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AN INVESTIGATION OF NYLON FIBER AS A PAPERMAKING MATERIAL

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Dissertation Submitted to the Faculty

of

Western Michigan College

by

Robert J. Thiessen

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In Partial Fulfillment of the Prerequisites for the Degree

of

Bachelor of Science

June 1956

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Abstract

Investigations were made to determine whether or not a practical method of bonding nylon-wood blend paper could be devised in which advantage is obtained from the strength qualities of the nylon fibers in the finished sheet. Two unsatisfactory methods were tried before a satisfactory method was found. By this method 25- to 50-per cent nylon content sheets were dipped in a five- to ten-per cent solids water emulsion of a polyamide resin. The sheets were then dried, free of surface support, and then heat pressed to complete the bonding. Paper of high strength and durability was thus produced.

AN INVESTIGATION OF NYLON FIBER AS A PAPERMAKING MATERIAL

Introduction

NYLON, A PROMISING PAPERMAKING FIBER

Nylon staple, a synthetic fiber with high strength, toughness, abrasion resistance and flex endurance, shows promise as a papermaking fiber. At the present it is too expensive to compete with cellulose fibers in the fields where the properties of cellulose paper suffice. But in fields where the strength properties of cellulose paper are not sufficient, nylon fiber paper represents valuable potential products for the paper industry. All that is necessary to realize these potential products is a practical method for making nylon or nylon blend paper which takes full advantage of the strength characteristics of the nylon fibers. Once such a method is devised, the large tonnage of nylon staple for papermaking purposes, as a result, should lower the unit price of the nylon staple and so increase its potential as a papermaking material.

FIBER-TO-FIBER BONDING

Because of the haphazard arrangement of the fibers in paper, there must be bonding at their crossover points in order to obtain structural strength in their network.

With cellulose fibers, the fiber-to-fiber bonding is quite handily effected. It is necessary only to beat the fibers mechanically in the medium of water, form a web of the water suspension of the fibers, and

then remove the water. Because of the fibrillous structure of the cellulose fibers, beating action frays the fibers, thus greatly increasing the contact area of the fibers. Also, the chemical relationship of cellulose with water produces hydrogen bonding of the fibers at their points of contact upon removal of the water. The main disadvantage of this type of bonding is that the addition of water destroys the hydrogen bonding.

With nylon the problem of fiber-to-fiber bonding must be solved differently. Nylon fibers do not fray upon mechanical beating and they do not form hydrogen bonds upon the simple removal of water. Also, in order to take advantage of the strength properties of nylon fiber, a much stronger and more permanent type of bond would be necessary.

Two methods of synthetic fiber bonding have been described by James K. Hubbard, Franklin H. Koontz, John R. McCartney, and Robert A. A. Hentschel¹. The first they call "salt bonding" in which the bonding agent comes from the fiber itself. The second they call "synthetic polymer bonding" in which a second bonding constituent is added to the waterleaf paper and serves to hold the fibers together.

SALT BONDING

In salt bonding, they make use of the fact that water solutions of certain salts at high concentrations are swelling agents or solvents for the plastic fibers but become nonsolvents when dilute. When a waterleaf paper of the pure fiber is impregnated with a dilute solution of an appropriate salt and the water subsequently evaporated, the salt solution concentrates, and tends to localize preferentially at the cross-over points of the fibers by capillary action, ultimately becoming

sufficiently concentrated to lead to solution or gelatinization of a small portion of the fiber surface at these crossover points. When the water is completely evaporated, the polymer reprecipitates and the bonding is complete. The excess salt is then washed off. The salts mentioned for use with nylon are calcium bromide, lithium bromide, zinc bromide, calcium thiocyanate, and magnesium thiocyanate.

SYNTHETIC POLYMER BONDING

In synthetic polymer bonding, nylon waterleaf paper was impregnated with a five- to ten-per cent dispersion of a specifically formulated polyamide binding polymer. On evaporation of the water, the polymer tends to concentrate at the crossover points of the fibers by capillary action, being deposited at these points and remaining there after removal of the carrier. The bonding is then completed by heat and pressure.

