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SIZING AND ITS EFFECT ON PAPER-POLYMER ADHESION

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ABSTRACT

joint-strength measurements, Using simple. the bond between printing papers and various thermoplastic-polymer formulations is defined. It is demonstrated that the bond strength to the paper-polymer interface strongly depends on processes with which the paper has been treated the sizing during manufacturing. Increased amounts of rosin sizes and synthetic, cellulose reactive sizing agents both lower the paper-polymer adhesion. However. the effect is more pronounced for synthetic-sizing agents. as also Swanson demonstrated by and others for extrusion-coated papers and board. The decrease in bonding strength with correlates decrease а in paper wettability. as measured by capillary penetration of organic liquids of suitable surface tension, defined or as by surface energetics using the elution gas-chromatography technique.

INTRODUCTION

Plain. uncoated. printing papers are treated with sizing agents to enhance their resistance to penetration by liquids, for instance water or printing inks. However. several undesirable characteristics may be acquired in the for example, those caused process, by the debonding properties of the rosin/alum sizing system.In addition, we have recently raised the issue of using the cellulose reactive sizes for the sizing of papers used for electrophotographic copiers and printers, where adequate

bonding strength must be created between the polymeric toner particles and the underlying paper fibres (1,2.)

The adhesive bonding between cellulosic materials and synthetic, thermoplastic polymers has been studied by many researchers, most notably by McLaren and Hofrichter (3,4) and by Swanson and co-workers (5,6). Using cellulose films, McLaren and Hofrichter (3) found that the polymer polarity, specifically that created by the carboxyl content of and adhering polymers, would affect the joint strength of cellulose-polymer bonds. Impregnating the cellulose film with organochromium compounds of the Werner type would make the cellulose water-repellent and This nonadhering (4). finding demonstrates the detrimental effect on cellulose-polymer adhesion of a cellulose reactive-sizing treatment, similar to that currently used for alkaline-made printing papers, as described next.

The effect of sizing on paper-surface energetics and role in the adhesion mechanism between polymers and its paper have been stressed by Swanson and co-workers. Swanson and Becher (5) found that polyethylene adhesion to bond-type papers decreased with decreasing critical surface the paper substrate (Fig.1). tension of Fatty acids molecules, similar to structural segments in the alkv1 ketene dimmer (AKD) and alkenyl succinic anhydride (ASA) sizes described below, were found to affect the wettability of cellulose films (7). Fredholm Westfelt and (8) demonstrated that the polarity modifications of AKD sizes could improve polyethylene adhesion without the paper its basic sizing characteristics, that is, its losing resistance towards liquid penetration.

PAPER-POLYMER ADHESION

In practice (considering the extrusion coating of papers and boards or the electrophotographic copying and thermoplastic toner polymers), the main printing with criteria for adequate paper-polymer bonding have been related to either adequate flow characteristics of the molten polymer (9) or adequate wettability of the paper Many observations, including those in this substrate (5). paper, indicate that both flow and wettability should be

considered properties that affect the bonding process, and that direct joint-strength measurements are desirable to characterize the strength of polymeric bonding. Modern adhesive theories stress importance molecular the of compatibility between the molten and the solid polvmer and the similarity in mechanisms between adhesive and cohesive bond failures, for example, breakage either at the interface or in the bulk of either polymer (10).



Fig 1—The effect of the critical surface tension of the paper substrate on the adhesion of polyethylene. From (5), reproduced with permission.

characteristics, defined flow Adequate as by polymer rheology, and wettability criteria, such as contact-angle capillarity, or surface energetics measurements, values. prerequisites, for adequate paper-polymer adhesion and, are in many cases, correlate with paper-polymer joint-strength measurements, as shown in the next section.



Fig 2—Electrophotographic image illustrating toner particles on the paper's fiber surface. The bottom photograph shows the framed area of the top photograph at increased magnification. From (11), reproduced with permission.



Fig 3-Heated fuser mechanism. From (11), reproduced with permission.



Fig 4-The effect of fusing temperature and time on fuse performance.

