Measuring the water retention of coating colors

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ABSTRACT *A new method for determining the water retention of coating colors is presented. The method is based on pressure filtration and involves the gravimetric determination of the aqueous phase penetrating through a filter into a paper. The contact time can be varied, and the influence of external pressure can be studied easily. The gravimetric method is sensitive to changes in coating color formulation. Because the procedure is simple and the measurement system is well defined, the method shows good reproducibility.*

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Analysis
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The dewatering of a coating color is an important aspect of an industrial coating process. Many quality and runnability problems originate from the interaction between the base paper and the water phase of the coating color. Unfortunately, the existing theory of water retention mechanisms does not provide enough information about the various factors that determine the extent of dewatering. One reason for our inadequate understanding is the lack of a reliable laboratory method for measuring the water retention of a coating color.

Methods for measuring water retention of coating colors

One of the most commonly known instruments for measuring water retention, the S.D. Warren test, was introduced by Stinchfield *et al. (1)* in 1958. The method is based on the conductivity measurement of a paper, which is in contact with coating color. The S.D. Warren test is fast and simple to use, but is has some disadvantages. The method is sensitive to structural defects in the test paper, and the thickness of the test paper also affects the result. Because conductivity is measured, coating colors with differences in electrolyte content cannot be compared. The viscosity of the coating color may also influence the amount of color remaining in the space between the paper and the electrode. If the viscosity is low, this amount is probably insufficient to act as a bulk source for dewatering.

Another test method based on conductivity measurements was developed by Thomin *et al*. A major difference between this procedure and the S.D. Warren test is that the coating color is applied to the test paper from both sides simultaneously. The measuring electrodes are not in direct contact with the test paper, and points of poor contact or short circuits between electrode and paper can be avoided.

Taylor and Dill (*3*) presented a sonic test for measuring water retention. Sound waves are continuously sent through a paper sheet, and their speed is recorded. The interaction between the drained water and paper lowers the speed of sound in the plane of the sheet. The sonic velocity is measured from the moment the coating color contacts the paper surface. The percentage of the original sonic velocity remaining after a measured period of time is taken as an indicator for water retention.

An optical method, originally developed by Stephan at BASF, was presented by Hentschel and Bischof (*4*). A water-soluble dye is added to the coating color. The dyed color is poured into a measurement cell, and a porous filter paper is brought in contact with the color. The change in light remission of the filter paper, caused by the penetrating aqueous phase, is measured with a photometer, and the time required to decrease the remission to 40% is suggested to constitute a value for the water retention. The measurements are made in a well-defined system, and the reproducibility is therefore good.

Beck *et al.* (*5*) presented a test method in which a cake of coating color is drained on a ceramic plate, thus changing the gloss of the coating color surface. When gloss is optically measured as a function of time, curves of gloss versus time can be plotted. Water retention of the color indicated by the time when the curve starts to fall off.

All these methods are indirect methods – that is, they are all based on measuring physical quantities other than the amount of drained water. The main disadvantage of all these methods is that they do not quantify the amount of water drained to the substrate.

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A film splitting technique, the KNP method, has also been presented (*6*). A coating layer of the thickness equal to that of the coating in the coating method is applied onto a smooth piece of glass plate using an applicator with variable slit width. The base paper is brought into contact with the coating layer at a given pressure. The contact time after which the split coating layer has reached the critical pigment-volume concentration (i.e., the solids contents at which only enough water is present to fill the voids in the consolidated coating) is taken as a measure for the water retention. This method differs from many others in that it demonstrates the role of solid contents in determining how fast the immobilization of the coating color is obtained.

In a development of the KNP method (*7*), the coating color is applied on a glass plate with a wedge-shaped groove. The depth of the groove increases from 0 µm to 100 µm. A test paper is brought into contact with the color and is removed after a given period of time. The depth at which the critical pigment volume concentration has been reached is taken as a measure of water retention. This modification makes the method less time consuming than its prototype.

