laboratory experiments or from pilot plant investigation. Two each were prepared from the laboratory or pilot plant recovered Phoresin III by adding fresh monomer to make a 50:50 solution of Phoresin III - beta chloroethyl ether, polymerizing the solution with 2% benzoyl peroxide until the viscosity had risen four-fold and then precipitating the solid partial polymer with either toluene or ethyl acetate-hexane. The four solutions of solid partial polymer dissolved in 30/70 ethanol/ethylene dichloride were tested in the Fairforest Finishing Company's laboratory and found to be equivalent from the standpoint of flame resistance imparted to cloth. There was slightly better retentions after laundering in the cases of the ethyl acetate precipitated product.

From the above list of probable causes and laboratory results it was concluded that laboratory results alone would not solve the scale-up difficulties. The effect of such variables in the process as precipitation solvent, effect of viscosity change, time of polymerization and amount of recycled Phoresin III employed, could best be studied on the pilot plant level.

The last contract extension was negotiated with the understanding that the variables could be studied while supplying ten 10 gallon lots of solution under service order contracts. The contract was modified after two 10 gallon lots were supplied to enable Victor to ship the solution directly to the applicator and circumvent the delay caused by shipping first to Natick, Massachusetts. One-half of the second ten gallon lot was delayed several days in transfer from Natick, Massachusetts to Spartanburg, South Carolina and prevented adequate evaluation.

EXPERIMENTAL DATA

General Procedures

1. Preparation of monomer

A 1.5% solution of sodium allylate in allyl alcohol was prepared by dissolving metallic sodium in allyl alcohol at 20-25° C. This catalyst solution was added to an equimolecular solution of diallyl phosphite and acrylonitrile at such a rate that the reaction temperature could be maintained at 30-32° C with circulating water. A 10% excess of catalyst solution was added after the exothermic reaction ceased. The solution was stirred for one hour and then saturated with CO_2 gas to destroy the excess catalyst. The allyl alcohol was stripped off at reduced pressure to 100° C/5 mm. The monomer was then slurried with an equal volume of 10% $NaHCO_3$ solution at 60° C for one hour. The bicarbonate layer was drawn off and the monomer stabilized by heating to 100° C at 1 mm pressure. A representative reaction consisted of 25.8 pounds of acrylonitrile and 78.9 pounds of diallyl phosphite. An over-all yield of 81% was realized for five charges. The finished monomer was required to meet the following specifications: $n_{25}D$ 1.4655 - 1.4700; Be°/25°C 15.2 - 15.5; less than 0.2% reduced phosphorus as phosphite, less than 0.4 ml of 0.1 N NaOH per gram of monomer acid titration and less than 0.25% H_2O.

2. Preparation of flameproofing solution

Seventy pounds of material was used per polymerization charge. Each charge consisted of equal weights Phoresin III and chlorex (beta chloroethyl ether). The charge was heated to 85° C under nitrogen gas in a 25 gallon reactor. The relative viscosity at 85° C was then determined by measuring the rate of flow through a metering device. While monitoring the metering device the solution was polymerized with 2% benzoyl peroxide until the rate of flow decreased to one-fourth the original value. The solution was cooled rapidly to 30° C and precipitated in two portions with fifty pounds of toluene while vigorous agitation was maintained with a Cowles agitator. The powder thus precipitated was twice reslurried with 35 pounds of toluene and then centrifuged dry. The dry powder was evacuated overnight at room temperature to less than 2 mm pressure in an adapted ribbon crystallizer. A 35-40% conversion to powder was realized in all cases. The powder was dissolved in 30/70 ethanol/ethylene dichloride solution in the Cowles Dissolver and neutralized with triethylamine.

