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COOPERATOR Institute
REPORT NO1
DATESeptember 23, 1950
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PAGE TO TO SIGNED Richard Us. Thickens
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FURTHER STUDIES ON RECENTLY DEVELOPED BONDING STRENGTH TESTER

A new series of fluids have been investigated to replace the castor oils formerly used and found to satisfy the criteria of lower temperature viscosity coefficients, resistance to thermal decomposition and resistance to aging.¹ Several materials were investigated with the most satisfactory results given by polyisobutylene (=200-5000 poises at 23° C.)

SAMPLE BACKING STUDIES

The effect of backing material (adhesive employed to fasten the sample to the sample wheels) was the first phase in the present series of studies. For all of the preliminary work the samples were fastened to the sample drum with double-faced scotch tape (Minnesota Mining and Manufacturing Company Tape No. 400.) The specimen was prepared in the following manner. The exposed side of the tape was stretched out face down and pressed into intimate contact with a clean glass sheet and then the backing strip was removed. The paper strip was then pressed into contact with the tape making sure that no wrinkles were introduced, and the strip with its backing then fastened to the drum again taking pains to introduce no wrinkles. It was felt that since

^{1.} Institute of Paper Chemistry, Research Bulletin, July, 1950.

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the early work had been done by glueing the specimen to a glass plate,² a new variable might have been introduced. Therefore, the effects of different backing materials on the VV (velocity viscosity) product were investigated. For the first series of tests, a commercial doublefaced coated stock was used because it gave a more abrupt end point and left less room for operator judgment to influence the tests. The tests on the coated stocks were conducted as follows. A layer of tape was laid down, the sample fastened to it, as described above, and tested. For the second series, there were two layers of tape backing and for the third three layers. The data are shown below with simultaneous measurements made of compressibility using the Federal gauge.

TABLE I

EFFECT OF TAPE BACKING THICKNESS ON VV PRODUCT AS COMPARED WITH COMPRESSIBILITY DATA

No. of Layers		Compressibility
of Tape	VV Product	Inches
-	Cm. Kilopoises/sec.	x 10 ⁻⁴ /02.
l	83.3	0.38
2	77.7	0.75
3	61.2	1.3

It appears that the oil wheel makes a larger and larger area of contact as the combination gets softer and thereby allows a larger section of the specimen to be stressed at any instance with consequent earlier rupture.

Institute of Paper Chemistry, Instrumentation Studies IV. Paper Trade Journal 123, No. 18, 24, 26, 28, 29; No. 9; 24, 26, 28, 30, 32, 34. October 31 and November 7, 1946.

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Two further tests were also run. In one series, only the ends of the specimen were fastened down with scotch tape; in the other, the specimen was fastened down in accordance with the procedure followed in Instrumentation Report LV (3M trim cement). The results were 81.6 and 101.3 kilopoise cm./sec. respectively. This series of tests was made with a film thickness of $5.7 \ge 10-4$ inches. A similar series was run on newsprint P-6A on the felt side with similar results. After considering the difficulty involved in cleaning up the equipment after each test, the old method was discarded in favor of the single layer of scotch tape. The only effect is to change a machine constant.

OIL WHEEL LOADING STUDIES

The next variable investigated and tied down was the load applied to the oil wheels. The work done on Instrumentation Studies LV indicated that the test was rather insensitive to the applied loads as predicted by dimensional analysis. This has turned out to be true within certain limits. On a coated stock, there appeared to be a tendency for the VV product to &crease with increasing load. This tendency becomes quite evident after studying figure one. It has been suggested that the falling off of the VV product is the result of disturbing the base stock by the compressive forces transmitted from the coating and by cracking the coating. All of the failures on this particular paper were body stock failures. When the same test was repeated for a newsprint (P6a), the VV product varied approximately 5.3%. This can be seen from Table II.

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TABLE II

THE EFFECT OF OIL WHEEL LOADING ON VV PRODUCT FOR NEWSPRINT P6A

Load 1b.	VV Product ¹
53.1	103.4
78.8	109.0
104.6	105.4
130.4	104.0

¹ The average of at least five determinations.

It was decided that as long as the newsprint values of VVP were not influenced by the load to any great extent while the coated paper appeared to be that the load on the oil wheels should be reduced to 75 lbs. and the test would then be done on the flatter part of the curve. The variability of the data at the higher loadings (79 lbs. and up were questioned considerably. It was thought that the variations might be due to differences in moisture contents of the paper because the tests were performed on different days, and at one time the humidity in the controlled rooms rose to 75% when the power went off following a storm. It was known that tear is quite sensitive to moisture changes and it was felt that the same might be true for the internal bonding strength test. Consequently two sets of samples were taken from a roll in such a way as to minimize the paper variability and exposed to 11% R.H. and 86.5% R.H. for three days. The samples were taken one from each atmosphere and immediately tested. The elapsed time between opening the desicators averaged and completion of the tests was 111 sec. The sheets conditioned at 11% gave an average VVP of 75.9 and those from

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the 86% gave 74.1, hardly a significant difference. Ideally this series of tests should be conducted again with a wider range of papers under properly controlled conditions.

Although the values given for the VV product do not change appreciably with a changing load the characteristics of the failure do change. This change applies especially to the newsprint. Above 50 lbs., the failures have largely been blisters which tail off to a complete rupture of the surface as the speed increases; below 50 lbs., the failures become pick failures. This is presumably due to insufficient pressure to force complete contact between the paper and the fluid. This effect is reasonable when the viscosities are considered. With a rough surface sheet such as the newsprints sufficient time is not available at low pressures for the fluid to flow into the interstices of the sheet so that an intimate contact can be made. However, small, weak patches of the surface can be pulled off. This effect did not appear on the coated sheet because the surface is obviously much smoother.

