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Flocculant Screening Method at Lab Scale for Application in Disc Stack Centrifuges with Hermetic Design

Chemicals produced using biotechnological methods like fermentation processes are obtained as complex diluted aqueous mixture, which still contains the production organism. Centrifugation is a commonly used technology for biomass separation. By flocculation, the settling velocity of microorganisms can be increased. Here, a laboratory flocculant screening method tailored for the separation of flocculated biomass in a fully hermetic disc stack centrifuge is described. The specific requirements of this process, namely, floc formation, floc stability, sliding behavior in the disc stack, and flowability of the sludge, were transferred to lab scale and validated in pilot tests. The qualitative results of the laboratory screening were in agreement with the processes at industrial scale.

Keywords: Biomass separation, Disc stack centrifuge, Flocculant screening method, Flocculation, Scale-down

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1 Introduction

A key challenge in introducing renewable raw material into the value chains of the chemical process industry is the cost- and energy-intensive water removal and product recovery process [1, 2]. Chemicals can be produced using biotechnological methods, e.g., fermentation processes. After fermentation, the value product is present in a complex diluted aqueous mixture, which still contains the production organism in an active or inactivated status. As part of the downstream processing, the biomass is very often first separated from the liquid via mechanical solid-liquid-separation. In addition to mechanical solid-liquid separation of the biomass by means of membrane technology, filtration, e.g., in filter presses, or centrifugation in decanter- and disc stack centrifuges are commonly used separation technologies [2].

This work focuses on biomass separation using disc stack centrifuges. Mechanical separation of biomass in disc stack centrifuges is often limited by the small size and small density difference between the microorganisms (solids) and the fermentation broth (liquid). The resulting low settling velocity of the microorganisms (MOs) means that only low throughputs can be realized, requiring higher overall investment and energy costs. The settling velocity of the MOs (solids) can be increased by flocculation. In contrast to disc stack centrifuges, flocculants are broadly applied when using decanters, e.g., in waste water treatment [3, 4]. A major hurdle for the application of flocculants inside disc stack centrifuges is the breakup of formed flocs in the shear-intensive inlet flow of nonhermetic centrifuges. Additionally, conventional output designs create throughput limitations with respect to both liquid and solid discharge.

The complexity of the fermentation broth composition prevents prediction of suitable flocculants and, to the best of the authors' knowledge, the selection is based on trial and error [5–9]. Different established laboratory methods for the screening of the broad variety of flocculants can be found in literature depending on the targeted application [5, 10, 11]. The stated laboratory methods often require big sample amounts, limiting the applicability as big sample amounts typically cannot be provided in early stages of the fermentation process development. Recent research and developments address this limitation in sample volume and the need for automation to achieve an efficient high-throughput screening [12–24].

As part of the EU project PRODIAS (processing diluted aqueous solutions), BASF SE (Ludwigshafen, Germany) and Alfa Laval (Tumba, Sweden) jointly developed a laboratory flocculant screening method. The screening is tailored for the biomass separation using a disc stack centrifuge (separator) with a fully hermetic design in combination with inline flocculation and the restriction of small test sample quantities. This method was validated with pilot-scale experiments in a disc stack centrifuge. The requirements on the screening were derived from the subsequent process steps in an industrial disc stack centrifuge, operated in a fully hermetic mode with a her-

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metic inlet and a continuous discharge. The following criteria were defined to select suitable flocculants:

- Complete flocculation: No single primary particles (single MOs) or fragments of particles may remain after flocculation, as the smaller settling velocity relative to the flocculated matter would cause a turbid clear phase when running the separator at the aimed throughput.
- Adequate floc size: To ensure a fast settling velocity inside the separator and enable a high throughput and, therefore, an efficient operation of the separator, a 5–10 times increase of the floc size should be achieved (material system-dependent).
- Shear stability of the formed flocs: The flocs must withstand the shear stress in the hermetic inlet of the separator or at a minimum a sufficient floc size should remain after passing the inlet.
- Sliding behavior of the biomass and the formed sediment: The flocculated biomass must slide off the discs in the disc stack and the sludge space of the separator (small angle of repose; Pentometer test).
- Rheological behavior of the formed sediment: Low yield stress and a strong decrease in the dynamic viscosity with increasing shear rate (shear thinning behavior) are necessary to ensure a good flowability and transportability of the sludge out of the separator bowl.

It should be noted that the stated criteria are not easy to fulfill as they can interact in an opposing manner. For example, capturing all primary particles and particle fragments could be assured by raising the added flocculant amount. However, the increase of added flocculant very often has a negative effect on the rheological behavior of the sediment, as stronger and wider networks of flocculant and particles are formed.

Fig. 1 sets out the main steps of the screening method, as derived from the stated requirements. First, the relevant broth characteristics, e.g., dry biomass content, pH value, conductivity, particle size distribution, and settling velocity of the untreated MOs, are determined. Second, the performance of different flocculants and necessary flocculant dosages are screened. A standardized procedure, which analyzes the resulting settling velocity of the flocculated broth and the absence of not flocculated fines, is applied for the second step. Provided that a suitable flocculant can be identified, the shear stability of the formed flocs and the sliding and rheological behavior of the respective biomass sediment are checked in the next steps. Pilot tests in a scalable separator are recommended if a satisfactory flocculant can be identified in the laboratory screening.

