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DETAILED INSIGHTS TO LIQUID ABSORPTION AND LIQUID–PAPER INTERACTION

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ABSTRACT

We present a method which provides detailed insights to the dynamics of the water absorption process and water-paper interaction, based on transmittance measurements of ultrasonic beams. We found that the water absorption process of an uncoated paper-sheet comprises two consecutive time regimes. The underlying mechanism that governs the regimes' shift is the combination of fibre surface modification by water and the recreation of the fibres lumen after wetting. In the first regime, water advances along the dry pore surface, which is hydro neutral, and the water forms a solid column inside the capillary (pore), while in the second regime, moving along the primed (wetted) surface of the capillary is a more favourable path as the surface becomes hydrophilic when wetted. Consequently, the water may not necessarily fill the entire capillary when the capillary expands in volume due to hydro-expansion and hence forms a hollow water column. We propose a model that enables us to determine/predict the depth of water absorption by the dry pore structure of the paper which is often the case for ink-paper interaction during printing. The results of our studies suggest that the depth of water penetration along the dry pore surfaces can very well be described by the Bosanquet model.

1. INTRODUCTION

Absorption of ink, ink vehicle, or colorants by a paper substrate is important for the final print quality, such as print gloss, print density, print mottle, print through etc. The major paper properties that influence the ink absorption include topological characteristics, surface energy, porosities, and pore-size distributions (Alam et al, 2007; Kettle et al, 2010; Lamminmäki, 2012). The major ink properties for the ink absorption are ink viscosity and surface tension. When it comes to ink–paper interaction, contact angle is the most important parameter. Understanding the dynamics of the ink absorption into the papers pore structure forms the base for understanding the ink-paper interaction and the print quality. In addition to printing, liquid–paper interaction has been identified as important for other types of applications, e.g., paper-based sensors (Liana et al, 2012).

There are a number of experimental methods often used for studying the liquidpaper interaction and the liquid absorption. The contact angle measurement indicates the trend for liquid spreading or wetting on the surface, as well as liquid absorption (von Bohr et al 2000). The Micro Drop Absorption Tester (MicroDAT) estimates the rate of liquid absorption by the paper substrate (Ström et al 2008). Both methods rely on detecting the contour of a liquid droplet above the substrate surface. Therefore, they are more applicable for studying coated grades than uncoated grades, whose rough surface and strong absorption are often problematic in measurement accuracy. Other methods like that of Bristow Wheel and Cobb Tester (TAPPI 1998; Aspler et al. 1987; Elftonson and Ström 1995; Swerin et al. 2008; Bristow 1967) are also frequetly used methods, with which the volumes of liquid absorbed by the paper substrate are measured. These techniques provide information about the total absorption volume over a long period of time or for a large area, which might not be relevant for printing. Moreover, they provide rather little insight into the dynamics of the liquid-paper interaction, for instance how the liquid moves in time inside the substrate paper and how the papers structure responds to the liquid absorption.

Physical models play an important role in understanding experimental observations and bring insights into a sophisticated ink-paper Winteraction process. The Lucas-Washburn (L-W) model (Washburn, 1921) is often used for studying ink (liquid) absorption, despite its singular problem at t=0 (Ridgway and Gane, 2002a). The Bosanquet model is an extension of the L-W model which has also been used in modelling capillary-force driven absorption processes (Bosanquet, 1923; Schoelkopf et al, 2000; Ridgway et al, 2002b). This model has recently been extended to a printing situation where a time dependent nip pressure is engaged (Yang 2013). Darcys Law describes the flow of fluid through a porous medium, which has also been used in studying the interaction between the flexographic ink and the paper (Xiang, Y. and Bousfield, 2000). All of the

above-mentioned models can be derived from the more general theoretical framework, i.e. the Navier-Stocks Equations. Modelling and simulation of the liquid–paper interaction with the Navier-Stocks equations has recently been demonstrated by the researchers (Dubé et al 2008, Holmvall et al 2011).