. Testing procedures

a) Laboratory

At Victor Chemical Works, the final flameproofing solutions were applied to 20 x 2 1/2" strips of 8.2 ounce cotton twill or

8.5 ounce cotton sateen. A 15-20% add-on was obtained by immersing the cloth for five minutes in the appropriate dilution of the final flameproofing solution. The excess solution was removed in a clothes wringer and the cloth dried for five minutes at 100° C and cured at 180° C for 5 minutes. One-half of the treated cloth was laundered at 140° F for 15 minutes with 0.5% Igepon T-73 detergent solution in a Launderometer. Both the laundered and unlaundered halves were tested for flame resistance by the Vertical Bunsen and 1/4" Strip Match Test methods. In all cases the solutions imparted excellent flame resistance to the cloth.

At the Quartermaster Research and Development Laboratories at Natick, Mass., a slightly different test procedure is employed. The cloth is treated by multiple passes through a laboratory padder at 40 lbs./sq.in. air pressure on the squeeze rolls. The drying of the cloth was accomplished by heating at 100° C for 1 1/2 to 5 minutes. A similar procedure was used to cure the cloth at 180° C for the same time intervals as used for drying. These larger samples of treated cloth were subjected to fifteen laundry cycles in the Wash Wheel using Method 5556 Federal Specifications CCC-T-191b. The QMC tests at Natick, Mass. were satisfactory at 20% add-on in regard to the permanence of flame resistance. The cloth also possessed the desired hand if 4% Celluflex plus 2% oleic acid were incorporated as plasticizers for the resin. The unplasticized stiffness returned, however, on "soaping" at 160° F. The ultimate plasticizer for Phoresin III finishes has not been discovered, but such compounds as Victor Softening Agent E5182, Santicizer 141 and Celluflex CEF, did impart acceptable hand to cloth treated with Phoresin III. The exact function of the plasticizer in the Phoresin III system has not been determined.

b) Plant scale

All plant scale applications were conducted at Fairforest Finishing Company, Spartanburg, S.C., in consultation with the Quartermaster Corps personnel who were present. Cloth samples of between 7 1/2 and 152 yards were treated in a manner similar to that used by the Quartermaster Corps. The 9 oz. and 8.5 oz. cotton sateen were padded by a 1 dip + 1 nip process at least twice with and without drying between padding operations. The cloth was dried by either passage through an overhead oven or a tenter frame at 220-250° F for 3/4 to 5 minutes. The finish was always cured at 320 - 350° F for 1 1/2 - 5 minutes in another overhead oven. In almost all cases the cloth was very stiff after curing and required soaping on a jig wash to soften the hand. The degree of softening on a plant scale was never as great as obtained in the Quartermaster procedure. The cloth was then Sanforized and Zelanized.

Following is a description of each of the ten lots of solution supplied for pilot plant application and the results of those applications.

Lot No. 1 (E3319)

This ten gallon sample was shipped to Natick, Massachusetts on July 11, 1955. The solution contained 22% polymer when tested at Victor and found to be representative. It had been prepared from recycled chlorex-Phoresin III solution with sufficient fresh monomer to replace the precipitated solid partial polymer from the previous cycle. The fresh monomer was obtained by purification of a monomer of questionable quality. Tests at Natick on a laboratory scale indicated that 20-25% of the resin washed out during the "soaping" step.

A pilot plant application of the solution was conducted at Natick using the coating equipment available to the Functional Finishes group. The cloth thus treated had satisfactory permanent flame resistance qualities. Its hand was stiffer than desirable and it seemed to have abnormally high loss in tear strength.

Lot No. 2 (E3596)

The seven gallons of this solution were sent to the Fairforest Finishing Company, Spartanburg, S. C., on July 25, 1955. This solution also contained 22% solid Phoresin III and was prepared in a manner similar to that used to prepare Lot 1 with the exception that a greater percentage of fresh monomer was employed to increase the size of a polymerization charge.

On August 2, a 7 1/2 yard piece of 9 ounce cotton sateen was padded with the solutian by a 1 dip + 1 nip process and dried at 260° F for 1 1/2 minutes to gain 15.5% add-on as determined by curing a small sample in the laboratory oven. Retreatment to increase the add-on resulted in a spotty surface due to the condition of the padder squeeze rolls.