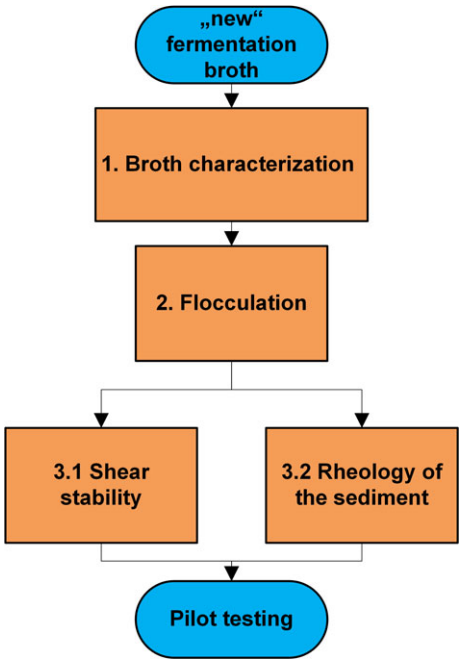

Figure 1. Overview of the laboratory screening method.

2 Materials and Methods

2.1 Fermentation Broth

In all experiments, a fermentation broth containing *Corynebacterium glutamicum* microorganisms was used as model system. The broth was inactivated after fermentation by a heat treatment. The dry biomass content varied between 1.4 and 2.2 wt % corresponding to 5.6 and 8.8 vol % of wet biomass in the broth depending on the batch. Volume percentages were determined from centrifuged broth samples by dividing the resulting volume of wet biomass by the total broth volume. The broth contained approx. 10 wt % γ-amino butyric acid (GABA) and some residuals of used trace elements and mineral compounds. The pH of the inactivated broth was 6.6 at 10 °C and the conductivity 24.6 mS cm^{-1}.

2.2 Flocculants

Different flocculants and coagulants, referred to as flocculants in this work, from the BASF product portfolio were used without further treatment. The flocculants used in the presented work are listed in Tab. 1 with the respective characteristics. Flocculant solutions with a mass fraction of active ingredients between 0.1 and 0.5 wt % were produced by diluting the liquid-grade flocculants or dissolving the solid-grade flocculants with the respective amount of tap water (pH 8.1; conductivity 500 µS cm^{-1} at 24 °C). The mixing was conducted with a propeller stirrer at 400 rpm. The flocculant solutions were stirred afterwards at 100 rpm for ripening.

2.3 Fermentation Broth Processing

2.3.1 Flocculation Studies

In general, 1.4 g of flocculant solution was added to 10 g fermentation broth. The active content of the flocculant solution was varied to achieve the wanted dosage of x kg active ingredient per ton dry biomass. The fermentation broth was stirred with a magnetic stirrer ($L^{1)}$ = 20 mm) in a 25-mL beaker at a

1) List of symbols at the end of the paper.

Table 1. Overview on flocculants. N/A, not available.

Name	Type	Charge	Charge density	Molecular weight	Structure	Delivery form
Catiofast VFH	Polyvinylamine	Cationic	High	N/A	N/A	Liquid
Magnafloc LT37	PolyDADMAC	Cationic	High	N/A	N/A	Liquid
Polymin P	Polyethyleneimine	Cationic	N/A	High	N/A	Liquid
Zetag 7109	Acrylic homopolymer	Cationic	High	Low	N/A	Liquid
Zetag 7587	Polyacrylamide	Cationic	High	Medium	Linear	Solid
Zetag 8187	Polyacrylamide	Cationic	High	Ultra-high	Linear	Solid
Zetag 9049FS	Polyacrylamide	Cationic	Ultra-high	Very high	Structured	Liquid

rotational speed of 200 rpm. The necessary amount of flocculant solution was added dropwise while stirring the broth. After adding the whole amount, the mixture was stirred for a further 2 min and finally transferred to the analytical centrifuge as fast as possible to characterize the sedimentation behavior.

2.3.2 Sedimentation Behavior

A multisample analytical centrifuge (LUMisizer, LUM, Germany) was used to determine the sedimentation kinetics of untreated and flocculated broth samples without requiring any material data or dilution prior to characterization. Based on the time- and space-resolved detection of light and transmission (STEP technology), velocity distributions were determined from recorded transmission profiles at a constant position. The cumulative velocity distributions were always determined in a region of the transmission profiles where effects of the formed sediment on the settling velocity could be excluded. A detailed description of the applied analytic method is given in literature [10, 25–31]. This analysis was performed at constant centrifugal force up to 2300 *g*. The applied centrifugal force was adjusted to the respective settling behavior in preliminary tests. Measurements were carried out in rectangular measuring cells (polycarbonate; width 10 mm; depth 2 mm). Before filling the measuring cells, the flocculated broth was shaken gently.

2.4 Spinning Disc Device

The shear stability of the formed flocs was evaluated using a spinning disc device (SDD). The SDD was provided by Alfa Laval AB. The design was adapted from Hoare et al. [13, 15, 16, 19, 20, 32, 33] with modification. The SDD consists of a circular small disc, which spins with high rotational speed in a cylindric cell. The SDD was made of stainless steel. The cylindrical chamber had a diameter of 50 mm and a height of 25 mm. The rotating disc had a diameter of 40 mm and a thickness of 1 mm. The chamber was cooled with tap water. The suspension was pumped through the SDD continuously with a volume flow of 200 mL min^{-1}. The SDD was operated at a maximum speed of 10 000 rpm. The volume flow and rotational speed were derived from CFD simulation combined with particle tracking simulations. This was done to correlate the shear treatment and the passage probability of particles through the zone of high shear in the SDD with the conditions in the inlet of industrial disc stack centrifuges with a hermetic inlet design. For details, see [34].

The effect of the applied shear on the shear-sensitive flocs was evaluated by measuring the particle size distribution directly after the SDD by means of an inline measuring device. To guarantee the necessary dilution for the particle size measurements, the feed of the SDD was diluted 1:500 with demineralized water in the feeding vessel of the SDD, with the positive side effect that only small sample amounts were needed for the tests. The particle size distributions of the untreated, flocculated as well as sheared fermentation broths were analyzed by means of static light scattering using a Mastersizer 3000 (Malvern Instruments Ltd, UK) with a measurement range between 0.01 and 3500 µm. For the measurements the Fraunhofer model was applied.

