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ENVIRONMENTAL STABILITY OF PAPER PRODUCTS

TECHNICAL REPORT

By

William F. Garland

November 1967

U. S. ARMY WEAPONS COMMAND

ROCK ISLAND ARSENAL

RESEARCH & ENGINEERING DIVISION

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ABSTRACT

This study covers the investigation of some fifteen paper additives (including oxidized and cationic starches, neoprene and urethane latices, a water soluble polymer, a fluorocarbon resin and aqueous and nonaqueous fungicides) for possible use in upgrading kraft type paper. It was desired to improve oil, water, aging and fungus resistance properties of the paper.

Good oil resistance was obtained using the fluorocarbon resin even though "alum" (aluminum sulphate) interfered with its deposition on the pulp fibers.

The nonaqueous solutions of fungicides provided excellent fungus resistance but also required an extra immersion step after the paper had been formed.

The best water resistance was obtained utilizing a water soluble polymer (Kymene 557).

In a "composite" hand sheet formulation (made from the base formulation plus additives giving the best individual properties) some properties were improved, i.e., tensile breaking strength and folding endurance and some were downgraded, i.e., tear and pH.

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PROBLEM

This study concerns the investigation of the addition of chemical additives to a base pulp formulation for the purpose of obtaining improved oil, water, aging and fungus resistance properties of a kraft paper (UU-P-268e).

BACKGROUND

Paper as a usable material has many drawbacks. Several, as far as the Army is concerned, are poor oil, water and heat aging properties and susceptibility to microbiological attack.

Normal paper is a matted or felted sheet of vegetable fibers.(1) In the course of preparation for manufacture into sheets, the fibers are treated in water by the mechanical process of beating. The fibers absorb water and there is formed on the surface of the fibers a gelatinous film which when dry, acts as a cement to bond the fibers together at all points of contact. When the paper is wetted, the "cement" again absorbs water and reverts to the gelatinous condition thus allowing the fibers to fall apart.(2)

Untreated paper products are highly susceptible to deterioration by oils and greases.(3) Penetration as "bleed thru" of oils and greases cause swelling and degradation of the fiber bond. Conversely, oil and grease resistance is attained by reduction of porosity of paper which has the effect of sealing or filling the surface pores.

Paper, when wetted with water, loses strength and coherence almost completely. It has been found that paper made under normal or ordinary conditions retains only 5 to 10 percent of its dry strength when wetted with water.(4) The extent of this loss is such that ordinary paper is almost useless in the wet state. It has been said that as much as 75 percent of some military items (during World War II) arrived at overseas destinations in a damaged or otherwise unusable condition.(5) Failure of paper products (boxes and packages) due to water damage contributed heavily to this percentage.

For several obvious reasons there is very little exact information on the aging of paper over long periods of time.(6) Many of the test methods for paper have been in existance for relatively few years. Therefore it is impossible to compare the present properties of old papers with the original values. Also, neither the original conditions of manufacture nor the conditions of storage are known exactly. The aging of paper is then a property of considerable importance. However, it is equally obvious that there is no universal agreement as to the cause of the deterioration of paper with age. Studies at this Research Laboratory have shown the paper components of barrier materials (MIL-B-117 and MIL-B-121) to become brittle and increase in acidity after both natural[7] and accelerated[8,9] aging.

Paper and paper products, because of the nature of their components, are normally susceptible to microbiological attack.[10] The attack on papers is carried out by one or several enzymes produced by microorganisms. These enzymes degrade cellulose, lignin, proteins, starches and other paper making materials. Not all, nor even a majority of the fungi and bacteria have the ability to utilize cellulose or lignin as a carbon source but when all the components that go into specialty paper grades are considered, a larger variety of microorganisms should be recognized as potentially able to degrade paper products. Certain species of organisms are widely distributed in nature and may easily innoculate papers, needing only suitable foods plus the proper environment to initiate the degradation process.[11,12]

APPROACH

A previous study[13] concerning the improvement of the stability of paper products showed only one manufacturer supplying a sample of upgraded kraft paper. The latter as well as various tub sized kraft papers did not meet the target requirements for an upgraded kraft paper. Due to the above reasons, it was decided to attempt the addition of chemicals and treatments to paper using Rock Island Arsenal Research Laboratories paper making equipment (see figures 1 and 2).

