

**IMPACT OF INK/PAPER INTERACTIONS ON PRINTABILITY
OF AQUEOUS PUBLICATION GRAVURE INKS
PART II. Sorption of Liquids by Paper During Ink
Setting and Drying**

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Abstract: Liquid transport behavior of water and water based varnishes in contact with paper was studied using different experimental techniques (electrical, optical and the Fibro 1100 DAT Dynamic Absorption Tester). It was found that the Dynamic Absorption Tester delivers very useful information about liquid spreading, but not liquid sorption for very short times of contact ($t < 1$ sec). Interactions of polymer ink binders with paper substrate are of great importance for printability and ink drying. The amount of water drained from the wet ink film into the substrate during the time needed for the ink to travel from the nip into the dryer depends very strongly on the chemistry of the system (ink and paper). Filtration theory can be used to describe the process of water drainage from a liquid ink film into a substrate.

Introduction

Solvent (water) release from a printed ink film is usually associated with drying and achieving ink film hardness sufficient to pass undamaged through the press rollers and, eventually, to meet the final scuff and other resistance properties.

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In the very first moment of ink transfer in the printing nip, the two processes responsible for solvent (water) removal start simultaneously: evaporation and sorption into the substrate. These processes run at varying rates. Between the nip and the dryer sorption usually prevails; but later, evaporation becomes dominant. Substrate porosity, oven temperature and air exchange rate, and solvent affinity to solid components of the ink (often referred to as solvent release property), all contribute to ink film drying. In the case of solvent publication gravure inks, the interactions between ink and paper are physical in nature. With water based inks, the process is further complicated by the chemistry occurring at the ink/paper interface.

Since the precise mathematical description of such a system is unattainable, interpretation of sorption experiments is usually based on rather abstract models. One of the most popular is the Lucas-Washburn theory, which correlates penetration distance with time and the system parameters.

$$l^2 = \frac{(br) \cdot t \cdot \gamma \cdot \cos \theta}{2 \eta} \quad [1]$$

where: l - distance of penetration; (br) - bed porosity; t - time; γ - surface tension; θ - contact angle; η - viscosity.

In essence, this equation describes transfer of a liquid from an infinite reservoir through a cylindrical capillary of a constant diameter (Ross and Morrison, 1988; Rosen, 1978; Danino and Marmur, 1994). The resemblance of such a model to a paper sheet which represents an anisotropic system of capillaries varying in shape and size is only remote, at best. Consequently, in the literature, demonstrations of the utility of the Lucas-Washburn model are as frequent as examples of its irrelevance to the paper/water system. Nonetheless, attempts have been made to modify the Lucas-Washburn model to account for paper swelling and other irregularities of the real life systems (Hoyland, 1978).

In fact, some literature dealing with liquid transfer in porous media other than paper (Kissa, 1981; Danino and Marmur, 1994), is helpful in understanding the phenomena occurring in systems of our interest. However, most of the literature on the subject is related to manufacturing of coated paper (Salminen, 1988; Eklund and Salminen, 1986; Beazley et al, 1970), and only a few articles deal with sorption as related to the printing process (Bannerjee et al, 1993; Jeske, 1990). Certainly, many similarities exist between these two processes. In both cases, a dispersion is applied onto the paper surface, and solvent (or water) eventually has to be removed from the system. Both processes are carried at a high speed, and for a short period of time a high pressure is applied to the paper sheet in a nip. However, the differences between printing and coated paper manufacturing are profound and numerous enough to seriously limit the usefulness of too far reaching parallels.

The literature describes the following techniques for measuring penetration of liquids into porous media: (1) Electrical conductivity measurements (Hoyland, 1978; Mark, 1969); (2) Optical measurements, using Kubelka-Munk theory (Anderson and Higgins, 1985); (3) Bristow Tester (Lyne and Aspler, 1982); (4) Modified Bristow Tester, (Eklund and Salminen, 1986); (5) Gloss decay measurements (Young et al, 1993); (6). Fluorescent indicators (van den Akker and Wink, 1969); (7) Ultrasonic measurements (Pan et al, 1988); (8) Dynamic Absorption Tester (Fibro 1100 DAT).

Four of the above methods can be used for evaluation of the liquid penetration rate within time periods shorter than 1 second (2,3,4,7). However, some phenomena start with the first contact of the liquid ink with the paper and proceed to completion largely within 0.5 second. Unfortunately, evaluation of the sorption phenomenon within such a short period of time is difficult. Furthermore, investigating these phenomena after a longer period of contact is irrelevant to many aspects of print quality. Therefore, in the course of this study every effort was made to pay

attention to phenomena taking place immediately after the liquid had been brought in contact with paper.