2.5 Viscosity Measurements

Dynamic viscosities were determined on a rotational shear rheometer (MCR 102, Anton Paar GmbH, Germany) using a Searle geometry (CC27, Anton Paar GmbH, Germany). During the measurements, the applied shear rate was controlled. Sludge samples for the viscosity measurements were produced by spinning 1 L of fermentation broth in a laboratory beaker centrifuge at 5346 *g* for 10 min. The supernatant was decanted and the sludge, which was concentrated to 100 vol %, was transferred to the rheometer.

2.6 Angle of Repose of Solids

The sliding behavior of the settled solids on the discs in the disc stack centrifuge was qualitatively characterized in a so-called Pentometer test using a lab swing-out beaker centrifuge. A metal insert, shaped like a dissymmetric open cone, and a lower cylindrical sediment chamber were placed in the beaker of the centrifuge. Both parts were connected by an axial passage. The incline of the open cone varied from 10 to 50° around its circumference. The Pentometer was placed in a laboratory cen-

trifuge and filled with suspension, then spun at 1240 *g* for 10 min. During spinning, the solids were forced to settle on the open cone walls and, depending on the sliding behavior, slide down via the central passage into the sediment chamber or accumulate on the open cone in dependence of the cone incline. After centrifugation, the conical surface of the metal insert was checked for biomass residuals and the respective angle of repose determined. For details of the characterization method, refer to [35].

2.7 Pilot Test Unit

The laboratory results were validated using a continuous-flow pilot-scale disc stack centrifuge (type Explorer, Alfa Laval AB, Tumba, Sweden). The pilot disc stack centrifuge had a completely hermetic design. The suspension was fed at the bottom via a rotating hollow spindle. The two separated streams left the separator via a hermetic clear phase outlet and heavy phase outlet close to the center axes. The hermetic design enables the fermentation broth to be gently accelerated to the speed of the bowl ensuring air is excluded from the inlet zone. Additionally, this design allows an energy-efficient operation of the disc stack centrifuges, especially at industrial scale [36, 37].

In this design, the accumulated sludge had to be transported against the centrifugal force by the applied pressure of the feed pump. A progressing cavity pump (Netzsch NEMO, Netzsch, Germany) was employed for this purpose. The fermentation broth was flocculated by dosing the respective amount of flocculant directly in the disc stack separator inlet spindle using a pipe-in-pipe setup. The disc stack separator was equipped either with 40 or 35° discs and operated at 10 000 rpm giving an area equivalent Σ of 1696, 2605, 2848, or 3049 m^2 and KQ values of 40, 61, 66, or 71, respectively. Σ in m^2 and the semi-empirical KQ values were calculated according to the following equations [38]:

$$\sum = \frac{\pi\omega^2}{g}\frac{2}{3}N\left(r_2^3 - r_1^3\right)\cot\alpha \tag{1}$$

$$KQ = \frac{280}{10^6}\left(\frac{n}{1000}\right)^{1.5}N\left(r_{out}^{2.75} - r_{in}^{2.75}\right)\cot\alpha \tag{2}$$

where r_2 is the maximal disc radius (m), r_1 is the minimal disc radius (m), r_{out} is the maximal radius of disc (cm), r_{in} is the minimal radius of disc (cm), N denotes the number of discs, α is the half-cone angle of the disc, ω means the angular velocity, g is the acceleration due to gravity (9.81 m s^{-2}), and n is the bowl speed. The feed flow rate was varied between 60 and 1000 L h^{-1}.

3 Results and Discussion

3.1 Flocculation

To meet the stated criteria, the flocculant screening starts with the screening of flocculation aids focused on the formation of flocs and the resulting impact on the settling behavior. A detailed flow chart of the established screening step is presented in Fig. 2. To keep the necessary sample volume small, 10 mL fermentation broth were always mixed in a small beaker with the required amount of active ingredient using a magnetic stirrer. It is recognized that the mixing conditions have a dominant effect on the flocculant performance.

Given the limited sample volume, the typical lab setup of parallelized propeller stirrers was inappropriate. For this reason, mixing on a vortex mixer and mixing by intensive shaking in a 50-mL flacon were tested in addition to mixing with a magnetic stirrer. Mixing with a magnetic stirrer showed the best flocculation performance in regard to the remaining fines and was comparable to the mixing with a propeller stirrer. It is known that magnetic stirrers can have a compacting effect on the formed flocs, but this effect is considered tolerable in the targeted application. The amount of flocculant or, more precisely, the amount of active ingredient in the flocculant solution, is normally referenced to the amount of dry biomass in the suspension. In case of the used model system, 10 kg active ingredient per ton dry biomass proved to be a good starting value for the screening, but this can vary from broth to broth. The mixing conditions and subsequent ripening time were kept constant (see Sect. 2).

In case of the used model system, an increase of the particle size by the factor of 5–10 would be sufficient to achieve the desired throughput increase. This targeted improvement of the separator operation does not require macroscopically visible flocs. Therefore, an analytical centrifuge (Lumisizer, LUM GmbH, Germany) was used for the evaluation of the floc formation. Fig. 3 displays the measured cumulative velocity distribution as a function of the settling velocity for the untreated broth sample in comparison to flocculated samples. Different flocculants were applied at a constant dosage of 10 kg active ingredient per t dry biomass. Additionally, the calculated distribution of an increase of the original particle size by a factor of 5 (velocity increase by a factor of 25 according to the Stokes settling velocity) is plotted in gray as a benchmark. The adequate rotational speed of the analytical centrifuge is floc size-dependent and must be determined in experimental trials. In case of the used model system and the tested flocculants, a rotational speed of 584 rpm (40 *g*) for 25 min yielded good results with regard to the interpretability of the transmission scans. As can be seen, different flocculants caused different floc sizes and respective settling velocities. Based on these results, suitable flocculants for the floc formation can be selected for the following screening steps.