Conclusions to the Loading Tests:

- With the newsprints, load variations from 50-130 lbs. make no difference.
- 2. With loads less than 50 lbs., pressures are insufficient to force an intimate contact with the surface with the result that the failure characteristics change.
- 3. With the coated stocks, regardless of the large spread of data, there is a consistent falling off of the VVP as the loading is increased.

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- 4. This falling off might possibly explain why some printers havetrouble with a specific paper while others may print easily and well on the same paper. Press pressures could have a very important effect.
- 5. The optimum load appears to be between 50 and 80 lbs.

DISCUSSION OF THE END POINTS

For the early work (Initial Report on Bonding Strength Studies, A.N.P.A. Report No. 10), the end points were taken as that place at which a complete body stock failure took place (see Figure II. P-6A, Report No. 10). On further study it was noticed that the body stock failure was in most cases preceded by a blister failure; i.e., the body stock separated from the surface but the surface remained intact. It was felt that once a blister had started the original conditions had been upset; the resulting system therefore was not the same as the original. Accordingly, a microscopic study was made of a large number of newsprints samples and it was noted that the failures even on the felt side were more gradual than originally suspected. The mechanism of failure is roughly as follows. Starting from the point of zero, velocity and following down the sheet the failures would first appear as small bundles of fibers being raised slightly from the body stock. These bundles would still be firmly fixed to the body of the sheet. This failure might correspond to dusting. The loosening wood become more and more prevalent until a blister actually started. The blister would usually start on one edge of the oil wheel track--probably because of local stress intensification--and spread over the whole track. Then, finally the stress

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would become large enough so that the sheet would actually have its surface pulled off. This was the end point formerly used but as explained above, the sample had been previously ruptured. It was decided, therefore, for all further work to make the end point that point at which the area of two mm. square was raised from the surface of the sheet.

FILM THICKNESS STUDY

A short study of the effect of film thickness on VVP was made using the standard coated sheet. As would be expected from a consideration of the hydraulic forces involved, the force necessary (VVP) to produce rupture increased as the film thickness increased. This is true only in the range tested. There is obviously a limiting thickness beyond which rupture will not take place because all of the shearing will be done in the film and very small stresses will be applied to the sheet. Similarly, there is another limiting thickness below which flow, limited by time and viscosity, will not take place. There will be no adhesion to the sheet and hence no rupture. The importance of adhesion has been shown experimentally by employing a silocone, whose adhesion (tack) is known to be low, as the stress transfer medium. With this silocone fluid of the same viscosity as the polyisobutylene, no rupture could be produced on a sheet normally easily torn. The results of the film study are plotted in Figure 2, and show the expected increase in VVP with increasing film thickness.

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A brief study has been made of the effect of tub sizing on bonding strength as a function of film thickness. An Oxford coating raw stock was sized with starch. The additions ranged from 0 to 5% of basis weight. All papers were coated on the felt side only. The 0% sheet was treated in the same manner as the starch coated sheets except that there was no starch added. The starch was put on the sheet and then spread out by means of a draw bar. These draw bars are tightly wrapped with different sizes of wire which determine how much coating is left behind. Unfortunately, the coating was not too well down so that in some cases it was possible to see that the sheet was not evenly coated. The uneven coating may well be the reason that the VV products taken on the five per cent coating with 1.17 and 1.55 mil. films and the product on the four percent coating will the 1.55 mil. film are low. This can be seen in Figure 4. The raw data are quite consistant internally and previous experiments would indicate that with increasing film thickness the VV product should go up, not down. It is interesting to note that with a coating as small as 3% the bonding strength has been almost doubled and further addition to 5% down not seem to make any appreciable difference. Because of the relatively wide sample variations it might be well to spot check the tubsize data to verify the observed trends.

CORRELATION STUDIES

An attempt has been made at correlating the bonding strength tester with the other physical tests (tear, tensile, mullen and Dennison)

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but in light of the data that have been taken so far, it hardly seems justifiable to state that there is or is not any correlation. The papers tested did vary over a range of about two to one in the VV product but it is to be remembered that a range of from 75 to 150 in the VV product is a very small section of the range of the bonding strength tester. The data in Table III are the results of this series of tests. However, they indicate that the investigation must be of a much wider nature than was first realized.

TABLE	III
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		TA	BLE III			
Paper	Burst,	Tensile	Tear	Dennison Pick	VVP	/ sec.
No.	pts./100	#/in./100#	Factor	Critical Wax	Kilopoise cm.	
13	60.0	63.9	.99	18	125	
14	48.9	54.3	.93	17	94.2	
15	34.3	56.5	.90	17	95.8	
16	46.0	51.7	1.15	11	73.2	
17	44.6	47.3	.91	10	82.3	
20	57.8	52.5	1.50	13	160	
24	67.9	58.7	1.21	13	364	
28	61.7	83.1	2.12	18	171	
P-6 P-7 P-8 P-9 P-10 P-11 P-13 P-14 P-15 P-17 P-18	20 23 19 21 21 25 23 21 22 19	25.0 30.7 28.5 27.7 26.0 32.4 34.2 24.1 29.3 25.4	.47 .49 .46 .52 .51 .58 .54 .46 .48 .51	7 86 7 8 8 8 8 6 7 7 7 7	86.1 102 107 127 146 127 131 105 99.6 95.6 106	Pro

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THE BONDING STRENGTH TESTER

This report covers work done to date on the bonding strength tester since report no. 1 to 1508 written in September 1950. This report also describes any change in the technique used in operating the tester. It is interesting to point out that the data presented in this report in many places disagree with data previously reported and in some instances the data are contradictory.

