

Water-based adhesive penetration into paperboard and coated paperboard

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ABSTRACT: The setting of water-based adhesives in contact with paperboard is important in the production of boxes and other packaging, but the topic has received little attention in the literature. The penetration of the adhesive into the paper surface is important to get good bond strength through mechanical interlocking. The influence of the process conditions and the paper properties on this penetration is lacking.

A water-based adhesive was applied to an uncoated and coated paperboard. The coating layer had two latex levels. Samples were characterized in terms of air permeability, void fraction, average pore size, and coat weight. The adhesive was applied to the samples in a mechanical press, squeezing a drop between two samples. The penetration of the adhesive was characterized with a silicone oil absorption method that measured the decrease in pore volume after the adhesive had been applied to approximate the pore volume taken up by the adhesive. The bond strength was measured with a peeling test.

The penetration depth into uncoated paper did not depend on the application method parameters such as pressing time, pressure, or initial solids of the adhesive. The penetration depth ranged from 35–40 mm. The penetration into 10 pph latex content coated paper was similar to the uncoated samples, but reduced penetration was observed into the 40 pph latex content coatings. The results were compared to the limiting amount of adhesive that was obtained from the weight gain of the samples and to a simple model that is based on flow in porous media. Peel tests revealed that good penetration was needed, as well as a strong coating layer to obtain high peel forces.

Application: The penetration of water-based adhesives into paperboard is important to understand in the converting of coated paperboard into boxes and other packaging applications. In this work, we look at the penetration of an adhesive into coated and uncoated paperboard and report how various parameters influence the results. The test methods and analysis may be of value to others working on this topic.

The penetration of water-based adhesives into a porous substrate is important in the setting rate and performance of water-based adhesives in the production of paper-based packaging. Aqueous adhesives are widely used alternatives to hot melt glues because of their costs and ease of use. Typical water-based adhesives that have been used for labeling are dextrin, starch based-jelly gums, ice-proof caseins, non-caseins, polyvinyl alcohols, formulated vinyl acetate ethylene copolymers, and acrylics. [1]

Adhesives are important for producing several products, such as corrugated boards and paper cartons. Water-based adhesives are set by water absorbed by fibers depositing the polymer in the joint and by the evaporation of water. When latex is used, as water leaves, the polymer forms a continuous film in the joint. This process increases adhesive viscosity as time progresses and forms a solid material as water evaporates. The setting of aqueous adhesives in the paper is unique since it may involve the adhesive changing properties as it penetrates due to water absorption by the fibers. While this is a common process and system, little theoretical work has been reported in the open literature on the parameters that control the setting rate and the final bond strength.

Adhesive penetration depths can be measured with cross-sectional images from microscopic techniques. Nin-

ness et al. showed how the aqueous adhesives setting is influenced by the coating layer and measured penetration of this adhesive using scanning electron microscopy and confocal laser scanning microscopy [2]. Various binders and different binders and levels have been used in the coating layer to see how that could impact the percentage of fiber-tear in the glued samples, but the penetration depth was not reported [3,4]. The setting of dispersion adhesives on paperboard was also studied: different adhesives with various viscosities were applied to a board while it connected to a tensile tester that measures force as time progresses. This study concluded that the rheological properties of the adhesive and the structure of the board surface are the key parameters that influence the setting process of dispersion adhesives [5]. Several papers are available around the penetration of adhesives into wood substrates, but less is reported for paper.

METHODS AND MATERIALS

Uncoated 336 g/m² bleached wood free paper supplied by SAPPI North America (Boston, MA, USA) was used in all experiments. Paper coatings were prepared using Acronal S 504 na styrene acrylate latex (BASF; Ludwigshafen, Germany) and a medium size particle kaolin pigment (Premier, Imerys; Paris) at 60% solids. The

	Thickness, mm	Basis Weight, g/m ²	Void Fraction	Permeability, m ²	Contact Angle, degree
Uncoated	430	336	0.5	3.0 × 10 ⁻¹⁴	118
10 pph	446	352	0.48	4.8 × 10 ⁻¹⁷	80
40 pph	446	352	0.44	1.4 × 10 ⁻¹⁷	84

I. Uncoated and coated paperboard properties.

paperboard (432 mm × 305 mm) was coated on one side with latex amounts of 10 and 40 pph (parts per hundred pigment) using a rod drawdown coater, followed by drying in an oven maintained at 100°C for 5 min. The coat weight for samples was about 15–17 g/m². The Aquence BG 0214 adhesive supplied by Henkel (Düsseldorf, Germany) was diluted with distilled water in some tests to determine the effect of adhesive solids on penetration; solids levels of 45% and 55% were studied.

