

New rheological test methods to simulate the processing of paper coatings

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ABSTRACT

Two methods are presented which provide new insight into the complex rheological behavior of paper coatings under process conditions. Firstly the use of a highly dynamical rheometer is used to measure the structure recovery, i.e. the thixotropy. Secondly an immobilization cell is described which provides information on the immobilization kinetics.

INTRODUCTION

New processing conditions like higher speeds and the demand for ongoing quality improvements in the paper industry lead to the need of a deeper understanding of the rheological phenomena involved in the coating of papers. Two methods are presented which provide new insight into the complex rheological behavior of paper coatings under process conditions. The first experiment describes the use of a rheometer with a highly dynamical transient response to measure the structure recovery, i.e. the thixotropy, of paper coatings. The second method describes an immobilization cell which in combination with a rheometer yields information about the immobilization kinetics. The goal of both tests is the simulation of the real processing conditions as close as possible.

When applying paper coatings via applicator rolls, the aim is to produce a homogenous and uniform coating thickness. In order to achieve this goal the paper

coatings need to have certain physical-chemical properties. Surface tension has great influence on the behavior during coating, as do the internal interaction forces, which determine the rheology of a substance.

Standard rheological experiments using oscillatory shear stress or shear strain amplitude and frequency sweeps provide information on the characterization of the paper coatings at rest. Although these experiments are important for the characterization of the coating itself and give valuable information on parameters like storage stability or sedimentation, they do not provide any relevant information on the processing characteristics like misting, waviness or blade runability.

A more important parameter for a better understanding of the practical processes is the thixotropy which in rheological terms can be defined as the characterization of the structure strength decrease under a shear load and the increase of the structure strength after the cessation of the shear.

Another parameter defining the quality of the final product is the immobilization kinetics of the paper coating after the coating is applied to the paper. The immobilization cell introduced by Willenbacher et al.¹ allows to investigate the influence of parameters like base paper, coating and various process conditions on

the immobilization kinetics and therefore on the quality of the final coated paper.

In the following two methods are described which use rheological tests for the determination of structure recovery and immobilization kinetics and allow more practically relevant investigations. Both tests can be used in research and development for improvements in coating formulations as well as a fast and easy way to perform quality control measurements. The new methods can serve as a tool to reduce the amount of expensive and time consuming machine trials significantly.

STRUCTURE RECOVERY -THIXOTROPY

In order to acquire more detailed information about processing characteristics, the process of applying the mixture to the paper substrate can be divided into three fundamental steps. Using rheological tests these can be simulated as follows:

- Slow movement in the storage tank (low shear rate)
- Transferring the mixture over the applicator rollers (high shear rate)
- Coating the paper, whereby the paper web has more or less the same speed as the rotating speed of the applicator rollers (low shear rate)

Taking these sections, we have a 3-step test in which there is a constant shear rate in each measuring section. Estimating the shear rates which occur during the process reveals that a difference of up to 9 decades between the low and high shear rates is possible. As the structure of paper coatings regenerates very fast after high shear, it is necessary to use a highly dynamical rheometer which is capable to cover a wide range of shear rates and to adjust to the various speed steps in a very fast way. This test which measures the structure decomposition at a high shear rate and the structure recovery after the cessation of this high shear load can be used as a

measure of the thixotropic behavior of paper coatings.

Samples and Instrument

Three paper coatings are used: SM I and SM II have a starch derivative as a rheological additive, whereby SM II has a higher additive concentration than SM I. SM III contains a rheological additive with an inorganic base. The measurements were conducted using a Physica UDS 200 rheometer, the temperature was set to 20°C and a cone/plate measuring system (\varnothing 50 mm, 2°) was used. The Physica UDS 200 is equipped with an electronically commutated synchronous motor (EC-drive) with a highly dynamical transient response, which allows controlled stress (CS) and controlled strain or controlled strain rate (CR) tests on the same rheometer². The general dynamic behavior of the EC-motor is demonstrated in Fig. 1 where the transient response of the EC-motor is shown by speed steps in air from 0.001 rpm to 1000 rpm and back to 1 rpm and 0.01 rpm. In general a speed step can be conducted in less than 100 ms without any speed overshoot independent of the sample viscosity.

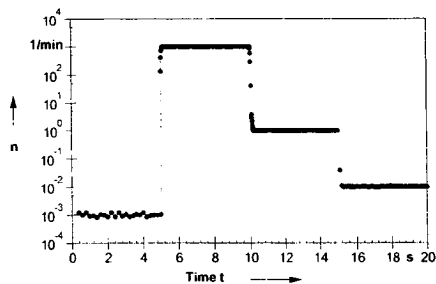


Figure 1. Speed steps in air.

A rheometer with such a high transient response is needed in order to adjust steps in shear rate fast enough and therefore enabling structure recovery measurements on paper coatings.

Results and discussion

The flow curves of the three paper coatings are presented in Fig. 2. It can be seen that the flow curves for coatings SM I and SM II display a similar course, the only difference being that one curve shows higher measurement values. Therefore a higher solids content only results in a thickening of the coating and has no effect on the course of the flow curve. At smaller shear rates coating SM III behaves like coating SM I; at higher shear rates like coating SM II. During application tests it was discovered that coating SM III shows less misting behavior than coatings SM I and SM II. This means that the steady state flow curve alone is not representative for the practical behavior.

