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ACTIVATED SLUDGE FLOCCULATION DYNAMICS: ON-LINE MEASUREMENT METHODOLOGY AND MODELLING

DYNAMICA VAN ACTIEF-SLIB FLOCCULATIE: ON-LINE MEETMETHODOLOGIE EN MODELBOUW

door

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Summary

The activated sludge process is one of the most widespread biological wastewater treatment method. For an effective process, the solid-liquid separation represents a crucial step. In order to obtain a successful solid-liquid separation and to meet the latest regulations regarding effluent quality, the microorganisms must form flocs, which settle and compact well without leaving a high suspended-solids concentration in the effluent. The activated sludge flocculation, however, is a very complex process in which many factors interact and may have an influence. Since a large number of these influences are poorly understood, the characteristics related to the floc-formation are still difficult to be predicted and controlled. Hence, improving knowledge on the flocculation step is an essential requirement for an optimal biological wastewater treatment.

The work described in this thesis was conducted in the framework of the "Sedimentation and Flocculation" (*SediFloc)* project conducted through several PhD studies. The objectives of this project were to quantify and model the flocculation process in secondary clarifiers by accounting for the influence of different physico-chemical parameters. Therefore, putting together the information concerning the flocculation dynamics and its modelling may represent one step forward in process understanding and a more accurate prediction of effluent quality.

This thesis aimed at developing a methodology for the on-line evaluation of the flocculation dynamics and at investigating the process response to variations of different physico-chemical factors. Finally, the joint effect of these parameters was investigated. To this end, Design of Experiment (DOE) and Response Surface Methodology (RSM) approaches were used. The knowledge resulting from this systematic study was used to explain the behaviour of a set of process characteristics.

The possibility of on-line monitoring of the activated sludge flocculation dynamics was demonstrated for the first time by Biggs (2000). Starting from this method, an important part of this work was devoted to the development of techniques that allow for the direct evaluation of the relationship between different operational parameters, the flocculation process and the effectiveness of the clarification step.

Initially, attention was paid to the possibility of measuring the floc morphological properties (e.g. the floc size and size distributions and fractal dimension). Due to the fragile biological nature, irregular structure and heterogeneous composition of the flocs, the measurement procedure may affect the results and lead to a misinterpretation of the data. Following a comprehensive literature study of the available sizing devices, a MastersizerS (Malvern, UK), a CIS-100 (Ankersmid, Belgium) and an automatic image analysis system (IMAN) developed in house in LabView (NI, USA) were selected for measurements. These devices were selected because of their different measurement principles, which allows obtaining much (sometimes complementary) information regarding the process dynamics, as well as because of their on-line analysis capabilities and similar sample preparation and manipulation requirements. The devices performance for activated sludge floc size and size distributions measurements was systematically evaluated and compared. To this end, the influences of some set-up

components, the concentration range and the possibility to in-line dilute the samples were investigated in order to find the operating conditions creating minimal floc disturbances during measurements. Moreover, the influence of floc shape and measurement principle on the size measurements was evaluated and the results were compared with those obtained for inorganic particles (round glass beads and irregular sand particles). It was found that all techniques used turned out to be fast and reliable methods for direct quantification of the floc size distributions.

For a correct evaluation of the influence of different physico-chemical factors on the flocculation process, the sludge samples should first be standardised. For this purpose, a Sequencing Batch Reactor (SBR) was built and operated under stable environmental conditions. An intensive monitoring procedure allowed for the evaluation of the sludge stability. In order to carry out this task, a procedure that linked the sludge settling performance with the evolution of the floc size and size distributions and microbial community dynamics (using DGGE and microscopical observations) was developed. It was found that sludge stability can be achieved only for relatively short time periods (e.g. of approximately one month) and, therefore, the experimental duration for performing the flocculation experiments should be restricted as well.

For evaluating the flocculation dynamics, an experimental set-up (FlocUNIT) was built. It consisted in a five-liter reactor in which the flocculation process took place. Different environmental conditions were applied in the reactor and their effect was monitored on-line by using specific sensors and off-line at the end of each experiment. A number of 36 experiments was performed by following an orthogonal and rotatable DOE. The influence of five relevant factors (temperature, shear stress, dissolved oxygen concentration, sludge concentration and calcium concentration) on a set a selected process responses (floc size, SVI, supernatant turbidity and suspended solids concentration, zeta potential, pH, conductivity) was evaluated.

Initially, the effect of each of the considered factors on the floc size dynamics was investigated separately. Among these factors, the most significant contribution on the size originated from the shear stress and the calcium concentration. High shear stress values produced deflocculation. However, calcium addition allowed the flocs to partially reaggregate even at high shear stress values. At low shear stress values, aggregation dominated and resulted in a slight and continuous increase of the floc size. The high affinity of the SBR sludge for calcium was not observed when different sludge samples collected from a WWTP were analysed. It was therefore concluded that the response of the activated sludge sample to the calcium addition is highly dependent of the initial calcium content of the sludge. At a higher solids concentration a tendency to form larger flocs was observed. Larger floc sizes were also observed when the temperature decreased in the reactor, while the dissolved oxygen concentration did not have a significant short-term effect on the floc size dynamics.

Finally, the joint effect of all five factors on the considered responses was investigated. The responses were evaluated by using a quadratic polynomial model, constructed from the experimental data after each factor underwent a linear or logarithmic scale normalisation. Accurate statistical models were obtained when the measurements were performed by using on-line sensors. The off-line analyses were influenced by the additional errors due to the sampling and measurements procedure, leading to model predictions with a moderate to poor statistical quality. Among the considered factors, the highest influence on the floc size dynamics came from shear stress. Similar operating conditions were found to be responsible for both larger floc formation and lower SVIs, demonstrating the link between the settling properties and floc size. The destabilisation process occurring during the (de)flocculation processes is not entirely due to charge effects and can not be fully described by using zeta

potential. Anaerobic conditions and low sludge concentrations led to an increase of the supernatant turbidity. However, a poor statistical model accuracy was found for models aimed at predicting the supernatant suspended solids concentrations and turbidity. It was suggested that the selected quadratic models can not predict the behaviour of these responses accurately. Conductivity showed, as expected, a direct relationship with the calcium concentration present in the system, while temperature and sludge concentration only had a small influence. Under the imposed factors, the pH did not change sufficiently to affect the flocculation process.

A process optimisation by using RSM was performed for the particular case of SBR sludge. Its goal was to find the conditions necessary for obtaining good settling properties (large floc size and low SVI) and low suspended solids concentrations of the supernatant ("zero" values for turbidity, suspended solids concentrations and zeta potential). It was found that, in order to obtain the imposed targets, the optimal operating conditions corresponded to the following factor values: temperature of approx. 7-8 ºC; as low as possible shear stress values (even lower than the minimum value (15 s^{-1}) considered in these experiments); a calcium concentration of 14-18 meq/l; a dissolved oxygen concentration of approx. 1 mg/l and a sludge concentration of around 3.2 g/l.

In conclusion, this dissertation focused on various aspects related to the measurement, quantification and optimisation of the flocculation process. As a result, a methodology and empirical modelling by using DOE/RSM was developed and used for on-line characterisation of the flocculation dynamics and of its response to the effect of five physico-chemical factors. The conducted research also demonstrated that for a better prediction and control of the flocculation process, an integral approach, in which the cumulative effect of different factors is accounted for simultaneously, should be considered. It was shown that with a relatively low number of experiments, it is possible to investigate the short-term effect of changing environmental conditions and to find the factor values that would optimise the process. Moreover, the obtained results may be further used for a more elaborated modelling of the flocculation process dynamics by using a population balance formulation.