Properties of the coated and uncoated samples were measured, including basis weight, thickness, void fraction, contact angle, and air permeability. The void fraction was determined using a silicone oil absorption method that measures the weight increase in the sample after saturation with oil, the weight increase divided by the density of the oil, and the sample area; the sample thickness gives the void fraction. The permeability was calculated from the Gurley porosity test time, knowing the thickness of the sample. The adhesive was characterized in terms of solids and viscosity. The viscosity was a strong function of solids and was reported for this adhesive by Khlewee [6]. More details regarding the materials, properties of the samples, and these tests methods can be found in Khlewee [6]. **Table I** reports the measured paper properties. The coated paperboard samples were slightly thicker, and the air permeability was three orders of magnitude lower than the uncoated sample.

The Model C carver press tester (Carver USA; Atlanta) was used to press the water-based adhesive between two paper samples. In this test, a syringe was used to apply a 0.4 mL spot on the bottom sample at ambient temperature. Another paper was placed on top of the spot, and the press was activated to a known pressure of 0.15 or 1 MPa and press times of 1, 10, 30, and 60 s. These two pressures were used after accounting for the cross-sectional area of the cylinder and the sample. The adhesive was pressed into the paper and squeezed radially. After release, the sample was removed and dried. After drying, a cut region from the center of the samples was taken with dimensions of 30 mm × 20 mm. This central region should be free of any edge effects. The cut regions were weighed to obtain the adhesive amount per area and then soaked in silicone oil to measure the change in the void fraction. From this change in void fraction, the adhesive penetration depth L was calculated as:

$$L = \frac{\varepsilon_p - \varepsilon_s}{\varepsilon_p} \delta_p \quad (1)$$

where ε_p and ε_s are the void fraction of the original paper and of the final sample, respectively; and δ_p is the paper thickness before the adhesion test. The silicone oil absorption method was compared with three other methods and was found to be a repeatable and convenient method to measure the void fraction of a sample [7]. Since there are two paper thicknesses in the experiment, δ_p was twice that of a single paper, and L is divided by two to get the penetration depth into one paper. The experimental steps are summarized in **Fig. 1**.

The press was also used to bond samples at 50.8 mm wide and 101.6 mm long. These samples were measured with a peel wheel in a mechanical tester (Model 5564, INSTRON; Norwood, MA, USA). Unbounded regions of 2.54 cm were clamped into the grip of the mechanical tester on one side and a peel wheel with tape on the other side. This tester was used to detect bond strength by stretching the length of the strip, and the glued samples were tested at the desired angle and direction. The peeling occurs at an approximate angle of 90° as the paper-glue region is peeled from the paper at a rate of 50.8 mm/min. The actual angle of peeling is lower because of the stiffness of the paperboard. **Figure 2** depicts this test.