The completeness of the flocculation is important in addition to the size of the formed flocs and the interrelated settling velocity. To realize high throughputs in a separator, the applied concentration of flocculant must be able to flocculate all fines. Fines can be detected by comparing the final transmission of the flocculated samples with the transmission of a particle-free, filtered broth. Additionally, visually checking the cuvettes directly after spinning is recommended as some flocculants may cause residuals on the cuvette surface which affects the transmission profiles.

Remaining fines as well as the resulting floc size depend on the added flocculant amount. Thus, the flocculant amount can be increased to achieve a complete flocculation and to enlarge

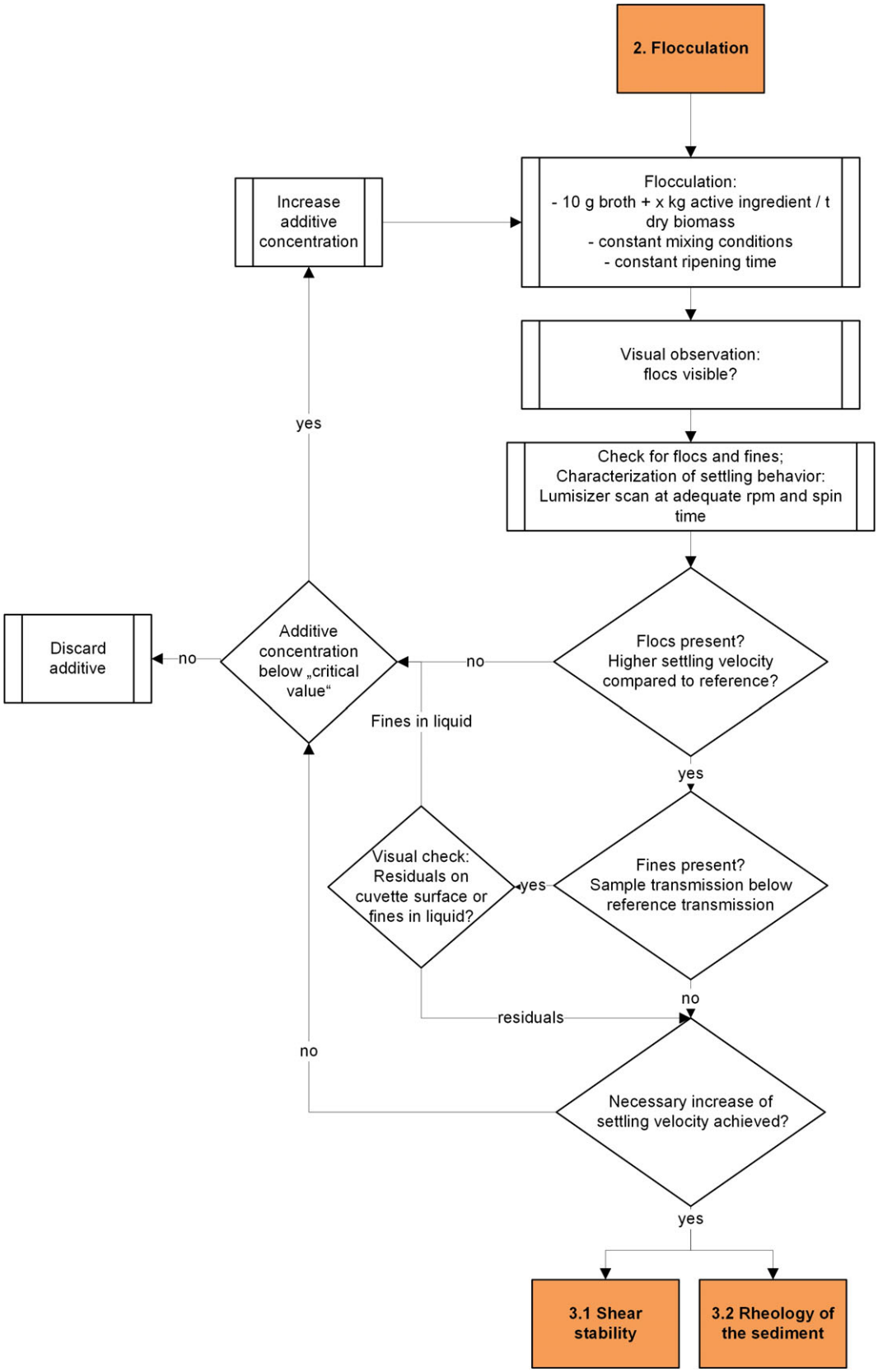

Figure 2. Detailed design of the flocculation step.