Half of Lot No. 1 was delayed in transit from Natick, Mass., to Spartanburg, S.C. This prevented an evaluation of E3319 on a pilot plant scale. The remaining six gallons of Lot No. 2 plus the five gallons of Lot No. 1 were combined and applied to 75 yards of 9 oz. combed sateen by a two pass system (1 dip + 1 nip) with drying for 1 1/2 minutes in an oven between passes. The cloth was dried at 260° F for 1 3/4 minutes after the second pass and then cured at 350° F for five minutes. The cloth was very stiff because a plasticizer had not been incorporated in the formulation to soften the finish. Experience had shown that incorporation of a plasticizer results in larger "wash out" losses. Since it was doubtful whether eleven gallons would adequately treat the 75 yards of cloth if two pass system were necessary, it was decided to not use a plasticizer. As a result it was necessary to "soap" the cloth for 5 hours on a jig was to improve the hand. This soaping operation removed 50% of the resin applied. Further tests demonstrated that after soaping the treated cloth was no longer adequately flame resistant.

Lot No. 2A (F1668)

A 5 gallon drum of solution plus a 2 quart sample of the solution which had been concentrated to 36% solids was submitted to the QMC on September 2, 1955. Tests at Natick, Mass., indicated that this was an inferior solution because the cloth treatment was not laundry resistant. The tests at Victor were unable to show significant differences between E3596 and F1668.

Lots 3 and 4 (E4079)

The exact reason for the inability to duplicate laboratory success on a pilot plant scale has not been determined. One variable, however, was eliminated by adopting the present pilot plant process for making Phoresin III. Without changing the procedure another twenty gallons of solution were prepared at 30% solid Phoresin III concentration.

On October 12, 1955 a quart sample was tested by the QMC procedure. The tests using 5% Triton K60 as a softening agent indicated that the solution was very good. At least 70% of the resin remained after 15 laundry cycles and the cloth was adequately flame resistant.

On November 8 and 9, 1955, 80 yards of 9 ounce combed sateen was treated in a two pass process at the Fairforest Finishing Company plant. The solution was modified with 5% Triton K60. After the first dip and nip the cloth was dried for two minutes at 226° F. For the second pass the solution was diluted with one-third its weight of 30/70 ethanol-ethylene dichloride. After the second pass the cloth was dried at 226° F for 1 minute and cured at 340-370° F for 5 minutes. The cloth was very stiff. Even after 10 "ends" jig washing the cloth was much stiffer than the laboratory sample. The tests at Natick on the final treated cloth were not encouraging. Only 12% add-on remained after the soaping operation which is not sufficient for adequate flame resistance.

Lots 5, 6 and 7 (E4693)

Another larger supply of flameproofing solution was prepared at a concentration of 32% solids. The procedure was the same as described for the preceding four lots of solution. On January 24,1956 a quart sample of this solution representing 30 gallons, was tested by the QMC procedure. Subsequent tests indicated that a single pass (1 dip + 1 nip) would give approximately 20% add-on and that incorporation of a plasticizer did not soften the treatment significantly. The treatment, however, did not pass the strip match test after 15 laundry cycles.

A meeting was held on March 12, 1956 at Spartanburg, S.C. with the intended purpose of preventing useless repetition of previous results. It was decided to test only 20 yards of cloth and then finish the study of plasticizers before treating a large quantity of cloth. It was found that an oil treatment such as Penetron S would increase the wettability of the cloth toward Phoresin III solution. The 210 yards of cloth to be treated with E4693 solution were first treated with Penetron S and dried on a tenter frame. The twenty yard sample was removed and given a single pass (2 nip + 2 dip) application, dried at 250° F for 4.5 minutes and cured at 350° F for 5 minutes. The treatment seemed, for the first time, to parallel laboratory results insofar as 20% add-on was realized with a single application. The cloth was jig washed for 10 ends and softened with Abcovel G. Subsequent analysis showed the original phosphorus analysis to be in error. The final analysis of the cloth which had been Zelanized showed only 16% Phoresin III content. The cloth was considered acceptable.