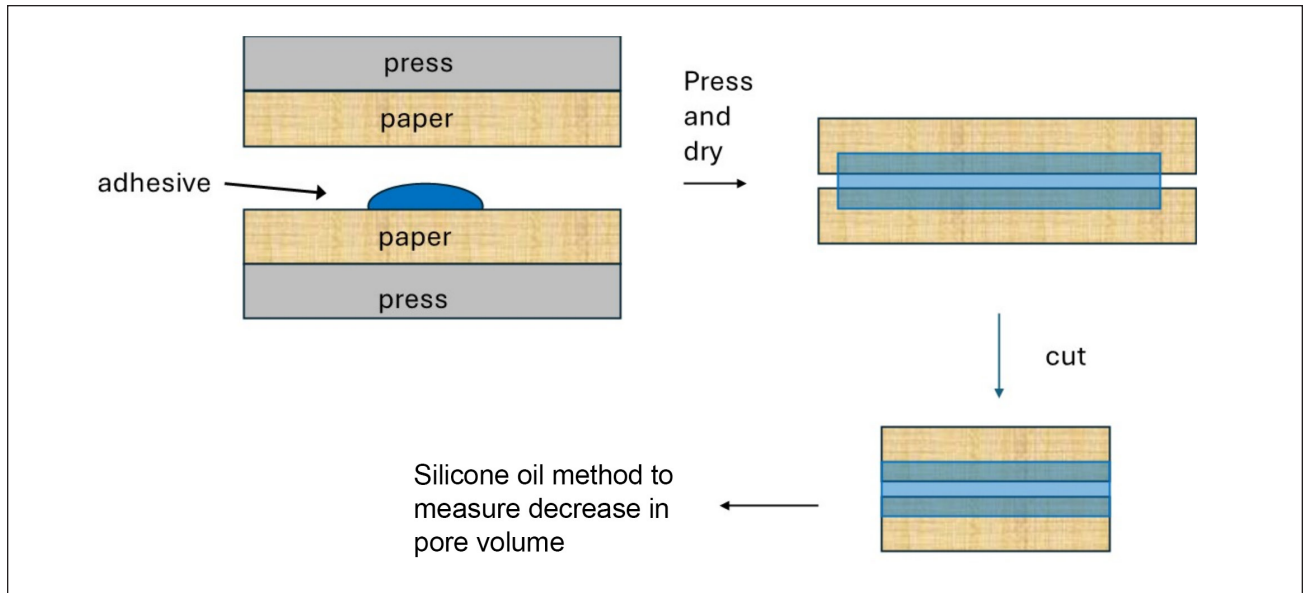
MODEL OF PENETRATION

A possible expression for the adhesive penetration rate based on Darcy's law for a single layer is:

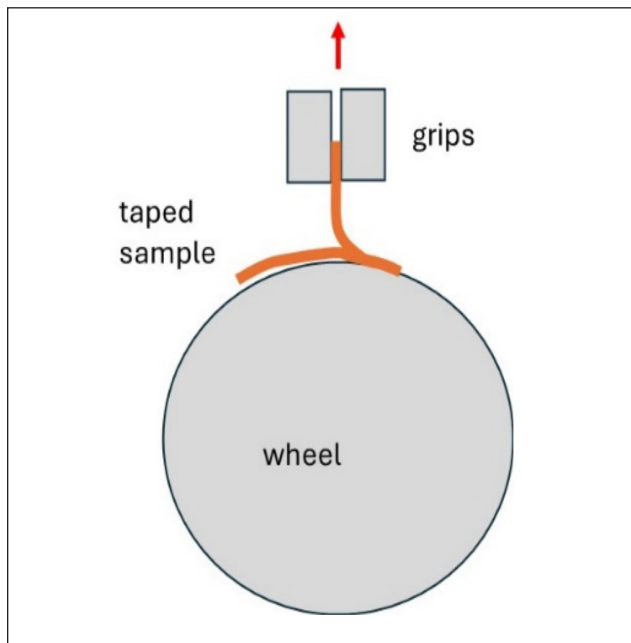
$$\frac{dL_p}{dt} = \frac{K_c \Delta P (1 - \frac{L_p}{L_{max}})}{\mu \varepsilon_c L_p} \quad (2)$$

where L_p is the instantaneous penetration depth of the adhesive, t is time, μ is viscosity, ΔP is the pressure driving force, K_c is the permeability of the coating layer, ε_c is the porosity of the coating layer, and L_{max} is maximum penetration depth based on the available adhesive. The value for permeability is obtained from air flow measurements through the sample, the void fraction is from silicone oil absorption of the sample, and viscosity is measured at various solids of the adhesive with a Brookfield viscometer. The term $(1 - L_p/L_{max})$ is used here to limit the penetration

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1. Adhesive film is pressed against paper that is uncoated or coated for a known time and press pressure. The sample is cut and soaked in silicone oil for penetration depth determination.



