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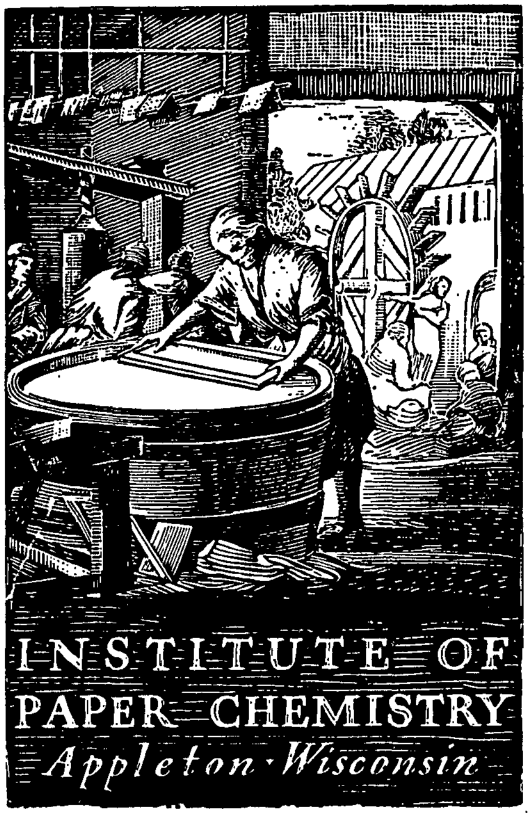

FUNDAMENTAL STUDY OF ADHESION OF CORRUGATED BOARD

Project 2696-4

Report Three

A Progress Report

to

FOURDRINIER KRAFT BOARD INSTITUTE, INC.

November 10, 1970

FORM LETTER: FKI Mailing

THE INSTITUTE OF PAPER CHEMISTRY
Appleton, Wisconsin 54911
Phone 414/734-9251

November 30, 1970

Project 2696-4

TO:

Enclosed is a copy of Progress Report Three, Project 2696-4, entitled "Fundamental Study of Adhesion of Corrugated Board," dated November 10, 1970. Report Three summarizes results obtained in efforts to optimize the surface chemical and surface physical properties of corrugating medium for adhesion.

Experimental medium having a range in receptivity, roughness, and porosity was prepared on Institute pilot equipment. The medium was subsequently corrugated using a conventional two-step starch adhesive and a modified one-step adhesive. While no difficulties were experienced in the corrugating operation, the internal strength of the medium proved to be weak and failure in the pin adhesion test occurred within the medium which was corrugated at speeds of 200 f.p.m. and higher. Hence, satisfactory analysis of the effect of surface properties on adhesion could not be made although some useful information was obtained from board corrugated at low speed in which case less adhesional failure occurred on the smooth side of the medium than on the rough side. Also, a trend for lower incidence of adhesional failure was found among the receptive surfaces than among the nonreceptive surfaces when using the conventional adhesive. The modified starch adhesive did not provide a consistent advantage. The fact that no adhesional failure occurred at higher corrugating speeds was interpreted to mean that surface properties may become of secondary importance when the internal bonding strength of the medium is low.

Exploratory efforts were subsequently made to improve internal bonding strength of the experimental medium by incorporating higher percentages of kraft pulp and, while acceptable results were not obtained, available information from 100% kraft medium indicated an advantage for smooth surfaces at corrugating speeds of 50, 200, 400, and 600 f.p.m.

Since optimization of surface properties for adhesion have not been fully attained, recommendations for continued work are presented in the report.

Yours very truly,

R. C. McKee, Chairman
Container Section

RCM/mkm
Enclosure

GENERAL

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

FUNDAMENTAL STUDY OF ADHESION OF CORRUGATED BOARD

SUMMARY

In an effort to optimize the surface chemical and surface physical properties of corrugating medium for adhesion, experimental medium was prepared and corrugated on Institute pilot equipment. The medium was prepared on the continuous web former which permits the formation of pronounced smooth and rough surfaces. Receptivity was varied through the internal addition of low percentages of sizing agent and porosity was varied in one case by changing the extent of beating.