Among the previous studies, the work presented by Daun (2006) is particularly interesting because of its close relationship to the present work. In his PhD thesis, the author described in a great detail, the principle of the ultrasonic measurement device called Mutec PDA (penetration dynamics analyser) module PEA. He also presented the physical model of liquid penetration into one-side sealed pores and validated the model by comparison with measurements using model porous materials. These model materials were membranes of identical pore sizes corresponding to the pore radius 0.2 µm, 0.4 µm, 1µm and 3µm. He observed a transition of the pseudo-attenuation coefficient (to the ultrasonic beams) with respect to time from a lower level in the short time regime (t<5 s) to a higher level in the longer time regime (t>10s). This corresponds to a transition from a higher to lower ultrasonic scattering cross-section. However, when studying other porous materials, e.g. coated and uncoated paper samples, the opposite phenomenon was observed, namely, the scattering cross-section exhibited a transition with time from lower to higher scattering cross-section. The transition time was found to be between 0.1-1 s, which is significantly shorter than that for the membranes. The author claimed that the capillary suction is important for the initial penetration which however, is too fast to be measured with the device and the measurement captured by the device is actually diffusion-driven penetration.

Despite the profound progresses achieved in both experimental and theoretical fronts, there is still a lack of detailed knowledge surrounding the liquid-paper interaction. Technically speaking, how to obtain a detailed insight into the liquidpaper interaction remains as a challenge. One of the difficulties originates from the strong time-dependent nature of the interaction process. Moreover, paper is a composition of different materials, i.e. fibres, fillers, starch, retention aid. These materials form a porous network with convex and concave surfaces having regions of varying surface energies. Furthermore the interfaces that are in contact with the liquid undergo continuous modifications throughout the process (von Bahr et al 2003). A typical example is that the contact angle decreases rapidly after droplet deposition on an uncoated paper substrate and this declining trend continues with time. The strong time dependence of the interface properties posts a number of questions on the viabilities of the commonly adopted assumptions employed when studying liquid absorption or the liquid-paper interaction, which have so far not been appropriately investigated. For example, how reasonable is it to assume a constant surface energy of an interface (such as pore surface), or a constant contact angle between the liquid and the interface, or a constant pore structure of the paper despite its direct contacts with liquid? Is it appropriate to assume that a capillary-force is present and dominates throughout the liquid-absorption process? The answers to the proceeding questions form the base for the viability of the theoretical simulations dealing with the liquid absorption.

In this article, we propose a method that enables us to characterize the detailed dynamics of the liquid absorption and the liquid–paper interaction by combining the experimental measurements with the theoretical means.

2. METHODOLOGY

In this section we first explain the instrument setup and the measurement principle. We present then a physical model with which the measured values (ultrasonic transmittance) are converted to the depth of the liquid absorption. Finally, we describe how the depth of the liquid absorption can be parameterized into two or three parameters that are the collective representations of the paper and the ink properties, based on the Bosanquet model.

2.1 Measurements of the liquid-absorption dynamics

The Emtec Penetration Dynamics Analyser (PDA) module HVL consists of an ultrasound generator and a receiver, between which there is a vertically movable sample holder and a liquid booth beneath the sample holder (see Fig. 1, left). The paper sample is mounted to the measuring head by an adhesive tape. When the sample is in contact with the liquid (water in the present study), liquid absorption begins. The measuring head receives an ultrasonic beam with frequency 1 MHz, which propagates through the water booth and the paper. The signal that transverses the paper and the water is received by the measuring cell. The liquid is taken up by the paper sample thanks to the capillary force (see Fig. 1, right). While the liquid fills the pores, the scattering of the ultrasonic beam decreases and consequently the transmittance increases. The transmittance is sampled throughout the whole measurement process, from 0.05 s after the release of the sample's holder and at a minimum possible time interval of 0.003 s. Readers who are interested in the principle of ultrasonic measurement may find the PhD thesis a useful reference (Daun, 2006).

The paper samples used in the study are three commercial copy papers, coded as A, B, and C. Their grammage and thickness (caliper) of these papers are summarized in Tab. 1. Additionally, the time for the transmittance to reach its maximum (t_{max}) is also listed.