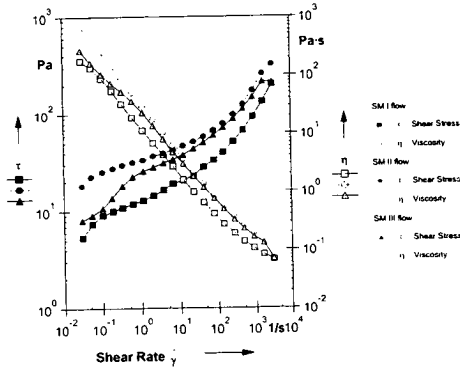


Figure 2. Flow curves of the paper coatings

This situation can be easily explained by examining the step shear rate test, which is shown in Fig. 3.

For the first and third interval a shear rate of 0.06 s^{-1} and a measuring duration of 1 and 0.3 seconds for each measuring point were taken as the test parameters, respectively. In the second interval the parameters were set to a shear rate of 600 s^{-1} with 10 measuring points in 5 seconds.

As can be seen in Fig. 3 SM III recovers from high stress much faster than coatings SM I and SM II due to the different additive. The viscosity at the transfer point from the applicator rollers to the paper web has an important influence on the formation of

drops during misting. Since the structure of coating SM III regenerates much faster, the viscosity increased to higher values during the time it takes for the coating to transfer from the applicator rollers to the paper web than in coatings I and II. Coating SM III therefore tends to a less pronounced misting behavior.

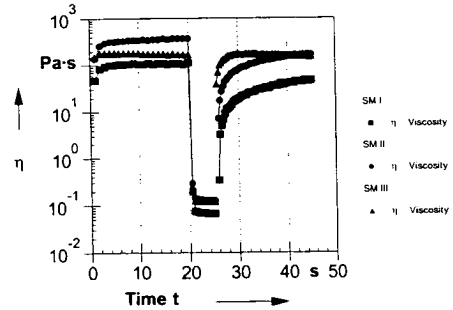


Figure 3. Time behavior and structural regeneration of paper coatings

Moreover it can be seen that increasing the solids content in coating SM II has an influence on the value of the viscosity only and not on the time dependent structural regeneration.

IMMOBILIZATION

A new laboratory test method using a rheometer equipped with an immobilization cell allows to characterize the kinetics of immobilization and the immobilization time of paper coating colors (pat. pent. BASF AG)¹. In the immobilization cell a base paper is placed into the rheometer and a vacuum pump forces the liquid phase of the coating to penetrate the paper. Varying the gap size, the pressure, the kind of base paper and the coating allows a simulation of the coating process under different conditions. The dewatering or immobilization can be observed under different rheological test conditions like constant rotational shear stress or constant oscillation.

Measuring with a constant gap and a constant shear stress

The coatings were placed onto the base paper. A vacuum and a constant shear stress of 100 Pa were applied. The measuring tool was a plate/plate system with 50 mm diameter, the gap was fixed at 0.5 mm. A Physica MC1 rheometer was used and a constant shear stress of 100 Pa was applied simultaneously with the onset of the vacuum. Reproducibility was found to be good. Fig. 4 shows results on four different coatings. It can be seen that three of the samples show immobilization after very different times, whereas the fourth coating does not show a significant amount of immobilization with the used paper. For quality control purposes the time where the viscosity reaches 100 Pas can be evaluated easily by a software analysis module.

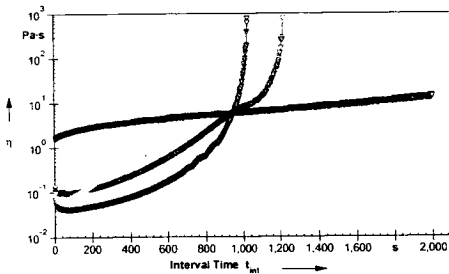


Figure 4. Immobilization test on different coatings.

Measuring with constant normal force and oscillation

By using a research rheometer a more advanced test can be performed. An EC-motor based Physica MCR 300 rheometer was used for this test. Again the coating is put onto the base paper and a pump is started to generate a vacuum below the paper. Simultaneously with the onset of the vacuum an oscillatory test with a constant amplitude and frequency is started. During the experiment the normal force is set to zero

and the change in the gap is recorded. Since the dewatering leads to a sample shrinkage a small negative force is acting on the measuring plate. A zero normal force acts against this force and results in a decreasing gap size. In Fig. 5 an example of such an experiment is shown. After the start of the test both the storage G' and the loss modulus G'' increase slightly. Between 250 s and 320 s G' increases very strongly and finally reaches a plateau value at 340 s, whereas G'' shows a maximum at 320 s. The maximum in G'' indicates large structural changes immediately before the total immobilization of the sample and can be used as a characteristic point of the immobilization kinetics. Simultaneously with the increase in the storage and the loss modulus the gap size decreases from 0.5 to 0.28 mm.

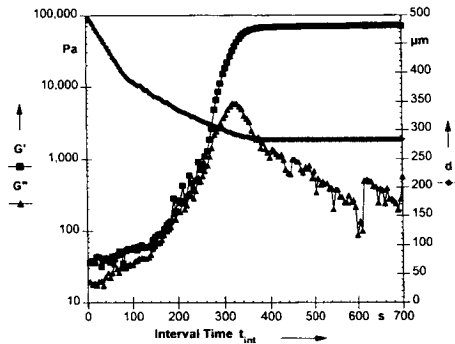


Figure 5. Immobilization test with constant normal force.

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