The purpose of this study was to investigate penetration of various liquids into three different types of paper substrates: supercalendered (SCA), light weight coated (LWC) and 60# heavy weight coated (HWC). The liquid media used were water, solution and dispersion water based varnishes, as well as finished water based publication gravure ink. Penetration time was measured using the electrical conductivity and optical methods and, in case of pure water, using the Fibro 1100 DAT instrument.

Experimental

Materials

Three types of substrates commonly used in publication gravure printing were selected for this study: (1) 35 lb supercalendered (SCA) from Myllykoski Paper Co. (Finland); (2) 40 lb light weight coated (LWC) from Champion International Corporation; (3) 60 lb coated from Westvaco.

These substrates were described in detail elsewhere (Sprycha and Hruzewicz, 1994). Additionally, handsheets of paper of different thickness, prepared of the same feedstock (bleached Kraft pulp) were obtained courtesy of International Paper Co. These samples were used to verify the performance of our in-house designed laboratory instrument for studying penetration of water through a paper substrate, as well as for evaluating the contribution from the surface vs. the bulk penetration of the sheet. The instrument readings are based on measurements of electrical conductivity. The paper samples were stored in a controlled environment chamber at temperature 20°C and 50% relative humidity.

Methods

The Fibro 1100 Dynamic Absorption Tester, by FIBRO System ab, was used to measure the spreading of water droplets on the paper surface and

the following sorption into the paper. It appeared that this instrument facilitates accurate measurements of contact angle and water spreading within very short periods of time, and sorption phenomenon within longer periods of contact time, e.g. complete sorption of a water droplet of a given volume. It fails to accurately measure sorption within very short times of contact.

The time of an aqueous solution's penetration across a paper sheet was determined by detecting local changes in the electrical conductivity of the paper. Mark (1969) described a conceptually similar method; however, he needed some 10 grams of fluid vs. 1 droplet (ca. 8 mg) needed in our method. This small amount of liquid made our method much less sensitive to "pinholes", (Beazley and Climpson, 1970) always present in a sheet of paper, and remarkably improved its accuracy compared to that of Mark's method. Both methods rely on electrical conductivity of the penetrating fluid. The circuitry consisted of two pairs of electrodes connected to an electronic timer. The first pair would switch the timer on at the time $t = 0$, i.e. at the very moment when the droplet was brought into contact with the paper surface, and the other would switch the timer off, at the moment when the liquid arrived at the other side of the sheet. The current needed to control the timer was about $10 \mu\text{Amps}$ at a resistivity of $10^5 \Omega$. The accuracy of the measurements was ± 0.01 second.

Most of our measurements, within a range of longer penetration times, were reproduced using an optical method, which employed changes in the paper reflectivity. Photometers are often used to clock the end point of penetration, but we found a visual method using a microscope with a video camera quite satisfactory. The visual method is not applicable for short-time measurements.

There is also a convenient method of determining the initial rate of solvent (water) release from a coating or ink film based on gloss differences between the wet and (semi-) dry film

(Young et al, 1993). Usually, an automatic glossmeter is used to follow the gloss changes upon the water loss. It was determined in the course of this work that the visual observation was accurate enough provided that the semi-dry ink film was distinctively flat. The ink film was applied using a series of Mayer rods, from #2 to #13, and a basic electronic stop watch was used to measure the elapsing time.

Results and Discussion

The contact angle of a water droplet on different paper substrates vs. contact time, as measured on the Fibro 1100 Dynamic Absorption Tester, is presented in Fig.1.

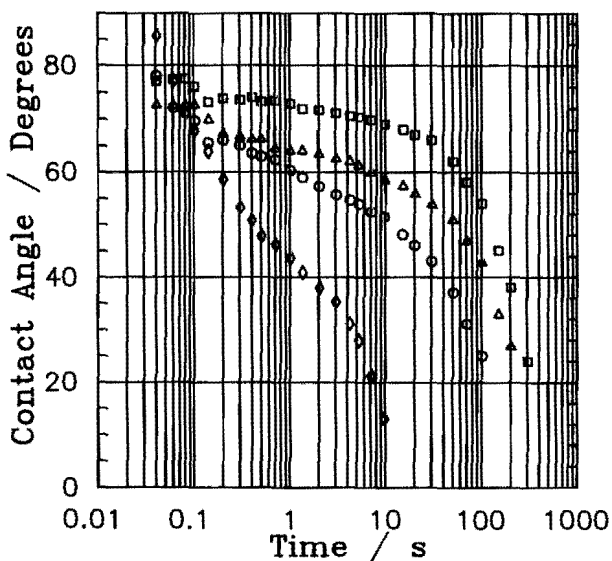


Fig. 1. Contact angle vs. time; Fibro 1100 DAT data for water. O 35 lb SCA; Δ 40 lb LWC; \square 60 lb HWC; \diamond 45 lb handsheet.

