CONTRIBUTION TO THE STUDY OF FLOCCULATION OF DIGESTATE

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Abstract

The paper deals with the intensification of separating the solid phase of digestate using flocculants only. The separated solid phase should subsequently be used in agriculture for fertilising. Flocculants (polyacrylamides) are difficult to biodegrade. In this respect, they should not deteriorate the properties of the solid phase and the flocculant dose must be as low as possible. The research aimed to identify the optimal cationic flocculant and its application procedure which would enable a dosage that would be both economically and ecologically acceptable. We tested 21 cationic flocculants of different charge density and molecular weight and 1 mixture of two selected flocculants (Sokoflok 53 and Sokoflok 54) with the aim to discover the lowest possible dose of flocculating agent to achieve the effective aggregation of digestate particles. The lowest flocculant doses were obtained using the mixture of flocculants labelled Sokoflok 53 and Sokoflok 54 in 4:1 proportion, both of a low charge density and medium molecular weight, namely 14.54 g/kg of total solids for a digestate from the biogas plant Stonava and namely 11.80 g/kg of total solids for a digestate from the biogas plant Vrahovice. The findings also reveal that flocculation is most effective during two-stage flocculant dosing at different mixing time and intensity.

Keywords: whole digestate, flocculant, charge density, molecular weight

INTRODUCTION

Anaerobic digestion results in biogas as a major product and whole digestate as a by-product. Biogas is further used for the generation of electric power and heat. Even if the production of digestate is not desirable, it makes an inseparable part of the process. Digestate is predominantly disposed of on agricultural land under the conditions stipulated by relevant legislation in each country.

Digestate is a stabilised material comprising of non-decomposed residual materials from digestion and necrotic microorganisms. Its properties are largely influenced by the type of the processed biomass. Most frequently we come across digestate made from agricultural feed materials, and which is subsequently used for fertilisation. However, there are several problems we perceive in the issue of digestate, for example, lying in the enormous volumes of the produced digestate to be used in agriculture, a low content of total solids (2-4 %), or rather low contents of nitrogen, phosphorus or degradable organic matter. This is related to the principle of digestate formation, when during anaerobic fermentation well degradable organic matter had already decomposed and been converted into biogas (Kolář et al., 2010). Low alkalinity of the digestate (pH about 7-8), caused by a high proportion of ammonia nitrogen, predetermines digestate to be used on acidic or neutral soils. The transportation and disposal of large volumes of digestate increases economic costs, potential risks of soil waterlogging and pollution of surface and ground water. To get the idea about the digestate volumes we are talking about, a biogas plant (BGP) with the output of 1MW produces on average 100 m^3 of digestate per day. More details are provided by Heviánková et al. (2014) who describe options and methods of digestate disposal, including rare methods or those verified in the laboratory scale (Heviánková et al., 2014).

Digestate contains high amounts of colloid and fine particles, and a negative charge prevails in the suspension (Sievers et al., 1994). Therefore, the separation of solid phase using conventional methods, such as sedimentation, filtration (pressure or under-pressure) or centrifugation, is difficult. Even if coagulating and flocculating agents may be used to intensify the separation of solid particles from digestate, their consumption is very high. At the same time, using coagulants, the separated solid phase contains high concentrations of iron or aluminium, and the filtrate is loaded with high concentrations of sulphates or chlorides in dependence on the type of used coagulant. This makes the further use of separated digestate phases difficult or even impossible.

Some methods of solid phase separation combine the use of coagulants, flocculating agents, agents to modify the pH value and defoamers. Such methods are effective but very difficult as for the technology and questionable in terms of utilising the products of such separation procedures. Another bulk of scientific works focuses on the intensification of the solid phase separation using flocculating agents and materials improving the filterability of sludge formed during mechanical dewatering (Qi et al., 2011). The materials mentioned above may be coal fines (Albertson and Kopper, 1983; Sander et al., 1989), char (Albertson and Kopper, 1983), wood chips (Lin et al., 2001), bagasse (Benítez et al., 1994); fly ash (Chen et al., 2010), (Benítez et al., 1994; Qi et al., 2011), cement kiln dust (Benítez et al., 1994), gypsum (Zhao, 2002; Zhao, 2006), and bentonite (Alvarenga et al., 2015).

As mentioned above, the intensification of the solid phase separation has been dealt with in many studies using commercially available flocculating agents of various types and different suppliers. Their base is formed by acrylic amide and its derivatives containing anion or cation groups of varying molecular weights, charge densities and molecule shapes.