Besides the gloss method, Beck *et al.* (*5*) also presented a pressure drainage method. A rubber bulb is used to press the coating color against a filter of 0.2 µm pore size with an external pressure of 20 atm. Water penetrates through the filter under the influence of this pressure, and the coating color is drained. Water retention is determined by the volume of the outcoming filtrate water in a given period of time. The applied pressure of 20 atm probably does not correspond to the industrial pressure situations, and the method accentuates therefore the importance of effects that are not related to the actual coating process.

Another pressure drainage method primarily used for determining the immobilization point for coating colors, was developed by Lamprecht. The method was presented by Reinbold and Ullrich (*8*) and is based on the same principle as the method developed by Beck *et al.* (*5*). External pressures from 0 atm to 3.5 atm and contact times from 20 min to 120 min have been studied.

Aschan developed a scraping-off technique, later used by Engstrom and Rigdahl (*9*), in which the wet coating is scraped off the base paper at different positions on a pilot coater. Jones and Hetherington (*10*) presented a dynamic water retention testing device using a laboratory coater of the puddle blade type. The increase in solids content of the color is a measure for the water retention. Two different colors can be tested simultaneously, and the speed can be varied from 3 m/min to 75 m/min.

Experimental methods

The gravimetric method

This method has been developed at the University of Abo Akademi. The method is based on the same principle as the methods developed by Beck *et al.* (*5*) and Lamprecht (*8*). The measurements involve gravimetric determination of the quantity of aqueous phase penetrating through a filter into a paper during a certain contact time and under a certain external pressure.

Figure 1 is an illustration of the apparatus. A filter of well-defined pore size and an absorbing paper are placed under a hollow cylinder made of aluminum. The system is tightened against the rubber blanket, and the coating color is poured into the cylinder. After a Teflon plug has been inserted in the cylinder, a weight corresponding to a targeted external pressure is put on top of the plug. Teflon has been chosen because of its low surface energy and low friction. This arrangement gives us a tight plug that moves smoothly.

The measurement procedure is as follows:

1. The absorbing paper is weighed on analytical scales.
2. The absorbing paper, the filter, and the cylinder are tightened against the rubber blanket.
3. The coating color is poured into the cylinder, and a timer is started.
4. The plug is inserted, and the weight is put in place. (This takes 8 s from the moment the color is applied.)
5. After a certain amount of time, the weight is removed, the apparatus is untightened, and the absorbing paper is detached from the filter. (This takes 8 s.)
6. The test paper is reweighed, and the aqueous phase transferred is registered.

The area of the cylinder is 8.00 cm^2, and 10 mL of coating color is used in each measurement. Whatman 17 Chr paper has been used as absorbing paper. The measurements have mainly been carried out with hydrophilic Millipore® polycarbonate filters, pore size 5µm, but additional experiments have also been made with Millipore 0.8 µm and Nuclepore® 5 µm. Three different weights are used to obtain external pressures of 0.05, 0.25, and 1.0 atm. The contact time has been varied from 60 x to 400 s.

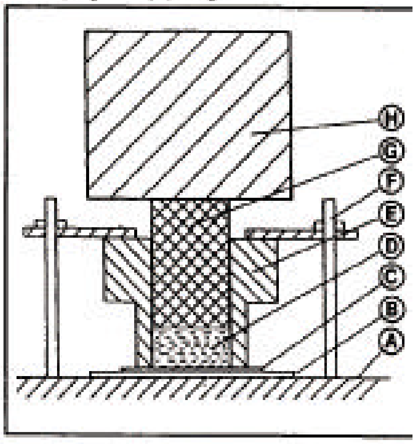
Results

Figure 2 compares the dewatering of coating colors based on clay and on ground calcium carbonate. The clay colors show a slower dewatering rate than colors based on coarse ground calcium carbonate. **Figure 3** shows the dewatering curves for ground calcium carbonate with the addition of three types of CMC (carboxymethyl cellulose) of different chain lengths. (CMC 1 represents the shortest chain length, while CMC 3 is the longest.) The dewatering rate increases with shorter chain length because of the lower viscosity of the aqueous phase of the color. **Figure 4** illustrates the influence of coating color solids contents on dewatering.