Mr. Emery, of Du Pont's Textile Fibers Department, has mentioned another method of synthetic polymer bonding called "thermoplastic bonding"². With this method, fibers of the resin, having a lower melting point than the fibers to be bonded, are blended with the base fibers in the slurry before sheet formation. Bonding is then accomplished by hot pressing of the dried resultant sheet at a temperature sufficient to melt the bonding fibers but not the base fibers.

With both salt bonding and synthetic polymer bonding, extremely high flex endurance and tear strength were obtained¹. Sewn joints and heat sealed joints showed about 70 per cent of the base strength of the sheets.

NYLON-WOOD PULP BLENDS

The foregoing experiments pertain to paper made of 100 per cent

nylon fibers. Knowledge of the performance of nylon fibers blended with wood pulp is also of practical value.

Tests were made on a series of sheets prepared with varying percentages of nylon using no bonding agent³. When the strength properties of the sheets were plotted against the nylon content, it was found that the strength decreases very rapidly and approaches a low asymptote as the nylon content is increased.

When the same experiment was repeated using "Zytel resin" as a bonding agent, the strength properties were found to decrease from the straight wood pulp sheet, go through a minimum at an intermediate nylon content, and then rise rapidly to a very high maximum at 100 per cent nylon. It was reasoned that this was to be expected since the bonding agent apparently acts only on the nylon and does not begin to exert its influence until sufficient nylon fibers are present in order to form a continuous network which can be bonded together³.

The Zytel resin is a Du Pont product and can be applied as a beater additive, or by dipping the dried sheets in the resin emulsion and then re-drying. If used as a beater additive, the resin is precipitated with alum. Best results were obtained by the dipping method.

FIBER DISPERSION

Nylon, like most of the other synthetic fibers, is hard to disperse properly. It resists wetting and foams when stirred vigorously, floating a large portion of the fibers in the foam. This condition is, of course, highly undesirable for papermaking. In order to be mixed evenly with other fibers and in order to be spread evenly on the wire of the paper-machine, the nylon fibers must be uniformly dispersed throughout the

stock. Also, the fibers should be uniformly dispersed with lack of foam or bubbles for proper circulation through pumps and other circulation devices. Proper dispersion is therefore important to the success of nylon fiber as a papermaking material.

Experiments with various synthetic fibers have been made on paper machinery using sodium carboxymethylcellulose as a fiber suspension and bonding agent⁴.

In the experiments on pure nylon fiber, sodium carboxymethylcellulose was also used to form the fiber suspensions prior to the sheet forming. After the sheets were made, the sodium carboxymethylcellulose was washed out with water.

In a set of experiments with Vinyon HH staple⁵, it was found that "Triton K-60", a cationic wetting agent of the alkyl quaternary ammonium type, dispersed the fibers satisfactorily but caused considerably increased foaming. When "Nopco KK", an anti-foam agent, was used in conjunction with it, a good fiber dispersion with no foam resulted. The optimum concentrations for both of the agents were from 0.01 per cent to 0.05 per cent consistency.

FIBER LENGTH

Experiments have shown² that short fibers make a better slurry and give a better fiber formation in the finished sheet, while long fibers improve the physical properties of the finished sheet. Fiber length of about one-quarter inch appears to give the best balance between physical properties of the finished sheet and ease of handling.

The nylon fibers used in my experiments were one-quarter inch staple which the Du Pont de Nemours and Company supplied. The fibers

had been treated with a special finish to promote their dispersion in water.

PURPOSE AND BASIC SCHEME OF PROCEDURE

After preliminary experiments made on various combinations of nylon and bleached kraft fibers, I decided to concentrate my efforts on experimenting with bonding methods of fiber mixtures containing up to 50-per cent nylon with wood fibers. Mixtures of over 50-per cent nylon content resulted in sheets which, while wet, were so weak that they seemed impractical to make on a standard paper machine. Although the ultimate strength of the finished sheet would be much higher for higher nylon contents³, the presence of at least 50-per cent wood fibers resulted in sheets that seemed to have enough wet web strength to be capable of being handled without too much difficulty on the wet end of a paper machine.

