

## An Approach to Rheological Evaluation of Dynamic Water Retention Measurements for Paper Coating Suspensions

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### ABSTRACT

The dewatering behaviour of three paper-coating suspensions with different thickener systems was investigated. Experiments were performed both on a laboratory coater and with a controlled shear stress rheometer and an immobilization cell. It seems that the permeability of the immobilised layer is – at least partly – governing the dewatering kinetics.

### INTRODUCTION

In the Dynamic Water Retention method (DWR), a laboratory coater for paper is used as a tool for achieving information regarding the water releasing ability of coating suspensions in contact with a base paper<sup>1</sup>. The method is said to well reflect the behaviour of coating suspensions and base paper in an industrial process. The method is based on a base paper being coated at a machine speed of 70 m/min during which samples of the suspension are taken from the tray at different times after start-up of the trial. The mass fraction solid material for the samples is measured gravimetrically, thus giving a measure of the dewatering between application and the subsequent metering. The immobilization cell, which was first described by Willenbacher et al.<sup>2</sup>, has been presented as a tool for measurement of dewatering of suspensions in terms of the time needed to reach immobilisation. The methods for measurements with the cell

have been further developed by other workers<sup>3, 4</sup>.

In this study, results from DWR measurements are put in relation to those obtained with a controlled shear stress rheometer. It was evident that the slow dewatering observed in DWR trials could not be explained merely in terms of liquid phase viscosity. Also the structure of the immobilised layer formed at the interface between wet suspension and paper surface has to be taken into account.

### EXPERIMENTAL METHODS

In the present study, three coating suspensions of similar solids content and pigment/latex system but different and increasing levels of thickener were used. The pigment system used was a 70:30 mixture (by weight) of calcium carbonate (Hydrocarb-90, supplied by Omya Pflüss-Stauffer AG) and clay (Century, supplied by Kaolin International BW). The latex used was a styrene-butadiene quality supplied by DOW Europe S.A. Three different thickeners were chosen, one carboxymethyl cellulose (CMC, Finnfix 30 supplied by Noviant Oy), one alkali-swelling emulsion (ASE, Sterocoll HT supplied by BASF AG) and one polyvinyl alcohol (PVA, Mowiol 20/98 supplied by Clariant Oy). In addition an optical brightener agent (OBA, Blanophor P supplied by BASF AG) was added to all three suspensions. The recipes are pre-

Table 1. Recipes and characteristics of the three coating suspensions used

Suspension (type of thickener added)	CaCO <sub>3</sub> :Clay [pph*]	Latex [pph*]	thickener [pph*]	OBA [pph*]	Initial solids content [% by mass]	Dewatering by ÅA- GWR [kg/m <sup>2</sup> ]
CMC	70:30	11	0.7	0.2	64.6 %	99
ASE	70:30	11	0.35	0.2	64.5 %	98
PVA	70:30	11	1.4	0.2	64.5 %	72

\*pph = parts (by mass) per hundred parts of dry pigment.

sented in Table 1 along with results from measurements of static pressurised dewatering. These measurements were performed according to the standardised method<sup>5</sup>, but with 0.2 bar pressure during 2 minutes.

The DWR measurements were performed on a laboratory size coating machine at a speed of 70 m/min. Samples of the metered-off suspension were taken from the tray between application and metering with intervals of 1 minute. Roll feeding of the base paper was used along with nip application and blade metering<sup>6</sup>. An uncoated base paper (80 g/m<sup>2</sup>) with a high degree of water sorptivity (Cobb<sub>60</sub>-value (SCAN P12): 100 g/m<sup>2</sup> and Cobb<sub>30</sub>-value (SCAN P12): 99 g/m<sup>2</sup>) was used. The same base quality was used for all coating colours. The suspension samples were analysed gravimetrically with respect to mass fraction solid material.

For the rheological measurements, a controlled shear stress rheometer (MCR 300, Physica Messtechnik GmbH, Germany) was used. Flow curves of the suspensions were taken at shear rates up to 2500 s<sup>-1</sup> with plate-plate geometry.