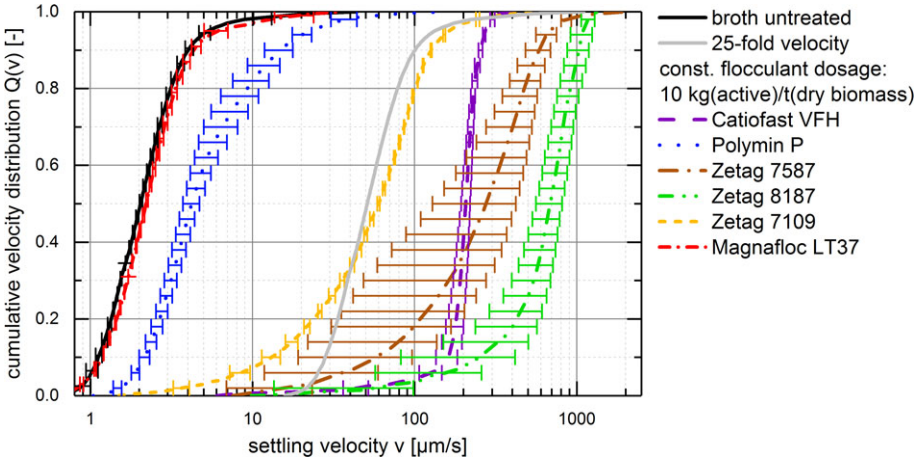

Figure 3. Cumulative velocity distribution of different flocculants in comparison to the not flocculated fermentation broth and to the velocity distribution calculated for a 25-fold increase in settling velocity (= fivefold increase in particle size) of the untreated broth. Standard deviations are based on three experiments.

the floc size under the applied mixing conditions. The maximum critical flocculant amount is process-specific and depends on various parameters, e.g., the effect on further downstream process steps or flocculant costs. Fig. 4 illustrates three examples of the effect of added flocculant amount on the resulting settling velocity. The targeted increase in settling velocity can be reached for Zetag 7109 and Polymin P in contrast to Magnafloc LT37 in the tested flocculant amount range up to 30 kg active ingredient per t dry biomass.

It should be noted, that the mixing procedure performed at lab-scale mimics long mixing and ripening times between MOs and flocculant (approx. 2 min), which cannot be realized at industrial scale when the flocculant solution is directly dosed into the inlet of the separator (at retention times below 1 s). Despite this obvious discrepancy between the screening method and the conditions at industrial scale, the comparison of the results at laboratory and pilot scale demonstrated that the presented method can be used to qualitatively identify suitable flocculants. The validation of the laboratory screening method is indicated below (see Sect. 3.4).

Further improvements could be realized by detailed simulation and characterization of the mixing conditions in the hermetic inlet of an industrial disc stack centrifuge to derive the design of a lab-scale mixing device. Overall, in our opinion, the presented laboratory screening step provides an efficient method for assessing the performance of different flocculants. Strong performers in this screening step can then be tested in the consecutive screening steps. Both, the shear stability of the formed flocs and the flowability of the formed sediment (sludge) inside the separator, are important to achieve the targeted throughput increase.

3.2 Shear Stability

The targeted increase in the throughput cannot be achieved if the flocs are too weak and break up into primary particles or undersized flocs because of the applied shear stress in the inlet of the separator. Therefore, it must be assured that the selected flocculant forms shear-stable flocs such that the remaining floc size will still be bigger than a critical particle size and that no primary particles are produced because of floc erosion. The shear stress in a hermetic inlet of a disc stack centrifuge was scaled down to laboratory scale using an SDD. As mentioned in Sect. 2, the used settings of the continuously operated SDD were derived from simulations.

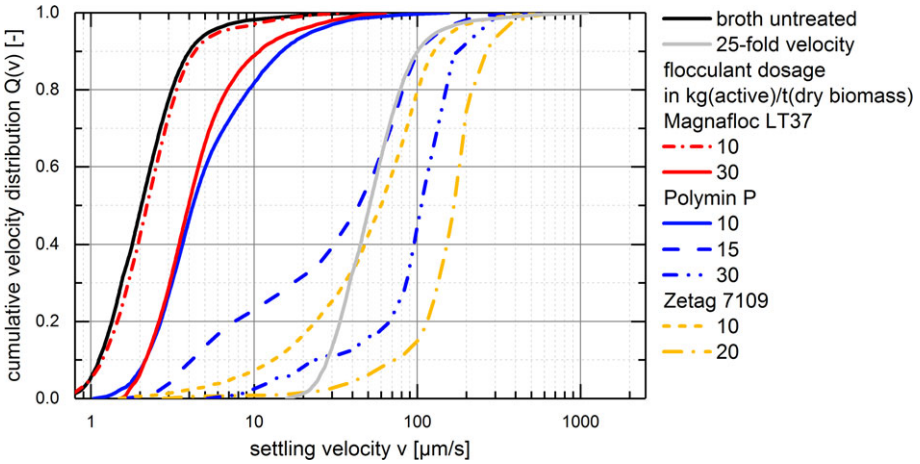

Figure 4. Effect of the applied flocculant amount on the resulting settling velocity distribution (samples spun at 40 *g* for 25 min).

Fig. 5 shows exemplarily the effect of different rotational speeds of the SDD and corresponding shear stresses on the formed flocs for one tested flocculant. As expected, the particle size distributions are shifted to smaller sizes for higher shear stresses.

The mean particle size of the measured volume sum distribution of flocs formed by different flocculants and different flocculant amounts is indicated in Fig. 6. The mean particle sizes of flocculated, not sheared broth are compared with the corresponding results after shear treatment. As in the analytical centrifuge data, the differences in floc formation performance can be clearly seen for the unsheared

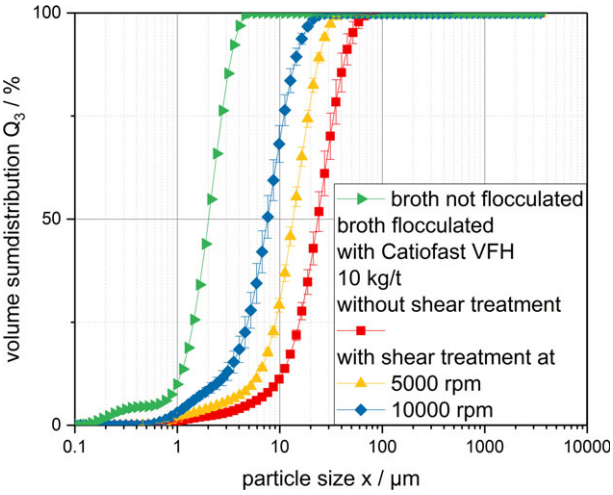

Figure 5. Resulting particle size distribution after a shear treatment in the SDD with different rpm (flocculant: Catiofast VFH with 10 kg active ingredient per t dry biomass).