Because the nylon fibers supplied by Du Pont de Nemours and Company were treated for ready dispersion, the problem of dispersing the fibers in slurries was solved for me.

Also, because synthetic polymer bonding methods are proving to be more practical than salt bonding², I concentrated my investigations on the use of synthetic polymers in bonding my paper samples.

Mainly, my experimental work was centered upon investigating the effects of bonding sheets of varying nylon content with synthetic polymers. "Zytel 61" nylon resin, which was recommended by Roy P. Whitney, Research Associate at The Institute of Paper Chemistry, was supplied by General Dispersions, Inc. in the form of "Genton 110", an aqueous dispersion of the resin. This was investigated as to its bonding effects,

applied both to fiber slurries and to dried, unbonded sheets.

"Plaskon", a urea-formaldehyde resin, was also tried as bonding agent in conjunction with sodium carboxymethylcellulose. The two were precipitated together in the fiber slurries.

In all cases, bleached kraft pulp, which was supplied by The Kalamazoo Vegetable Parchment Company, was used as the wood fiber stock in my investigations. The KVP Company also supplied the "Plaskon" resin for the thesis work.

Equipment

A Valley laboratory beater was used in all cases to prepare the wood pulp stock, using a full weight load on the counterpoise.

Slow speed laboratory stirrers were used to disperse the nylon fibers in the wood pulp stock and also to provide agitation to the stock during the addition of resins and their precipitation with alum.

British sheet molds and Noble and Wood sheetmaking equipment were used to form the test sheets.

A Carver laboratory press was used to press sheets at an elevated temperature. A standard sheet press and pump were used for pressing at room temperature.

Drying rings and plates were used to dry the sheets at room temperature. A Noble and Wood steam cylinder drier was used for quick drying at 120 degrees centigrade.

The testing equipment used were a Mullen bursting strength tester, an M.I.T. folding endurance tester, an Elmendorf tearing tester and a Schopper tensile breaking strength tester.

Experimental Procedure

STRENGTH VS. UNBONDED NYLON CONTENT

Sheets of nylon fiber and kraft pulp were made with nylon contents of zero per cent, 12.5 per cent, 25 per cent, and 50 per cent. The nylon fibers were mixed with the kraft stock, which had been beaten separately to about 300 Canadian Standard Freeness, until uniformly dispersed and sheets were then made from the slurry in a British sheet mold. The sheets were pressed for five minutes at 50-pounds per square inch and conditioned in drying rings in a constant humidity room. They were then tested for physical strength characteristics.

UREA FORMALDEHYDE RESIN WITH CMC PRECIPITATED IN FIBER SLURRIES

Sheets of from zero to 25 per cent nylon fiber content with "Plaskon" resin and sodium carboxymethylcellulose as bonding agents were formed on a Noble and Wood sheet mold. The sheets were dried on the steam drying cylinder at 120 degrees centigrade, conditioned in a constant humidity room and tested for physical strength characteristics.

The bonding was accomplished by precipitating the resins in the fiber slurries at a pH of 4.5 with alum. Three per cent "Plaskon" and five per cent CMC, as based on dry fiber weight, were used.

The use of CMC in higher concentrations than five per cent of fiber weight resulted in too slow draining stock. Three per cent urea-formaldehyde resin was used because it is generally considered the upper limit for retention of softness of a finished sheet.

"GENTON 110" RESIN PRECIPITATED IN FIBER SLURRIES

Genton resin was precipitated in fiber slurries containing from zero

to 25 per cent nylon fibers. Ten per cent and 20 per cent resin based on sheet weight were used in these experiments, being precipitated by alum at a pH of 4.7.