MACHINE REPAIR

While doing some preliminary work prior to making a study of coated papers it was noticed that the oil track on the sample was not complete. The oil wheels are 1/2 inch wide and the track on the sample was at some points considerably less than 1/2 inch wide. When this effect continued through repeated tests it was concluded that the oil wheels were not contacting the sample evenly. This conclusion was confirmed when a strip of aluminum foil was passed between the wheels and the resulting track showed that the oil wheels were riding on one edge. As it happened, both wheels were riding on their inside edges. By measuring with a micrometer it was found that the sample wheels were

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slightly conical rather than perfectly cylindrical as they are supposed to be. The sample wheels are one inch wide and the diameter of one of them varied 0.002 inch from one edge to the other edge. The other wheel varied 0.001 inch from one edge to the other. The wheels were therefore reground to the original specifications. After the grinding operation the wheels were again mounted in the tester (care being taken to align the axles of the sample wheels and oil wheels so they were parallel to each other). Aluminum foil was again passed between the wheels and it was noticed that with the wheels as close to perfection as a standard machine shop could make them there still were variations in the track. The sample wheels, by design, start and stop in a predetermined position, but the oil wheels can be started in any arbitrary position. After starting the oil wheels in several positions a position was found which produced the best track through the testing range and the oil wheel was thereafter always started in this position. While this was the best track it was not necessarily perfect. There is at least one point in the track where only one edge of the oil wheel is in contact with the sample wheel; however, at no point is the elevated edge more than 0.0003 inch from the sample wheel. It is probably safe to accept this irregularity in the track since most paper thicknesses vary at least this much.

VISCOSITY MEASUREMENT OF OILS

Since the final result (VVP) is directly dependent on the viscosity of the oil used it is necessary to know this viscosity exactly. Also these oils may be susceptible to aging so it is necessary to recheck the viscosities periodically. Data from two such checks are included in this study.

The viscosities of the polyisobutylenes used with the bonding strength tester were determined by the falling ball method. The ball used was a 3/32-inch steel ball bearing which was timed through a free fall of 14 to 15 cm. Care was taken to use a containing vessel whose diameter was in the order of 20 times that of the ball and also to be certain that the ball had reached terminal velocity before reaching the timed interval. Stokes Law applies directly. The oil containers were placed in a temperature-controlled water bath whose temperature was controlled to 5.21° B., 3.45° B., and 0.46° B. $\pm 0.01^{\circ}$ (22.00° C = 2.09° B). The Beckman thermometer used in the bath is the same one used on the bonding strength tester. The four polyisobutylenes (Indoil H-100, Vistac no. 1, no. 2, and no. 4) which had been used in testing were measured.

In the course of testing papers a need developed for a polyisobutylene whose viscosity is a little more than half of the viscosity of Indoil H-100. Also polyisobutylenes were needed whose viscosities were midway between the viscosities of Indoil H-100 and Vistac no. 1 and midway between the viscosities of Vistac no. 2 and Vistac no. 4. To obtain these desired viscosities two polyisobutylenes were mixed and the proportions were changed until the desired viscosity was obtained. There was some concern as to whether a mixture such as this would behave as a pure polyisobutylene. To check this a very low viscosity polyisobutylene, Indoil L-100, was mixed with the highest viscosity polyisobutylene, Vistac no. 4, so that the resulting viscosity was equal to that of Indoil H-100. These two, the mixture of Indoil L-100

and Vistac no. 4 and the Indoil H-100, were then tested against each other on the bonding strength tester. The results of this test were so nearly the same that the mixtures were assumed to behave exactly as the pure polyisobutylenes so the other mixtures were made. The viscosity data are presented in Table I. From this table it is evident that the oils are quite stable with respect to aging. The oil whose viscosity changed the most over the six-month period was Indoil H-100 and the change was a 1.2% increase over the January viscosity measurement. The general trend as shown in Table I indicates that the effect of aging is to increase the viscosity a little. Figure 1 shows the oils in the order of their viscosity and demonstrates how well these oils cover the testing range.

SPECIMEN BACKING STUDY

The following requirements were set up to determine the proper specimen backing; it should approach the type of backing used in the printing operation, its use should produce consistent results, and it should be quite easy to use. With this in mind four different papers were tested using five different backings. The results of these tests are shown in Table II. The trim cement is a product of Minnesota Mining and Manufacturing Company. It was applied by rotating the sample wheels in a trough containing a dilute solution of trim cement. The trim cement was diluted with petroleum ether and toluene until it was of such a consistency that it flowed on the sample wheels in an even film just

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TABLE I

VISCOSITY DATE (JANUARY, 1951)

Viscosity Viscosity Viscosity at 20.37°C. at 25.12°C. at 23.36°C. Polyisobutylene 265.2 221.9 Indoil H-100 355.8 Vistac no. 1 737.8 868.9 1151.2 Vistac no. 2 1669.5 1953.1 2604.7 Vistac no. 4 4990.6 5833.4 7757.4 Mixtures Indoil L-100 and Vistac no. 4 71.4 129 Indoil L-100 and Vistac no. 1 563.9 360.9 Vistac no. 2 and Vistac no. 4 2617 4095

VISCOSITY DATA (JUNE, 1951)

	Viscosity	Viscosity	Viscosity
Polyisobutylene	at 25.13°C.	at 22.52°C.	at 19.94°C.
Indoil H-100	224.6	289	371.9
Vistac no. 1	740.3	942.7	1201
Vistac no. 2	1670	2135	2731
Vistac no. 4	5013	6394	8202