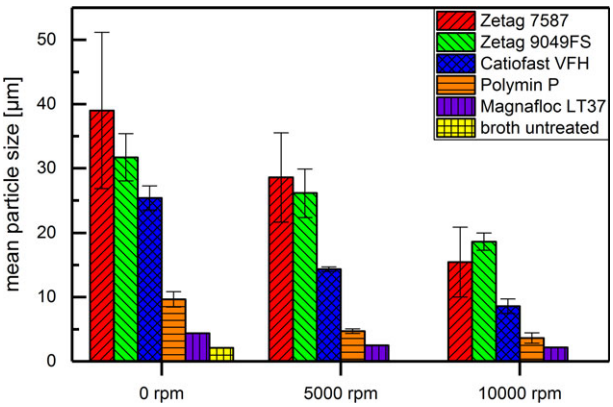

Figure 6. Comparison of different flocculants regarding the shear stability (flocculant concentration in kg active ingredient per t dry biomass: Zetag 9049FS: 10; Zetag 7587: 10; Catiofast VFH: 10; Polymin P: 15; Magnafloc LT37: 30).

samples (0 rpm). Furthermore, the floc sizes decreased with higher rotational speed of the SDD consistent with increasing shear stress. Hence, under the chosen conditions, the effective shear rates and the residence time in the SDD are high enough to break up the formed flocs.

As can be seen, none of the flocculants formed completely shear-stable flocs. The flocculants differ significantly in the floc size before and after shear treatment, but all mean particle sizes decrease with increasing rotational speed. Under the chosen conditions, Zetag 9049FS, Zetag 7587, and Catiofast VFH produced flocs with a remaining particle size bigger than 5 µm after a shear treatment with 10 000 rpm in the SDD. In contrast, the flocs formed by Polymin P and Magnafloc LT37 were broken up nearly to the primary particle size at the same shear treatment. Floc stability caused by different flocculants can be qualitatively characterized and compared. The results can be used to further reduce the number of suitable flocculants.

3.3 Rheological Characterization

3.3.1 Pentometer Test

To continuously operate the disc stack centrifuge, the settled biomass must slide down on the discs in the disc stack and the pointed sludge space of the separator under the influence of the centrifugal force. At industrial scale, the incline of the discs in the disc stack has a significant influence on the settling area that can be placed in the same size apparatus. From an investment cost perspective, discs with a disc angle of α = 35° are preferable to discs with a bigger angle. The required inclination of the discs and the bowl geometry (the angle of repose) can be identified using a Pentometer, established by Alfa Laval. Generally speaking, the lower the angle of repose determined in the Pentometer test, the better. Fig. 7 shows exemplary pictures of the conical Pentometer surface. It can be observed that the incline of the cone to which the biomass accumulated depends on the applied flocculant. Tab. 2 gives an overview of selected results.

The applied flocculants strongly influence the angle of repose of the accumulated biomass. The flocculants which were tested successfully in the floc formation and floc stability screening steps, generally showed higher angles of repose than those with a poor flocculant performance. Zetag 7109 and Polymin P exhibited an angle of repose slightly bigger than 35°. Using these flocculants, 40° discs in the disc stack should be feasible. Zetag 7587, Zetag 9049, and Catiofast VFH showed a bigger angle of repose around 45°. Therefore, discs with an angle greater than 45° should be applied, lowering the realizable settling area. Additionally, the measured high angles of repose are indicative of an insufficient flowability of the formed sediment. Overall, the Pentometer test gives a qualitative comparison, which enables to narrow down relevant apparatus geometry parameters.

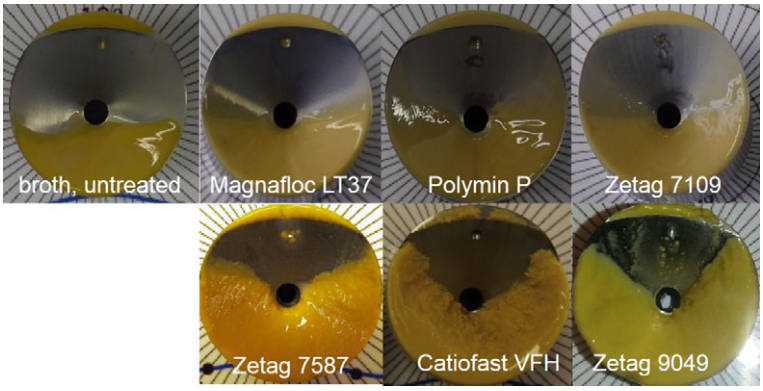

Figure 7. Exemplary results from the Pentometer test comparing different flocculants. The highest angle on the cone point upwards, the lowest downwards in the image (flocculant concentration in kg active ingredient per t dry biomass: Magnafloc LT37: 30; Polymin P: 15; Zetag 7109: 20; Zetag 7587: 10; Catiofast VFH: 15; Zetag 9049: 45).

Table 2. Overview Pentometer test results (standard deviation based on multiple experiments).