Four series of pilot runs were made in the course of the program. The first series utilized commercial semichemical pulp with 5% of long-fiber kraft but the medium produced was found to be nonuniform and was not tested further. The second series utilized 90% waterleaf broke (medium) and 10% of long-fiber kraft pulp. The resulting medium provided an acceptable range in smoothness, receptivity, and porosity and was subsequently corrugated utilizing a conventional two-step and a modified one-step starch adhesive. While no difficulties were experienced in the corrugating operation, the internal bonding strength of the medium proved to be weak and failure in the pin adhesion test occurred within the medium corrugated at speeds of 200, 400, and 600 f.p.m. Hence, satisfactory assessment of the effects of surface properties on adhesion could not be made. However, some information was obtained from board corrugated at 50 f.p.m. Without exception, less adhesional failure occurred on the smooth side of the medium than on the rough side in cases where such comparisons could be made. Also, a trend was indicated for lower incidence of adhesional failure among the receptive surfaces than the nonreceptive surfaces when conventional adhesive was utilized. The modified

adhesive formulation which contained some highly swollen starch granules but no carrier did not produce a consistent advantage over the conventional adhesive in the limited tests available. The fact that failure occurred within the medium among board corrugated at speeds in excess of 50 f.p.m. was taken as an indication that the surface properties of the medium and some properties of the starch adhesive may be of secondary importance when the internal bonding strength of the medium is low.

The third and fourth web former trials involved the preparation of unsized medium from furnishes comprised of 1) a 70:30 blend of broke and long-fiber kraft, and 2) 100% long-fiber kraft. These trials were made in an effort to produce medium with improved internal bonding strength. Failure in the pin adhesion test occurred primarily within the medium prepared from the 70:30 blend whereas some failure occurred at the medium-adhesive interface in the case of the 100% kraft medium. The smooth side of the kraft medium showed less adhesional failure than the rough side at all corrugating speeds.

Since the goals of the current program were not fully attained recommendations for further work are included in this report.

INTRODUCTION

Progress Report Two summarized results obtained in an examination of starch adhesive component penetration into calendered corrugating medium. The starch- and water-component penetration and the physical condition of the starch at the surface and within the medium were of primary concern in this study. The volume of adhesive accepted by the medium in short-time intervals was found to depend upon the receptive nature of the medium. However, regardless of the receptive character of the medium, an initial classification of adhesive components occurred in the application process wherein starch was rejected in favor of water (liquid). A further classification of starch and water occurred as a function of depth of penetration wherein most of the starch was retained within 1.2 mils of the surface whereas water (liquid) penetrated in greatest amounts beyond the 1.2-mil depth. Hence, water migrated away from the uncooked starch in all cases leaving the starch in a "starved" condition at the surface. The nonreceptive medium retained a higher proportion of water with the starch at the surface but the total amount of adhesive accepted by the nonreceptive medium was notably lower than that accepted by the receptive substrate.

Examination of the treated medium under the microscope revealed that the uncooked starch was held primarily at or near the surface although some granules were lost in large depressions in the medium surface. Most of the starch granules evident in the samples were small enough to enter surface cavities and thereby become unavailable for bonding. Considerable dispersed starch was associated with the uncooked starch in the surface regions but some of the dispersed material penetrated away from the granules into the interior of the medium.

The aforementioned results were interpreted to mean that neither a highly sized nor a completely wettable medium would provide optimum adhesion from the reference starch adhesive. It was postulated that what is needed in so far as the medium is concerned is a reasonably smooth surface but one which has some sizing so that it accepts the adhesive uniformly and does not lose water rapidly in the surface regions. With respect to the adhesive, a starch suspension containing moderately but uniformly gelled granules was considered desirable. The present report pursues these aspects of the adhesion problem. It was the goal of this phase of the program to establish optimum surface physical and surface chemical properties for good adhesion.

EXPERIMENTAL

PRELIMINARY STUDIES

Preliminary laboratory tests were conducted to establish approximate conditions for producing corrugating medium having a range in water receptivity. It was planned to subsequently produce sufficient medium at each condition to utilize on the Institute's corrugator. Of particular importance was the matter of providing a range in receptivity at a forming pH of 7.5-8.0. Rosin-alum sizing cannot be utilized under these conditions and alkyl ketene dimer (Aquapel) is generally less effective in this type of furnish. Surface treatments designed to produce sizing were not considered appropriate in this case because of possible influence on porosity and surface bonding strength.

Three series of handsheet preparations were made during the course of the experimental program. The first of these utilized a commercial furnish comprised of 92% semichemical pulp and 8% of cuttings. This furnish was used in combination with 5% of freshly beaten softwood unbleached kraft pulp. The freeness of the blend was approximately 500 cc. C.S.F. The second series utilized waterleaf broke (medium) to which was added 10% of long-fibered kraft pulp. The freeness of the combined pulps was 540 cc. C.S.F. The third series utilized 70% of waterleaf broke and 30% of long-fibered kraft pulp. The pulps were beaten to yield a freeness in the blend of 310 cc. C.S.F. Aquapel 360X was added in amounts ranging from 0 to 0.4% (dimer based on fiber) in combination with equal amounts of Kymene 557 as a retention aid. The Aquapel and Kymene were added separately allowing five minutes agitation between additions. Handsheets equivalent to 26-lb./1000 ft.^{2} were formed at pH 7.5-8.0 on a Noble & Wood mold and, after drying on a steam drum and aging several days at 73°F., 50% R.H., receptivity was measured by the water drop test. Results are recorded in Table I and receptivity as a function of addition level is shown graphically in Fig. 1.

