

Pre-Selection of Flocculants by the Separation Analyser Lumifuge 114

By

T. Sobisch

Distributed by

 **infoClearinghouse.com**

Download or publish at InfoClearinghouse.com

©2001 T. Sobisch
All Rights Reserved

PRE-SELECTION OF FLOCCULANTS BY THE SEPARATION ANALYSER LUMIFUGE 114

T. Sobisch

L.U.M. GmbH

Rudower Chaussee 29 (OWZ), D-12489 Berlin, Germany

<http://www.lum-gmbh.de>, e-mail: info@lum-gmbh.de

The operation of high performance dewatering machines such as high pressure filter presses and decanters relies heavily on the use of flocculants for sludge conditioning. Due to the varying composition of wastewater sludges pre-selection of promising flocculants in front of field tests is a frequent task. To this end, a lot of different laboratory methods have been developed, which are in part very sophisticated or on the other end may be very simple but strongly based on individual judgement and experience.

The focus of our company is on separation stability of disperse systems. We develop methods and equipment for control and solution of related practical problems (see www.lum-gmbh.de). The aim of our ongoing work is to offer a solution based on our separation analyser LUMiFuge 114. For further applications of the separation analyser see references [1 - 4].

The Lumifuge 114 - principle of measurement / application

The separation analyser (Fig. 1) is an analytical centrifuge with an integrated optoelectronic sensor system which allows to trace the local and temporal changes of light transmission during rotation.


LUMiFuge™ 114 Separation Analyser	Classification of Stability and Demixing of Dispersions
	<ul style="list-style-type: none">• optical detection of separation processes during centrifugation• up to 2500 times faster• 8 samples simultaneously• concentrated and diluted dispersions• documentation and evaluation of measured data• rapid prediction of sample separation stability

Fig.1

Up to eight samples can be investigated simultaneously. A centrifugal acceleration in the range of 12 to 1200 x g is used to speed up the separation process, thereby reducing the time necessary for investigation. The measuring scheme is depicted in Figure 2.