Electrophotographic Imaging

Our studies of paper-polymer adhesion pertain to electrophotographic printing, where the image is created by micron-sized, polymeric toner particles heat-sintered (fused) onto paper fibres of the surface of uncoated. plain, bond-type papers (see Fig.2) (11). The process has been analyzed, on the basis of toner rheology, by Lee (9) for noncontact thermal fixing and by Prime (12) for both noncontact thermal fixing and hot-roll fusing. The latter process is characterized by pressure assisting the image fixing, that is, the paper passes through a heated roller assembly, like that shown in Fig.3, which is a fusing mechanism widely applied in high-speed copier and printer designs.

a given paper, the fixing process for For both noncontact and pressure-assisted fusing, is dominated by the flow characteristics of the toner. For noncontact time, fusing, the heat exposure dictates the t, temperature, T, at which softening and bonding occur, and this, to some degree, also governs the machine design choice (flash, hot roll, or heat plate in Fig. 4). The relationship between temperature and time is deemed to be the main reason why laboratury-measured adhesion correlates well with the fuse degree of printed output, as demonstrated in the next section.

The effect of pressure, p, was found to be equivalent to that of the square of the exposure time. Further mathematical relationships between fuse fix, toner rheological characteristics, and process parameters (p,t and T) have been given by Prime (12)

ADHESION TESTING

The testing of adhesion should relate to the image fix of the final print, that is, how well an image like that shown in Fig. 2 withstands efforts to remove it, intentionally or unintentionally (for example, cold offset in a stack of sheets or deliberate erasure for subsequent fraudulent copying of security print).

The Scratch Test

The scratch test is performed on the final print and, therefore, relates directly to the resistance towards print removel using simple actions, such as those of knife edges or fingernails, that simulate the action of the tool (12).



Fig 5—Scratch-test device for measuring fuse quality. The enlarged view at the bottom shows the preloaded blade scraping at the image. From (12), reproduced with permission.

plane" (see Fig. 5) loaded with a carbide Α "toner blade calibrated to a predetermined value is stroked over until a visible breakout the print occurs. Data is reported as number of strokes (fuse-quality strokes) using sampling techniques to average over the print nature and Typically. the paper surface. stroke values range from for synthetic-sized, alkaline-made, printing paper below 10 for slack-rosin-sized. acid-made types to 15 - 20bond papers, used similar to those in xerographic, cut-sheet brands. Figure 6 illustrates the variation paper in paper-polymer adhesion when different paper types are processed through a high-speed laser printer. such as the 3800 Printing Subsystem (11). TBM The fuse grade of synthetic-sized paper gradually decreased from 9 to nearly when 5 fuse-quality strokes about 1500m (two cartons) of in paper were processed the printer. Α change to rosin-sized gradually increased the fuse paper grade to more than 10 fuse quality strokes.



Fig 6- Fusing variation for synthetic- and rosin-sized papers. From (11), reproduced with permission.

Laboratory Bonding Test

Scratch testing requires printed paper that often is for assessing not available paper types for electrophotographic-printing use. In such cases. simple joint-strength measurements of heat-bonded paper to polymeric may define whether paper-polymer toner the adhesion is sufficient. The technique relies on simulating bonding-process time in the the short printer through a slower heating process in the laboratory on a hot plate or in an oven at a lower temperature, assuming that the relationship between time and temperature is the same as measurement technique (2), given in Fig.4. In our that toner was extruded into filaments that were bonded between paper strips on а temperature-controlled hot plate. Subsequently, the bond was sheared in a tensile tester. as 7a. Later improvements demonstrated better shown in Fig. "T" mode reproducibility for testing in the (Fig.7b--bond shearing perpendicular to the sheet's surface). The force range for well-bonded joints was in the range equivalent to 30-50 10 - 3kg load. All measurements were required to produce interface breaking without paper being fibers pulled from the sheet's surface (that is, the strength of the paper's surface is greater than the paper-polymer adhesion).



Fig 7—Laboratory paper-polymer adhesion testing. The extruded toner filament bonded between paper strips is strained in the direction of the arrows; (a) shear mode, (b) "T" mode.

Table 1 illustrates measurements obtained for four toner formulations ; two rosin-sized, one ASA-sized, and The filament only covers a one AKD-sized paper types. limited area on the sheet (the filament is 1.5mm long). Consequently, the nonuniform application of paper sizing significant variation creates a in strength values (significant standard-deviation values for each set of ten measurements in Table 1). Nevertheless, this technique is good enough to define the decrease in fuse-quality strokes for increasing additions in the sizing level (Table 2), as well as the performance characteristice of different paper types (Table 1).