PROCEDURE

Two samples of sulfate pulp (one bleached and one unbleached) were obtained for evaluation as a base sheet for a standard paper formulation. Both types of pulp were made up into hand sheets after various beating times and tested for the properties indicated in Table I.

The general overall properties of the bleached pulp were higher than those of the unbleached pulp. The bleached pulp with a beating time of 20 minutes was selected as the base material for a standard paper formulation.

The standard paper formulation used to evaluate the various additives is shown in Table II.

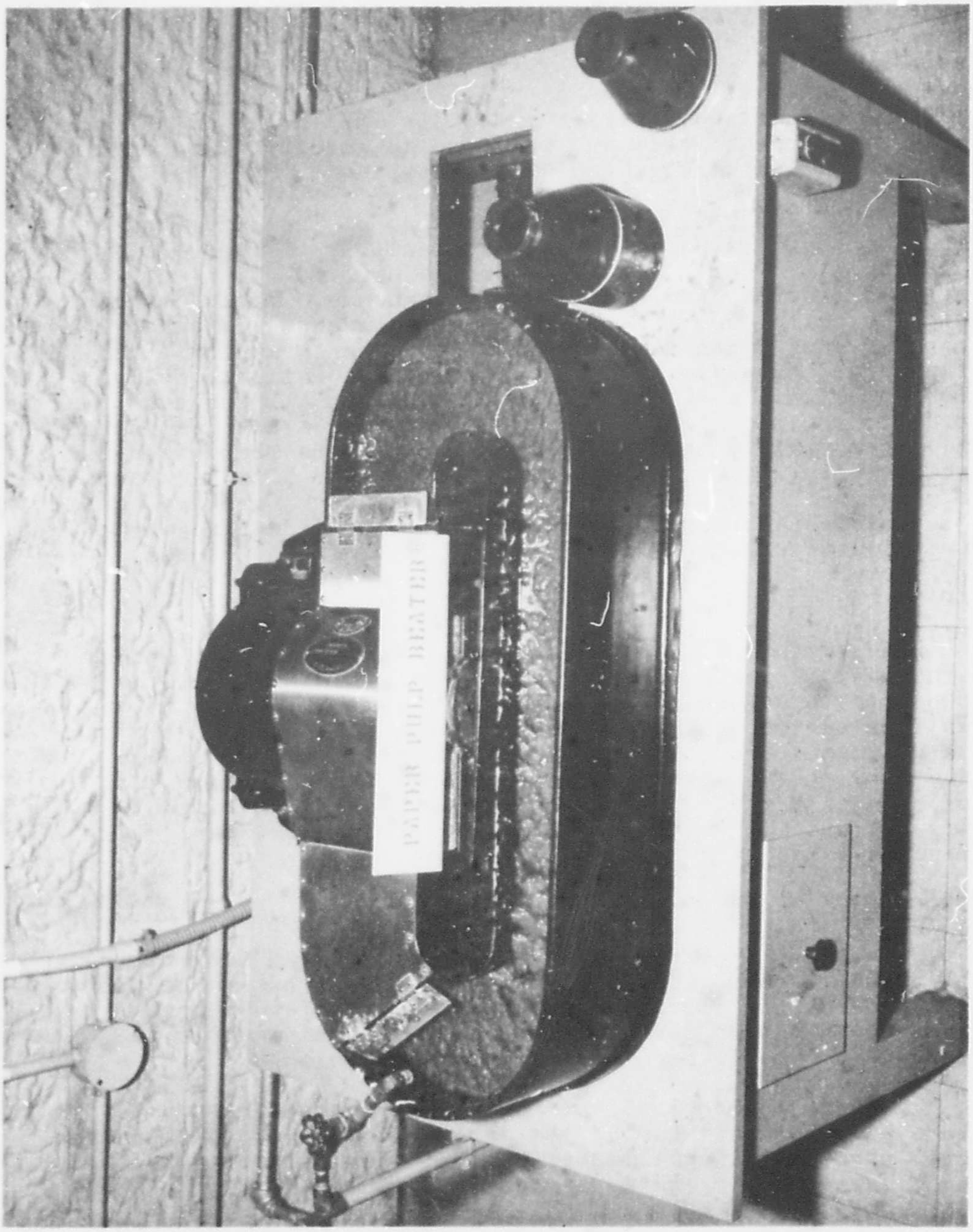

FIGURE 1 VALLEY 1/2 LB. BEATER

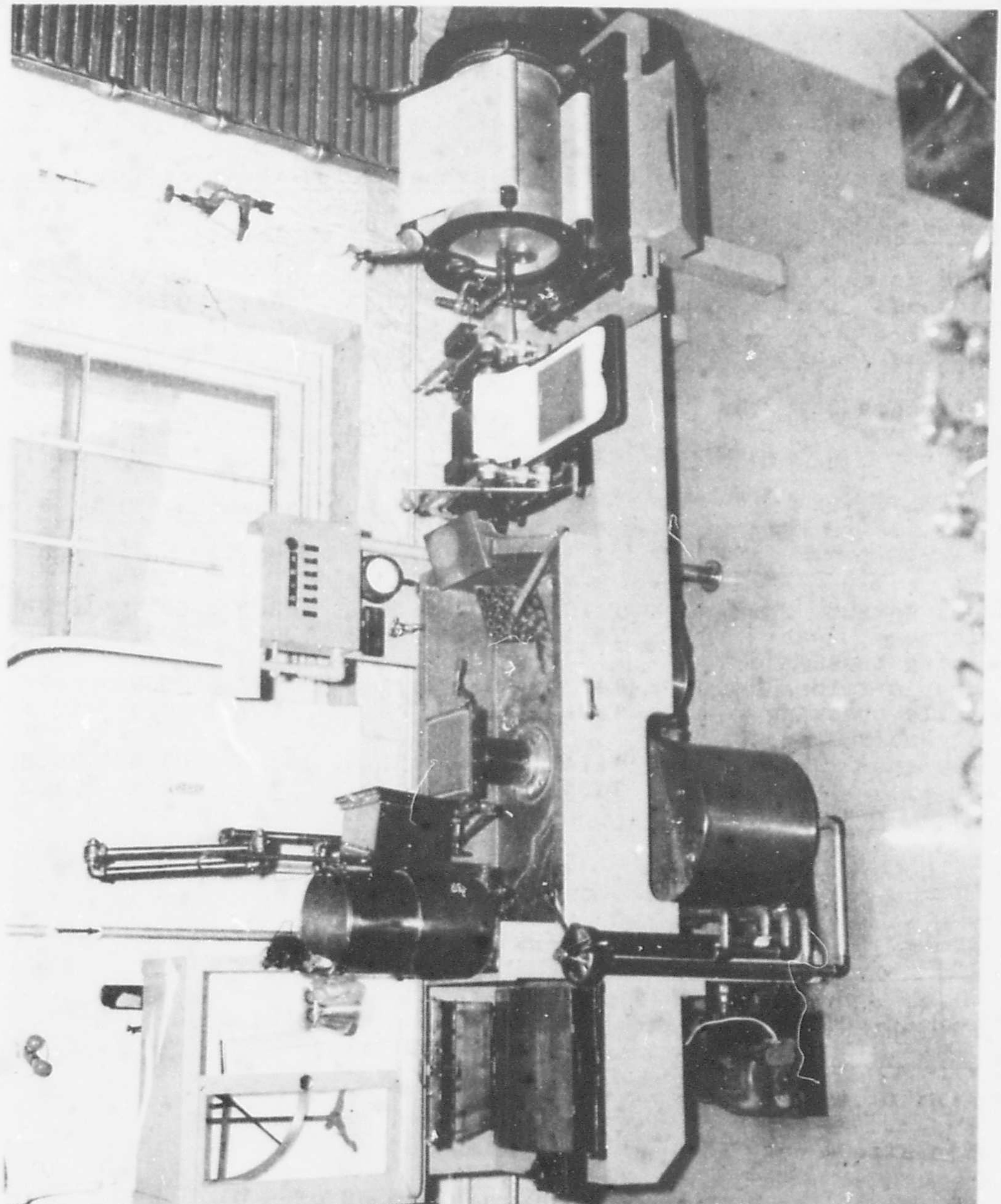

FIGURE 2 NOBLE AND WOOD SHEET MACHINE

TABLE I

PULP EVALUATION

Property	TAPPI Test Method	Bleached Pulp - Beating Time, Minutes 0	10	20	30	60
Basis Weight, g/m^2	T410	60.7	60.8	59.9	59.6	59.4
Thickness, inch	T411	.0054	.0049	.0044	.0042	.0039
Folding Endurance, double folds	T423	1	58.8	780.4	1390	1482
Tensile Breaking Strength-Dry, lbs	T404	2.4	6.0	9.1	12.5	14.2
Tear, gms	T414	56.4	120	78.8	56.8	43.2
pH, unit	T435	7.2	7.2	7.1	7.2	7.1