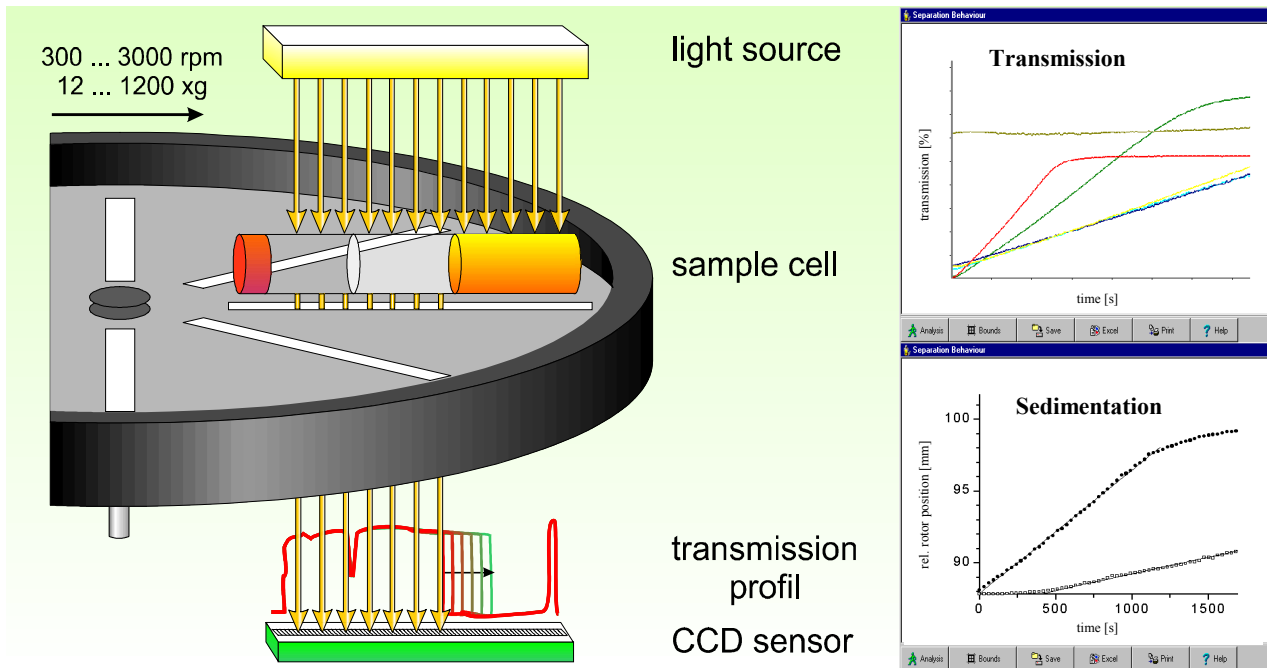


Fig. 2: Measuring scheme of separation analyser LUMiFuge 114

In preset time intervals the local transmission is determined over the entire sample length simultaneously whereby transmission profiles are generated. The transmission profiles change during centrifugation according to the progress of separation. The separation process can be depicted as time course of the relative position of the boundary between supernatant and sediment (Figure 2 – feature Sedimentation) or of the transmission averaged over a chosen part of sample length (Figure 2 – feature Transmission).

The essential new feature is that the overall separation process can be traced. Therefore the dynamic behaviour of the sludge under centrifugal acceleration can be investigated.

What do we see if we investigate sludge samples with or without flocculants?

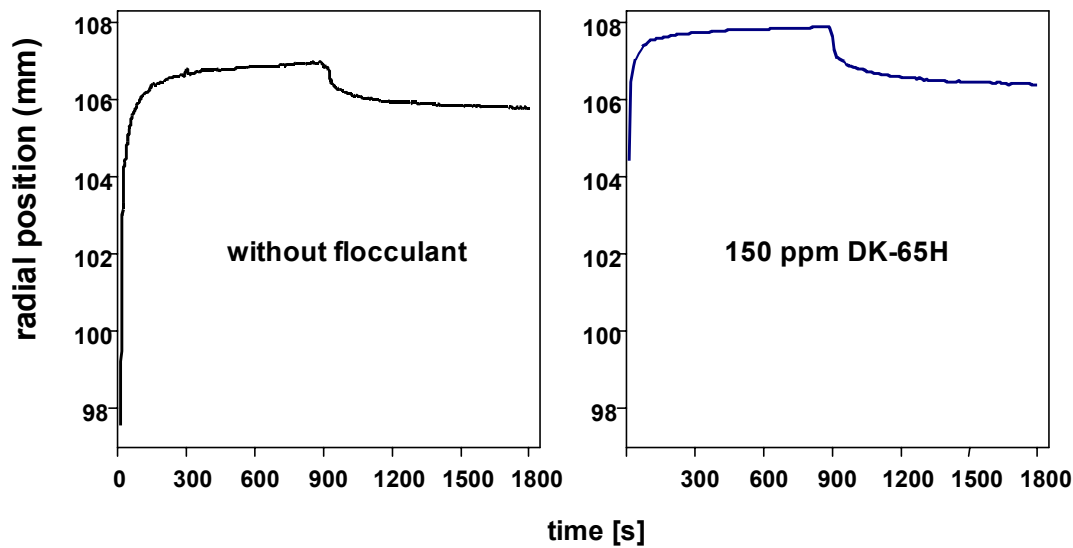


Fig. 3: Dewatering behaviour of a sludge during centrifugation without and with flocculant: time course of centrate height (measured as distance to the centre of rotation), first 900 s at 1200 x g (compression) then 900 s at 12 x g (dilatation)

Figure 3 shows the typical behaviour of sludge during a compression – dilatation cycle. The change in sludge volume is depicted as time course of the radial position of the phase boundary corresponding to the increasing height of the centrate. First the samples were compacted at high centrifugal acceleration afterwards the acceleration has been reduced. Due to flocculant addition a faster and higher compaction is obtained. From the picture it is evident that common centrifugation tests predict to low values for sludge dewaterability because the sediment volume increases again after switching-off the centrifuge.

Two parameters were derived from these kind of measurements to evaluate the performance of flocculants. First the relative sludge volume per total solids in the compressed state, second to characterize the dewaterability of the sludge cake during the first seconds of centrifugation the ratio of the initial sludge volume reduction in relation to the residual value.

General procedure

Of course, evaluation of flocculant performance is not quite so simple. Not only the dynamic behaviour of the sludge under centrifugal acceleration has to be addressed adequate modelling of shear forces acting on the conditioned sludge is also a key requirement. This can only be managed by adapting the sample pre-treatment, so that similar changes take place in the sludge samples as in practice.