Paper-Toner Bonding Strength (10-3 kg)

Toner Rosin-Sized (A) Rosin-Sized (B) ASA Sized AKD Sized

1	17.0 ± 5.9	14.1 ± 4.7	12.7 ± 3.4	8.4 ± 6.6
2	18.6 ± 3.6	17.6 ± 7.6	13.7 ±2.3	15.0 ± 4.1
3	19.4 ± 9.7	23.4 ± 7.5	8.5 ± 3.6	12.8 ± 4.3
4	32.6 ± 9.7	29.1 ± 9.2	18.9 ± 3.4	19.1 ± 3.8

Table 1. Bonding Strength of Four Toner Types Using Different Paper Types.

Size Level (%)	Strength Bonding (10- ³ kg)	Fuse Quality Strokes	Contact Angle* (degrees)	Surface Energy** (mN/m)
0.1	24	1-2	75	20
0.05	37	6	62	28
0.05	42	20	53	33

* Using methylene iodide

** Comparative value only--see "Contact Angle" below.

Table 2. Bonding Strength of AKD-Sized Papers

In all cases, the adhesive level of the synthetic-sized paper types was lower than that for the rosin-sized types. This rating is typical for printing papers that are currently marketed for electrophotographic use; synthetic-sized papers are more likely to show lower adhesive bonding strength than rosin-sized papers when tested under identical contitions, either in the laboratory or after printer or copier imaging (see Fig. 6)

Toner-Resin Evaluation

Toner formulations consist of polymeric resins chosen on the basis of physical properties, such as softening chargeability, and brittleness (13). The temperature, adhesiveness of toner components may be evaluated via the laboratory bonding test by substituting extruded toner with extruded toner components and adjusting the bonding temperature to the range at which bonding starts developing (Table 3).

The date shown in Table 3 was obtained for ASA-sized Generally, paper-polymer adhesion paper. varies significantly depending on the sizing type for pure resins and resin mixtures (see Table 1). An analysis of resin-flow characteristics is sufficient not to predetermine the bonding characteristics of printing papers that are currently used in electrophotographic copying and printing

WETTABILITY AND PENETRATION

majority of Today, the synthetic-sized. plain, bond-type printing and writing papers are sized with so-called cellulose-reactive sizes of the AKD or ASA types (see Fig.8). Hydrophobic tails of the reacting molecules (the "R" groups in Fig.8) are sought to create the sizing effect, possibly assisted by the molecular mobility of the tails to "sweep" over a large area of untreated fiber surface (7). This explains why relatively small sizing amounts create very effective sizing levels. The details of the reaction mechanism have recently been investigated, as described elsewhere at this symposium (14,15).

Resin Type E	Nonding Temperature (°C)	Bonding Strength (10- ³ kg)
Styrene/butyl	95	0.0
methacrylate	100	10.5 ± 2.2
	105	22.0 ± 9.4
Polyvinyl butyral	115	0.0
	120	8.1 ± 1.1
	125	19.5 ± 6.2
	130	23.4 ± 5.5
Polyamide	70	0.0
-	75	11.9 ± 6.5
	80	45.2 ±15.7
Butyl methacrylate	/ 105	0.0
methyl mathacryla	te 110	12.9 ± 5.0
	115	20.8 ± 5.9
Styrene/allyl alco	hol 85	0.0
• • •	90	7.3 ± 3.2
	95	10.5 ± 2.3

Table 3. Bonding Strength of Toner Components

Contact Angle

Wettability assessments through contact-angle measurements are sometimes carried out to estimate the level of sizing applications. Unfortunately, the variety in sizing treatments of commercially available bond-type papers creates a variation in angle response that strongly depends on sizing type, liquid type, and other experimental conditions, such as drop size, contact time, and paper structure. This makes it difficult to analvze data in terms of sizing-application levels when differently sized papers are compared.