Sheets were formed, pressed, and dried on Noble and Wood sheet-making equipment. The dryer was operated at 120 degrees centigrade. The sheets were then conditioned in a constant humidity room and tested for physical strength characteristics.

Remnants of the sheets containing 20 per cent resin (as based on sheet weight) were pressed for 30-seconds at 160 degrees centigrade in a Carver laboratory press at a pressure of 1,000-pounds per square inch. These sheets were then conditioned in the constant humidity room and tested for physical strength characteristics.

"GENTON 110" RESIN APPLIED BY DIPPING OF UNBONDED SHEETS

In one set of experiments, sheets made on a British sheet mold containing from zero to 25 per cent nylon fibers were pressed for five-minutes at 50-pounds per square inch and then dried with drying rings and plates. They were then dipped into a five percent solids emulsion of Genton 110 resin, allowed to drain, and then dried in drying rings (minus the plates). They were then pressed at 160 degrees centigrade in a Carver laboratory press under a pressure of 1,000-pounds per square inch. Then they were conditioned in a constant humidity room and tested for physical strength characteristics.

In another set of experiments, sheets containing zero, 25, and 50 per cent nylon were formed, pressed, and dried on Noble and Wood sheet-making equipment. A set of pure kraft sheets were set aside to use as a control set. The rest were dipped in a ten percent solids emulsion

of Genton 110 resin, allowed to drain, and dried in drying rings (minus the plates). These were then pressed at 160 degrees centigrade in a Carver laboratory press under a pressure of 1,000-pounds per square inch. All of the sheets were then conditioned in a constant humidity room and tested for physical strength characteristics.

Results and Discussion

The tests made on the unbonded sheets (unbonded with respect to the nylon portion) showed that the physical strength of the paper falls off rapidly as the nylon fiber content is increased (as indicated in Table I). This can be explained by the fact that the nylon fibers hinder the fiber-to-fiber bonding of the cellulose fibers and do not supply any bonding themselves to the paper structure. While the tear factor shows an impressive increase up to about 25 percent nylon, the decrease in folding endurance, burst factor, and breaking length greatly outweigh that increase as far as durability is concerned.

Because the tear resistance of the unbonded sheets increased with nylon content, an increase, by itself, of tear resistance of the bonded sheets with increasing nylon content can not be considered as an indication of increase in overall physical strength of the paper.

Nylon fibers, used with the urea-formaldehyde resin and carboxymethylcellulose as a bonding agent, did not show any impressive gains in strength over the use of nylon fibers with no binder (Table II). This might be explained by the chemical difference of the resins as compared to nylon.

My attempts in bonding nylon fiber content sheets with "Genton 110"

by precipitating the resin in the fiber slurries did not produce very good results (Tables III and IV). The decrease in folding endurance and tensile strength indicate that the synthetic polymer bonding was insufficient to make up for the loss of cellulose fiber-to-fiber bonding caused by the presence of the nylon fibers. I believe that the poor bonding shown by this precipitation method is caused by the fact that the resin is spread more or less over the whole surface of the fibers—not concentrated at the crossover points. Therefore, in order to obtain a given degree of bonding at the crossover points of the fibers, relatively more resin must be used.

The same resin dispersion used as a bonding agent by dipping the nylon content sheets in it produced much better results (Tables V and VI). Although the tensile strength continued to decrease with increasing nylon fiber content, the folding endurance and tearing resistance suddenly jumped up at about 25 per cent and greater nylon fiber content.

The amount of resin retained in the sheet has much bearing upon the balance of strength characteristics of the sheet. At 25 per cent nylon fiber content, the folding endurance was doubled by doubling the consistency of the resin dispersion and the tensile strength was also increased slightly while at the same time the tearing resistance was decreased to almost one-half.

At 50 per cent nylon fiber content using a ten per cent solids dispersion of the polyamide resin, an extremely tough sheet was obtained. The folding endurance was almost fifty times as great as the untreated kraft base paper. The tearing resistance was over six times as high as the best untreated kraft sheet that I made.

