New Rheological Test Method to Determine the Dewatering Kinetics of Suspensions

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Abstract:
Rheology plays an important role in dewatering processes. It is therefore interesting to analyze the dewatering process and the rheological behavior of a suspension simultaneously. An exact determination of the immobilization point at the maximum of the loss factor as well as the immobilization time can be attained using an oscillatory time test with preset strain. The degree of dewatering is determined via normal force controlled gap setting. This report offers an insight into the theory of the dewatering of liquid supersaturated suspensions and shows how the kinetics of dewatering can be determined using paper coating as an example.

Zusammenfassung:
Bei Entfeuchtungssvorgängen spielt die Rheologie eine nicht ganz unbedeutende Rolle. Was ist naheliegender als den Entfeuchtungsvorgang und das rheologische Verhalten einer Suspension gleichzeitig zu messen. Die messtechnisch exakte Erfassung des Immobilisierungspunktes beim Maximum des Verlustfaktors und der Immobilisierungszeit, ist mit Hilfe einer deformationsgesteuerten Oszillations-Zeitmessung möglich. Der Immobilisierungsgrad wird über eine normalkraftgesteuerte Spaltnachführung ermittelt. Dieser Bericht gibt einen kurzen Einblick in die Theorie der Entfeuchtung von flüssigkeitsübersättigten Suspensionen und zeigt an dem Beispiel einer Papierstreichmasse wie die Kinetik der Entfeuchtung rheologisch erfasst werden kann.

Key Words: Immobilization cell, dewatering by pressure difference, degree of dewatering, immobilization point, immobilization time, degree of immobilization, filter cake formation.

1 INTRODUCTION

In the past, it was only possible to consider the dewatering of suspensions and their rheological properties separately, i.e. either the dewatering process or the rheological behavior was examined. Using an immobilization cell it is possible to combine both measurements [1]. This simultaneous measurement of the dewatering kinetics and the corresponding rheological properties provides application-specific information and aids in process and product optimization. Rheological oscillation tests reveal unattained information about the kinetics of the filter cake formation. Furthermore the method can be used to measure the water retention of suspensions and to evaluate the coating base paper [1].

The immobilization-point as well as the immobilization-time are important factors in the optimization of the drying process. Drying times that are too long, for example, result in high process costs without significantly improving the product. Inhomogeneous products with a rough surface structure are often the result of material systems that are rheologically unbalanced. The rheological characterization of materials aids problem solving in technical applications.

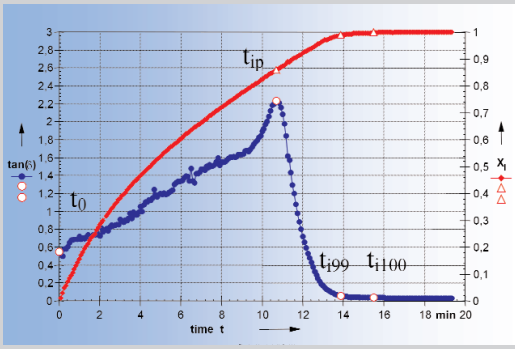
2 THEORETICAL CONSIDERATION

2.1 DEWATERING

The dewatering of a suspension can be compared to squeezing out a sponge. At the beginning, the liquid can be squeezed out easily. The tighter the sponge is squeezed, the less liquid comes out and the more force is required. After complete "compression" (immobilization), the rheologically interesting part of the dewatering is completed.

One option for reducing the liquid part of a suspension is dewatering with gas pressure [2, 3]. At the beginning of the dewatering process, a gas pressure difference is applied to the sample. After a certain period of time, the sample is dewatered to the extent that the suspension reaches a saturated condition: all space between the particles is still filled with liquid but the particles touch each other and so the sample is immobile. A filter cake is produced. In many technical processes, thermal drying follows immobilization to achieve complete drying.