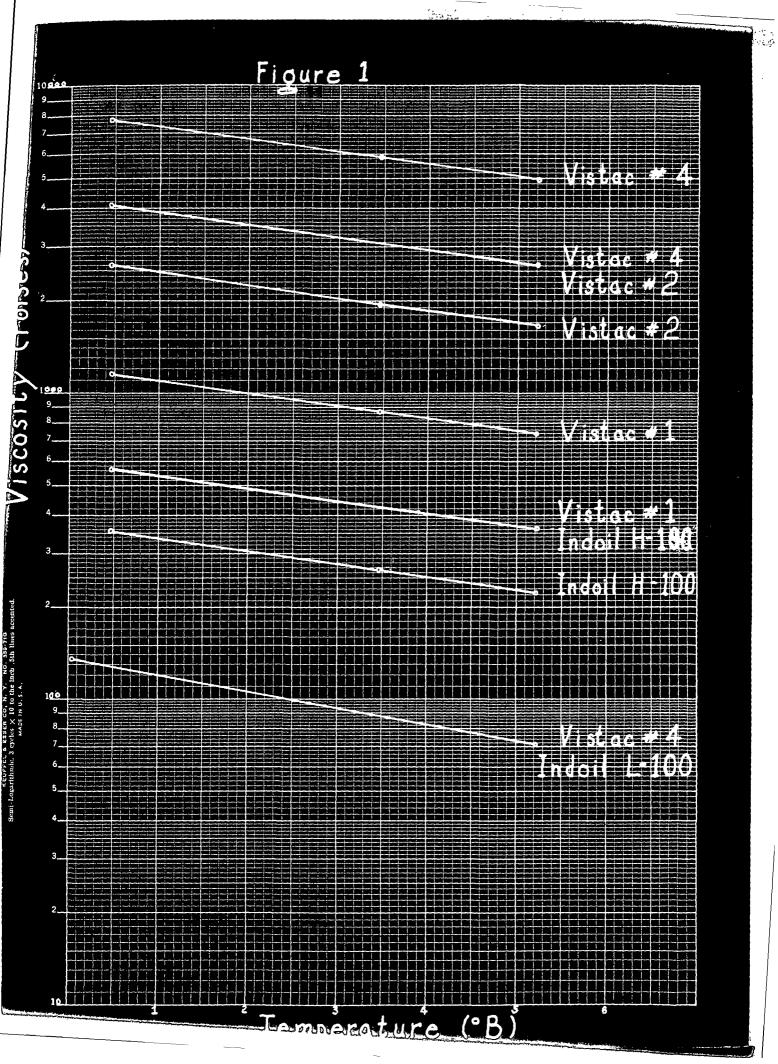


TABLE II

EFFECT OF SPECIMEN BACKING

Type of failure		Appleton Coated VVP	Vacuum dried newsprint VVP	Regular newsprint VVP	100% rag bond VVP
Complete rupture	Trim cement	93•3	92.6	160	612
Complete rupture	Tape no. 666	. 89.3	128	224	59 5*
Complete rupture	Tape no. 400	93.0	119	218.5	562
Complete rupture	Tape 2 layers	81.4	122	207	536
Complete rupture	Tape 3 layers	76.2	118	202	557
Blister	Tape no. 666	53	74.7	143	235*
Blister	Tape no. 400		70.9	140	253
Blister	Tape 2 layers	27.5		132	263
Blister	Tape 3 layers	29.5		115	232

* Results of a single measurement. All others are averages of 6 or more measurements.

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thick enough to hold the sample securely. The tape no. 666 is a doublefaced, pressure-sensitive cellophane tape and the tape no. 400 is a double faced, pressure-sensitive paper tape. The multiple-layer backings were of tape no. 400.

As the result of the data of Table II it was concluded to continue to use the tape no. 400. Some of the reasons for this conclusion are the following: (1) The VVP-values did not vary much from one backing to another, so from that point it did not make much difference which backing was used. (2) The data obtained with the trim cement varied considerably from one specimen to another, indicating a variation in the cement (the ether used to dilute the cement evaporated quite rapidly which made it necessary to redilute the cement before each application and the resulting different concentrations could account for the variation in VVP). (3) The oil track on the sample of Appleton Coated was irregular and incomplete when using tape no. 666 as a backing. This irregular track results when the oil wheel does not make uniform contact with the paper sample. These non-uniform tracks can only be due to the irregularities in the paper itself and apparently the reason they showed up when using tape no. 666 is that this tape is not compressible enough to absorb these paper irregularities and since this difficulty was not encountered with tape no. 400 another vote was cast in its favor. (4) The multiple tape layer backings were eliminated for reason no. 1 above and also because of the possibility of obtaining erroneous results when testing coated papers. If the backing is too soft and compressible the coating may become cracked at or near the point of contact with the oil wheel, and this would certainly affect the bonding strength measurement.

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CHANGES IN OPERATING TECHNIQUE

A few changes have been made in the technique of operating the bonding strength tester since the last report was written. For the most part these changes were made to eliminate variables which affected the final VVP. The order in which the changes appear in this report is not necessarily the order in which the variables were discovered.

When testing a very strong paper on which it is necessary to apply a large stress to obtain a rupture the backing tape (no. 400) would separate from the sample wheel (see Figure 2). This caused a stress intensification which resulted in an early failure of the paper and consequently an erroneous VVP. To counteract this the sample wheel was made slightly tacky with a very dilute solution of trim cement before the strong paper specimen was applied. After becoming aware of what was happening it was possible to know whenever the backing tape lifted by the way the specimen came off the sample wheel.

A very large variable was found and eliminated when the effects of fingerprints was discovered. These effects are shown very strikingly in Figure 3. In all cases the specimens were prepared as described by Thickens (<u>1</u>), except that cloth gloves were worn while preparing the first specimen. The second and third specimens were prepared in the

(1) Report No. 1, Project No. 1508, September 23, 1950.