It is important to remember that, in spite of its commercial name, this instrument measures only the height and base of the droplet - from which its computer calculates the respective contact angle values. These, in turn, may or may

not be related to absorption. Even on the supercalendered substrate, the amount of liquid absorbed from an 8 microliter droplet within the first 0.5 second was less than 2% of its volume. Such a small change in a droplet's volume has a negligible effect on the contact angle value, which is measured with an accuracy of ± 1 degree. In general, to incur a measurable change in the contact angle value, the volume of a droplet has to change by at least 3%. Depending on the substrate, it may take up to several seconds for such a volume change to occur due to the liquid sorption into the substrate. Therefore, the contact angle changes within these shorter contact times, measured with precision by the Fibro 1100 instrument at 0.02 second intervals, are related to dynamic wetting phenomenon and are not influenced by sorption at all (Sprycha and Hruzewicz, 1994). Of course, this is not to say that no sorption occurs at the same time: both sorption and wetting start in the moment of the liquid's contact with the substrate. However, in terms of impact on the contact angle value, the wetting and spreading phenomena prevail, at first. Then they quite rapidly achieve equilibrium, at which moment sorption starts taking over. Moreover, the penetration rate in the paper will depend on the direction, due to the anisotropic properties of this medium.

During the publication gravure printing process, the opportunity for sorption of the solvent (or water) is practically limited to the time period when the print travels between the printing nip and the dryer, i.e. 0.1 to 0.2 second. An illustration of droplet spreading and sorption, within such short contact times, can be seen in Fig. 2 (changes of contact angle vs. contact time, and those of droplet base vs. contact time are presented in Fig. 2a and Fig. 2b, respectively).

These data are in a good agreement with those obtained earlier using the Rame-Hart goniometer coupled with a video camera (Sprycha and Hruzewicz, 1994). On coated papers, there is a rapid initial change of contact angle related, mainly, to the wetting and spreading phenomenon and then the curve levels off. The slope of the latter

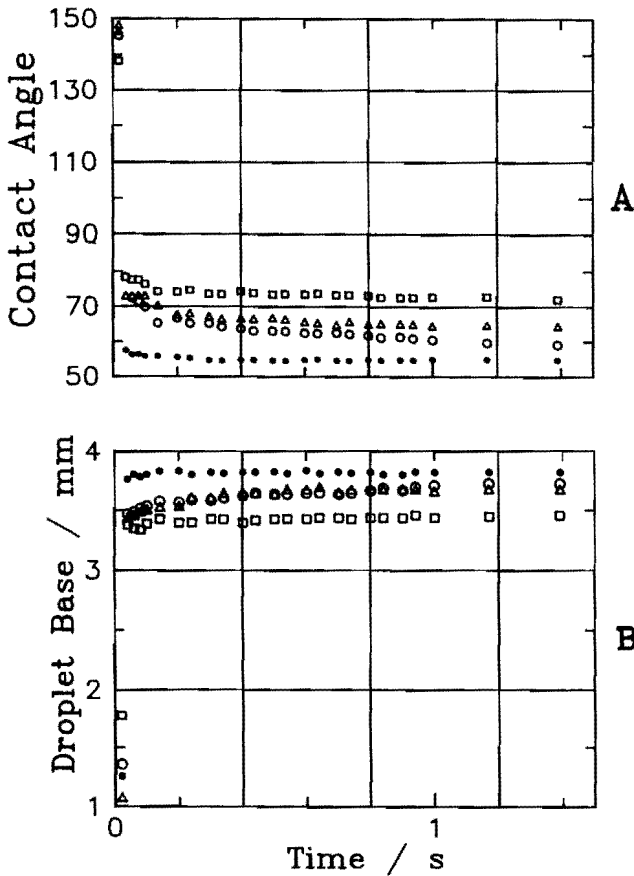


Fig. 2. Fibro 1100 DAT data for water; A) Contact angle vs. time; B) Droplet base vs. time; ○ 35 lb SCA; △ 40 lb LWC; □ 60 lb HWC; ● Leneta board.

part of the curve is steeper for supercalendered substrate due to its higher absorptivity. On non-absorptive substrates, like Leneta board, the end part of the curve is horizontal, which further confirms its close relationship to sorption.

As can be clearly seen in Fig. 2, droplet spreading is a very fast phenomenon. The changes in the droplet base size become negligible within 0.1 second on Leneta Board and 60# West-vaco paper, and within 0.25 second and 0.5 second on LWC and SCA substrates, respectively. Then, after some transition period, small and

steady contact angle changes occur which are related to the liquid sorption with no changes in the droplet base. On the Leneta board, contact angle is constant at this point. Finally, as the liquid in the droplet becomes depleted due to sorption, the droplet base becomes smaller and smaller, and the droplet disappears.