Three plasticizers from the many studied softened the Phoresin III finish to almost the desired degree in the laboratory. The most effective plasticizer was a mixture of tris-beta chloroethyl phosphate and oleic acid. This treatment was undesirable only insofar as soaping caused stiffness to return. Another was Victor Softening Agent E5182 which was unaffected by soaping but only imparted fair softness to the Phoresin III finish. The last plasticizer was diphenyl octyl phosphate. These three plasticizers gave approximately equal softness to the Phoresin III finish after the soaping procedure.

On May 16, 1956 the remaining cloth was treated by a single pass (2 dip + 2 nip) process. 152 yards were treated with E4693 solution containing 4% Victor Softening Agent E5182. A 30 yard portion was treated with E4693 containing 4% Celluflex CEF and 2% oleic acid. The cloth was dried at 250° F for 1 1/2 minutes and cured at 320-340° F for 1 1/2 minutes. The 152 yards were very stiff but on jig washing became as acceptable as the latter 30 yards treated with the solution containing Celluflex CEF. The cloth satisfactorily met the flame resistance requirements of both the Vertical Bunsen and Strip Match tests. Hand and water repellency still were not acceptable.

Lot No. 8 (E4637)

This solution was prepared in 38% concentration by the recyclization procedure. A quart sample was tested by the QMC procedure after July 25, 1956. Using 4% Celluflex CEF and 2% oleic acid as additives the solution was applied to 9 oz. sateen by a one pass (1 dip + 1 nip) process. The cloth was dried on a tenter frame at 290-310° F for 1 1/2 minutes and then cured at 350° F for 1 1/2 mins. At 20% add-on the cloth did not possess the desired hand but was satisfactory in all other respects including the Vertical Bunsen Flame resistance test. It did not however, pass the strip match test after the 9th laundry cycle. The water repellency was also not completely satisfactory but much better than obtained with E4693. It would appear that penetration of the cloth with 38% solution is insufficient.

Lot No. 9 (E4602)

This solution was prepared using one grade of fresh monomer. Recycled Phoresin III was excluded entirely. The powdered partial polymer was dissolved in sufficient 30/70 ethanol ethylene dichloride to make 32% concentration. The solution was applied to 9 ounce sateen by a single pass process (2 dips + 2 nips) after 4% Celluflex CEF and 2% oleic acid had been incorporated as plasticizers. The cloth was dried at 290-310° C on a tenter frame for 1 1/2 minutes and cured at 350° C for 1 1/2 minutes. The hand was much improved after soaping; in fact, except for failure to pass the strip match test after the first laundry cycle, the finish came closest of all treatments to meeting the requirements. It even passed the Vertical Bunsen Test after 15 laundry cycles.

Lot No. 10 (E4566)

This 32% solution was prepared using another grade of fresh monomer without incorporation of any recycled Phoresin III. As in the cases of Lots 8 and 9 this solution was applied to 9 ounce sateen by a one pass (2 dip + 2 nips) process on August 15 and 16, 1956 at Fairforest Finishing Company. It had been modified with 4% Celluflex CEF and 2% oleic acid. The treated cloth was dried on a tenter frame at 290-310° F for 1 1/2 minutes and cured at 350° F for 1 1/2 minutes. The finish was not permanent to laundering.