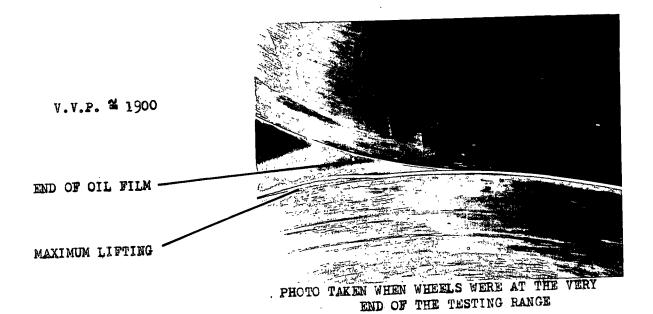
FIGURE 2

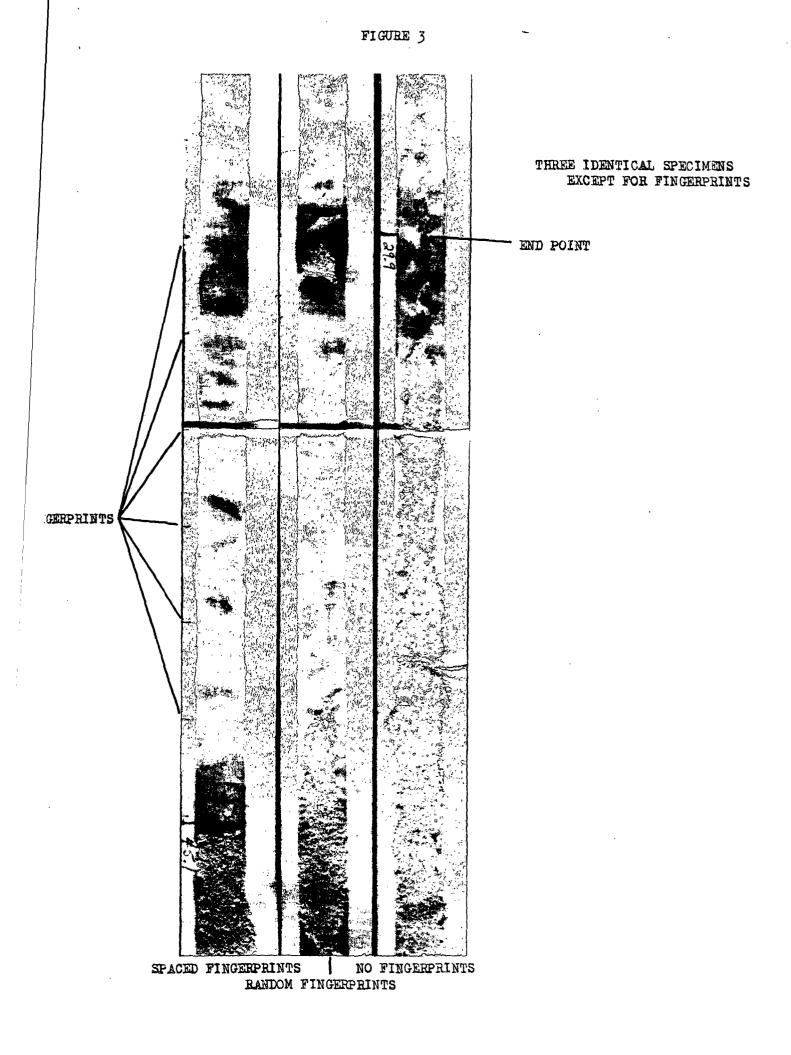


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prescribed manner except that while preparing the third specimen an effort was made to apply the fingerprints at one-inch intervals. Using the results of specimen no. 1 as the true VVP, the VVP of the third specimen is in error by 22.5%. The fact that the distance (and therefore the velocity) is greater for the third specimen and that a rupture can be stopped once it has started as in the second specimen indicates that the presence of a fingerprint somehow weakens the oil connecting link between the steel oil wheel and the paper specimen. Apparently it is the oil in a fingerprint that causes this effect. This oil is either of a much lower viscosity than the polyisobutylenes, or else it is thixotropic, or both.

Another quite important change in the technique is the one made to eliminate slippage between the oil wheel and the specimen. The bonding strength tester is designed so that the accelerating force is applied directly to the sample wheels and the sample wheels transmit this force to the oil wheels through the oil film. The accelerating force is quite large and since the oil film is fluid it is very possible that some shearing or flowing takes place. The amount of slippage was measured and was found to be in the neighborhood of 15% when using an oil whose viscosity is 276 poises at 23.0°C. If the bonding strength tester is to reproduce to action of a printing press, all slippage must be eliminated. This was done very easily and satisfactorily by applying a layer of friction tape to one of the sample wheels and then using the other wheel only for testing specimens.

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Some difficulty was encountered while testing some very strong semichemical paper. The oil wheel was not making good contact or at least the oil track on the paper appeared as though the paper was not being wet properly. This occurred while using the most viscous oil (6000 poises) at the highest velocities (300+ cm./sec.). To correct this situation it was decided that an increase in the loading weight on the oil wheels would force the oil and sample wheels into more intimate contact. Some work had been done to determine the effect of varying the loading weight (2) on the VVP but since so many changes had been made since then this study was done again. The load was weighed with a spring scale in the following manner; the load applied by the apparatus itself without any additional weights is 38 lbs./linear inch. The load applied using one weight is 57 lbs./linear inch. All of the auxiliary weights are the same so the oil wheel load is n (57-38) + 38 where n is the number of auxiliary weights. Two papers were used in this study: one was a 100% rag bond and the other was a laboratory coated paper whose coating contained 12% casein and was quite fragile. Tests were made using loading weights of 76, 95, 114, and 133 lbs./linear inch. The results of these tests are plotted on Figure 4. The increased loading weight seems to have very little effect on the VVP which is as it should be. Figure 4 indicates that the VVP decreases slightly as the loading weight increases which could be the result of a better contact. Since the increased loading weight has no detrimental effect on the VVP and better contact is made with the specimen it was decided to use a loading weight of 133 lbs./linear inch instead of the 95 lbs./linear inch that had been used.