The dynamic transmittance of paper B along the liquid absorption process is shown in Fig. 2, wherein the values have been renormalized by setting the initial



Figure 1. Illustration of the Emtec PDA measurement device module VHL (left) and the measurement principle (right): the transmittance of the ultrasonic beam that propagates through the paper depends on the degree of pores filled by the liquid.

| Paper sample | Grade | Grammage (g/m²) | Calibre (µm) | $t_{max}(s)$ |
|--------------|----------|-----------------|--------------|--------------|
| A (King Sun) | uncoated | 80 | 99.4 | 0.518 |
| B (Hx Sun) | uncoated | 80 | 98.3 | 1.35 |
| C (Jinqiu) | uncoated | 80 | 101.1 | 0.626 |

| Table 1. | Descriptions of | the | paper | samples |
|----------|-----------------|-----|-------|---------|
|----------|-----------------|-----|-------|---------|



Figure 2. The dynamic transmittance of paper B in response to liquid (water) absorption. The transmittance initially increases with time then decreases after reaching its maximal transmittance at t= t_{max} . The initial value of transmittance at t=0 is set as unity, namely T(0)=1 or 100%.

value at t=0 as unity, i.e. T(0)=1 or 100%. The plot exhibits the typical characters for the uncoated paper grade. The evolution of the transmittance can be divided into two time regimes separated by the time when the transmittance reaches the maximum. We name the short term regime (t<t_{max}) capillary-force driven absorption and the long term regime (t>t_{max}) diffusion/wetting driven absorption. The time corresponding to the maximal (t_{max}) transmittance of each paper is listed in Tab. 1.

2.2 Determination of the depth of liquid absorption

Propagating in the paper sheet, an ultrasonic beam is attenuated because of absorption and scattering. Scattering occurs at a location where there is a discontinuity in material distribution in the wave path inside the paper sample, such as the surface of a pore. The scattering decreases while the pore is filled by the liquid. Hence the transmittance improves.

Let E_{dry} and E_{wet} be the attenuation coefficients of the ultrasonic beams in the paper sheet in its dry and wet states respectively, and D_0 be the thickness of the paper sheet in its dry state. The renormalized transmittance of the dry paper sheet may be expressed as

$$T_{drv} = T(0) = A\exp(-E_{drv}D_0) \tag{1}$$

where A is a constant that renormalizes the transmittance at t=0 to T(0)=1. While the liquid penetrates the paper, it gradually fills the pores. Hence the transmittance increases because of reduced scattering (attenuation). Assuming that in the regime 1 (t<t_{max}), the hydro expansion of the paper sheet is negligible when the maximum transmittance is reached, we have,

$$T_{wet} = T(t_{\max}) = A \exp\left(-E_{wet}D_0\right) = \exp\left[\left(E_{dry} - E_{wet}\right)D_0\right]$$
(2)

where T_{wet} is the renormalized transmittance of the thoroughly wetted sheet. From equation (2), we have,

$$E_{wet} - E_{dry} = \log(T_{wet})/D_0 \tag{3}$$

As the thickness of the paper sheet, D_0 , can be separately measured, the difference in the attenuation coefficients of the sheet in its wet and dry states can then be determined from equation (3).

Assuming the depth of the ink penetration in the first regime is X(t) (0<t<t_{max}), then the portion of the sheet remains dry is $D_0^-X(t)$. The corresponding (renormalized) transmittance can be expressed as

$$T(t) = A \exp\left[-E_{dy}\left(D_0 - X\right)\right] \exp\left[-E_{wet}X\right] = \exp\left[\left(E_{dy} - E_{wet}\right)X(t)\right] \quad t \in (0, t_{max}).$$
(4)

The depth of the liquid absorption can then be estimated from the measured transmittance, namely

$$X(t) = \frac{\log(T(t))}{(E_{dry} - E_{wet})}, \qquad t \in (0, t_{max})$$
(5)

2.3 Parameterizations of the depth of liquid absorption

According to the Bosanquet model (Bosanquet 1923, Schoelkopf et al 2000), the depth of liquid absorption as a function of time t can be expressed as,

$$X(t) = \left(\frac{2b}{a^2}\exp(-at) + \frac{2b}{a}t - \frac{2b}{a^2}\right)^{\frac{1}{2}},$$
(6)

The quantities a and b are constants defined in equation (7), which may be regarded as the collective properties of the paper and the liquid, namely

$$a = \frac{8\eta}{R^2 \rho}, \qquad b = \frac{P_c}{\rho} + \frac{2\gamma \cos\theta}{R\rho}, \tag{7}$$

where *R* is the radius of a capillary tube, ρ the mass density of the liquid column, η the viscosity of the liquid, γ the surface tension of the liquid and θ the liquid– paper contact angle. and finally P_e the external pressure exerted on the liquid ($P_e=0$ in the present study). The Bosanquet model has been used to study liquid absorption when the external pressure is constant or zero (Schoelkopf et al 2000, Ridgway et al, 2002a,b). When applied to a printing situation, the quantity *b* is strongly time dependent because of the time dependence of the external (nip) pressure. The solution is then more sophisticated which was recently worked out by the author (Yang, 2013).