Flocculant	Angle of repose [°]
Untreated broth	32 ± 3
Magnafloc LT37, 30 kg t^{-1}	34 ± 1
Polymin P, 15 kg t^{-1}	36 ± 2
Zetag 7109, 20 kg t^{-1}	38 ± 2
Zetag 7587, 10 kg t^{-1}	45 ± 2
Catiofast VFH, 15 kg t^{-1}	45
Zetag 9049FS, 45 kg t^{-1}	44

3.3.2 Rheological Characterization

In a fully hermetic design, the formed sludge must be flowable to such an extent that it is possible to force it out of the separator bowl by solely the feed pump pressure. Ideally, the separator can be operated without intermittent discharge, which would limit the throughput. Nevertheless, the operation of the separators could be improved if operated in fully hermetic mode for most of the time. Periodic discharges with long intervals would only be needed to completely empty the sludge space and prevent the sludge from growing into the disc stack. Commonly, concentrated microorganism suspensions show a shear-thinning behavior. The addition of flocculants and related formation of flocs and networks can increase the viscosity in a broad shear rate range and, therefore, complicate the transport of solids out of the separator. Fig. 8 presents examples of 100 vol % consolidated sludge treated with different flocculants. The appearance varies from crumbly and rubber-like to toothpaste-like.

Based on experiments by Alfa Laval, the rheological behavior of a baker's yeast suspension, consolidated to 100 vol %, was taken as benchmark (reference) for the flowability of the sludge inside the separator with a hermetic outlet design. In general, the biomass sludge should show a low yield stress, a pronounced shear-thinning behavior, and resultant low dynamic viscosities at shear rates > 100 s^{-1}. It should be noted that the formation of the sediment and the resulting rheological properties inside the separator are particularly difficult to precisely transfer to lab scale. The time scales for settling, sediment formation, and sediment consolidation/compression are very short in an industrial separator. In contrast, lower *g* force and batch operation mode in the lab procedures create longer retention times. Fig. 9 compares the measured dynamic viscosities at low shear rates of sludge prepared according to the laboratory method and sludge taken from the separator bowl during pilot testing. In both experiments, the same flocculant and flocculant dosage were applied.

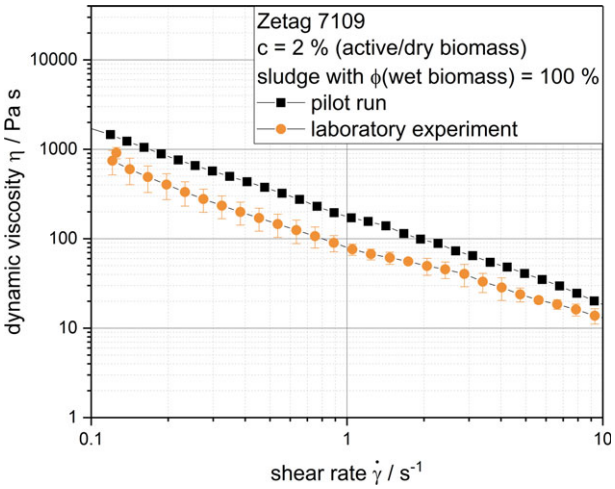

Figure 9. Comparison of the measured dynamic viscosity as a function of the applied shear rate of the biomass sludge produced at laboratory vs pilot scale.

The dynamic viscosity of the sludge sample from the laboratory experiment is slightly smaller over the measured shear rate range. Nevertheless, the magnitude of the dynamic viscosity and the shear-thinning behavior are comparable. In our opinion, the applied preparation method at lab scale is suitable to scale down the relevant behavior for the targeted qualitative rating of different flocculants. The effect of different flocculants on the flowability of the sludge is depicted in Fig. 10. All sediments show shear-thinning behavior, but the viscosity slopes of some of the samples are shifted to higher values in the measured shear rate range. Taking the benchmark into account, the flocculants, which showed good performance in the floc formation and floc stability, Catiofast VFH and Zetag 7587, generated sludge with too high viscosities and, thus, an insufficient flowability.

It should be noted that some of the flocculants caused a very crumbly sediment or rubber-like texture. The measurements of the dynamic viscosity of these sediments are challenging. From a scientific point of view, the accuracy of these measurements is questionable, but the extracted results are in accordance with the visual observations and the results of the pilot testing (see below). Thus, in our opinion, the applied method is suitable to compare the effect of different flocculants on the rheological behavior in a qualitative way and rate the suitability of different flocculants based on these data.

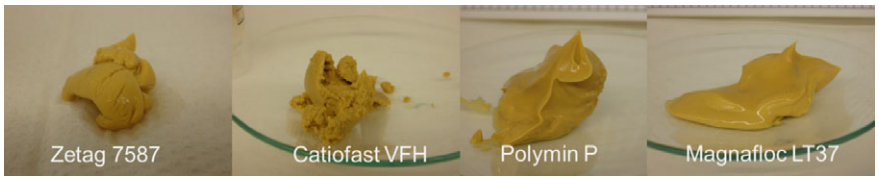

Figure 8. Examples of the resulting sediment dosing different kinds of flocculants (flocculant concentration in kg active ingredient per t dry biomass: Zetag 7587: 10; Catiofast VFH: 10; Polymin P: 15; Magnafloc LT37: 30). The same laboratory procedure was applied.