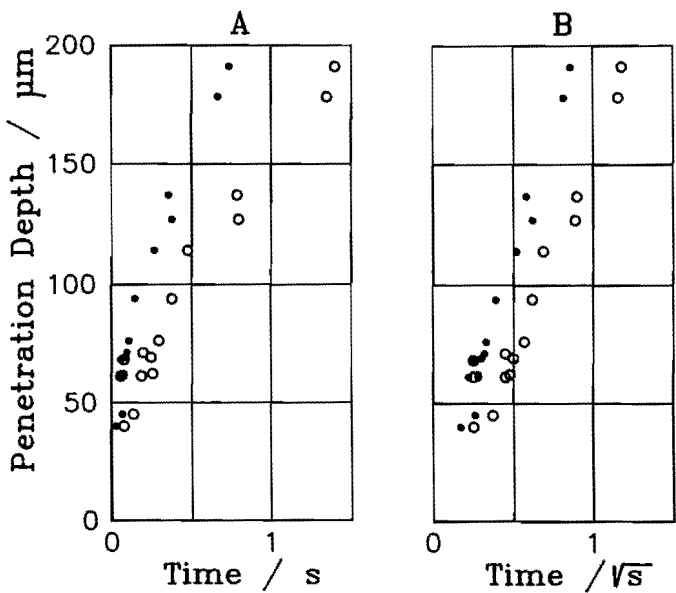


Fig. 3. Penetration depth vs. time A) Penetration depth vs. square root of time. B) O handsheet metal plate side; ● wire side.

The relationship of penetration depth vs. time obtained for both wire and metal plate sides of the IP handsheets is presented in Fig. 3. The linear and time square root relationships are presented in Fig. 3a and Fig. 3b, respectively. The penetration time was measured using the electrical conductivity instrument designed by us for the purpose of this study. The time square root curve fits well the Lucas - Washburn equation. The run of curves in Fig. 3b obtained for the metal plate side of the substrate may indicate occurrence of, so called, "wetting delay" phenomenon, existence of which has been argued for (Lyne and Aspler, 1982; Jeske, 1990) and against

(Salminen, 1988; Eklund and Salminen, 1986) in the literature. We do prefer using the term, "sorption delay", instead (Sprycha and Hruzewicz, 1994) for the following reasons: It is quite conceivable that the adhesion forces may require a certain amount of time to fully develop upon the contact of a liquid with the paper substrate. The delay in wetting would have to be very short, perhaps much less than 0.001 seconds, as evidenced by excellent ink transfer occurring routinely on high speed publication gravure presses. It would be quite impossible to detect such a short delay by any of the techniques described in the literature. Therefore, it is safe to assume for all practical purposes that the wetting delay is equal to zero. As the surface properties of paper substrate are very different from its bulk properties, however, the rate of the liquid penetration through the surface layer can be quite different, and some sorption delay can, most certainly, occur.

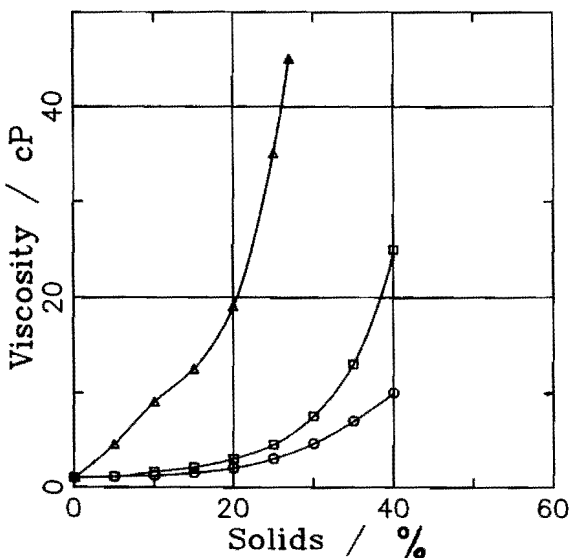


Fig. 4. Viscosity vs. polymer concentration; \circ modified polyester; Δ polyamide; \square sulfonated polystyrene.

Fig. 4 represents the viscosity of polymer solutions vs. their concentrations; the relationship of penetration time of the same solutions vs. their viscosity are presented in Figs. 5 through 7. The data were obtained using electric conductivity (Figs. 5a through 7a) and optical methods (Figs. 5b - 7b). Other properties of the polymer solutions are given in Table 1.