2. Peel wheel test with sample taped to the free rotating wheel. The grip attaches to an unglued region of the paperboard.

to the amount of adhesive applied in the nip; as L_p approaches L_{max} , the rate goes to zero to represent the limitation of the available adhesive. The integration is performed using a mathematical software tool. The L_{max} is known from the weight of the adhesive applied per unit area and is calculated as:

$$L_{max} = \frac{W_a}{\rho \varepsilon_p} \quad (3)$$

where W_a is the weight of the adhesive per unit area, r is the density of the dry adhesive, and e_p is the void fraction of the paper.

If the adhesive flows through the coating layer into the paper, a two-layer model can be used similar to Eq. 1, but with addition of resistance to flow through the coating layer:

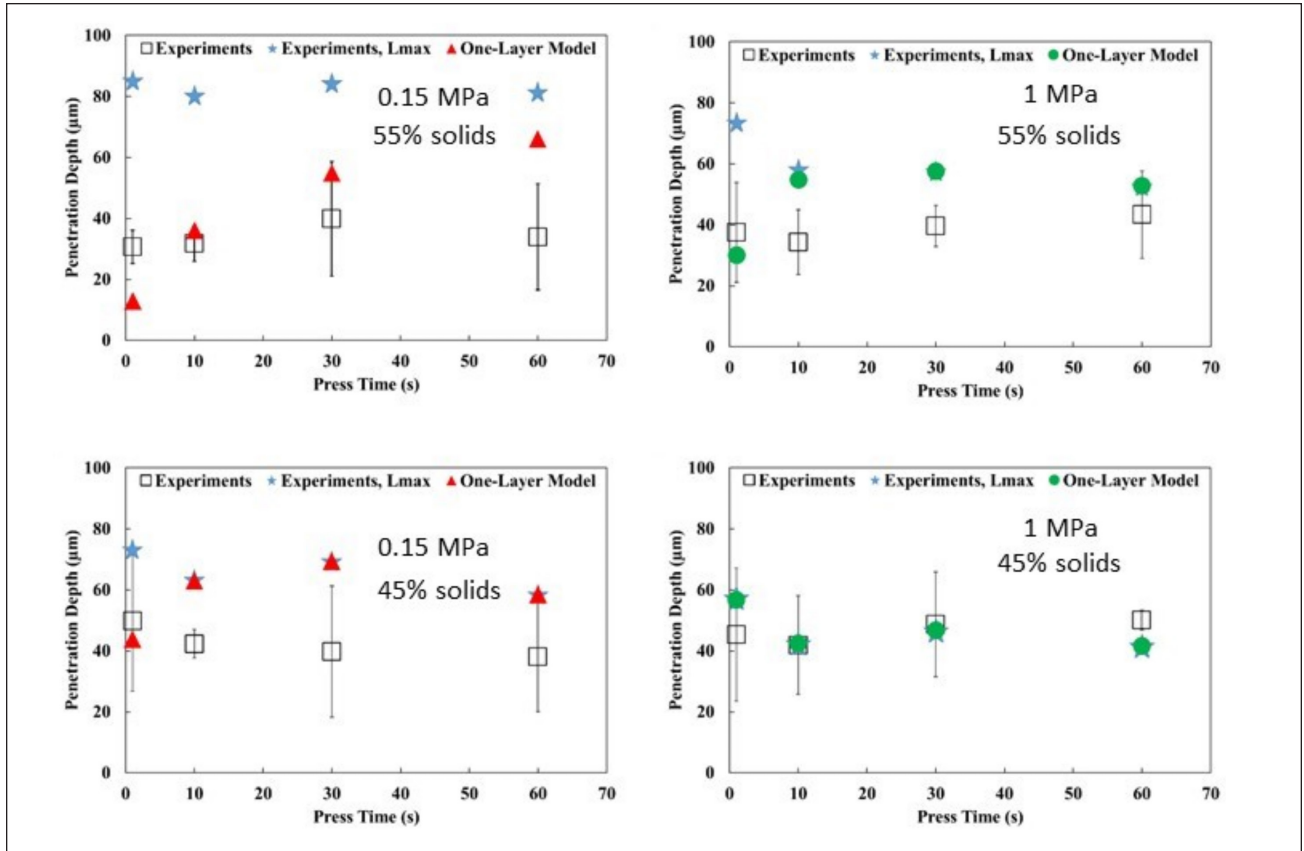
$$\frac{dL_{p1}}{dt} = \frac{\Delta P \left(1 - \frac{L_{p1}}{L_{max}}\right)}{\frac{L_{p1} \mu \varepsilon_p}{K_p} + \frac{L_c \mu \varepsilon_c}{K_c}} \quad (4)$$