The kinetics of the dewatering is dependent on the total flow resistance R [m^{-1}], the filter surface A [m^2], the viscosity of the liquid phase η [Pas]



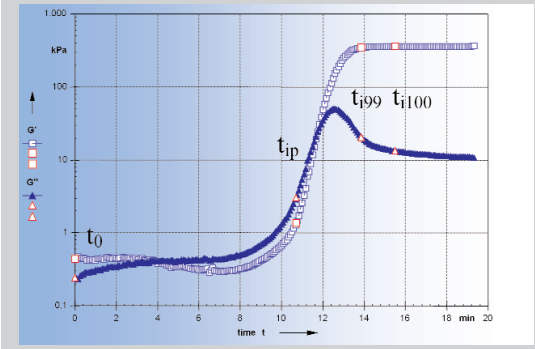


Figure 8 (left): Transition from the supersaturated suspension to the immobilized condition.

Figure 9 (right): Immobilization of a paper coating.

matrix liquid becomes less important. Similar to the glass transition point in temperature tests on polymers, a quasi phase transition (the immobilization point t_{ip}) can be determined at the maximum tan δ (Figure 8; t_{ip}). The immobilization point marks the transition between liquid and solid behavior. The maximum is reached in this example at the immobilization point after t_{ip} = 10.7 min.

Particle/particle interactions increase with progressive dewatering. The substance therefore has an increasingly elastic character, i.e. the substance becomes less mobile. The flow character is lost and the solid character dominates, i.e. tan δ << 1 (Figure 8; t > 11 min).

5.3 RANGE 3: (LIQUID) SATURATED & IMMOBILIZED SAMPLE

The time, at which a further dewatering using the gas pressure difference method is no longer useful, can be given as the immobilization time t_i. The immobilization time for the examined paper coating is 15.5 min at p = 70 kPa and with a 1 mm sample height at the start of the test. The loss factor tan δ moves towards zero and the storage modulus G' is now considerably larger than the loss modulus G'' (Figure 9; t > 14 min).

However, the dewatering is not yet completed. As already mentioned, the filter cake is in a saturated condition. Now the capillary dewatering phase begins. This cannot be determined via the dimension of the measuring gap. Although the gap now remains constant, a further decrease in G'' can be observed (t > 15.5 min).

Several specific points can be analyzed from the rheological material functions which have been measured. Table 1 lists the loss factor tan δ (ratio: viscous to elastic), the storage modulus G' (elastic), the loss modulus G'' (viscous) and the value of the complex viscosity $|\eta^*|$ at the start of the test t_o, at the immobilization point t_{ip} and on reaching the immobilization time t_i.

The immobilization time t_i was determined at a degree of immobilization of 99% and 100%. The immobilization time t_{i_99} can be regarded as an effective drying time because the dewatering by pressure difference is to a large extent already complete. A subsequent thermal drying step would be definitively more effective than continuing with the immobilization process. The analysis of the degree of immobilization is performed after the measurement is completed. An analysis using the loss factor is a usable method for the online evaluation of the immobilization time, e.g. $t_{i\_tan\,\delta}$ at tan δ = 0.05.

6 CONCLUSION

Precise information on the kinetics of dewatering can be attained using an immobilization cell and a suitable rheometer. The corresponding rheological properties provide information about the filter cake formation and give an insight into the solid content profile between measuring plate and base paper. The oscillatory time test with preset strain and normal force control has several advantages over conventionally used methods.

A complete measurement of the immobilization process was not possible using a shear stress controlled rotational time test. The method presented here, however, determines the degree of dewatering as well as the immobilization point at the maximum of the loss factor, the immobilization time and the viscoelastic character of the sample at each point of the measurement. The measurements can also be continued in the solid state. The measuring gap adjusts itself to the sample volume due to the normal force control. This enables an alternative measurement of the dewatering kinetics.

The measured rheological values also reveal information about the suspensions viscous-elastic behavior, i.e. with consideration on surface

Table 1: Specific points in the dewatering process of a suspension.

	Time [min]	**tan δ** [-]	**G'** [Pa]	**G"** [Pa]	**|η*|** [Pas]
t_o	0	0.55	420	320	50
t_{ip}	10.7	2.24	1360	3050	334
t_{i_99}	13.9	0.057	349000	19900	35000
t_{i_100}	15.5	0. 037	361000	13200	36100

leveling and sedimentation behavior. Additionally the results can be used to evaluate the filter medium. Varying the gap and the pressure can attain further information.

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