According to equation (6), the liquid absorption can ultimately be characterized by a pair of parameters, a and b. This makes it possible to actually obtain these parameters by fitting the measured values (calculated with equation (5)) to the Bosanquet model (equation (6)). The agreement between the measured values and the fitted ones provides the evidences about the applicability of the model to the liquid absorption problem.

One can even obtain the contact angle by replacing the expression for *b* in equation (7) with the following, assuming that the external pressure $(P_e=0)$ is zero,

$$b = b_0 \cos \theta, \qquad b_0 = \frac{2\gamma}{R\rho}.$$
 (8)

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In this way, the contributions from the materials properties represented by the parameters, a and b_0 , are separated from that from the liquid–paper interaction, namely the contact angle. More details on the determinations of the parameters, a and b or a, b_0 and θ are given in Sec. 3.2.

3. RESULTS AND DISCUSSIONS

Figure 3 depicts the time dependence of the measured transmittance values upon liquid absorption for all of the three papers. The transmittance values have been renormalized by setting their respective initial values to unity. Despite the different times needed to reach the maximum for each of the papers (t_{max} is shown in the parenthesis in the legends in figure 3), there are clear similarities between these curves and all of them have two time regimes. The more detailed characteristics between the curves can probably be attributed to different paper chemistries. For example, the paper B has a longer t_{max} because of it is more heavily sized than the other papers.

The explanation of the behaviour in the first regime $(0 \le t \le t_{max})$ where the transmittance improves upon liquid absorption is rather straight forward, i.e., because of the filled pores by the liquid. But why the transmittance drops and continue to decline after the maximal transmittance in the long term regime $(t \ge t_{max})$ is not as



Figure 3. The dynamic transmittance values of the papers, the time (in second) corresponding to the maximal transmittance values are shown in the parentheses in the legends. The transmittance values of the papers are normalized to their respective initial values at t=0.

obvious. This will be the focus of Sec. 3.1. In Sec. 3.2 we will show the depths of liquid absorption obtained from the transmittance measurements and their comparisons with theoretical calculations.

3.1 Understanding the transmittance measurement and the liquid–paper interaction dynamics

When propagating in the paper, an ultrasonic beam attenuates due to absorption by the papermaking materials and due to scattering by the empty voids or pores. When the paper sheet is dry there are many voids or pores which scatter the ultrasonic beam. Upon liquid absorption, the voids and pores are rapidly filled by the liquid. The scattering decreases and consequently the transmittance increases. Hence the behaviour in the first regime is more or less expected. However, the behaviour observed in the second regime is largely unexpected because, intuitively, one would have expected that the transmittance would stay at the level of maximal transmittance as the paper sheet would have become saturated by the liquid. It is therefore surprising that the transmittance decreases with time and can even reach a lower level than the initial transmittance of the dry sheet. Considering the fact that the total amount of physical materials involved, paper and water, are constant throughout the measurement, it is reasonable to expect that the absorption attenuation of the ultrasonic beam is also constant. This implies that the reduced transmittance is very likely due to the increased scattering of the ultrasonic beam, which is in line with observations reported by Daun (2006)

Both the structure and the properties of the paper sheet undergo continuous modifications due to water up-take, which is probably the underlying mechanism that causes the increased scattering of the ultrasonic beams. There are a few possible mechanisms that probably contribute to the increased scattering resulting in transmittance reduction in the second regime. The fibre-wall swelling and hydro expansion of the paper sheet are the natural parts of the modification process, i.e., swelling of the fibres renews the volume of their lumens which were collapsed in their dry state. This is one of the possible origins that causes increased void space inside the paper hence enhanced scattering of the ultrasonic beam. The second possible explanation to the increased scattering is related to the modification of the fibre-surface chemistry by water. It is well known that water modifies the surface chemistry of the wood fibres, leading to decreasing contact angle of the liquid with the wall of the capillary with time. It implies a transition of the surface energy of the capillary wall from a lower state to a higher one. The time needed to reach its maximal transmittance, t_{max} is a measure of the transition time. Figure 4 illustrates the possible scenarios of the water-paper interaction in different periods of time. For simplicity we consider a capillary that is connected to a water bath of infinite supply. According to our simulation shown Sec. 3.2,