Table 1. Physical Properties of Polymer Solutions

	Sulfonated <u>Polystyrene</u>	Modified <u>Polyester</u>	<u>Polyamide</u>
% solids	30	40	60
Viscosity (cP)	110	10	260
pH	6.5	7.5	8.5
Conductivity (mS/cm)(5% solutions)	15.0	5.3	2.2

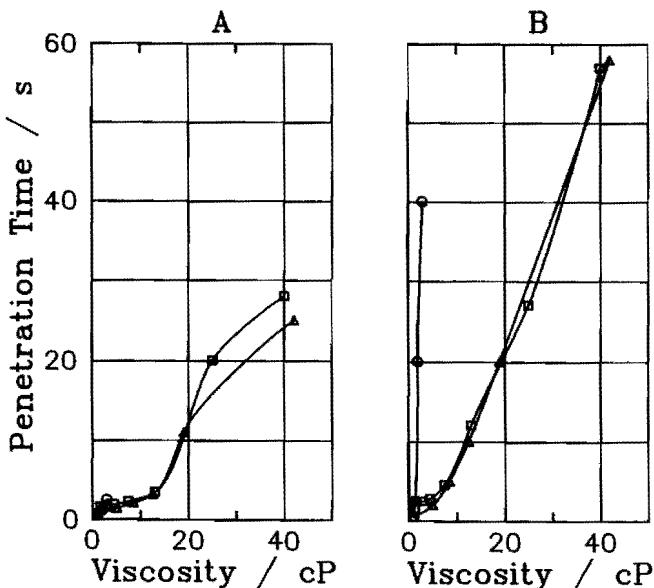


Fig. 5. Penetration time vs. polymer solution viscosity; A) electrical; B) optical; 35 lb SCA paper. Symbols are the same as in Fig. 4.

It is apparent that the optical methods yielded penetration times consistently longer than the electrical, even though no differences

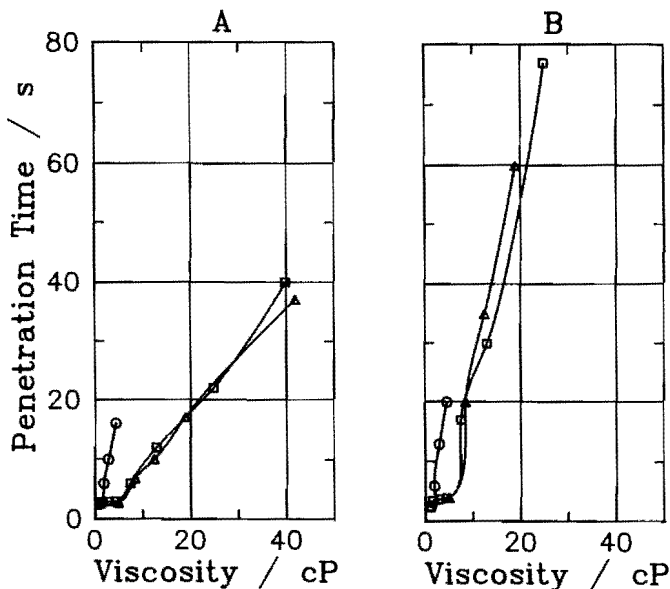


Fig.6. Penetration time vs polymer solution visc. A) electrical; B) optical; 40 lb LWC paper. Symbols are the same as in Fig. 4.

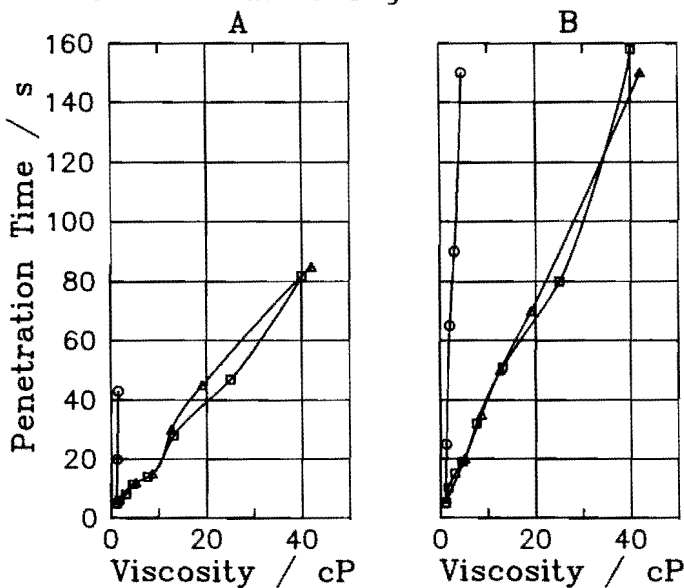


Fig.7. Penetration time vs polymer solution visc. A) electrical; B) optical; 60 lb HWC paper. Symbols are the same as in Fig. 4.

were noticed when penetration of pure water was measured. This was perhaps related to the elevated electrical conductivity of the varnishes in comparison to that of water. As a result, much less of the solution was required on the other side of the substrate to switch the electronic timer off.