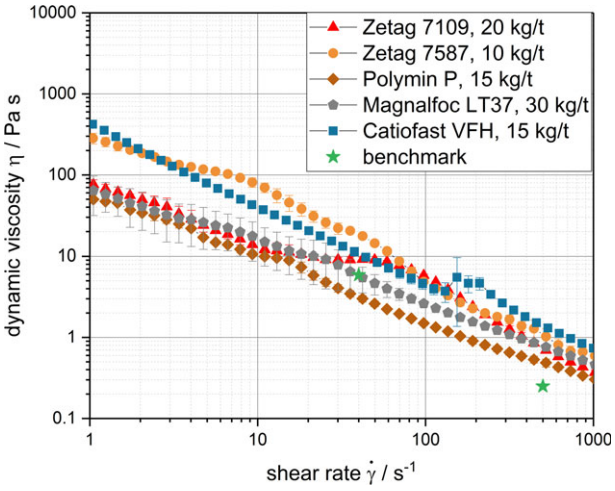

Figure 10. Rheological behavior of biomass sludge dependent on the applied flocculant type.

3.4 Pilot Tests

The screening steps were validated by testing different flocculants in pilot scale as described in Sect. 2. The criteria floc formation and floc stability were validated by evaluating the achieved separation efficiency in dependence of the realized load factor (throughput per KQ). The Pentometer test was checked by testing different disc angles. The flowability of the formed sediment was validated by comparing the discharge intervals in which the pilot separator was operated stably in fully hermetic mode. Fig. 11 shows the achieved separation efficiency based on the detected solids in the clear phase vs the load factor in L h^{-1} clear phase flow per KQ of the pilot separator.

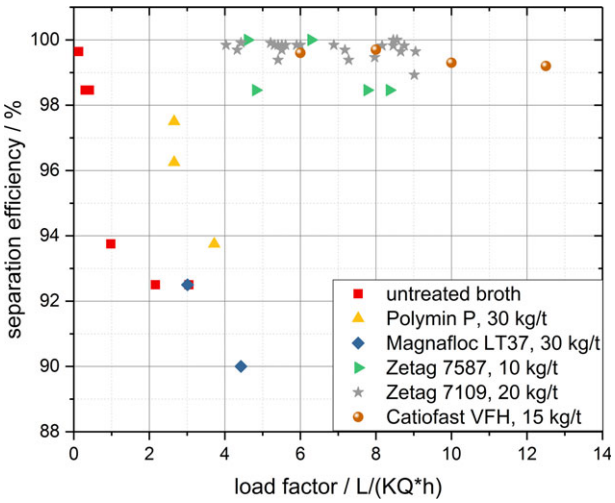

Figure 11. Comparison of the flocculant performance regarding the separation efficiency in the pilot tests.

For the used untreated model system, a satisfying separation efficiency above 99 % could only be achieved for very low flow rates and corresponding load factors < 0.5 L KQ^{-1}h^{-1}. The load factor could not be significantly increased by using Polymin P or Magnafloc LT37 as flocculants. As predicted in the laboratory screening method, Magnafloc LT37 and Polymin P were not able to flocculate the used model system sufficiently or the formed flocs were not stable enough to yield an adequate remaining floc size. In contrast, a satisfying separation efficiency (> 99 %) up to a high clear phase flow rate of more than 500 L h^{-1} and corresponding load factors above 8 L KQ^{-1}h^{-1} could be realized by using Zetag 7587, Zetag 7109 or Catiofast VFH. These results are in accordance to the qualitative evaluation in the laboratory screening. In summary, the SDD, operated as stated, is suitable to predict the floc breakup in a hermetic inlet design of a disc stack centrifuge and qualitatively characterize different flocculants in regard to their floc stability.

The Pentometer tests could be validated as the pilot separator could be operated with 40° discs but not with 35° discs in case that Zetag 7109 was taken as flocculant (angle of repose = 38 ± 2).

With regard to the flowability of the formed sludge, none of the flocculants which yielded satisfying separation efficiency could be used without requiring regular discharges. However, the achieved discharge intervals varied. Tab. 3 indicates the necessary discharge intervals obtained for the tested flocculants. In case of Catiofast VFH (15 kg t^{-1}), the formed sediment demonstrated such poor flowability that the heavy phase outlet blocked and no operation in fully hermetic mode was possible. Using Zetag 7109 (20 kg t^{-1}), discharge intervals around 30 min could be realized in contrast to shorter discharge intervals of approx. 5 min when Zetag 7587 (10 kg t^{-1}) was applied at a comparable flow rate. These differences can be correlated satisfactorily to the rheological behavior in the laboratory method.

Table 3. Necessary discharge intervals in dependence of the applied flocculant.

Flocculant	Discharge interval
Zetag 7587, 10 kg t^{-1}	≈ 5 min at 600 L h^{-1} feed rate
Zetag 7109, 20 kg t^{-1}	≈ 30 min at 600 L h^{-1} feed rate
Catiofast VFH, 15 kg t^{-1}	Heavy phase outlet blocked

4 Conclusions

A laboratory flocculant screening method is presented, tailored for the specific requirements of the separation of flocculated biomass in a fully hermetic disc stack centrifuge. The consecutive process steps floc formation, floc shear stability, sliding behavior in the disc stack, and flowability of the sludge were transferred to lab scale. It could be shown in pilot tests that the qualitative results of the laboratory screening steps are consistent with the processes at industrial scale. The need for only small sample quantities allows the rapid and more effective search for suitable flocculants at an early stage of fermentation process development.

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Symbols used

g	[m s^{-2}]	acceleration due to gravity, 9.81
KQ	[–]	semi-empirical measure of centrifuge size (Eq. (2))
L	[m]	stirrer length
n	[rpm]	bowl speed
N	[–]	number of discs
r_1	[m]	minimal radius of disc
r_2	[m]	maximal radius of disc
r_{in}	[cm]	minimal radius of disc
r_{out}	[cm]	maximal radius of disc

Greek letters

α	[°]	half-cone angle of disc
ω	[rad s^{-1}]	angular velocity

Abbreviations

GABA	γ-amino butyric acid
MO	microorganism
SDD	spinning disc device

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