Figures 9 and 10 show contact angle measurements obtained using a variety of purified liquids that are often applied in contact-angle measurements and subsequent



Fig 8-Cellulose-reactive size formulations.

surface-energetics analyses of smooth, solid surface $(\underline{16})$. It is apparent that none of the liquids provide a stable, constant value at the onset of wetting on the rosin-sized paper (Fig. 9). For the ASA-sized paper, four of the eight liquids appear suitable for experimental readings (water, glycerol, thioglycol, and methylene iodide in Fig. 10). This is consistent with experience of AKD-sized paper using methylene iodide for sessile-drop measurements $(\underline{1,2})$ (see Table 2, columns 4 and 5).

Pure liquids and liquid mixtures have both been used past to assess the decrease in paper-polymer in the adhesion through an associated decrease in wettability This approach is helpful when dealing with sizing (2,5). levels rather than with sizing types (see Fig. 1 and Table determination of surface-energetics 2). The characteristics (Table 2, column 5) appears to be more valid when it is carried out on nonporous films than on gas-chromatographic technique paper. for which the described next is more suitable.



Fig 9-Contact-angle variations of selected liquids on rosin-sized, bond-type papers.



Fig 10-Contact-angle variations of selected liquids on ASA-sized, bond-type papers.

Capillary Penetration

The difficulties in defining wettability through contact-angle measurements may be alleviated by using liquids of lower surface tension that penetrate into the paper structure. Theoretically, the penetration rate into a paper strip suspended in the fluid (by wicking) may be described through the Lucas-Washburn equation, according to which :

$$h^{2} = (r \gamma \cos \theta t)(2 \eta)$$
(1)

where h = Liquid rise (height at time t)

- r = Effective capillary radius
- γ = Liquid surface tension
- η = Liquid viscosity
- θ = Contact angle between the liquid and the solid paper-fiber surface

Measurements of paper penetration have been carried out many times in the past. The TAPPI capillary test (TAPPI Method UM 451) was developed based on the so-called Klemm test used by the Forest Products Laboratory and described by Simmonds (17). Much later, Everett et a1. (18) at this symposium series described simple а gravimetric technique that allowed the continuous recording of the absorption rate using an electronic microbalance. liquid penetration into Back (19) measured the sorbing (unsized) papers using water and mineral oil.

Aberson (20) demonstrated how the static-capillarity test may provide both capillary size and contact-angle data. Using water without added surface-active agents for penetration measurements on the same wood pulp, he derived the contact angle for water, assuming that the only penetration rate that determines the parameter for water with an added agent was the effective capillary radius ($\cos \theta$ = in Eq.1). Based on this approach, we propose to use pairs of liquids for capillary-penetration measurements on the same printing-paper sample.

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Rewriting Eq.(1) for liquids 1 and 2 and deriving the ratio betweem the individual equations :

$$(h_1/h_2)^2 = (\gamma_1 \cos \theta_1 t_1 \eta_2)/(\gamma_2 \cos \theta_2 t_2 \eta_1)$$
 (2)

eliminates r.

Choosing liquid 2, for which $\cos \theta_2 = 1$ (complete wettability) provides the contact angle of liquid 1 as :

$$\cos^{\theta_1} = (h_1/h_2)^2 \left({\gamma_2 \ \eta_1 \ t_2} \right) \left({\gamma_1 \ \eta_2 \ t_1} \right)$$
(3)

Note that no assumptions are made regarding the effective capillary radius. However, it is required that neither liquid swell the fiber matrix to maintain constant r.

far, our data base indicates that the above So approach is better suited for characterizing printing paper than the direct measurement of contact angles of sessile drops on the paper's surface. Using benzyl alcohol $(\gamma_1=39.0 \text{ mN/m}, \eta_1=5.8 \text{ mNxs/m})$ and hexadecane $(\gamma_2=26.7 \text{ mN/m}, \eta_2=3.3 \text{ mNxs/m})$, and assuming that $\cos \theta_2=1$ hexadecane, the $\cos \theta_1$ ranged from 0.51 to 0.84 for 24 for rosin-sized paper types and from 0.23 to 0.77 for 18 synthetic-sized types. All papers analysed obeyed the theoretical requirement between penetration time and height given by Eq.(1). A lowering of $\cos \theta_1$ for both rosinand synthetic-sized papers corresponded to a decrease in adhesive strength, as measured by the laboratory bonding selected paper types that had been used for test for electrophotographic copying and printing (see Fig.11).