TABLE I

COMPARISON OF ETHYL ACETATE AND TOLUENE AS PRECIPITANTS

Sample Number	B8001			B8002			B8003			B8004		
Recycled Phoresin III source	Laboratory			Laboratory			Pilot Plant			Pilot Plant		
Precipitation solvent	Ethyl acetate			Toluene			Ethyl acetate			Toluene		
Treated at *	F	V	F	F	V	F	F	V	F	F	V	F
Plasticizer A	No	No	Yes	No	No	Yes	No	No	Yes	No	No	Yes
% Add-on Dried 100°C (Anal)	17.7	-	16.7	18.3	-	19.6	16.1	-	16.3	16.5	-	17.0
" Cured 180°C(wt)	19.5	13.3	22.4	25.0	16.6	26.1	21.6	13.0	19.6	23.0	13.5	25.7
" " " (anal)	16.3	14.7	16.9	17.0	17.0	19.4	16.1	15.3	15.6	16.3	16.1	17.0
% Retention (dried to cured) Anal.	92.5	-	100	92.8	-	99.0	100	-	95.8	99.3	-	97.2
% Add-on soaped (wt.)	19.0	-	13.8	20.8	-	15.2	18.4	-	16.1	17.6	-	19.4
" " (Anal.)	13.5	-	13.5	13.8	-	13.6	13.8	-	12.4	13.8	-	12.1
% Retention (cured to soap((wt.)	97.5	81.2	61.7	83.2	76.1	58.2	85.2	84.8	82.2	76.5	77.5	75.5
" " (Anal.)	83	93.0	80.0	81.2	92.3	70.2	85.7	92.0	79.6	84.7	87.1	71.2
% Add-on (15 launderings) (Wt.)	14.2	-	12.9	18.9	-	14.4	17.1	-	15.7	14.9	-	12.7
" (Anal.)	10.5	-	10.8	11.0	-	9.4	11.5	-	9.9	11.0	-	9.3
% Retention (Cured to 15th) (Wt.)	73.0	-	57.6	75.6	-	55.2	79.2	-	80.0	64.8	-	49.4
" (Anal.)	64.4	-	64.0	64.7	-	48.5	71.5	-	63.5	67.5	-	55.6
Char length, init. Ins.	2.2	3 3/4	2.3	2.3	3 1/2	2.1	2.2	4 3/8	2.1	2.1	3 3/4	2.0
" " ,1st cyc. "	3.2	4 1/2	3.8	1.9	4	2.1	3.1	4	3.1	3.2	4 3/8	3.1
" " ,5th " "	3.4	-	3.1	3.2	-	3.0	3.5	-	2.6	3.5	-	3.7
" ",10th " "	3.3	-	4.5	3.3	-	3.4	3.4	-	4.1	4.0	-	4.0
" ",15th " "	3.0	-	4.0	3.1	-	3.3	3.2	-	3.4	3.1	-	3.1

* F = Fairforest
V = Victor

TABLE II

EVALUATION OF TEN LOTS AT VICTOR

Lot Number	1	2	3 + 4	5,6,+7	8	9	10
"E" Number	3319	3596	4079	4693	4637	4602	4566
% Add-on, wt.	16.3	16.1	20.6	23.4	24.2	19.9	22.8
% Phos. anal.	2.46	2.36	2.82	3.22	3.20	2.82	2.98
% Add-on, anal.	17.1	16.4	19.6	22.4	22.2	19.6	20.7
% Retention, laundering 1 cycle, wt.	75.9	77.8	77.2	86.8	84.2	83.0	88.8
% Phos. anal. laundering 1 cycle	2.46	2.25	2.46	3.10	3.10	2.72	2.78
% Retention, laundering 1 cycle	100	95.5	87.4	96.3	97.0	96.5	93.3
Char length, cured only	3 7/8	4 5/8	3 5/8	3 1/2	3 7/8	4 1/4	3 5/8
Char length, laundered	4 3/8	4 5/8	3 5/8	3 1/2	3 3/4	3 7/8	3 1/2
1/4" Strip Match Test cured	+	+	+	+	+	+	+
1/4" Strip Match Test laundered	+	+	+	+	+	+	+