TABLE I

THE SIZEABILITY OF CORRUGATING MEDIUM FURNISHES

Series No.	Set No.	Fiber Furnish	Combined Canadian Freeness, cc.	Aquapel[a] Added, % based on fiber	Kymene[b] Added, % based on fiber	Water Drop, sec.
1	1	95% Commercial furnish[c]	500	None	None	29
	2	+ 5% long-fibered kraft	500	0.05	0.05	32
	3		500	0.15	0.15	273
	4		500	0.20	0.20	479
	5		500	0.40	0.40	600+
2	1	90% Broke[d] + 10% long-	540	None	None	12
	2	fibered kraft	540	0.03	0.03	14
	3		540	0.05	0.05	69
	4		540	0.08	0.08	488
	5		540	0.30	0.30	600+
3	1	70% Broke[d] + 30% long-	310	None	None	20
	2	fibered kraft	310	0.02	0.02	43
	3		310	0.05	0.05	198
	4		310	0.08	0.08	600+
	5		310	0.20	0.20	600+

[a]Aquapel 360X.
[b]Kymene 557.
[c]Furnish was comprised of 92% semichemical and 8% clippings.
[d]A waterleaf commercial medium.

On the basis of these results it was decided to utilize 0.03 to 0.06% of Aquapel to produce a low-to-moderate level of sizing and approximately 0.3-0.4% to yield a highly water-resistant medium in subsequent pilot-scale preparations.