The immobilization cell measurements were performed according to the procedure previously presented<sup>4</sup>, in which a 2.4 mm thick layer of suspension is dewatered under shear in a plate-plate geometry. As the normal forces are set to a constant value

during measurement, the vacuum driven dewatering of the suspension causes the upper rheometer plate to move downwards thus enabling the calculation of an apparent filter cake thickness as described previously<sup>4, 7</sup>.

The liquid phase of the suspensions were separated by first centrifuging at 6,466 g for 4.5 h, after which the supernatant was filtrated twice through polycarbonate filters with pore sizes of 0.4 and 0.2  $\mu$ m, respectively. The three filtrate phases were examined gravimetrically with respect to mass fraction solid material. Rheological measurements on the filtrates were performed using double gap geometry.

## RESULTS AND DISCUSSION

The results from the DWR measurements are presented in Figure 1, from which it can be seen that, for trial times above approximately 1.5 min, the suspensions denoted CMC and ASE have been dewatered to a greater extent than the suspension denoted PVA. Since all three coatings were performed using the same base paper, the probable reason for the difference is either differences in coating colour rheology (viscosity of the coating suspension or of the liquid phase) or differences in the structure of the layer formed at the interface between wet coating suspension and paper surface at application.

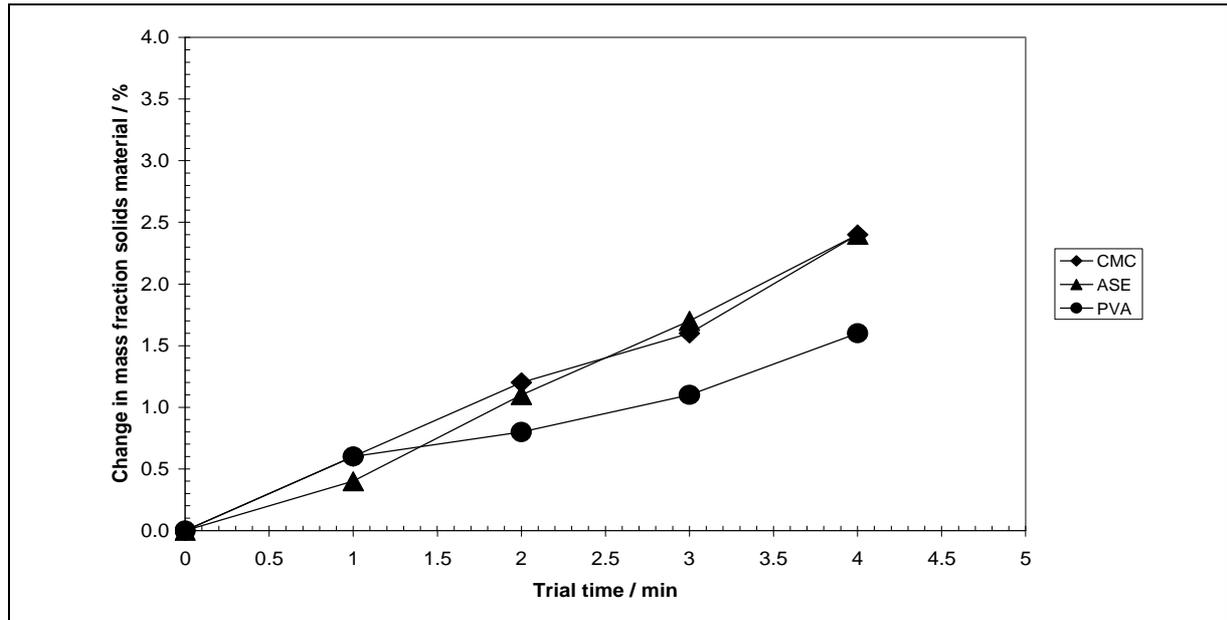


Figure 1. Results from the DWR experiments.