(2) Report No. 1, Project No. 1508, Sept. 23, 1951

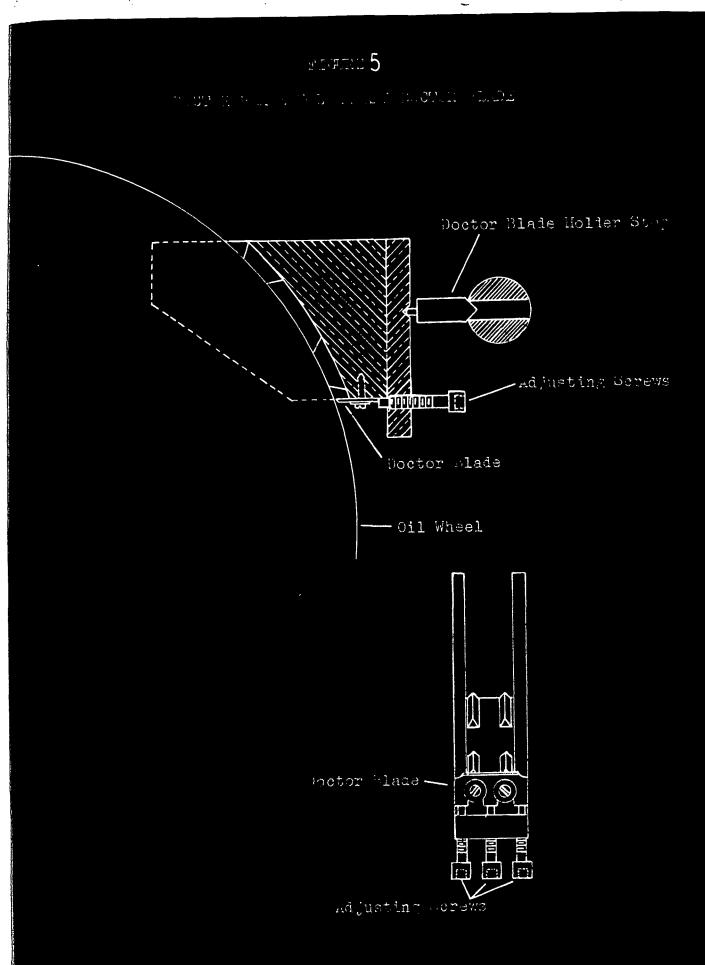
The only other change worth noting is the change in the doctor blade holder and the method of determining film thickness. The doctor blade holder is essentially the same as the one used previously. The main difference is the three adjusting screws which make the chore of adjusting the doctor blade to the proper film thickness much easier. These screws have considerably decreased the time necessary for adjusting to the desired film thickness and they could do much better still if screws with a finer thread were used. Another minor change was to remove the doctor blade holder stop that was on the tester and substitute for it the one shown in Figure 5. This stop has a double swivel action which insures proper seating of the doctor blade holder on the oil wheel.

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When applying the oil film to the oil wheel the doctor blade applies an even film of the proper thickness over the entire circumference of the wheel with the exception of a space 1 1/2 inches to 2 inches long directly under the doctor blade holder. In removing the doctor blade holder from the oil wheel the doctor blade holder is tipped forward until the doctor blade is in contact with the wheel so that when the doctor blade holder is pulled along on the wheel this uneven portion is scraped clean. To measure the film thickness this uneven portion is scraped very clean for a two-inch interval with a razor blade. Then the remaining even film is scraped off with a new razor blade, transferred to a piece of aluminum foil and weighed. The film thickness is: $T = M/(2.54)^3(C-2)$ WP

where T is the film thickness in inches, M is the film mass in grams to the nearest 0.1 mg, C is the wheel circumference in inches, W is the wheel width in inches, and **p** is the specific gravity of the oil being applied. Using the method outlined above it is possible to reproduce



film thickness quite consistently.

OIL FILM THICKNESS STUDY

The purpose of this study was to determine the best oil film thickness to use in testing a wide range of papers. Theoretically the VVP of any paper changes as the oil film thickness changes. To get the complete picture let's start with a film thickness of zero and proceed to some very thick film. At zero film thickness there of course is nothing to transmit the stress from the oil wheel to the paper specimen so the VVP is infinite. At zero plus some very small increment some of the stress is being transmitted but the oil film is so thin that only the very high points of the specimen are contacted and thus the stress transmitted for any unit of area is very small. As the oil film thickness increases the oil contact becomes better and more stress is being transmitted. This produces ruptures in the specimen at lower speeds and consequently the VVP is lower. This phenomenon continues until a minimum VVP value is reached. At this point a kind of balance condition exists. The stress produced by the separation at the nip of the wheels is transmitted or applied to the paper specimen by and only by the oil film. Since this oil is a fluid this connecting link is not a positive connection but an elastic connection. In any given oil film a certain amount of shear takes place and as the oil film increases this shear becomes greater. This shear dissipates stress which would otherwise be transmitted to the paper specimen. Now then, as the oil film was increasing the advantage gained by the better contact was being counteracted by an increased shearing in the oil film. It can be seen,

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therefore, that the minimum point in the VVP vs. film thickness curve is the point where there is nothing to gain by further increasing the film thickness because the small additional advantage gained by the better contact will be more than counterbalanced by the increased shearing in the oil film. As the film thickness increases beyond this point more and more stress is dissipated in the shearing in the oil film making it necessary to go to higher speeds and consequently higher VVP values to obtain failures. If the film thickness were allowed to continue to increase one can imagine that it would reach a point where all the stress would be dissipated in the oil and the VVP would again go to infinity.

The desired film thickness is that which will produce VVP's of about the minimum value. The tester will then be operating in the horizontal section of the VVP vs. film thickness curve and small errors in the determination of the film thickness will not appreciably affect the resulting VVP.

A group of papers was selected which would cover the desired range in VVP. These papers were then tested with film thicknesses of 0.0002 in., 0.0006 in., 0.0012 in., and 0.0018 in. Five specimens of each sample were tested at each film thickness and the averages of these five tests are plotted on Figure 6. This group of papers consists of newsprints, laboratory and commercially coated papers and a 100% rag bond. These papers were selected for their range in bonding strength and also for the variety of surfaces they presented. The film thickness to surface relationship has a definite effect on the bonding strength measurement (VVP). A very smooth surface such as that of a supercalendered coated sheet will respond to a much smaller film thickness than some rough

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surfaced sheet such as the rag bond that was used. This very problem makes it practically impossible to make valid comparisons between the VVP's of two papers whose surface characteristics are different. In fact this is true of any two different papers no matter how their surface characteristics compare. In other words, a comparison between the VVP of a rag bond and the VVP of a kraft paper is not necessarily valid even though the smoothness of each is the same because the failure produced by the bonding strength tester will vary in nature and at this point the human element of error will be introduced when an end-point is selected.