Our laboratory procedure consisted of the following steps: Preparation of stock solutions of flocculants in tap water, according to the recommendations of suppliers and field applications. The sludge samples were first mixed with the flocculant solution by moderate stirring (600 rpm for 1 minute) followed by additional shearing with a high speed Ultra Turrax homogenizer (8000 rpm for 90 seconds) to model the shear forces inside the decanter. The step of additional shearing was modified later on in that a baffled stirred reactor was used instead (2000 rpm for 1 minute). The dewatering behaviour of the samples stirred moderately and of the samples additionally sheared were compared for different flocculants and flocculant concentrations using the separation analyser at a centrifugal acceleration of $1200 \times g$.

Four cationic flocculants of the Floclair series (Münzing Chemie GmbH, Heilbronn) were studied in more detail.

The products compared were all of the emulsion polyacrylamid-type with a degree of charge of 65 %. They differed in molar mass and structure as follows.

Floclair DK-65S with medium molar mass and a high degree of branching.

DK-65H with high molar mass and a lower degree of branching.

DK-65 and DK-65N both linear with medium and low molar mass.

Case study I: flocculant selection - anaerobic sludge digested in a sludge reactor

As an example the following comprises the results of flocculant testing for dewatering of an anaerobic sludge (total solids 4 %, ignition loss 36 %), which has been digested in a sludge reactor.

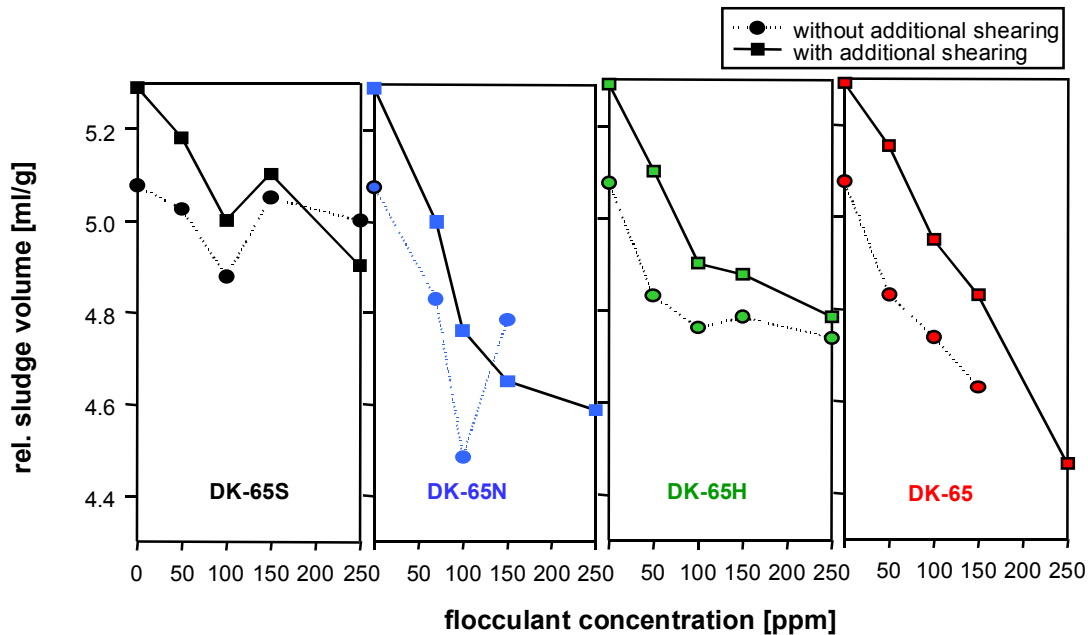


Fig. 4: Comparison of the performance of different flocculants without and with additional shearing – residual sludge volume

In relation to the residual values it can be seen that for all flocculants the additional shearing reduces dewaterability – the residual sludge volume is increased. For higher flocculant concentrations the two curves approach each other. The linear products exhibit a significant better performance than the branched flocculants. The medium molar mass DK-65 produced the best results, in this case the lowest values for the sludge volume could be obtained.

Figure 5 shows the comparison of the flocculant performance in relation to the initial dewaterability (during the first 30 s of centrifugation). In case of this sludge the same trend could be observed as in relation to the residual total solids of the sludge cake, so flocculant Floclair DK-65 resulted in the fastest compression of the sludge cake even at a concentration of 100 ppm.