More interestingly, the sorption curves depended very much on the chemistry of the polymers: those for a polyamide and a sulphonated polystyrene had quite similar runs, determined mostly by their viscosities, whereas a modified polyester curve indicated much slower sorption of the solution. Moreover, at higher concentration of the modified polyester, (in the case of SCA substrate even at the lower concentrations), the electrical conductivity method failed to measure the sorption, altogether. Although a detailed understanding of the chemistry of the system is lacking, we know that both acidic and salt forms of the polyamide and the sulphonated polystyrene polymers are soluble in water and, therefore, behavior of their solutions is rather insensitive to pH. An acidic form of the modified polyester, however, is insoluble in water. Therefore, on contact with the acidic paper, the polymer apparently precipitated from the solution and impregnated the surface with a gelatinous layer, hindering water penetration down into the substrate. This effect was the strongest on the SCA substrate, which was the most acidic of all. Importantly, this type of information on the substrate and polymer chemistry is very helpful to an ink chemist while formulating an ink for a specific substrate. In this case, it is quite obvious that a printing ink containing modified polyester will have better holdout properties, i.e. will prevent the pigment particles from penetrating into the substrate.

Analogous viscosity vs. concentration curves for a number of polymer dispersions and a clay dispersion are presented in Fig. 8, and penetration time vs. viscosity for the same varnishes are presented in Figs. 9 through 11. Other properties of the polymer dispersions are presented in Table 2.

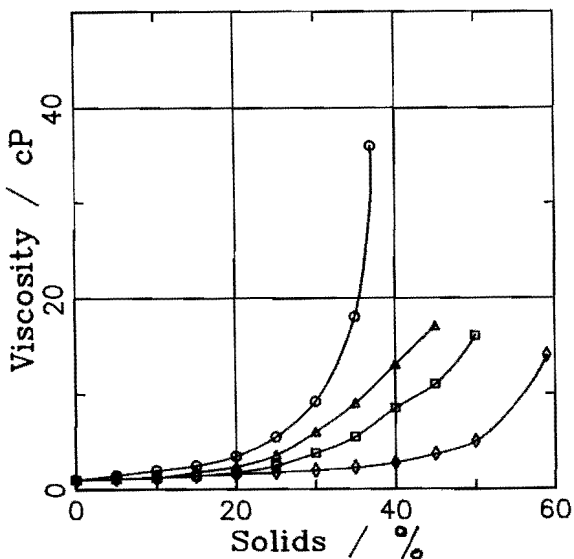


Fig. 8. Viscosity vs. dispersion concentration.
 ○ acrylic; Δ sulfonated polyester; □ carboxylated styrene latex; ◇ clay.

Table 2. Physical Properties of Polymer Dispersions

	<u>Acrylic</u>	<u>Polyester</u>	<u>Carboxylated Styrene</u>
% solids	49	44	49
Viscosity (cP)	500	17	16
pH	8.5	6.0	6.0
Conductivity (mS/cm)			
- 10% solids	5.0	2.3	3.0
- original varnishes	14.0	6.4	9.0
Particle size (nm)	130	110	130

Again, there were differences between the curves obtained by the electric conductivity (Figs. 9a - 11a) and optical methods (Figs. 9b - 11b), which could be related to the electrical conductivity of the varnishes (Table 2). Above and beyond these, the sorption rate was strongly

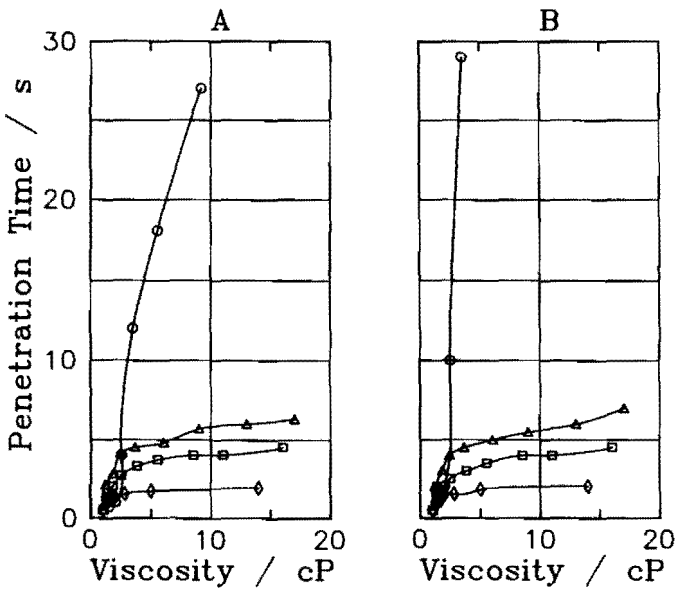


Fig. 9. Penetration time vs. dispersion visc.
 A) electrical; B) optical; 35 lb SCA paper. Symbols are the same as in Fig. 8.