TABLE III

SUMMARY OF PILOT PLANT APPLICATIONS

Designation No.	E8837	VFS No.20	E3319*	E3319-3596	E4079	4693	4637	4602
Compound	Br.TAP	Phoresin III	Phoresin III	Phoresin III	Phoresin III	Phoresin III	Phoresin III	Phoresin III
Soln conc.%solid	27	17.5	22	22	32	32	38	32
% Add-on, wt.	26.1	17.0	23.3	(a)	(a)	21.1	20.2	18.6
" , anal.	26.3	-	21.9	9.0	13.6	16.0	21.3	12.0
Tensile strength;								
untreated	151x162	-	170x174	-	-	176x172	176x172	176x172
initial	185x180	-	227x233	-	-	170x171	206x198	186x173
aft.1 1dry cyc.	177x163	-	194x197	-	-	199x197	201x200	186x180
aft.3 1dry cyc.	171x160	-	173x183	-	-	193x188	198x193	173x161
Tearing strength:								
Untreated	6.1x7.2	14.1x14.1	8.5x14.1	-	-	5.5x5.9	6.8x5.6	6.8x5.6
Initial	5.9x7.6	7.8x10.4	4.7x5.8	-	-	4.5x5.9	4.4x3.8	6.5x5.9
Aft.1 cycle	6.6x10.0	11.3x11.4	4.9x6.1	-	-	5.8x7.0	6.3x4.9	7.2x5.9
" 3 "	7.3x8.9	-	6.2x6.1	-	-	4.9x6.8	6.5x5.4	6.2x5.9
Flexibility,								
Untreated	0008x0007	0004x0004	0004x0004	-	-	0014x0008	0014x0008	0014x0008
Initial	0040x0035	0011x0009	0120x0090	-	-	0033x0063	0141x0120	0052x0074
Aft.1 cycle	0025x0013	0008x0010	0075x0075	-	-	0043x0035	0070x0042	0020x0018
" 3 "	0021x0012	-	0053x0035	-	-	0035x0048	0036x0026	0014x0013
H₂O resistance								
spray rating	90.0	-	-	-	-	0.0	50	50
C.L. Init.(Match)	5.7	4.4	4.4x4.0(+)	4.3x5.2(-)	-	4.2x3.9(+)	3.8x3.8(+)	4.4x4.5(+)
C.L. 1 cyc. "	4.8	4.2	3.9x4.1(+)	-	-	4.3x3.1(+)	3.8x3.9(+)	4.7x4.7(-)
C.L. 3 " "	4.8	-	4.6x4.2(+)	-	-	4.2x4.4(+)	4.1x4.4(+)	5.1x4.5(-)
C.L. 6 " "	4.6	-	4.7x4.9(+)	-	-	4.4x4.1(+)	4.3x4.2(+)	4.9x5.3(-)
C.L. 9 " "	4.6	-	4.9x4.6(+)	-	-	4.2x3.6(+)	4.3x4.4(-)	4.8x5.0(-)
C.L.12 " "	5.5	-	4.0x4.4(+)	-	-	3.8x5.1(+)	4.5x4.6(-)	5.4x4.9(-)
C.L.15 " "	5.3	-	4.1x4.6(+)	-	-	3.6x4.0(+)	4.6x4.7(-)	5.1x5.2(-)

*This pilot plant run was conducted at Natick, Mass. on coating equipment.

(a) The complete evaluation of these finishes were not disclosed to Victor by report. The unsatisfactory nature of the finish was born out by the field condition tests conducted with cloth.

Appendix

During a review of this report the following additional comments were obtained from Victor Chemical Works:

1. Data on the viscosity of flameproofing solutions

Some viscosity data are available, but were purposely excluded from the report because of its incomplete and confusing nature. The percentage of solids is contained in Table III. The degree of polymerization is not known but must be of the order determined by Dr. Esteve at Rhode Island University with triallyl phosphate.

2. Criteria for good flameproofing solution

No fixed quality control for the flameproofing solution was established. The precipitated partial polymer was considered good quality if it remained a free flowing solid which could be vacuum dried to powder form. This dried powder always gave a true solution when dissolved in 30/70 ethanol/ethylene dichloride. Therefore it should be concluded that the solution itself was criteria of quality. Off-grade gummy solid precipitate would result in gel formation upon dissolution.

3. Effect of solvents of precipitation

Paragraph 2 on page 4 and Table No. 1 were purposely included in the report to show that there was no significant difference between the products precipitated with toluene or ethyl acetate-hexane. Table No. 1 was the basis for the decision to use toluene to precipitate the prepolymer for each and every one of the ten 10 gallon lots.

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