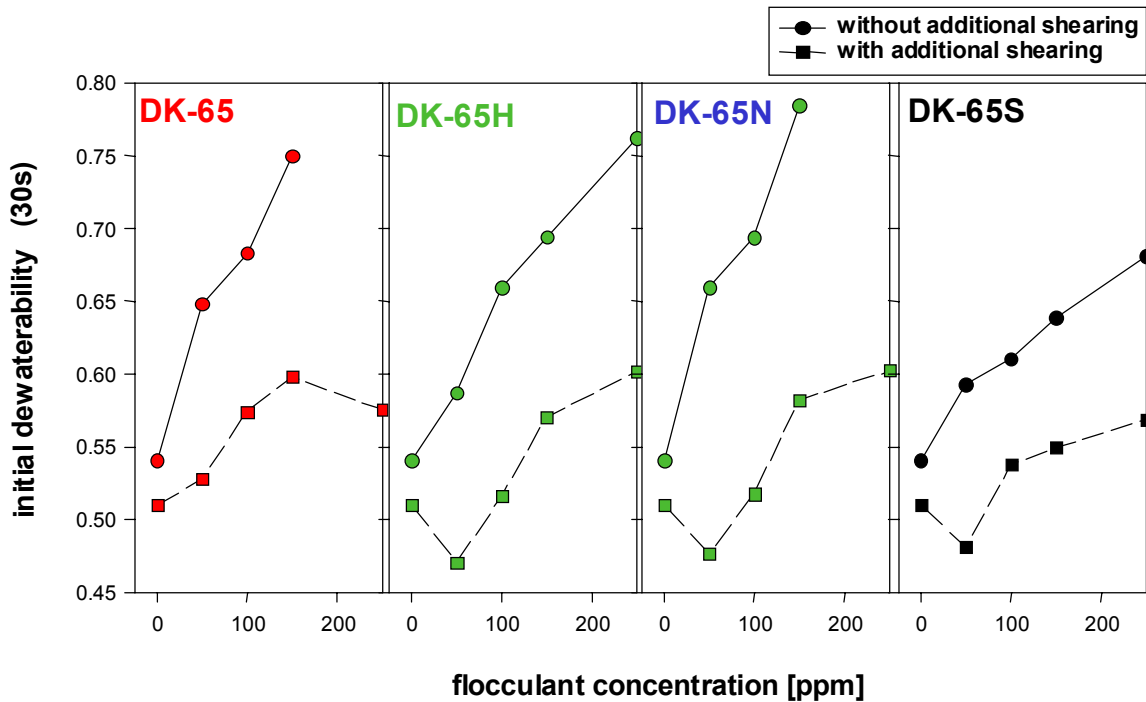


Fig. 5: Comparison of the performance of different flocculants without and with additional shearing – initial dewaterability

However, during dewatering of this sludge with a decanter under field conditions it was experienced that DK-65 and DK-65H as well are suitable flocculants, but DK-65N and DK-65S are not applicable. Further laboratory investigations revealed that dewatering results substantially deteriorated for DK-65N if sludge samples are strained mechanically prior to flocculant addition (stirring 5 minutes at 600 rpm). On the other hand with DK-65 and DK-65H only minor changes of performance were observed, after pretreatment of sludge samples similar results were obtained for the initial dewaterability. Therefore, it may be assumed that differences between laboratory and field results are due to the shearing of the sludge during transportation by pumps. For this reason changes of sludge structure prior to flocculant addition should be considered as an important factor in relation to flocculant performance.

Case study II: effect of mechanical preconditioning – anaerobic sludge digested in an old sludge basin

The laboratory procedure could not only be used for comparison of different flocculants for a special sludge to be dewatered, but also for the investigation of the influence of other parameter like sludge aging or mechanical stresses during pumping.

Figure 6 demonstrates the effect of mechanical conditioning on two anaerobic sludges from an old sludge basin (S1 - total solids 6 %, ignition loss 41 % - was obtained in autumn, S7 - total solids 5 %, ignition loss 39 % - was obtained in spring).

They were continuously pumped with a peristaltic pump in the laboratory and at different times the sludge dewatering behaviour was investigated for different flocculant concentrations. The total solids content of the sludge cake is depicted as function of treatment time. Superfloc E4208 is a linear product used on-site for dewatering of S7, SD2065 is branched.