Property	TAPPI Test Method	Unbleached Pulp - Beating Time, Minutes 0	10	20	30	60
Bases Weight, g/m^2	T410	61.2	59.7	62.5	60.4	61.6
Thickness, inch	T411	.0074	.0062	.0058	.0051	.0048
Folding Endurance, double folds	T423	0	11.5	137	559	879
Tensile Breaking Strength-Dry, lbs	T404	1.7	4.1	7.7	9.4	12.4
Tear, gms	T414	48.4	121.6	143.6	103.6	79.2
pH, unit	T435	7.4	7.3	7.3	7.4	7.2

TABLE II

STANDARD PAPER FORMULATION

Bleached sulfate pulp	-	360 grams, beaten 20 minutes in 23 liters tap water
Titanium Dioxide	-	40.2 grams
Rosin size	-	7.2 grams
Alum	-	4.0 grams (to pH of 5.0)
Additive	-	(added in proportioner)

A 2-1/2 quart measure of the pulp slurry was placed in the proportioner (see figure 2) diluted to the mark and hand sheets formed according to the manufacturers procedure (similar to TAPPI Method T205).

Additives of the types indicated in Table III were obtained in an effort to improve the oil, water, aging and microbiological properties of the kraft paper.

All additives (with the exception of A12-A15) were added at the proportioner as recommended by supplier. Fungicides A13-A15 were not water soluble (A13 and A14 soluble in methanol, A15 in naphtha). Hand sheets of the base formulation were immersed in 1, 2, 3 and 4% solutions of all four fungicides for 1 minute and dried at ambient temperature.

Approximately 25 hand sheets each were made from formulations utilizing the additives listed in Table III (with the exception of A12-A15). Prior to testing all sheets were conditioned for at least 48 hours at 72 ± 2°F. and 50 ± 4% R.H. All tests were performed according to TAPPI test methods with the following exceptions:

Folding Endurance (TAPPI-T423) - M.I.T. procedure

pH (TAPPI-T435) - Hot extraction procedure

Tensile Breaking Strength - Wet (TAPPI-T456) - 2 minute water immersion

Fungus Test (TAPPI-T487) - Similar to that specified in Specification MIL-B-3959

Tear (TAPPI-T414) - 1/2 width specimen

Oil Resistance (TAPPI-T462) - In addition ASTM #3 and MIL-L-46002 oils

Additives A1 through A5 were applied as 1, 3 and 5% additions based on the weight of the pulp, however, due to time and a shortage of bleached pulp only a low and high range of additions of additives A6 through A11 were investigated.

TABLE III

ADDITIVES

Additive	Type	Trade Designation	Manufacturer
A1	Oxidized Starch	Staysize 109	A. E. Staley Mfg. Co.
A2	Cationic Starch	Electra	Anheuser-Busch, Inc.
A3	Cationic Starch	StaLok 300	A. E. Staley Mfg. Co.
A4	Cationic Starch	Dasol C	Miles Chemical Co.
A5	Cationic Starch	Epic-N	A. M. Meincke and Son, Inc.
A6	Neoprene Latex	Latex 460	E. I. Dupont de Nemours, Inc.
A7	Urethane Latex	P-501	Wyandotte Chemical Co.
A8		Glyoxal	Union Carbide Corp.
A9	Water Soluble Polymer	Kymene 557	Hercules, Inc.
A10	Alkyl Ketene Dimer	Aquapel 360X	Hercules, Inc.
A11	Water Soluble Fluorocarbon	FX-806	3 M Co.
A12	Water Soluble Fungicide	Dowicide - Q	Dow Chemical Co.
A13	Fungicide	G-4	
A14	Fungicide	G-11	Sindar Corp.
A15	Fungicide	Cunilate 2174-P	Scientific Oil Compounding Co., Inc.

RESULTS

Results are as shown in Tables IV and VI.

DISCUSSION

Tensile Breaking Strength - Dry

The tensile strength test shows the resistance of paper to rupture when subjected to a longitudinal stress and corresponding strain imposed by an outside force. Tensile strength then is a fundamental property of paper.