where L_{p1} is the penetration depth into the paper layer in addition to the coating layer; K_p and K_c are the permeabilities of the paper and coating, respectively; e_p and e_c are the void fraction of the paper and coating layer, respectively; and L_c is the thickness of the coating layer and does not change with time.

As water from the adhesive is taken up into the cellulose structure of the fibers, the viscosity of the adhesive may increase as it contacts the fibers. At some depth of penetration, we can balance the water that is taken up by the fibers, the water that is still in the adhesive, and the initial water in the adhesive. An equation comes from that balance of the solids content in the paper pores:

$$S_{new} = S_o + \frac{(1 - \varepsilon_p) \rho_f S_w}{\varepsilon_p \rho} \quad (5)$$

where ρ is the wet density of adhesive, which is around 1 g/mL; ρ_f is the density of fibers, which is around 1.5 g/mL; S_w is the short-time water uptake by the fibers; S_o is the initial solids content of adhesive; and S_{new} is the new solids



3. Penetration depth measured and predicted for 55% solids (top figures) and 45% solids (bottom figures) for 0.15 MPa (left figures) and 1 MPa (right figures) for uncoated samples. Open squares are the experimental data, the blue stars are the maximum possible based on how much adhesive was applied, and the other filled symbol is the one-layer model prediction. The L_{max} is maximum penetration depth based on the available adhesive. For the high-pressure cases, the model predicts the maximum limit and the symbols are on top of each other.

content of adhesive that is in the porous network. This new solids level can be used to adjust the viscosity in Eq. 2 or Eq. 4, knowing the correlation between solids and viscosity.

RESULTS AND DISCUSSION

For the tables and figures that follow, all results were from the average of three values; standard deviations as high as 30% were common. This error likely comes from the uncertain removal of the surface silicone oil in the silicone oil test. **Figure 3** shows the experimental adhesive penetration depths compared to the model predictions for uncoated samples glued at 45% and 55% solids, 0.15 and 1 MPa pressure, and four press times. The maximum penetration depths are also shown in all figures as blue stars and were calculated from the mass of adhesive applied.

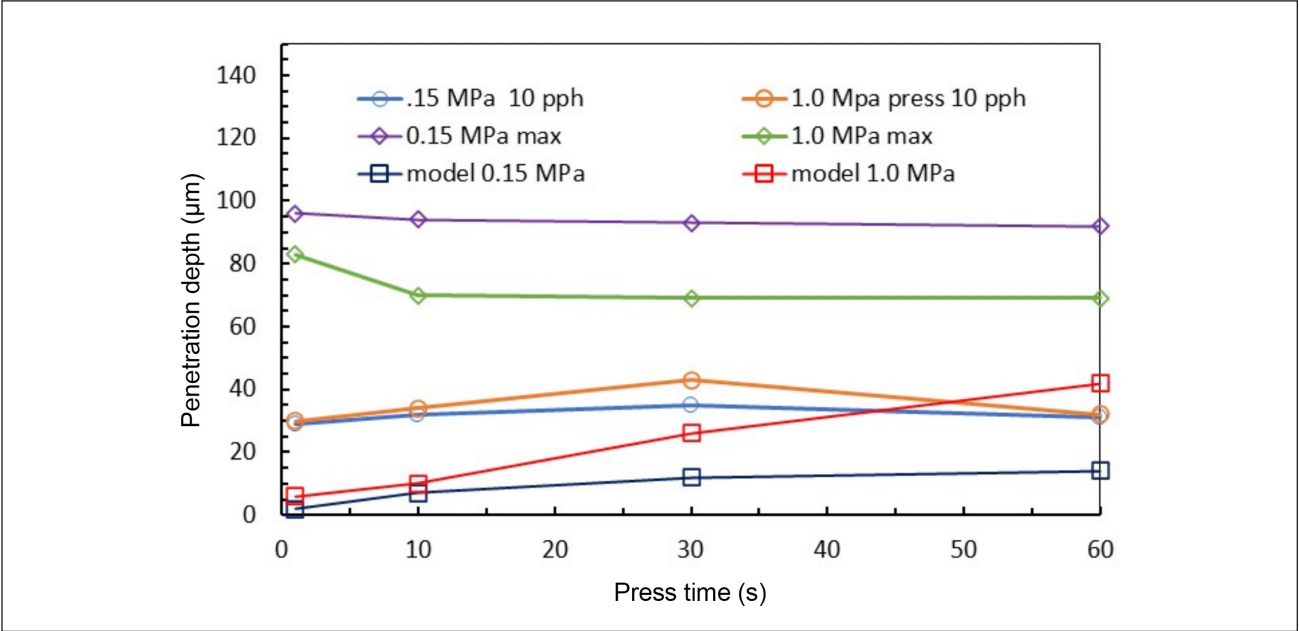
Surprisingly, the results on uncoated paperboard had no significant variations with pressing time nor for different solids or pressures. For comparison, on the other hand, the model predictions increased with time unless they were limited by the maximum parameter as would be expected from the equations. For the high-pressure cases for both high and low solids, the results matched the model and the maximum level possible; this result indicates that all the