Figure 4. Possible scenarios of the water-paper interactions in the capillary-force driven and wetting/imbibition driven regimes. Top: the capillary-force driven regime ($t < t_{max}$) wherein water advances along the dry pore surface that is close to hydro neutral. The contact angle ϕ -90° and water fills the entire section of the capillary. Middle: the transition stage ($t < t_{max}$) wherein a void (unfilled) space may be recreated in the section with primed pore surface due to the combination of expanding pore size (hydro-expansion) with modified surface chemistry, moving along the primed surface is a more favourable path for the liquid water. Bottom: the wetting/imbibition driven regime ($t > t_{max}$) wherein the surface has been turned to be hydrophilic. The contact angle is smaller than 90° (ϕ < 90°) and continuously decreases toward zero with time. Hence, the water may only partially fill the capillary.

at the very beginning the wall of the capillary (void) is hydro neutral as the water front has a contact angle about 90°. Hence the water can fill the entire section of the capillary and forms a solid water column. After the regime transition ($t>t_{max}$) the capillary surface is primed and has higher surface energy. Consequently, moving along the primed surface is more favourable for the liquid than filling in the void space of the capillary as the contact angle becomes remarkably smaller than 90°. A void is therefore created. The void expands with time as the capillary surface becomes more hydrophilic and the contact angle becomes even smaller as illustrated at the bottom panel in Fig. 4.

It is important to rule out other possibilities, such as creating air bubbles in the water by the ultrasonic beams, which in turn cause increased scattering of the



Figure 5. The dynamic transmittance values of the bopp and kbopp plastics films. The transmittance values of the films are normalized to their respective initial values at t=0.

ultrasonic beams. Measurements with bi-axially oriented polypropylene (bopp) and kbopp (bopp base with PVDC coating) films have been performed. As shown in figure 5, the transmittance values are constants throughout the measurement time. This implies that there is no remarkable generation of air bubbles in the water by the ultrasonic beams.

3.2 The depth of water absorption

The depths of water absorption are obtained from the PDA measurements employing equation (5). The calculations are performed only to the first half of the first regime ($0 \le t \le t_{max}/2$), in order to avoid the complexities associated with the hydro-expansion as well as the regime transition. From a printing point of view, this time length is sufficient as the volume of the water absorbed during this period is definitely larger than that of ink in a printing situation. This is confirmed as the depths of water absorption are very close to the full papers thicknesses (about 100 µm) for all the three papers, see Fig. 6.

The depths obtained from the PDA measurements have further been used to fit the Bosanquet model given in equation (6), by means of the Least Square technique offered by Matlab. As shown, the depth based of the PDA measurement (dashed lines) determined by employing the equation (5) and those (solid lines) from data fitting to the Bosanquet model (equations (6)–(8)) are in very good agreements. The fitting parameters, *a* and b_0 depend on the properties of the paper



Figure 6. The depths of water absorption into paper sheets derived from the PDA experimental (transmittance) data (dashed lines) using Eq. (5) and by fitting to the Bosanquet model (solid lines), using Eqs. (6)–(8), in the first half of the first regime $(0 \le t \le t_{max}/2)$.

| Fitting parameters | Paper A | Paper B | Paper C |
|--------------------|-----------------------|-----------------------|-----------------------|
| a | 7.340*10 ⁵ | 7.130*10 ⁵ | 7.263*10 ⁵ |
| b | 0.0083 | 0.0033 | 0.0079 |
| b_0 | 3.067 | 2.879 | 3.377 |
| θ | 89.70° | 89.88° | 89.71° |

Table 2. The collective characteristic parameters of the paper and ink

and the water, while the contact angle θ on the water-paper interaction as defined in equations (7) and (8). The values obtained from the data fitting are collected in Tab. 2. For convenience, even the parameter *b* defined in equation (7) is shown in the table. Considering the fact that the pore radius is about R=10⁻⁶ m and the quantities *a* and b_0 are proportional to R⁻² and R⁻¹ respectively (equations (7) and (8)), the obtained parameters seems to be in reasonable order of magnitude. Unlike the quantity *a* which is similar for all of the papers, the quantity *b* for paper B is less than half of those for paper A and C. This can be attributed to the fact that paper B is more heavily sized, for which the contact angle is closer to 90° than those of papers A and C.