Across the board increases in breaking strength occurred with all percentage additions of additives A1 through A5. All cationic starches were significantly better than the oxidized starch A1. The best of the starch additives A3 produced an increase of 60% in breaking strength at the 1% addition level. The latices only slightly improved the breaking strength (at the most, 20%). The chief contribution of the latices appeared to be a good "hand" (softness), flexibility, and an increase in elongation. The breaking strength improvement produced by A9 (water soluble polymer) approached that of the best starch.

Factors involved which could affect the breaking strength are cooking temperature of the starches (i.e., if the temperature is not high enough the starch granules swell but do not burst), control of the pH (especially with latices and resins), and order of addition of chemicals.

Tensile Breaking Strength - Wet

Practically any paper will become wetted eventually if soaking is sufficiently prolonged. Paper that has not been especially treated for wet strength shows a value in this property of from 5 to 10% of its dry strength. Normally, wet strength is imparted to paper by addition of various thermosetting aminoplastic resins (i.e., urea formaldehyde), latices, etc.

The base paper used in this study retained only 3% of its breaking strength after 2 minute water immersion and thus falls into the category of paper that has not been especially treated for wet strength. Only one of the starches, A4, produced any significant wet strength retention (16%) after 2 minute water immersion. The neoprene latex A6 and the water soluble polymer A9, respectively, when added to the base formulation produced the highest wet strength retention (30-35%) of any of the additives tested however, the latex also had very low dry tensile breaking strength initially.

Oil Resistance

The resistance of paper to penetration of water is greatly affected by surface treatment of the fibers with little or no effect upon the porosity, however, the penetration of oily materials is little affected by such treatments. Oil (and grease) resistance is attained by reduction of porosity of papers.

The three test oils were selected for their penetrating ability, i.e., castor oil, slow; ASTM #3 oil, medium; and MIL-L-46002, fast. As shown in Table IV all of the starches showed some resistance to castor oil but none to the ASTM #3 or MIL-L-46002 oils. Additives A6 through A10 had little resistance to any of the three oils. The fluorocarbon additive A11 had the best across the board oil resistance but fell far short of expectations. It is felt here that the alum used in the base formulation interfered with the proper orientation of the fluorocarbon on the fibers by forming a complex with the aluminum from the alum.

Tear

Tearing strength is primarily a property of the inner structure of the paper sheet, but surface treatment sometimes affects the values to a marked degree. Tearing strength (an empirical property in paper manufacturing) can sometimes be interpreted to reflect the general nature of the fiber present in the paper as well as the beater treatment to which it has been subjected. For example, the test is somewhat dependent upon fiber length and cementing action.

The starch additives in general produced no significant increase in tearing strength but rather the opposite occurred. Though in the case of additives A2, A4 and A5 a significant increase in bond strength has occurred (shown by an increase in the tensile strength) indicated by a lowering of tear results. The remaining additions do not fit this pattern.

Fungus Resistance

Under normal conditions, paper is not a good medium for the growth of molds.[14] Paper contains an abundance of food substance, but this food is not readily available because of the low moisture content and because most of the moisture is held by the fibers. The presence of such substances as starch, casein, glue, etc. does not mean that the paper will mold, because here again, the moisture content of the paper is usually so low that no growth takes

TABLE IV

PROPERTIES OF HAND SHEET FORMULATIONS
BASIC FORMULATION (CONTROL) PLUS PERCENT ADDITIVE

Property	TAPPI Test Method	0 (Control)	1% Additive #1	3% Additive #1	5% Additive #1	1% Additive #2	3% Additive #2	5% Additive #2	1% Additive #3	3% Additive #3	5% Additive #3	1% Additive #4	3% Additive #4	5% Additive #4	1% Additive #5	3% Additive #5	5% Additive #5
Tensile Breaking Strength, dry, lbs.	T404	6.4	7.6	9.5	6.9	8.7	10.7	11.9	10.2	11.4	12.5	8.3	8.1	10.6	9.3	9.4	11.3
Tensile Breaking Strength, wet, lbs.	T456	.2	.25	.3	.3	.15	.4	.3	.3	.4	.7	.3	.9	1.6	.6	.3	.4
Oil Resistance, sec.	T462																
Castor Oil		2	18	13	16	9	7	9	9	18	9	18	20	19	15	19	20
ASTM #3 Oil		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
MIL-L-46002 Oil		0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Tear, grams	T414	113	97	96	108	90	79	77	86	74	80	112	97	82	96	87	77
Fungus Resistance	T487	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail
pH - Original	T435	6.2	6.3	6.4	6.5	6.8	6.5	6.3	6.5	6.5	6.4	6.2	6.3	6.2	6.4	6.8	7.2
Aged (70 hrs @ 212°F)		5.4	5.2	5.3	5.4	5.8	5.8	5.6	5.4	5.6	5.6	5.6	5.4	5.4	5.6	5.8	5.2
Folding Endurance, folds	T423																
Original		144	293	397	309	428	1098	620	865	926	1222	280	320	783	319	604	1153
Aged (70 hrs @ 212°F)		104	128	173	214	507	993	547	501	1218	1476	251	453	938	229	1061	1219