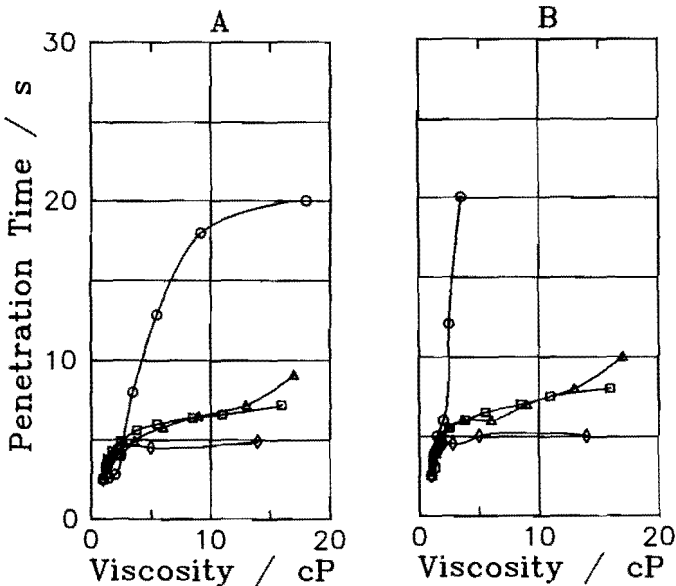


Fig. 10. Penetration time vs. dispersion visc.
 A) electrical; B) optical; 40 lb LWC paper. Symbols are the same as in Fig. 8.

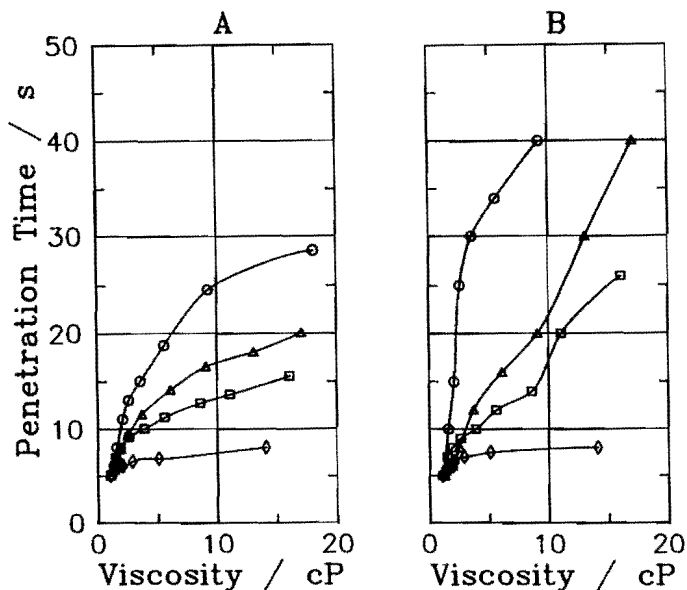


Fig. 11. Penetration time vs. dispersion visc. A) electrical; B) optical; 60 lb HWC paper. Symbols are the same as in Fig. 8.

dependent on the method of stabilization of a given dispersion.

The clay dispersion, stabilized primarily by the electrostatic forces, was the fastest to release water. A steric mechanism, on the other hand, is prevailing in stabilizing the acrylic dispersion and hence it released water much slower. Moreover, the acrylic dispersion precipitated at a pH below 7, which further slowed down water penetration into the paper substrate, more so with the more acidic papers (Figs. 9 to 11).

Printing inks are usually multicomponent dispersions. On contact with a paper, each of the components will penetrate into the substrate at its own rate: solvent is the fastest, and the bulky flocs of pigment and polymer are the slowest. Moreover, a sort of filtration cake is immediately formed which further slows the sorption process. All these processes can be very

detrimental or very beneficial to ink printability and drying, depending on their ever-shifting balance. In the case of solvent ink, all the phenomena are physical in nature, but with water based ink, chemistry further complicates the picture. The average pore size of uncoated paper is of the order of 1 to 2 microns, whereas that of coated paper is 0.02 to 0.48 microns (Zang and Aspler, 1995). Each of them will definitely require ink of distinctly different properties to provide adequate print quality.

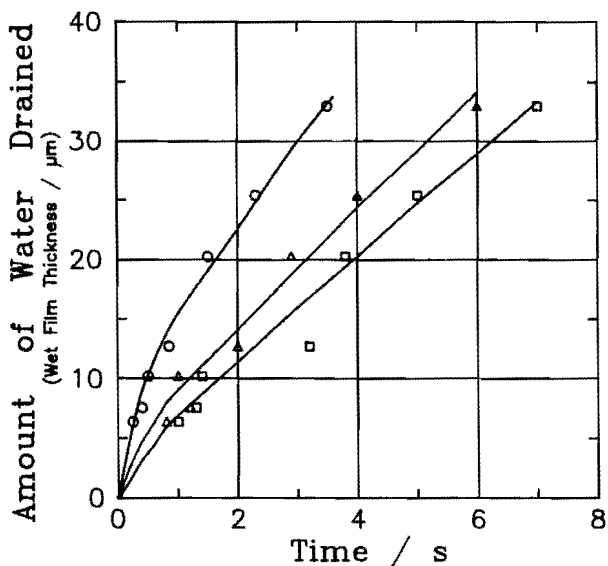


Fig. 12. Amount of water drained (expressed as wet ink film thickness) vs. time for experimental ink. \circ 35 lb SCA; \triangle 40 lb LWC; \square 60 lb HWC.