The build-up of an immobilised layer was investigated according to the immobilisation cell methods previously presented, and the values for apparent filter cake thickness ( $l_a$ )<sup>4</sup> are given in Table 2, which also includes the corresponding values for the so-called normalised apparent cake thickness ( $l_a^*$ )<sup>4</sup>, defined as  $l_a/V$ , where  $V$  is the volume of filtrate. The factor  $l_a^*$  is a structural parameter correlated to the void fraction in the cake but also to the ability of the cake to resist break-up due to the shear flow across the surface of the cake. The upper limit of this apparent cake is defined as the level at which the cohesive forces within the layer no longer can resist the shearing forces exerted by the upper rheometer plate.

Table 2. Values at 500 s trial time.

	$l_a$ / mm	$l_a^*$ $10^{-9}$ / m <sup>-2</sup>
CMC	$0.59 \pm 0.05$	$0.73 \pm 0.02$
ASE	$1.2 \pm 0.03$	$1.9 \pm 0.1$
PVA	$0.95 \pm 0.06$	$0.92 \pm 0.01$

In Table 2, values are given at the arbitrarily chosen trial time of 500 s. The discussion

concerning these results are given in more detail in a future publication<sup>8</sup>, but  $l_a$  at a certain point of time is suggested to be dependent both on liquid phase viscosity and the permeability of the immobilised layer. As can be seen from Table 2, the values are lower for CMC and PVA compared to ASE. To evaluate which one of the two parameters liquid phase viscosity and immobilised layer porosity gives the greatest contribution to  $l_a$ , the viscosities of the filtrates were measured. These viscosity curves are shown in Figure 2.

In Figure 2 can be seen that, at low shear rates, the apparent viscosity of the liquid phase is considerably more elevated for CMC than for PVA (and ASE). This leads to the conclusion that the lower values for CMC presented in Table 2 are mainly due to the high liquid phase viscosity at shear rates similar to those experienced by the suspension when passing through the immobilised layer.

The viscosity measurements on the coating suspensions at low to moderate shear rates did not provide any explanation to the differences in dewatering behaviour. The apparent viscosity – values which are not

presented here – for PVA was close to the values for both CMC and ASE in this shear rate region.

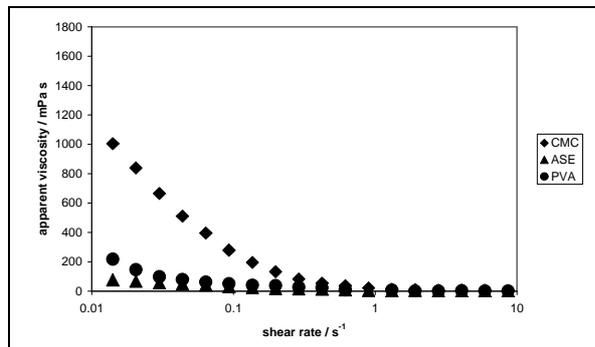


Figure 2. Viscosity curves of the filtrates

However, at more elevated shear rates, suspension PVA showed an increase in the measured viscosity. This viscosity increase (within paper industry often referred to as dilatancy) is most often an indication of poor performance of the suspension in an industrial coating process<sup>9</sup>. The shear rates at which this viscosity increase occurs are similar to those experienced by the suspension in the application nip used. However, since the dewatering from application to metering takes place at very low shear rates, the viscosity increase causes slow dewatering only if the process is irreversible.

## CONCLUSIONS

In measuring the dewatering properties of calcium carbonate/clay based suspensions, the suspension containing PVA as a thickener showed slower dewatering than did suspensions containing CMC or ASE.

The explanation for this behaviour was investigated through rheological measurements on the coating suspensions as well as on the liquid phases. These investigations showed indications of a less permeable structure being formed with PVA, which is the reason for the slow dewatering. It seems that the results from the DWR method are not a direct measure of coating suspension viscosity, indicating that other parameters such as immobilised layer structure influence

the dewatering and therefore should be taken into account.

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