These results are encouraging in view of the lack of sizing-test procedures suitable for the characterization of both clay-filled, acid-made, rosin-type printing papers and carbonate-filled, alkaline-made, ASA or AKD type papers. А inherent to capillary penetration is that bulk and weakness surface fibers are wetted, so that papers that have been coated give misleading heavilv surface-treated or can As for extrusion coating with polythelene (5), results. and cellulose-reactive sized affect polymer rosin both adhesion. This is evident from the use of an increasingly



Fig 11—Adhesive-strength variations of bond-type papers of varying contact angles as measured by liquid penetration.

greater variety of chemically different paper types in printers (see Table 4). Rosin-sized papers may create poor paper-toner adhesion (sample F in Table 4); cellulose-reactive sizes create а large variability in adhesion, as illustrated by the low-to-high degree of wettability (cos θ_1 for samples B-E and G-H in Table 4).

Sample	Size Type	Fuse Rating	$\cos \theta_1^*$
Α	Rosin	Very Good	0.84
В	AKD	Good	0.77
С	AKD	Good	0.64
D	ASA	Fair	0.65
Е	AKD	Poor	0.54
F	Rosin	Poor	0.51
G	ASA	Poor	0.35
Н	ASA	Very Poor	0.23

*As measured by capillary penetration of benzyl alcohol.

Table 4. Examples of Chemically Different Paper Types Used for Electrophotographic Copying and Printing

SURFACE ENERGETICS

surface energetics of cotton-filter paper can be The measured using the gas-adsorption technique described by Dorris and Gray (21). The technique provides the London dispersion-force contribution to the surface energy, which for cotton fibers was $48 \pm 3 \text{ mN/m}$, a good agreement with the expectations based on contact-angle measurements of solid Recently, Gray (22) has described the theory and films. the applicability of this technique to the reviewed characterization of cellulose and wood fibers.

have found that the technique is successful in We characterizing commercially made printing of papers low-to-moderate energy levels (30-45 mN/m), and that a majority of paper types used for electrophotographic that category (see Table copying and printing belong to 5). Extending the paper range to include high-quality, water-marked. cotton-bond, writing and printing papers gives an increase in the energy-level range, as shown in correlation Fig.12. However, the between surface energetics and adhesive strength is still apparent. Τn this case, high erasability ("Number of Corrected Characters" in Fig.12) was determined using resistive-ribbon, thermal-transfer technology (R2T2 technique), where character correction is measured after remelting and lift-off of the polymer from the paper (23).

oraring type

Surface Energy* (mN/m)

None	-	Whatm	an No.4 filter paper	50.3
Rosin	-	No.4	Xerographic (A)	45.5
Rosin		No.4	Xerographic (B)	42.5
Rosin		No.1	Xerographic (A)	38.8
Rosin		No.1	Xerographic (B)	38.7
ASA		No.4	Register bond	40.0
AKD	-	No.4	Bond	35.8

*Using C7/Cg absorption peaks.

Table 5. Examples of Printing Papers Suitable for Gas-Adsorption Analysis





Fig 12—Resistive-ribbon thermal-transfer correction of bond-type papers of varying surface energetics.

The of surface energetics measurement using gas absorption allows a more reliable analysis of both rosinsynthetic-sized types contact-angle and paper than or capillary measurements penetration. hydrocarbons The are more suitable than polar liquids for the analysis of а variety of sized papers and there is less ambiguity procedures. regarding validity of calculation the Unfortunately, technique time-consuming the is and. therefore, less suitable as a routine method in the paper mill.

CONCLUSIONS

The joint-strength measurements obtained illustrate the effect of sizing nature and amount of paper-polymer adhesion. Bond-strength development depends a great deal on the chemical nature of the paper substrate, and, for printing papers, it is best characterised by direct measurements on bonded paper strips.