Property	10% Additive #6	20% Additive #6	10% Additive #7	20% Additive #7	5% Additive #8	20% Additive #8	5% Additive #9	20% Additive #9	5% Additive #10	20% Additive #10	0.3% Additive #11	0.6% Additive #11
Tensile Breaking Strength, dry, lbs.	7.3	6.7	6.4	7.7	6.2	7.9	10.0	11.0	6.0	5.7	7.9	7.0
Tensile Breaking Strength, wet, lbs.	2.1	2.3	.6	.7	.45	.4	3.0	4.0	.75	1.3	.35	.3
Oil Resistance												
Castor Oil	0	0	11	14	2	2	2	3	3	3	25	45
ASTM #3 Oil	0	0	0	0	0	0	0	0	0	0	5	10
MIL-L-46002 Oil	0	0	0	0	0	0	0	0	0	0	0	0
Tear, grams	102	79	123	108	110	116	91	82	114	106	107	182
Fungus Resistance	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail	Fail
pH - Original	5.3	5.5	4.8	4.8	5.7	5.9	6.2	6.2	5.9	5.9	6.1	6.1
Aged (70 hrs @ 212°F)	5.0	5.2	5.1	4.9	5.4	5.5	5.0	5.2	5.3	5.4	5.0	5.5
Folding Endurance, folds												
Original	63	130	20	49	165	190	948	1434	241	9	97	88
Aged (70 hrs @ 212°F)	16	46	46	131	153	196	1587	1756	201	176	202	81

place under normal conditions. However, papers are sometimes used and stored at high moisture content so that mold growth is favored. Papers which come into contact with wet earth or other places where microorganisms are prevalent lose their strength rapidly, probably due to attack on the cellulose.

Paper hand sheets containing additives A1 through A11 had no resistance to fungus. Agar plate tests and actual soil burial tests gave identical results. Fungus susceptibility of starch treated papers increased with increasing concentration of starch (1, 3 and 5%). Additive A12 (a water soluble fungicide) did not effectively inhibit fungus growth.

The three remaining fungicides were effective in all concentrations (1-4%) in preventing fungus growth on the base formulation hand sheets. While effective, an obvious drawback to their use is the required immersion step after the paper is already made.

pH

The acidity (or alkalinity) in paper may be caused by the presence of residual chemicals left in the pulp by the process of manufacture, by chemicals added to the paper to obtain certain desired results, by chemicals absorbed from the air or by contact with the skin when handled. The sizing of paper, wet strength treatment and dye and filler retention usually depend upon the addition of acidic materials during paper manufacture. Hence, many papers are acidic to various degrees at the time of manufacture.

As shown in Table IV, the pH of the base formulation hand sheets is on the acid side. The starches with the exception of A2 and A5 had little effect on the original pH values of the papers. The pH of the A2 starch solution was approximately 4.5 probably contributing to increased acidity of hand sheets at higher starch concentrations. The pH of A5 solutions was approximately 8, thus apparently yielding more alkaline hand sheets. For the remaining additives, the pH of the additive solution did not necessarily define the pH of the paper hand sheets. For example, both latices (with pH's in the 7.5 to 12.5 range) produced definitely acidic hand sheets while additives A8 and A10 (pH of both materials is 3) yielded slightly less acid paper.

With the exception of additive A7 all hand sheet formulations using additives A1 through A11 were more acidic after aging for 70 hours at 212°F. For many years acidity has been considered a major factor in paper deterioration. It was found that a pH of about 7 is desirable for maximum aging resistance. In the current study only a rather high percent of additive A5 produced a neutral paper which, however, aged very poorly.