4. SUMMARY

We present a method that brings insights into the dynamics of the water absorption process and water-paper interactions. The surfaces of wood fibres undergo continuous modifications by water during the absorption process. Accompanying this modification there are two sub-processes on-going. First the wood fibres regain their lumens which were collapsed when they were dry. Second, the contact angle of water with the pore surface continuously decreases from initially 90° to smaller than 90° and then gradually toward zero. Because of these the water absorption process is divided into two time regimes. In the first regime, the pore wall is nearly hydro neutral and the contact angle is close to 90°. When advancing inside the paper, water fills the entire cross section of a void. On the contrary, in the second regime the pore surface becomes hydrophilic, the contact angle becomes smaller than 90° and moving along the pore surface is a more favourable path for the water. As a consequence water advances inside the pore only along the pore surface, leaving the pore space partially unfilled. When the contact angle further decreases with time, the unfilled void space expands. The combination of expanding unfilled pore spaces with regaining lumens of wood fibres lead to reduced transmittance for ultrasonic beams along the time.

We have also proposed a model that enables us to determine the depth of water absorption with time from the transmittance measurements in the first regime. We have found that the depth of water absorption by dry paper pore structure can very well be described by Bosanquet model.

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Transcription of Discussion

DETAILED INSIGHTS TO LIQUID ABSORPTION AND LIQUID–PAPER INTERACTION

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We have worked on printability research for quite some time, and of course, we have also looked at penetration of liquids. Would you go back to your slide where you show the wetting front travelling through the paper, and you have a straight line separating the wetted and unwetted parts of the paper? This is not what we have in paper. You are probably aware of the work of Pat Gane's group at Omya; they have demonstrated that we do not have a continuous wetting front travelling through the paper. We have a distribution of pore sizes and the small pores fill very quickly, and the larger pores fill more slowly, so what you actually have is some kind a fuzzy wetting front with very little wetting of the small pores at the leading edge and then more and more wetting as you travel back from there. So I am a little bit worried if this approach of a continuous front is really going to work in cooperation with the ultrasound measurements. Pat Gane and co-workers have shown that the distance between the initial front and the front where you have full wetting can be quite large, especially in the initial part of the wetting, and later on the large pores are picking up a little bit.

The other thing is, you showed that there are no bubbles generated by the ultrasound generator. We have also looked into the PDA measurements, we have filmed the imbibition process of the sheet, and what is actually happening as the water is pushing in, there are bubbles generated, not by the ultrasound, but by the air which is pushed out of the sheet. So what we actually have is a cloud of bubbles, which slowly moves upwards, and this interferes with the measurement in our experiments. We have seen very weird things with multiple peaks in the measurement, so we were really unsure how to interpret this result.

Discussion

Li Yang

Firstly, this measurement is over quite a large area, so it is more like an average. It does not trace the movement at each pore size. This model takes the average, including both small and large pores. Second, this approach incorporates very difficult to obtain printing relevant quantities like a contact angle and the effective pore diameter.

Torbjörn Wahlström Stora Enso

If we consider real printing conditions in a printing nip, would you say that there is a back pressure inside the sheet, under the liquid penetration, or can it be assumed that the pressure inside the sheet is atmospheric?

Li Yang

I think if you look at the ink penetration or liquid penetration, when you have extra pressure it is important which pressure is stronger and they compete with each other. I have presented an article on this last year in the Paper Physics Conference¹ where I introduced dynamic nip pressure, which is time dependent. I think in the normal printing situation, the nip pressure is higher but that is for a very short time that is even shorter than capillary driven processes.

¹Li Yang, "Ink dynamics and ink transfer under printing nip", In Proceedings of 2012 International Paper Physics Conference, Stockholm, pp 114–120, June 10–14, 2012.