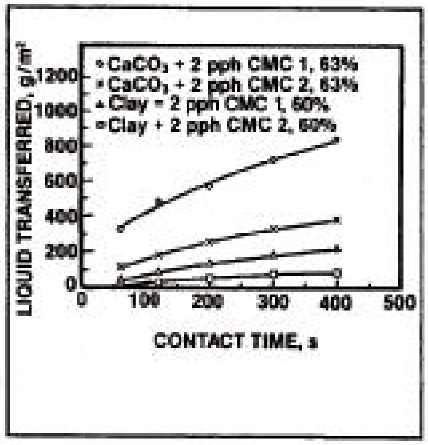
To relate the new method to an established method, we carried out parallel measurements with the BASF method, which is commonly known in the paper industry and is based on a different measurement principle. The correlation, presented in **Fig. 5**, is generally good. A longer time before reaching the 40% remission in the BASF measurements also occurs as a lower dewatering in the gravimetric measurements.

However, the two methods are in conflict in one respect. The triangles in Fig. 5 all represent colors with the same viscosity in the aqueous phase but with different solids contents. On the gravimetrical axis, the points fall in a logical order. The color with the highest solids content also has the lowest dewatering rate, while the points fall in the opposite order on the BASF axis. This difference can probably be explained by the fact that the water-soluble dye is added to the

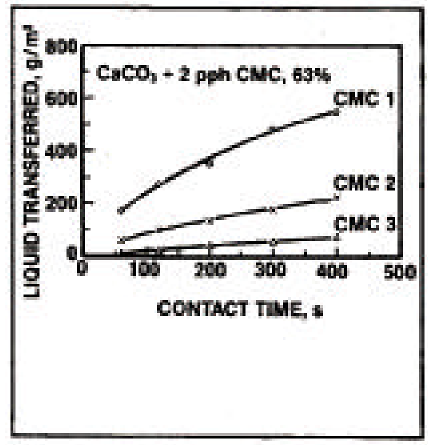
1. The apparatus, illustrating (A) rubber blanket, (B) absorbing paper, (C) filter, (D) coating color, (E) hollow cylinder, (F) tightening device, (G) Teflon plug, and (H) weight



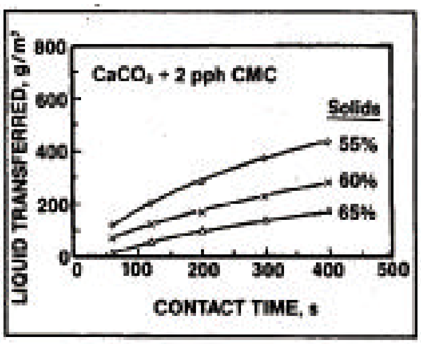
2. A comparison of dewatering of coating colors based on clay and coarse ground calcium carbonate (External pressure: 1.0 bar)



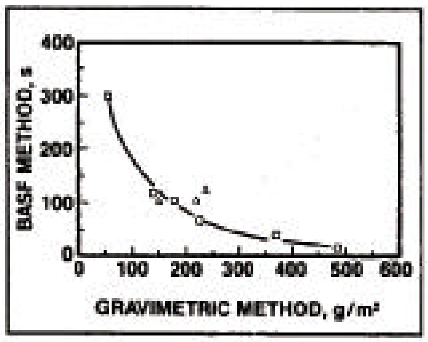
3. Dewatering of ground calcium carbonate colors with CMC of different chain lengths (External pressure 0.25 bar)



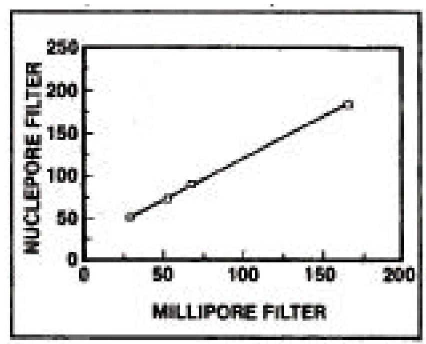
4. The influence of coating color solids on dewatering