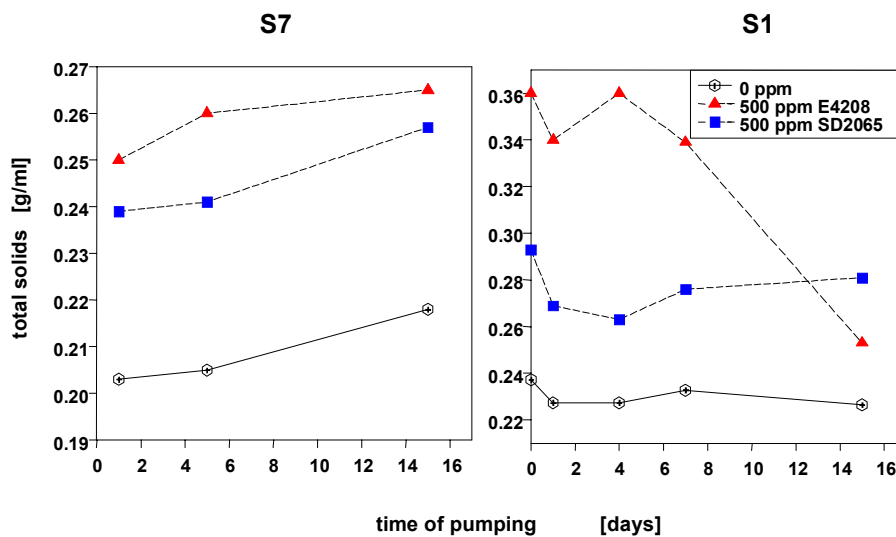


Fig. 6: Effect of mechanical preconditioning (pumping) on sludge dewatering comparison of linear and branched flocculant (Superfloc E4208 and SD2065) for anaerobic sludge samples from an old sludge basin (S7 with 6 month longer storage time)

As it was observed on-site, for sludge S7 dewaterability increased with prolonged mechanical conditioning (reduction in flocculant demand during the working week by 30 %!).

A quite different behaviour was found for S1. For the linear product dewaterability more and more decreased with pumping, however, for the branched product dewaterability improved after going through a minimum. So, after 15 days of pumping this product got more efficient than the linear one. There are at least two major counteracting effects, which influence the dewaterability. First, loose agglomerates are broken up, which would have not a sufficiently high mechanical stability - during flocculation more homogeneous flocs will then be formed. Second, mechanical stresses will shift the particle size distribution to lower values. The results show that the actual effect of mechanical preconditioning can only be deduced by experiment either in the field or in laboratory.

Relation between experimental results and dewatering performance

The parallel behaviour in assessment of flocculant performance based on residual total solids and initial dewaterability (see Fig. 4 and 5) is not found in every case. During experiments carried out on a number of sludges and flocculants it turned out there is a trend that based on residual total solids the performance of polyelectrolytes with lower molar mass or high degree of charge is often overvalued. Moreover, it was found that in many cases residual total solids gave the same ranking as the widely used capillary suction time.

These correlations are demonstrated by the following picture (Figure 7).

The relative statistical ranking for results based on capillary suction time, residual total solids and initial dewaterability are compared. A value of 100 would mean that the flocculant in question was the best for all sludges.

It can be seen that for DK-65N with lower molar mass CST and residual solids gave a substantial higher ranking than the initial dewaterability. On the other hand DK-65S with a high degree of branching exhibits a significant lower ranking by the CST-method.

Based on the general procedure presented a screening procedure has been worked out. For each flocculant only one concentration appropriate for field conditions was tested. Thereby, per measurement with the separation analyser 8 flocculants can be compared. Promising flocculants should produce high total solids and a high initial dewaterability as well.