Folding Endurance

Another test relating to the durability of paper is the folding endurance test. This test is a performance test and is indicative of a combination of physical properties of the paper sheet. It derives its importance and widespread use because of the frequency with which the ability to endure folding occurs in the use of paper products. It is affected by bonding strength, stretch and pliability. In general, as bursting strength (not considered here) and tensile strength increase, the folding endurance will also increase.

As shown in Table IV, all starch additives, glyoxal and kymene produced hand sheets with across the board increases in original folding endurance. However, both latices, the fluorocarbon and the alkyl ketene dimer (high concentration) produced sheets with folding endurance values even lower than those of the untreated base formulation. The rule of thumb that the folding endurance increases with increased tensile strength did not apply in the latter case.

Folding endurance results after aging 70 hours at 212°F. varied from a large decrease as in A6 (70%) to a large increase as in A10 (high concentration) (1800%). It is thought that the increases in folding endurance after aging are due to a curing effect and the decrease due to natural aging of materials not stable under the aging conditions.

Composite Hand Sheets

From the foregoing, it is apparent that paper which appears to be a simple structure actually may become quite a complex one. One property, even though important, cannot be overemphasized without loss of some property equally necessary to satisfactory performance.

In an effort to obtain an improved paper with a good balance of properties the following formulation was set up:

TABLE V

COMPOSITE HAND SHEET FORMULATION

Pulp	360 grams	)
TiO_2	40.2 grams	)
Rosin	7.2 grams	)-- base formulation
Alum	4.0 grams	)
Sta-Lok 300	1% based on pulp weight for dry breaking strength	
Kymene	5% based on pulp weight for wet breaking strength	
FX-806	1% based on pulp weight for oil resistance	

The results from hand sheets made from this formulation are shown in Table VI.

TABLE VI

COMPOSITE HAND SHEET PROPERTIES

Tensile Breaking Strength, Dry, lbs.	8.6
Tensile Breaking Strength, Wet, lbs.	3.5
Oil Resistance, seconds	
Castor Oil	>72 hours
ASTM #3 Oil	>72 hours
MIL-L-46002 Oil	2 - 3
Tear, grams	88.4
Fungus	Fail
(after immersion in fungicides A13-15)	Pass
pH (original)	5.8
(aged)	5.3
Folding Endurance (original)	635
(aged)	865

It will be noted in Table VI that the dry breaking strength, tear, folding endurance and pH properties are somewhat lower than the individual formulations from which the composite was taken. This is due to the fact that many of the variables are interrelated and certain additives tend to cancel each other out or compete during certain reactions, i.e., the starch (A3) and the water soluble polymer (A8) did not give as good results when added together in the composite formulation as they did in separate formulations. To pass the fungus test, the additional step of immersion in the nonaqueous fungicide solutions was still required.

CONCLUSIONS

The chief contribution of the starch additives over the base formulation properties appeared to be an increase in tensile breaking strength and folding endurance.

The two latices produced paper formulations with the poorest folding endurance and pH of any of the additives tested. These papers, however, were more extensible than any of the other papers tested.

Additive A11 (fluorocarbon) formulation had the best oil resistance of any of the formulations tested even though the alum of the formulation interfered with its deposition on the pulp fibers.

The three nonaqueous fungicide treatments were effective in preventing fungus growth (in all percentages tested, 1% - 4%) however, their use required an additional dipping procedure.

Compared to the base formulation, incorporation of additives into the composite formulation improved some properties, i.e., tensile breaking strength and folding endurance and downgraded others, i.e., tear and pH.

RECOMMENDATIONS

That other internal sizing agents, not requiring alum (i.e., fortified rosin sizes) be investigated for improving the oil resistance of paper.

It is recommended that water soluble fungicides be investigated for addition to the pulp prior to sheet formation.

It is also recommended that the techniques and methods used in this study be utilized in the upgrading of Specification MIL-P-10831 Target Paper.

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13. ABSTRACT

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The nonaqueous solutions of fungicides provided excellent fungus resistance but also required an extra immersion step after the paper had been formed.

The best water resistance was obtained utilizing a water soluble polymer (Kymene 557).

In a "composite" hand sheet formulation (made from the base formulation plus additives giving the best individual properties) some properties were improved, i.e., tensile breaking strength and folding endurance and some were downgraded, i.e., tear and pH. (U) (Author)

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Kraft paper						
Fungus Resistance						
Oil Resistance						
Heat Aging						