adhesive is pressed into the paper. For the high solids and low-pressure case, the results were much less than the maximum but do not seem to depend on the pressing time.

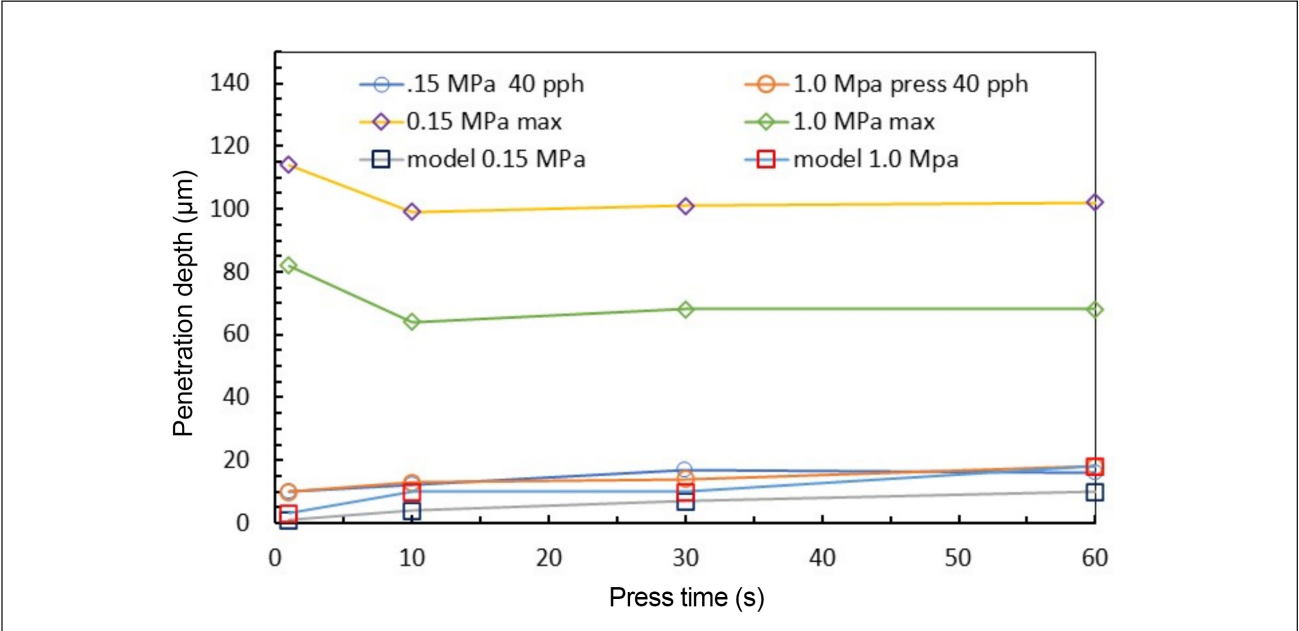
A few reasons can explain the uncoated paperboard results. Even with a short press time of 1 s, the adhesive could still penetrate the sample after pressing due to capillary absorption as the sample dries. Other tests not reported indicate that a plugging mechanism may limit the penetration depth, and more time does not help increase the penetration. Only for the high-pressure pressure does the model agree with the results, but they are also at the maximum penetration depth; penetration is limited by the amount of adhesive applied, not the flow of the adhesive into the sample.