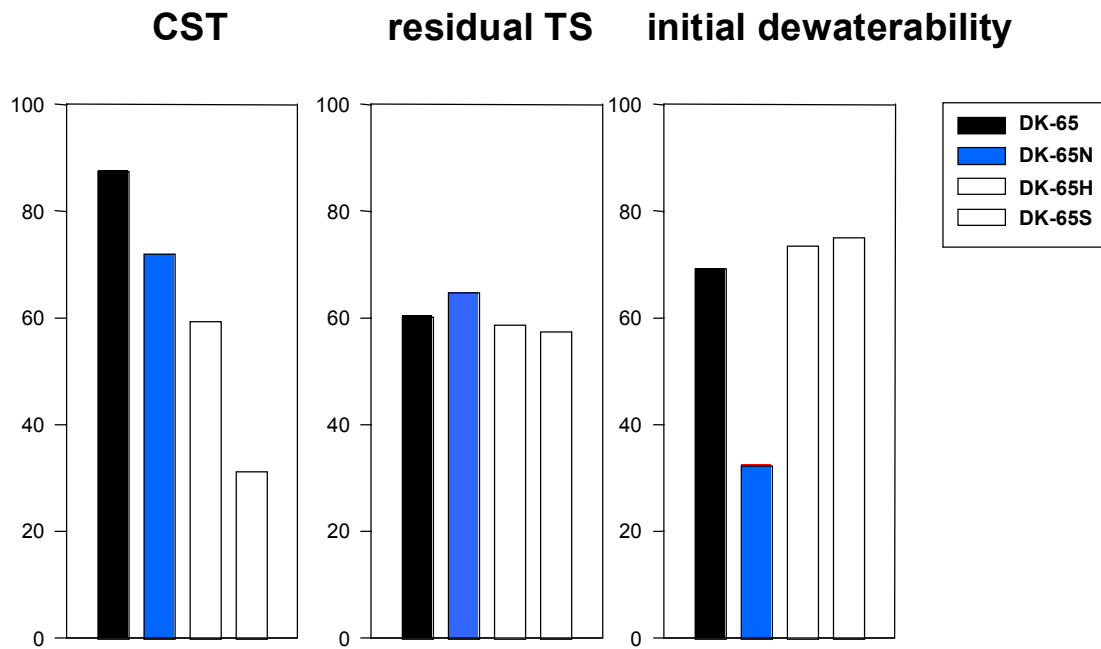


Fig. 7: Relative statistical ranking of flocculant performance for a range of different sludges – based on results obtained by CST-Method and Lumifuge

CONCLUSIONS

The separation analyser Lumifuge 114 is an efficient tool for the characterization of sludge samples and flocculant optimisation. The development of appropriate methods for sample pre-treatment is essential for getting relevant results for sludge dewatering under practical conditions.

Flocculant performance could be estimated by evaluation of stability against intensive shearing, total solids obtained and initial dewaterability of the sludge cake. Results were in good agreement with field results.

The beneficial or negative influence of mechanical pre-conditioning of sludges before flocculant addition could only be deduced by experiment. The separation analyser can be used to this end for model investigations.

A screening procedure has been developed. Efficient flocculants should produce high residual total solids and good initial compressibility as well.

Lab-scale investigations deliver more reliable results if the dynamic behaviour of the sludge under centrifugal acceleration is also investigated. The separation analyser Lumifuge 114 can provide results about the compression behaviour of sludges in the range between 10 and 100 s. So far no other method or device is known which can deliver such results.

References

[1] von Homeyer, A.; Krentz, D.-O.; Kulicke, W.-M.; Lerche, D.: „Optimization of the polyelectrolyte dosage for dewatering sewage sludge suspensions by means of a new centrifugation analyser with an optoelectronic sensor“, *Colloid Polym. Sci.* 277 (1999), S. 637-645

[2] Sobisch, T.; Lerche, D.: „Application of a new separation analyzer for the characterization of dispersions stabilized with clay derivatives“, *Colloid Polym. Sci.* 278 (2000), S. 369 - 374

[3] Sobisch, T.; Lerche, D.: „Untersuchungen zur Schlammentwässerung mit dem Entmischungsanalysator LUMiFuge 114“, in *Proceedings 14. Karlsruher Flockungstage 28./29.11. 2000 Münster, Schriftenreihe ISWW Universität Karlsruhe Band 103 S.107 – 125, Karlsruhe 2000, ISBN 3-9807000-6-2*

[4] Sobisch, T.; Lerche, D.: „Investigations on sludge dewatering with the separation analyser LUMiFuge 114“, *Wateronline* 22. März 2001, <http://www.wateronline.com/read/nl20010323/411274>

Acknowledgements

We gratefully acknowledge the support of the Bundesministerium für Wirtschaft und Technologie under grant number KU 0011101KTL 9 as well as of the following companies CIBA Speciality Chemicals (former Allied Colloids GmbH Hamburg); Cytec Industries B.V., Neuss; DNWAB Dahme-Nuthe Wasser-, Abwasserbetriebsgesellschaft mbH Königs Wusterhausen; HeGo Biotec GmbH Berlin; HÖRNER Schwedt; HWG Havelländische Wasser GmbH und Co.KG Potsdam; MÜNZING CHEMIE GmbH Heilbronn.