The resin retained in the dip bonded sheets ranged near 20 per cent,

more or less, of the total sheet weight. Thus, it is evident that the dip bonding was more efficient insofar as the amount of bonding agent used was concerned.

Although there is some degradation of the cellulose portion of the sheets in heat pressing then at 160 degrees centigrade, as shown by the decrease in folding endurance of the treated kraft sheet as compared with the untreated kraft sheet (Table VI), the increase of strength due to the successful bonding of the nylon content may far overshadow it.

Table VI also shows a great increase in wet strength with the use of the polyamide resin and nylon fibers in the paper.

Conclusions

The investigations showed that nylon fibers used in conjunction with a polyamide resin dispersion may be used with wood pulp to produce a sheet of unusually high strength and durability.

The polyamide resin appears to be best applied by dipping the nylon content waterleaf paper in a five- to ten-per cent water dispersion of the polyamide resin, drying the sheet out of contact with adsorbant or absorbant surfaces, and then heat pressing the bonded sheet at a temperature just above the melting point of the resin, and below the melting point of the base nylon fibers.

The best nylon fiber content for good balance between strength of the finished sheet and ease of handling appears to be from 25- to 50-per cent by weight of the waterleaf paper, using one-quarter inch nylon staple. The higher the nylon content, the higher the strength of the finished sheet, and the lower the strength of the wet web.

June 2, 1956


Robert J. Thiessen

APPENDIX

All tests were made in accordance with TAPPI Standards. The results are expressed in average values.

Table I. Properties of Unbonded Sheets

% Nylon Fiber	Fold, cycles	Burst Factor	Tear Factor	Stretch, %	Breaking length, meters
0	692	77	94	3.45	9150
12.5	350	64	163	3.25	6820
25	160	42	240	2.75	5610
50	1	16	177	2.70	1410

Table II. Properties of "Plascon" and GMC Slurry Bonded Sheets

% Nylon Fiber	Fold, cycles	Burst Factor	Tear Factor	Stretch, %	Breaking length, meters
0	500	66	69.2	2.1	10300
5	454	62	123	2.4	9400
10	268	58	182	2.2	8380
15	241	55	200	2.25	8280
20	152	48	235	2.0	6660
25	110	41	284	2.5	6160

Table III. Properties of 10% "Genton 110" Slurry Bonded Sheets

% Nylon Fiber	Fold, cycles	Tear Factor	Stretch, %	Breaking Length, meters
0	908	96	2.5	10550
5	883	142	2.35	8840
10	577	167	2.35	8330
15	655	193	2.5	7370
20	486	232	2.8	7070
25	160	232	1.9	5660

Table IV. Properties of 20% "Genton 110"
Slurry Bonded Sheets

% Nylon Fiber	Fold, cycles	Tear Factor	Stretch, %	Breaking Length meters
0	1210	93	2.8	10450
5	778	129	2.6	9400
10	447	160	2.5	8490
15	320	188	2.4	7230
20	470	240	2.9	6060
25	455	272	2.9	5710
0	900	95.5	3.15	11350
5	417	127	2.25	9450
10	417	150	2.0	7180
15	326	182	2.05	7930
20	435	212	2.75	7380
25			3.2	8080

AFTER
PRESSING

Table V. Properties of Sheets Dip Bonded in
5% Solids "Genton 110" Resin (Heat Pressed)

% Nylon Fiber	Folds, cycles	Tear Factor	Breaking Length meters
0	666	103	12000
5	698	142	12700
10	798	172	11860
15	693	260	9750
20	617	292	9300
25	3000	467	8180

Table VI. Properties of Sheets Dip Bonded in
10% Solids "Genton 110" Resin (Heat Pressed)

% Nylon Fiber	Fold, cycles	Tear Factor	Stretch, %	Breaking Length, meters condit'd	wet
0 (not bonded or heat pressed)	920	67	2.2	12000	580
10	717	74	2.3	12500	1520
25	6000	254	2.4	10450	2420
50	43200	616	4.6	6770	2520

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