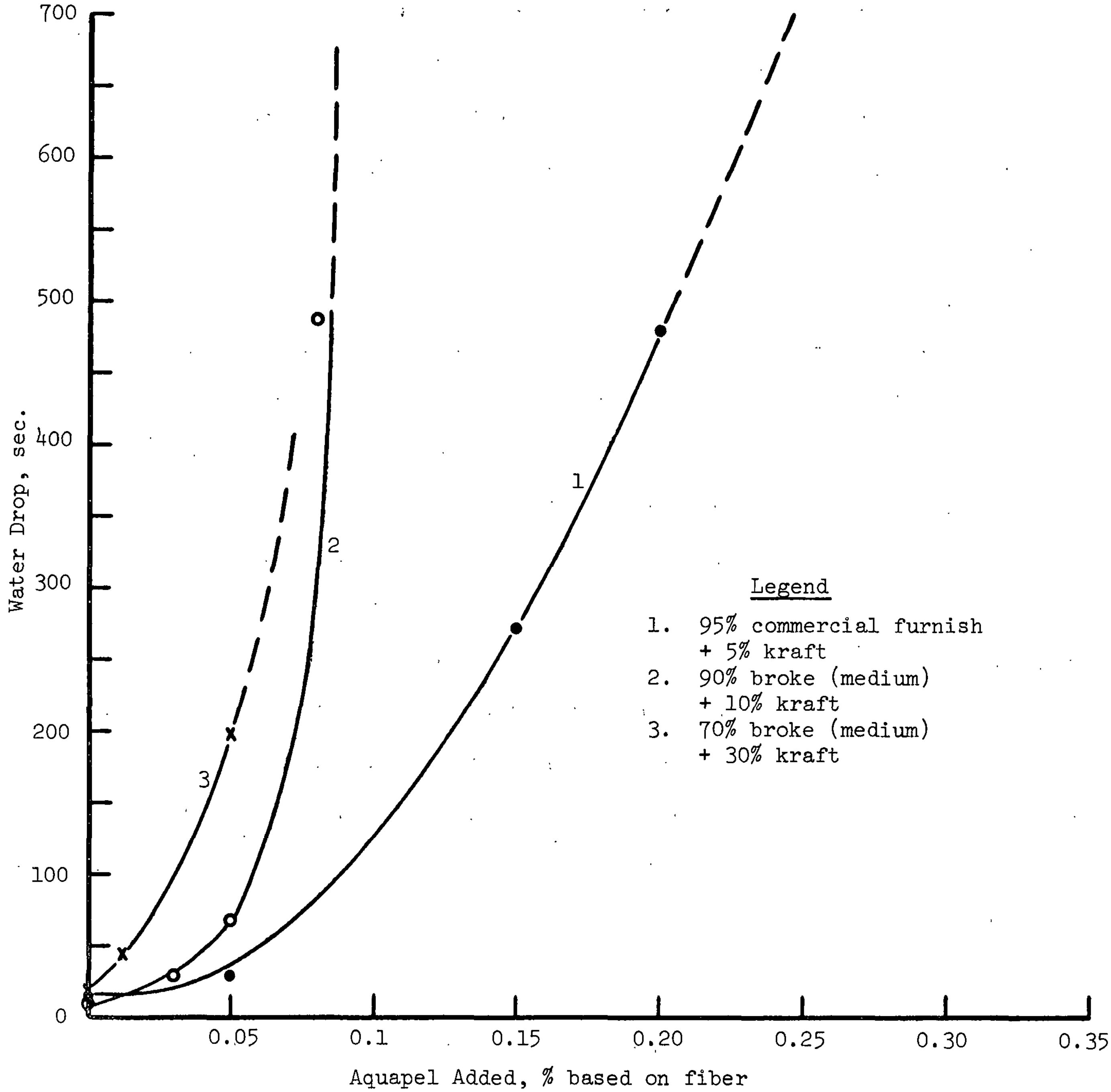

Figure 1. The Effect of Sizing Agent Addition Level on the Water Resistance of Experimental Corrugating Medium

PREPARATION AND TESTING OF CORRUGATING MEDIUM

The Institute's continuous web former was utilized to prepare 26-lb. medium for subsequent corrugator trials. The advantage of the web former in this application lies in the capability of producing a web with pronounced smooth and rough sides simultaneously. By operating at low speed most of the drying is accomplished on the first drier, thereby imparting a smooth surface to the side in contact with the drum leaving a relatively rough surface on the opposite side.

Series One

Medium was produced from a combination comprised of 95% of the commercial furnish and 5% of long-fibered unbleached kraft in the first web former series. The commercial furnish was slushed five minutes in a 5-lb. Valley beater and the kraft was beaten to 500 cc. C.S.F. The combined freeness was approximately 500 cc. Separate runs were made incorporating 0.0, 0.03, and 0.3% of Aquapel in combination with equal amounts of Kymene 557. The sizing agent and retention aid were added to the stock chest where the fiber consistency was approximately 1% and the pH was preadjusted to 7.5-8.0 with dilute sulfuric acid. Approximately 1000 ft. of medium was produced at each condition. While pronounced smooth and rough sides were evident in the experimental medium, subsequent inspection and testing revealed structural irregularities and nonuniform sizing. Since reasonable uniformity is a requisite in studies of adhesion, the medium was not corrugated or tested further.

Series Two

The second series utilized a blend of 90% of broke and 10% of long-fibered kraft. The broke and kraft were the same as those utilized in the handsheet studies. The broke was beaten to 350 and 450 cc. C.S.F. for separate web former runs and the kraft was beaten to approximately 500 cc. The combined freeness was 440-450 cc. in most runs and 360 cc. in one case designed to reduce porosity. The pH of the blended pulps was adjusted to 7.5 in the stock chest. Aquapel 360X was added in amounts of 0.08, 0.12, and 0.4% in combination with equal or lesser amounts of Kymene 557. Medium produced in the trial was conditioned to 73°F., 50% R.H., and subsequently tested for basis weight, caliper, apparent density, water drop, Bendtsen smoothness and porosity, and I.G.T. surface bonding strength. Results are recorded in Table II.

On the basis of the results in Table II, the decision was made to proceed with the corrugating operation. Two starch adhesives were used in corrugating. The first was a conventional two-step adhesive prepared according to the Stein-Hall procedure as had been used in previous studies on this project (1, 2). The second adhesive was prepared according to a modified one-step Corn Products procedure which was reported to provide more uniformly swollen starch granules compared to the two-step formulation (3). The following procedure was employed in preparing a six-gallon batch of the modified adhesive:

A starch suspension comprising 10.15 lb. of Bondcor C in 14,920 ml. of tap water was stirred in a 45-liter stainless steel drum contained in a 100-liter water bath adjusted to 115°F. When the temperature of the starch suspension reached 115°, a caustic solution comprising 142.3 g. of sodium hydroxide dissolved in 3910 ml. of tap water at 130°F. was added with stirring over a period of 4-5 min. The temperature of the suspension was maintained at 115°F. until the viscosity reached 25 sec. as measured by the Stein-Hall flow viscometer. At this point, 20.93 g. of alum dissolved in 125.6 ml. of tap water was added and stirred in for two minutes. Finally, 132 g. of borax was added with stirring.