The results where the adhesive is applied on the coated side of the paperboard are reported in **Fig. 4** for the 10 pph latex content coating layer. A big surprise was that the experimental penetration had a magnitude similar to the uncoated paperboard (35 mm) and does not seem to depend again on the pressure of the event nor the pressing time. The adhesive has a latex that may have a large particle size distribution. It seems like some significant component of the polymer in the adhesive can fit through the small pores in the

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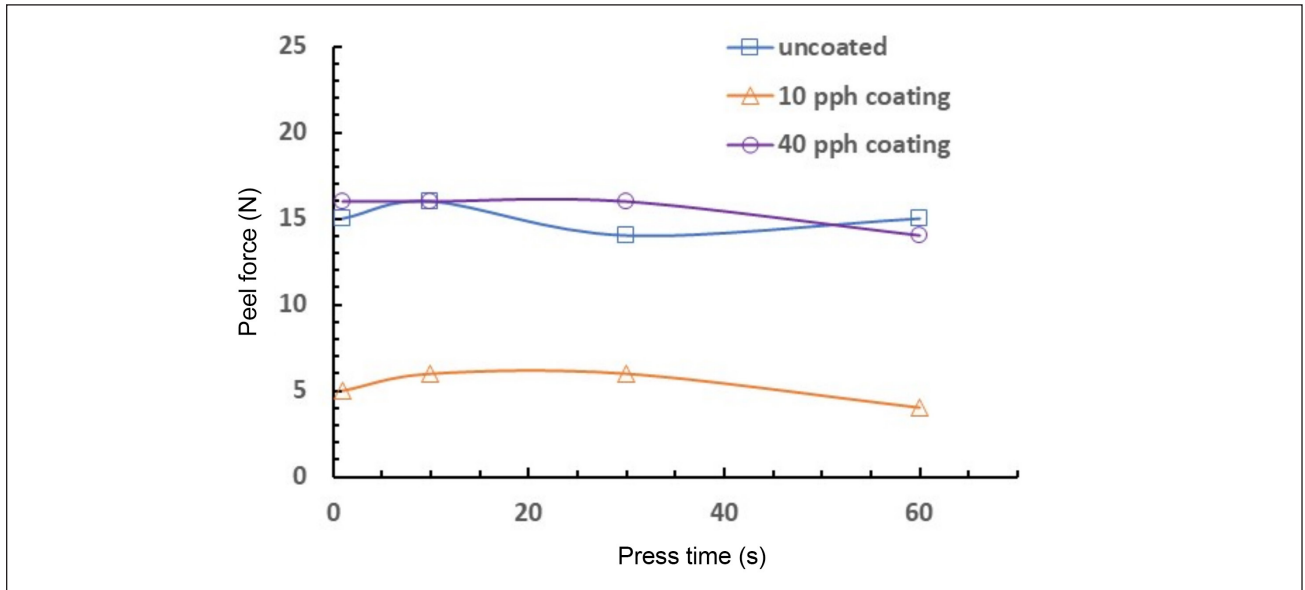
4. Penetration depth measured for adhesive in contact with a coating layer with 10 pph latex content for 0.15 and 1.0 MPa pressure and various pressing times. The model predictions are shown, as well as the maximum penetration possible based on the amount of adhesive available. The adhesive is at 55% solids.



5. Penetration depth on samples with a coating layer with 40 pph latex content for 0.15 and 1 MPa pressure for different pressing times. The model results show the trend when the viscosity adjustment is not made. The maximum values are shown based on the amount of adhesive applied per unit area. The adhesive is at 55% solids.

coating layer and fill in paper pores on the other side of the coating layer. There may be some issues with the method to measure the pore volume; therefore, these results should be taken with some caution. The results at 30 s and 60 s are in the range of the experiments for the high pressing pressure case but are low for the small pressure case. A key finding is that this adhesive seems to be able to penetrate the coating layer when the latex content of the coating layer is 10 pph.