5. Correlation between the gravimetric method and the BASF method



6. Correlation between measurements made with Millipore filters and Nuclepore filters



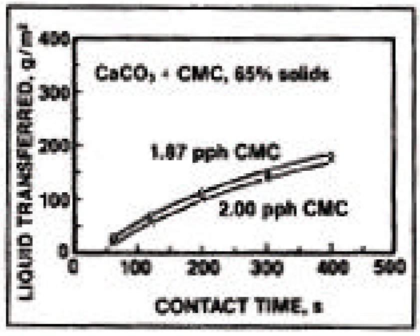
coating color in relation to the weight of the total color. This augmentation leads to a higher concentration of dye in the water phase of a color of higher solids contents. Thus, a smaller quantity of the aqueous phase is needed to reduce the remission to 40% for a color of higher solids contents. This presumably can be avoided by adding the dye in relation to the aqueous content of the color.

The filling of the pores in the filter requires some contact time, which delays the aqueous phase in reaching the test paper. When carefully detaching the filters from the immobilized color, we observed that colors based on clay and ground calcium carbonate showed different "saturation weights" in the Millipore filters. Calcium carbonate showed higher "saturation weights" than clay, which can probably be explained by the difference in their particle shapes. The plate-like clay particles can plug pores in the filter, leaving some of the pore volume unsaturated, while the irregular ground calcium carbonate particles probably do not cause the same effect.

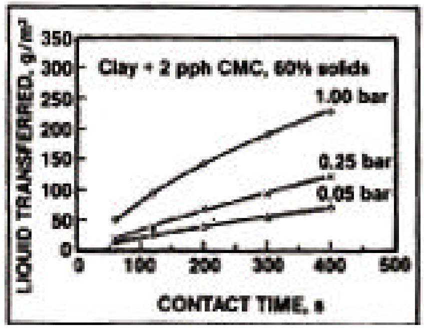
To try to control this, we made measurements with Nuclepore filters of the same pore size as the Millipore filters. The Nuclepore filters are thin, 15µm, compared to Millipore filters, 120 µm. However, the correlation between measurements with Millipore and Nuclepore filters proved to be surprisingly good, as **Fig. 6** shows. This means that results and conclusions made from Millipore measurements can be compared with measurements made with Nuclepore. Nevertheless, the incomplete saturation of the Millipore filter indicates that some caution is needed when comparing results from Millipore measurements for colors based on different pigments. A thinner filter decreases the importance of the variations in the saturation weight of the filter.

The reproducibility of the experiments is very good. The mean coefficient of variation is about 3%. Even the slightest difference in color composition can be seen clearly, as **Fig. 7** shows. **Figure 8** gives an example of the influence of external pressure on dewatering a coating color.

One of the main advantages of the gravimetric method is that the aqueous phase lost to the absorbing substrate is directly quantified. In other words, a direct measure of the dewatering of the coating color is obtained. The apparatus is portable, easy to use, and relatively fast (about 10 min per measurement). The measurement procedure is so simple that the experimental results can hardly be influenced by any unknown effects not related to water retention. A disadvantage is that measurements cannot be made at contact times corresponding to the actual situation in a coating process. Some time is required to fill the pores in the filter before the water front reaches the test paper, and the



7. An example of dewatering curves for two coating colors with small differences in composition (External pressure: 0.25 bar)



8. The influence of external pressure on the dewatering of a coating color

manual performance of the measurement also takes some time.

An interesting parameter is the test paper, which can also be ordinary coating base paper. This option allows a paper mill to determine the dewatering rate of the base paper for the color system. Practical experience indicates that the method is sensitive to the mixing procedure of the coating color before the measurements, especially which the mixing of clay colors. This indicates that the flocculation kinetics of coating colors could be an interesting area for further research in the field of water retention.

Conclusions

We have developed a new method, based on pressure filtration, for determining the water retention of coating colors. The amount of water phase penetrating through a filter into an absorbing paper is gravimetrically determined. The contact time can be varied, and the role of external pressure in the dewatering of coating colors can easily be studied. The apparatus is simple to use, and good reproducibility is obtained. The new method is sensitive for small changes in the coating color formulations.

The gravimetric method has some disadvantages, such as the lack of shear during the measurement. Furthermore, contact times do not respond to the real coating process. Nevertheless, the method could be a complement to the existing methods and will probably bring us a step closer to better understanding the water retention theory.

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