TABLE II

PHYSICAL PROPERTIES OF EXPERIMENTAL CORRUGATING MEDIUM
Series 2

Run No.	Additives, % based on fiber	Basis Wt., lb./1000 ft.^{2}	Caliper, pt.	Apparent Density	Water Drop, sec.		Bendtsen Smoothness, ml./min.		Bendtsen Porosity, ml./min.		I.G.T. Bonding Strength, kp.-cm./sec. (in direction)	
					Wire	Felt	Wire	Felt	Wire	Felt	Wire	Felt
1	None	28.1	12.6	2.2	14	11	1200	3180+	1280	1760	75	55
2	Aquapel 360X, 0.08 Kymene 557, 0.06	28.3	12.1	2.3	84	35	1240	3190+	1290	1550	81	59
3	Aquapel 360X, 0.12 Kymene 557, 0.12	26.0	11.6	2.2	393+	262+	1550	3190+	1210	1780	64	60
4	Aquapel 360X, 0.4 Kymene 557, 0.3	26.0	11.4	2.3	600+	600+	1690	3190+	2610	2810	59	71
5[a]	Aquapel 360X, 0.12 Kymene 557, 0.12	27.6	11.5	2.4	600+	600+	1770	3190+	773	868	60	59

[a]Stock beaten to lower freeness than in Runs 1-4.

The Stein-Hall viscosity of the modified starch adhesive was 23 sec. at 100°F., the solids content was 18.7%, the pH was 12.15, and the surface tension was 56.4 dynes/cm. By way of comparison, the conventional two-step starch adhesive has a Stein-Hall viscosity of approximately 32 sec., a solids content of 20.2%, a pH of 12.2, and a surface tension of 59-60 dynes/cm. Photomicrographs of the two adhesives are presented in Fig. 2 and 3.

The experimental medium was corrugated without difficulty at speeds of 50, 200, 400, and 600 f.p.m. utilizing the two starch adhesives. Pin adhesion tests were subsequently conducted and, contrary to expectations, the medium corrugated at speeds in excess of 50 f.p.m. failed internally (decapped) in spite of satisfactory surface bonding strength. The failure in some cases resembled a ply separation deep within the medium. Since no adhesional failure was found at the higher corrugating speeds, the pin adhesion values were of little interest to the present program and were, therefore, not tabulated. Results obtained at 50 f.p.m. are recorded in Table III where the percentage of adhesional failure reflects the approximate amount of failure at the medium-adhesive interface. In cases where no adhesional failure occurred, the pin adhesion values merely reflect the strength of the medium.

Series Three and Four

Efforts were subsequently made on the web former to produce medium having improved internal bonding strength. Two web former trials were made in this direction. Series Three utilized a blend of approximately 70% broke (medium) and 30% of long-fiber kraft. The broke was beaten to 300 cc.; the kraft to 360 cc. C.S.F. The freeness of the blend was 310 cc. The fourth and final trial in the current series utilized 100% softwood unbleached kraft at a freeness of 520 cc. C.S.F. These two furnishes were utilized without additives at pH 7.8 to prepare unsized 26-lb. medium having smooth and rough sides. The experimental mediums were subsequently