The curves of Figure 6 can be interpreted as follows: For the rougher papers (the rag bond and the newsprints) the increase in VVP as the film thickness decreases is very pronounced. This increase in VVP starts at film thicknesses of 0.0006 to 0.0007 inch. Apparently films whose thicknesses are less than this wet the paper surface very poorly. The smoother coated papers do not show this VVP increase at the same point in film thickness. It was predicted that thinner films would be required to show this increase. From the curves of Figure 6 it appears that thicknesses somewhat less than 0.0002 are necessary to show the predicted increase in VVP. The average curve has a minimum value at about 0.001 inch and for this reason the bonding strength tester will be operated at this film thickness hereafter or at least until a better reason is found for changing it.

COATING STUDY

This study was undertaken to determine the effect of coatings in general and casein content in particular on the bonding strength of the sheet. The test samples for this study were prepared by the physical chemistry group. Two series of papers were prepared; the first was coated with clay-casein mixtures by means of a no. 12 Mayer rod and the second was coated with various concentrations of cooked, converted starch with a no. 4 Mayer rod. An Oxford Paper Company coating raw stock was used in all cases. All coated papers were air-dried under a tension of 600 g, st room temperature for 15 minutes. The clay-casein coated sheets were conditioned at 50% relative humidity and 73°F. for 18 hours and were then sealed in Pliofilm bags and were taken to the laboratory of The Appleton Coated Paper Co. where they were supercalendered. For a summary of the coating color formulations see Table III.

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TABLE III

CASEIN COATING COLORS

de No.	% Casein based on clay	% Solids of color applied	Coating formula
10	6	_ 50	100 g. HT clay 75 ml. H ₂ 0 5 ml. 10% quadrafos 32 g. casein dispersion (6 g. 0.D.)
11	8	50	100 g. HT clay 70 ml. H ₂ 0 5 ml. 10% quadrafos 42.5 g. casein dispersion (8 g. 0.D.)
12	10	50	<pre>100 g. HT clay 63 ml. H₂0 5 ml. 10% quadrafos 53 g. casein dispersion (10 g. 0.D.)</pre>
13	12	49	100 g. HT clay 59 ml. H 0 5 ml. 10% quadrafos 64 g. casein dispersion (12 g. O.D.)
14	14	49	100 g. HT clay 57 ml. H ₂ 0 5 ml. 10% quadrafos 74 g. casein dispersion (14 g. O.D.)
15	16	48	100 g. HT clay 55 ml. H ₂ 0 5 ml. 10% quadrafos 85 g. casein dispersion (16 g. 0.D.)

TABLE III (Continued)

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STARCH COATED SHEETS (Superfilm no. 4 Starch)

Code No.	% Starch Applied	% Increase in wt.
16	3	1
17	5	. 2
18	7	3
19	9	4
20	11	5

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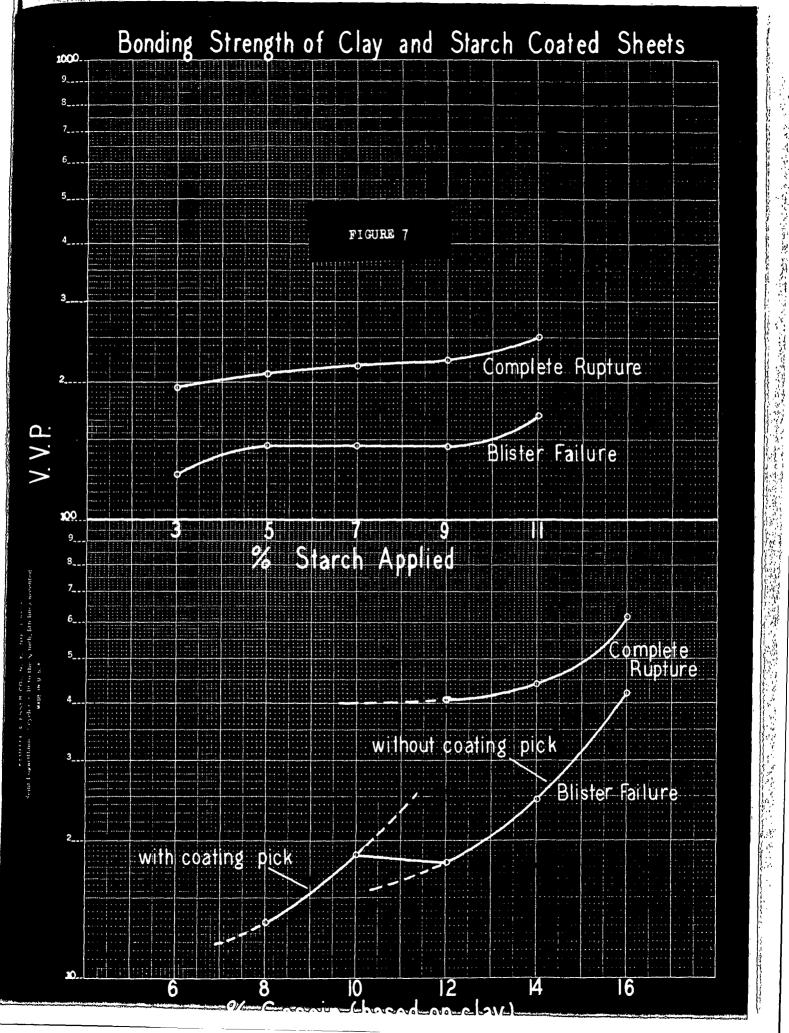
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CALENDERING DATA FOR CLAY-CASEIN COATED PAPERS