Wettability assessments through contact-angle measurements are of limited value, particularly of In contrast, wettability measurements rosin-sized papers. capillary penetration appear to be far better suited via and correctly predict a lowering of adhesive strength caused by poor wettability.

gas-adsorption technique applied by Gray The and Dorris for determining the London-force contribution to the surface energy of cellulosic pulp and papers is applicable to the characterization of a majority of chemically far more complex printing papers used for electrophotographic and printing. copying The technique is perceived as generating data that is independent of sizing type and. therefore is suitable for bond-strength predictions based solely on the lowering of surface energetics. Currently. however, this technique appears to be more appropriate for the research laboratory than for the quality-assurance testing laboratory.

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Transcription of Discussion

Sizing and its Effect on Paper-Polymer Adhesion by J. Borch

S.U. Hossain Kimberly Clark Corp., Neenah, U.S.A.

You reported that the surface energy of Whatman filter paper was 50 ergs/cm². Water has a surface energy of 72 ergs/cm² and yet water wets the filter paper very well. Would you like to comment on that?

Dr. J. Borch I would rather like you to refer to Dr. Gray's work on the effect of water on cellulose. Our value of $50 \text{ } \text{ergs/cm}^2$ is slightly higher than his value of 48 ergs/cm².

Dr. J. Mardon Omni Continental, New Westminster, Canada

Did you consider looking at the effect of different paper surfaces, e.g. the top and bottom of a Fourdrinier sheet or the two sides of a twin-wire formed sheet or on the effect of filler?

Borch You are probably referring to the contact angle technique which we did not pursue. We used the penetration method which does not distinguish between the surfaces. I should add a word of warning about using that technique on a material which is not a typical bond type paper where you have a slight amount of surface starch and 10-15% filler. In these cases, the penetration will not follow the Lucas-Washburn equation because you get inhomogeneous wetting on the surface compared with the interior of the sheet.

F.E. Masterman MacMillan Bloedel, Vancouver, Canada

Did you try any mechanical papers with no sizing?

Borch We are just in the process of doing that and initial results look promising in that penetration appears to follow the Lucas-Washburn equation. We have not done any work on mechanical papers using the more time consuming gas adsorption procedure.

Masterman I would just like to add one further point. We were talking about self-sizing and one of the interesting things is that when the temperature in your 3800 is somewhat over 400° F, the resins come out at such a level that they drip all over the place. So there are lots of possibilities for sizing even in unsized paper.

Borch Most papers fortunately do not behave that way in the 3800 because the resin levels are not that high.

Aspler The surface energy, as measured by Dorris and Grav's GC technique, is London or the non-polar contribution to the surface free energy of the cellulose. You also have to account for the polar contribution which for cellulose is extremely variable and is not very well understood at all. Have you considered the effect of surface roughness on the adhesion of these toner particles in the thermal transfer process? You would think that the range of surface roughness exhibited by commercial papers would have a very significant effect on the adhesion.

Borch The surface roughness appears to affect the adhesion according to the kind of fixing process you are using in the printer or copier. The Xerox Corporation have demonstrated that there is a roughness effect when you are using a hard fuser roll. The hardness of the roll will determine the contact between the roll, the toner and the With soft roll fusing, roughness seems to have far paper. less effect unless you go outside the normal roughness range expected from normal bond papers. The over-riding effect will be of sizing and surface chemistry.

Prof J. Marton Westvaco, Laurel, U.S.A.

The surface free energy influences not only the strength, but also the slipperiness of the paper adhesive surface. Slippery paper can, of course, cause quite a few problems and headaches for the papermakers. Would you care to comment on whether you found a connection between surface free energy, adhesive strength and surface slipperiness?

It is difficult to see correlation between Borch these properties because papermakers tend to modify the frictional characteristics of the paper by modifying the surface sizing. Using our gas adsorption technique, of do not see any difference in the energy values course. we for the surfaces only. You still get a low energy value which would correlate with the normal friction co-efficient in the range of 0.5 to 0.6 paper/paper friction.

Prof W. Scott Miami University, Oxford, U.S.A.

Your data showed that the synthetic sized papers have lower bonding strength than the rosin sized papers. Could you comment on that with regard to the end-use performance of these papers?

Borch Yes, you are right, on average, the synthetic sized papers would have a lower bond strength, but we have seen examples of rosin sized papers with lower bond strength too. It all depends on the magnitude of the strength loss. If the papermaker detects a loss, he will counteract it by adding less synthetic size.