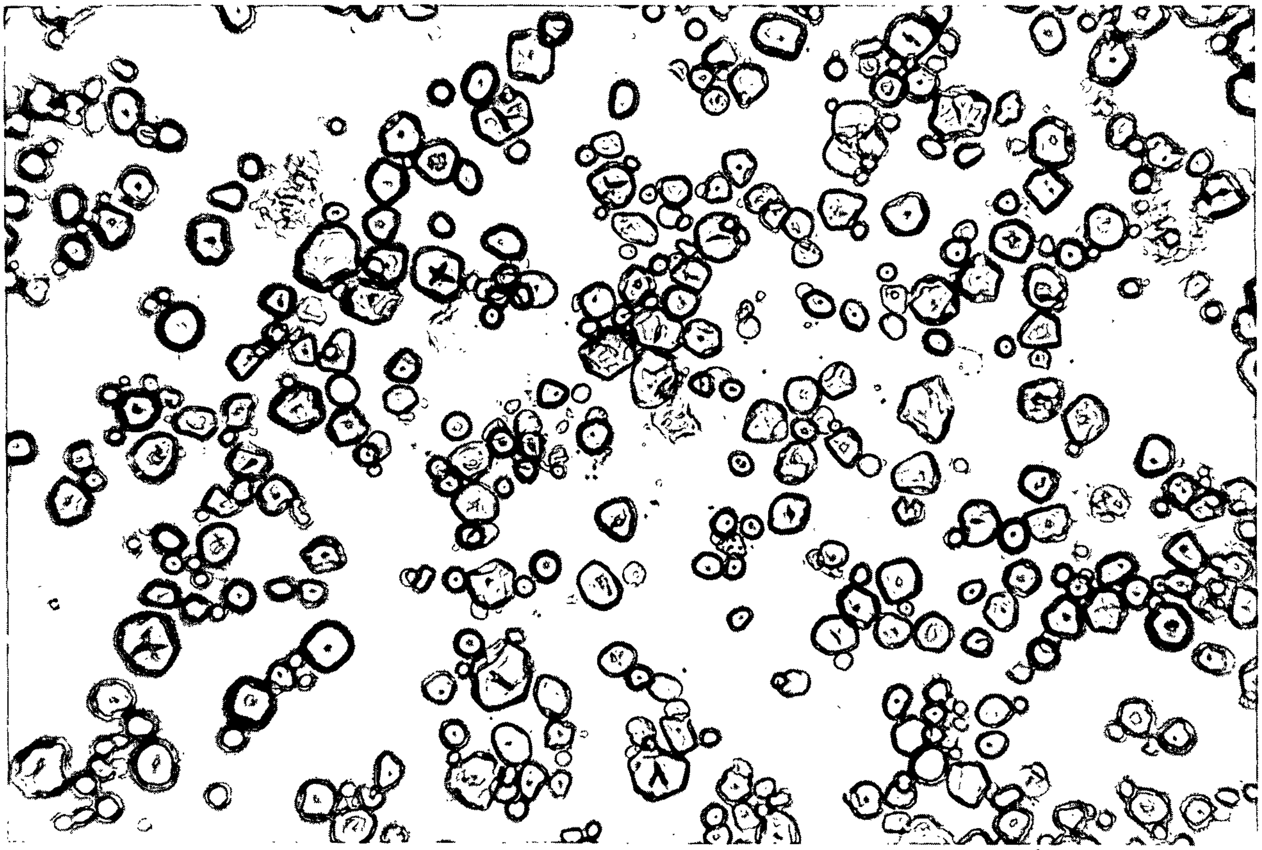

Figure 2. Conventional Two-Step Starch Adhesive, 365X

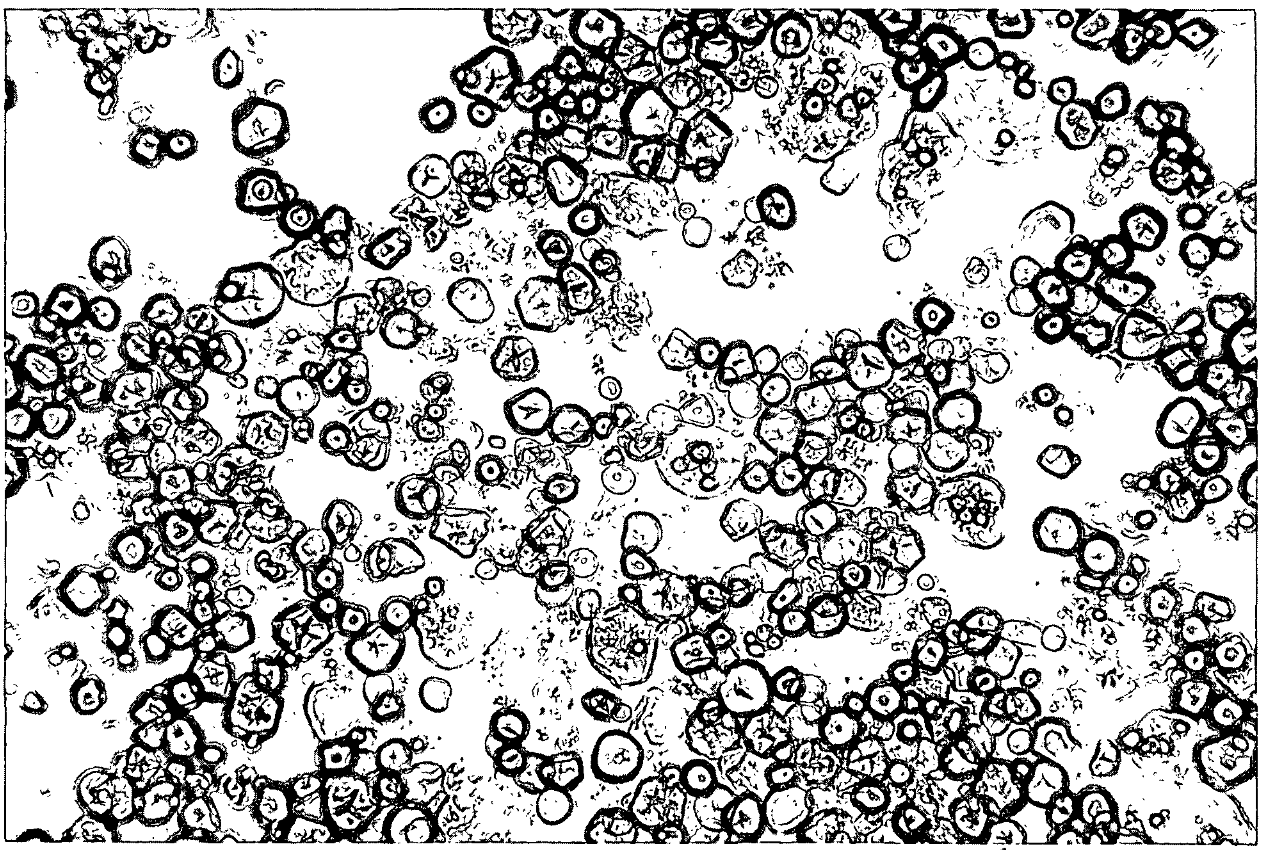

Figure 3. Modified One-Step Starch Adhesive, 365X

corrugated at speeds of 50, 200, 400, and 600 f.p.m. using the conventional two-step starch adhesive. The pin adhesion values for the 70:30 blend were low and, once again, failure occurred almost completely within the medium; hence, these results are not recorded. The locus of failure varied in board made from the kraft medium and the pin adhesion results are recorded in Table IV.

TABLE III

PIN ADHESION DATA FOR EXPERIMENTAL MEDIUM FROM SERIES 2
(Corrugated at 50 f.p.m.)

Run No.	Side	Water Drop, sec.	Porosity, ml./min.	Regular Adhesive Pin Adhesion	Regular Adhesive % Adhesional Failure[a]	Modified Adhesive Pin Adhesion	Modified Adhesive % Adhesional Failure[a]
1	Smooth	14	1280	59.2	None	57.2	None
	Rough	11	1760	56.0	None	60.4	20
2	Smooth	84	1290	59.6	None	59.8	None
	Rough	35	1550	59.0	None	58.2	20
3	Smooth	393+	1210	54.6	10	57.8	None
	Rough	262+	1780	50.0	80	54.8	None
4	Smooth	600+	2610	44.4	None	46.0	None
	Rough	600+	2810	45.2	20	42.4	None
5	Smooth	600+	773	60.2	40	64.0	None
	Rough	600+	868	62.8	50	59.2	60

[a]Failure at the medium-adhesive interface — all other failures occurred within the medium.

TABLE IV

PIN ADHESION DATA FOR UNSIZED 100% KRAFT MEDIUM

Corrugating Speed, f.p.m.	Rough Side			Smooth Side		
	Pin Adhesion, lb.	Adhesional Failure,[a] %	Cohesional Failure,[b] %	Pin Adhesion, lb.	Adhesional Failure,[a] %	Cohesional Failure,[b] %
50	50.4	40	0	52.0	0	20
200	56.2	50	40	55.2	10	50
400	45.4	10	70	45.0	0	90
600	44.0	70	10	49.8	20	80