% Casein based on clay	No. of passes thru supercalender	Gloss (B & L)
6	1	37
8	1	36
10	1	30
12	2	31
14	4	30
16	Lį.	33

The bonding strength tests were conducted under the ordinary operating conditions of 50% relative hunidity and 73°F. temperature. The film thickness used was .0012 inch and the oil wheel load was 95 lbs./ linear inch. The results of these tests can be seen on Figures 7, 8, and 9. For the most part the points that are plotted on these figures. are averages of five tests. Figure 7 shows the effect of increased amounts of casein and starch on the V.V.P. One of the most striking results shown on Figure 7 is the effect of paper surface. The starch coated paper was coated only with starch which was absorbed by the paper or at the most left a very thin coating on the surface (this coating was so thin that the coated side was indistinguishable from the uncoated side). This means that the coated surface of the starch coated paper was rough; very rough when compared with the supercalendered claycasein coating. If it can be assumed that the casein that is absorbed by the paper improves the bond in the paper about as much as the starch that is absorbed then the difference in VVP is the result of surface differences. The supercalendered clay-casein coating is very smooth and so the oil film makes very good contact with it. This improved contact allows a greater amount of the available stress to be transmitted to the paper which results in an earlier rupture and consequently a lower VVP. The curve showing the blister failure of the clay coated sheets is quite interesting. It is interesting because it is not a nice smooth curve but is a curve made up of two sections. These sections are labeled "with coating pick" and "without coating pick." The explanation of this curve is somewhat speculatory because all of the facts are not readily known. The writer's hypothesis is as follows:





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First of all an assumption is made with regard to the location of the separation in a blister failure. It is assumed that the blister failure occurs in the body stock and never at the coating-body stock interface. The coating itself appears to be brittle in nature and to have very little mechanical strength. It would appear then that if enough stress were applied to the specimen to separate the coating from the body stock the coating would immediately flake away and the failure would be a coating pick and never a blister. If then a blister develops it must be because the coating clings so tenaciously to the body stock that before a separation occurs between the coating and body stock the body stock itself gives way. The next thing to take into account when explaining the two-sectioned curve of Figure 7 is the two opposed effects resulting from a change in casein content. These two effects are the strengthening of the coating-body stock bond (which would tend to lower the VVP) and the strengthening of the fiber to fiber bond of the body stock (which would tend to raise the VVP), which are the result of an increase in casein content. Why does strengthening the coating-body stock bond tend to lower the VVP? Because if we start with a coating whose casein content is very low we can see that on the bonding strength tester this coating will immediately flake off and practically none of the stress will be applied to the body stock. Since we are looking for a blister failure. which occurs in the body stock it is obvious that we will never find If we were to increase the casein content we would reach a point one.

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where the coating-body stock bond was strong enough to transmit enough stress to blister the body stock. Therefore, increasing the casein content lowers the VVP. Why does increasing the casein content raise the VVP? Simply because the chemical fiber to fiber bond is aided by an adhesive bond which grows stronger as the casein content increases. With these things in mind we can see in Figure 7 that at 6% casein the coating body stock bond is not strong enough to transmit the stress necessary to blister the body stock and therefore there is no VVP data. At 8% casein nearly the same condition exists except that some blisters were produced. It appears as though 8% casein is approaching the transition point. For the most part the coating picks off but there are spots where the coating-body stock bond may be a little stronger or the body stock may be a little weaker because some blisters are produced. At 10% casein this condition is reversed. In other words. the blister failure now predominates and the coating pick is confined to small spots which are probably caused by some weakness in the coatingbody stock bond. As the casein content increases from this point there is no coating pick so all the stress from the oil is applied to the whole paper and the weakest spot appears to be in the body stock where blistering takes place. The effect of changes in the casein content can also be seen in Figure 8 where VVP is plotted against oil film thickness. The code numbers used on Figure 8 are explained in Table III. In addition R, B, and C mean complete rupture, blister failure, and coating pick respectively. The effect of coating pick on blister failure and vice versa can be seen in curves of papers 11 and 12 whose coatings contain 8% and 10% casein. Figure 9 presents a very nice family of curves which behave as predicted from the standpoint of added adhesive increasing

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the VVP and also with regard to oil film thickness. These curves are also of interest because of the fact that they are contradictory to similar previous data (3).

CONCLUSIONS

The following conclusions are based on the work presented in this report:

1. It would seem that all the possible variables in the bonding strength testing procedure have been identified and are now controlled so that variations in the testing results are caused primarily by variations in the paper specimens.

2. The resistance of the polyisobutylenes to aging is reassuring in that the value of VVP's will remain constant over a considerable period of time. It appears that a semiannual viscosity check will be adequate.

3. The bonding strength tester will be operated with an oil film thickness of 0.001 inch. This was concluded when the average curve of a series of curves of VVP vs. film thickness reached a minimum in the neighborhood of 0.001 inch.

4. The coating study showed very nicely how the bonding strength of coated papers is increased by increasing the casein content of the coating. This study has provided a little food for thought as to

(3) Report No. 1, Project No. 1508, Sept. 23, 1951 Fig. 4

how coated papers fail. The study seems to point out that clay-casein coated papers may pick if the casein content of the coating is less than 12%.

5. Fingerprints on the specimen have a very definite detrimental effect on the VVP. That is, fresh fingerprints are bad. If the detrimental effects of fingerprints are due to the thin film of oil that is applied it could be reasonably assumed that this oil would be absorbed in the specimen after a period of time and its effect might possibly disappear. A few specimens were tested for this effect and the result indicated that the effect diminished but did not disappear in a 24-hour interval. So far no adequate study has been made to determine if and when fingerprint effects disappear.

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