Fig. 12 represents the amount of water drained from the wet ink film vs. time. This relationship clearly depends on the type of paper and its porosity. The filtration rates drop with time due to the formation of a "filter cake" on the substrate surface. This is most evident on the SCA substrate, which is the most porous, to begin with, and the least on the 60# Westvaco.

After the "filter cake" had been formed, (or almost immediately for 60# Westvaco substrate), each curve acquires a linear course. The filtration rate is often described (Reed, 1988) by the Ruth form of Darcy's law:

$$\frac{Q}{A} = \frac{\Delta P}{\eta R} \quad [2]$$

where: $Q=dV_e/dt$ - volumetric flow rate; A - face area of the filtration bed; ΔP - applied pressure drop; η - viscosity; R - bed resistance.

The bed resistance consists of the medium resistance in series with the resistance of the deposited cake. For the printing ink/paper systems where the range of filtration times is very short it is reasonable to assume that Q is constant. Then the Equation [2] can be rearranged into the form:

$$\frac{V_e}{A \cdot t} = \frac{\Delta P}{\eta \cdot R} \quad [3]$$

where V_e is the volume of fluid drained from the ink film during the time t .

Multiplying and dividing the left side of Equation [3] by V_i (volume of ink applied) and substituting $V_i/A = x$ (the initial thickness of the ink film) and $V_i/V_e = K$, the Equation [3] becomes:

$$x = \frac{K \cdot \Delta P \cdot t}{\eta \cdot R} \quad [4]$$

The Equation [4] determines the filtration capability of the system and relates the thickness of the drained ink film to the time of filtration. It is evident from the equation [4] that if all the filtration parameters of the system are constant, the amount of printing ink drained by way of filtration will be a linear function of time.

The x vs. t relationship for 60# Westvaco (Fig. 12) is indeed almost linear over the whole range. This is easy to explain, as the paper coating layer on this substrate is quite thick and contains very fine pores. Therefore, the "filter cake" deposited on its surface from the ink film can very well be more porous and the paper coating prevails as the factor controlling the filtration rate. For the 40# LWC substrate, the relationship becomes linear in about 1 second after the ink film application. Apparently it takes about 1 second to plug the larger pores in the paper coating and establish a uniform porosity of the system. The same is true for the SCA substrate, except that formation of the filter cake controlling the equilibrium filtration rate takes longer with this substrate, due to its high initial porosity. The relationships presented in Fig. 12 allow for estimation of the amount of water drained from the ink film at different times, as well as the ink film thickness corresponding to the maximum water capacity of the substrate - information very important for predicting the ink drying behavior.

For example, for the systems studied here, ink film thickness which can be drained of water within 0.2 second is about 5, 2 and 1 micrometers for SCA, LWC and 60# Westvaco, respectively. Obviously, these numbers will vary depending on substrates and ink formulas. Zang and Aspler (1995) recently proposed the "cake formation" mechanism while discussing setting of oil based (offset) inks. They were able to successfully apply this model for coated substrates, but not for newsprint due to its irregular large pore structure.

Experiments reported here did confirm that the thickness of an ink layer drained was indeed a linear function of time, as predicted by Equation [4]. It is understood that for larger amounts of ink and longer filtration times, the filtration rate would eventually slow down and the curve start to deviate from a straight line.

Another aspect important for the gravure printing process which has been silently omitted

from this discussion, is the role of pressure in the sorption process. If the pressure is high enough, the capillary phenomena are assuming a secondary role in the sorption (Salminen, 1988). Accounting for the pressure in the printing nip would very much complicate this analysis and the difficulty is how to determine the effective pressure exerted on the ink being transferred. One certainly cannot use the impression pressure value, even upon correcting it for the nip width, because the ink meniscus in a gravure cell is concave (Jeske, 1990). The substrate roughness and the air present in the paper introduce further difficulties. It seems obvious that even a pressure impulse of 1 millisecond in the printing nip may have a significant impact on the ink sorption phenomenon. This, however, may become the subject of another study.

Conclusions

1. The transport of water in laboratory hand-sheets can be described with good approximation by the Lucas-Washburn theory.
2. The process of water drainage from a liquid ink film can be described using the filtration theory.
3. The Fibro 1100 DAT Dynamic Absorption Tester delivers very useful information about liquid spreading upon contact with paper for $t < 1$ sec., however, is much less accurate for sorption measurements within short time ranges.
4. Interactions of polymeric ink binders with paper substrates are of paramount importance for printability and ink drying. The amount of fluid drained from a wet ink film into the substrate during the time needed for the ink to travel from the nip into the dryer depends very strongly on the chemistry of the system (ink + paper).

Acknowledgments

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