[a]Failure at the medium-adhesive interface.

[b]Failure within medium.

Note: The Bendtsen smoothness values for the kraft medium were as follows: rough side 2440 ml./min.; smooth side 1500 ml./min.

DISCUSSION OF RESULTS

The information in Table I and Fig. 1 serve to indicate that the sizeability of the blended pulps used to prepare experimental medium improved as the percentage of kraft in the furnish increased. Aquapel provided a modest degree of water resistance at addition levels in the range of 0.03 to 0.06% and a high level of water resistance at additions of 0.1% or higher depending upon the furnish. While these results may be of little significance to practical medium production, the information was of importance to the experimental program.

The test results in Table II show a reasonable range in the receptivity, smoothness, and porosity in the experimental medium from the second series of web former trials. Water drop values fell in the desired range from slack sizing in Run No. 1 to a high level of water resistance in Runs 4 and 5. Runs 3 and 5 utilized equal amounts of Aquapel and Kymene but Run 5 produced notably higher water drop values, presumably due to lower porosity. Differences indicated in smoothness on the wire side may have resulted from changes in the release properties of the web on the first of the two driers on the web former. Reasons for the unusually high porosity in Run 4 are not apparent since no changes in machine operation were made. The I.G.T. surface bonding strength levels were of the same order as those measured previously on the commercial mediums and, hence, a problem with surface strength was not anticipated.