For the low-pressure case, the model underpredicts the penetration measured, either when the model accounts for viscosity change or not. This result may be caused by capillary penetration of adhesive after the pressing event while the sample is placed in the oven to dry. Again, this mechanism may also explain the independence of the results on pressing time and pressing pressure.



6. Peel force obtained for adhesives applied with 1 MPa pressure with various pressing times for the uncoated paper, 10 pph latex containing coating, and the 40 pph latex containing coating.

Figure 5 contains the results for the penetration into the 40 pph coated paperboard for 55% solids and the 0.15 MPa and 1 MPa pressures, respectively. Now the experimental values are smaller than the maximum but are still significant and on the order of the coating layer thickness. Again, the one-layer model adjusting the viscosity underpredicts the results; however, at longer times and not adjusting viscosity, the model predicts the results, especially for the high-pressure case. As expected from the permeability results, the adhesive has trouble penetrating a coating layer that would have fewer and smaller pores. The results in Fig. 5 show that a significant amount of the adhesive is not in the pore space but is between the two paper surfaces.

The average peel force after 1 MPa pressing at various times is shown in **Fig. 6**. Quite a surprising result is that the peel force on the 40 pph latex content coating layer results were larger than the results on the 10 pph latex content coating, even though the penetration on the 10 pph system was higher than the 40 pph. It is clear from looking at the peel surfaces of the 40 pph system and the uncoated paper that “fiber pull” is seen, while with the 10 pph there is no fiber pull. The coating layer having 10 pph latex is weak compared to the 40 pph coating layer. This results in peeling within the coating layer rather than fiber pull, as well as a peeling force for the 10 pph that is lower than the uncoated paper and the 40 pph latex content coating layer. The adhesive, even though it penetrates well into the 10 pph coating layer, does not reinforce the layer. The peel force of the uncoated sample and the 40 pph coating layers sample must be linked to the mechanical properties of the paperboard.

SUMMARY

The penetration of an aqueous adhesive into paperboard and coated paperboard was characterized using a silicone oil absorption method for various pressures, solids, and times.

- The penetration does not follow the expected Darcy law expressions, even after accounting for the increase in viscosity due to water absorption by fibers.
- Penetration depth into the uncoated samples does not depend on the pressing time or pressure.
- The penetration depth into the samples coated with a pigmented coating with 10 pph latex content was similar to the uncoated samples.
- For the 40 pph latex content latex, the penetration was on the order of the coating layer thickness.
- The peel force was determined by the strength of the coating layer and not the penetration of the adhesive into the samples.

ACKNOWLEDGEMENTS

We thank the sponsors of the University of Maine Paper Surface Science Program for their discussions, advice, and support. We thank Alice Cheung of Henkel for supplying the adhesive and for discussions. **TJ**

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ABOUT THIS PAPER

Cite this article as:

Khlewee, M., Desisto, W., and Bousfield, D.W., *TAPPI J.* 24(1): 48(2025). <https://doi.org/10.32964/TJ24.1.48>

DOI: <https://doi.org/10.32964/TJ24.1.48>

ISSN: 0734-1415

Publisher: TAPPI Press

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ABOUT THE AUTHORS

We studied this topic at the suggestion of sponsors of the Paper Surface Science Program at the University of Maine. This study complements previous research on the topic of water-based adhesives because it is linked to the settling of inks.

The most difficult aspect of this study was measuring the absorption of the silicone oil. We did discover that adhesives can penetrate coating layers on paper. This research can help mills with designing coating layers for good adhesive performance.

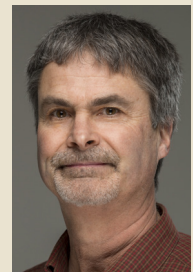
Khlewee was a graduate student, DeSisto is professor, and Bousfield is Calder professor and director of the Paper Surface Science Program in the Department of Chemical and Biomedical Engineering at the University of Maine, Orono, ME, USA. Email Bousfield at bousfld@maine.edu.



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