The photomicrographs (Fig. 2 and 3) reveal some notable differences in the conventional two-step and the modified one-step starch adhesives. The one-step formulation provided a large number of highly swollen starch granules although many granules were left in an unswollen or slightly swollen condition. Hence, the uniformity which was sought in the one-step formulation was not achieved. Mention should be made that the starch slurry was heated to 115°F. in the modified procedure, whereas the Corn Products procedure (3) specifies 100°F. The higher temperature was utilized in the present study when the viscosity remained unchanged after heating the starch slurry for 45 minutes at 100°F.

No apparent operational difficulties were experienced in the corrugating operation utilizing the experimental medium and the two starch adhesives and, while the results in Table III are limited to low speed only, several points of interest can be derived from the available data and from the locus of failure at higher speeds. First of all it becomes apparent that the surface characteristics of the medium and some properties of the starch adhesive, including degree of swelling and apparent viscosity, may become relatively unimportant if the internal bonding strength of the medium is low. This is indicated by the fact that all pin adhesion failures occurred within the medium among board corrugated at speeds of 200 f.p.m. or higher while the surface properties and porosity varied over a wide range. Since failure will occur at the weakest point, surface properties probably become of greater importance as the internal strength of the medium increases.

Results obtained with board corrugated at low speed (Table III) show that, without exception, the smooth side provided better adhesion than the rough side in those cases where some adhesional failure occurred. This also applies to the unsized 100% kraft medium in which case data are available at higher corrugating speeds (Table IV). With reference to receptivity (Table III), the more receptive surfaces tend to show lower adhesional failure than the nonreceptive surfaces when the conventional starch adhesive was employed. This excludes Run 4 because of notably lower internal bonding strength which may have resulted from the high level of sizing agent. Results obtained with the modified one-step adhesive do not support this trend but the available evidence is too limited in scope to be conclusive. Further comparisons at higher, more realistic corrugating speeds are needed.

The pin adhesion values for the unsized kraft pulp (Table IV) were unexpectedly low but some failure occurred at the adhesive-medium interface, particularly in board produced at higher corrugating speeds. Production of 26-lb. kraft medium which would fulfill the surface and porosity requirements of the current program presented operational difficulties on the web former and, as a result, it was necessary to use lightly beaten stock. The observation was made that a drop of water placed on the unsized kraft medium penetrated through the web spontaneously but was not able to spread rapidly in a lateral direction. Hence, the water drop value was approximately 75 sec. compared to 10-15 sec. for the unsized medium containing semichemical pulp. In consideration of these points it would not seem feasible to use 100% kraft in future efforts to produce experimental medium. However, combinations containing higher percentages of kraft may be suitable.

In review, while the goals set forth in the Introduction have not been fully attained, the current information tends to confirm the importance of smoothness to adhesion in the corrugating operation. However, smoothness attained by calendering a rough medium was previously shown (2) to result in a pronounced reduction in surface bonding strength which could lead to reduced pin adhesion values in spite of satisfactory surface adhesion. Smoothness attained prior to drying would presumably be more desirable. The current work also provides some evidence confirming that receptive medium produces better adhesion than nonreceptive medium when the standard two-step starch adhesive is utilized. However, optimization, in so far as receptivity is concerned, has not been achieved because the cohesive strength of the medium proved to be weaker than the adhesive strength of the bonds formed in corrugating.

FUTURE WORK

Considerable evidence has been obtained showing the advantage of a smooth medium over a rough medium in so far as adhesion is concerned. In order to provide a satisfactory base for optimization of surface receptivity for adhesion, medium of improved internal bonding strength will be required. In this direction, it is recommended that the current program be extended to permit preparation of experimental medium on laboratory equipment or possibly by modification of commercial medium. As indicated earlier, one possibility would involve higher percentages of kraft fiber in combination with semichemical pulp. Another possibility would involve modification of commercial waterleaf mediums by surface treatment with small amounts of low energy materials. As in the current series, the effect of starch adhesive modification on adhesion would be examined in an effort to provide satisfactory bonding over a wider range in the surface properties of the medium.

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THE INSTITUTE OF PAPER CHEMISTRY

Gerald R. Hoffman
Research Assistant

Joseph J. Becher
Research Associate

John W. Swanson, Director
Division of Natural
Materials & Systems

Robert C. McKee, Chairman
Container Section