The aim of the work is to search for such flocculating agent and its application procedure so that its consumption is economically feasible. We tested selected flocculating agents and we also prepared a mixture of two different flocculating agents.

With regard to the fact that the separated solid phase should be applicable in agricultural fertilising, it is important for the flocculating agents not to aggravate its properties. In general, polyacrylamides are very difficult to biodegrade, and thus it is desirable for the flocculating agent dose to be as low as possible.

Due to the varying flocculation characteristics of the dewatered materials, laboratory assays or performance tests must be carried out for each case separately to determine the required amount of the dosed polymer. Organic matter prevails in the digestate and, in general, it holds true that during the dewatering of sludge of an organic origin using machinery, cationic polymer flocculating agents are used, usually without added inorganic auxiliary agents, such as salts of iron or aluminium or lime. The doses of the polymer flocculating agents usually range from 2 to 8 g/kg of sludge total solids.

Our results so far (Souček, 2014) concerning the consumption of flocculating agents to separate the solid phase from digestate significantly exceed the usual dose range. For example, Popovic et al. (2013) experimentally verified that when adding the cationic flocculating agent Superfloc C-2260 (linear chain, high molecular weight, medium charge density) in doses from 200 to 500 ml of 0.1% polymer solution into 500ml of sample, no significant changes in the tested system were observed, or no effect on the separation efficiency of codigestate occurred. An increase in the separation efficiency was achieved by Popovic et.al. having added 0.5 g of biochar and 120 ml of chitosan solution.

Alvarenga et al. (2015) obtained positive results in dewatering digestate using cationic ZETAG 9014 (high molecular weight, low charge density), dosing approximately 19-25 g/kg TS of digestate. This resulted in good filterability of the separated liquid constituent without the gelling of filter cake. At the same time, the digestate had to be modified by adding a diatomite-bentonite mixture containing calcium oxide ranging from 0.8 to 1.6kg/kg TS of digestate.

MATERIALS AND METHODS

BGP digestate

For the experiments, we used the samples of digestate from two agricultural biogas plants in the Moravian-Silesian and Olomouc Regions. Both of them process cattle's liquid manure, silage maize and haylage. The samples were drawn from the storage site for the whole digestate which is permanently stirred. The samples were studied for the pH value, temperature, total solids (TS) and loss on ignition (LOI), in accordance with Standard ČSN EN 12879. For the input analysis, we used a Denver Instrument SI-234 A analytical balance (d=0.0001g), WTW pH meter (Model 330i) with automatic compensation for temperature, dryer and an annealing furnace.

Polymer organic flocculants

During the digestate flocculation assays, we tested a number of different polyacrylamide polymer flocculants SOKOFLOK (also referred to as Flopam) distributed by Sokoflok Sokolov (produced by SNF Floerger) and SUPERFLOC (produced by Kemira), see Tab. 1. The applied flocculants are in the dry form on the base of acrylamide-dimethylaminoethylacrylate copolymer. We prepared solutions of the dry flocculants, namely 0.1% solutions, which is the most common concentration under the plant conditions. For more information about the flocculants, see, for example, Bolto and Gregory (2015).

Tab. 1: The tested cationic flocculants (for more details, see the company brochures by SOKOFLOK accompanying the product of SNF Floerger and www.strykerchem.com/docs/SuperflocC-496TDS.pdf)

Commercial name	Name in Figures	Charge density	Molecular weight	**Commercial name**	Name in Figures	Charge density	Molecular weight
Sokoflok 53	**S 53**	low	medium	Sokoflok 67	**S 67**	medium	medium
Sokoflok 53 CN	**S 53CN**	low	high	Sokoflok 109	**S 109**	medium	medium
Sokoflok 53 GP	**S 53GP**	low	very high	Superfloc C 495	**C 495**	medium	high
Sokoflok 54	**S 54**	low	medium	Superfloc C 496	**C 496**	medium	high
Sokoflok 54 CN	**S 54CN**	low	high	Superfloc C 496 HMW	**C 496 HMW**	medium	very high
Sokoflok 54 GP	**S 54GP**	low	very high	Sokoflok 59	**S 59**	high	medium
Superfloc C 492	**C 492**	low	high	Sokoflok 61	**S 61**	high	medium
Superfloc C 492 HMW	**C 492 HMW**	low	very high	Sokoflok 65	**S 65**	high	medium
Sokoflok 55	**S 55**	medium	medium	Sokoflok 68	**S 68**	high	medium
Sokoflok 56	**S 56**	medium	medium	Sokoflok 62	**S 62**	very high	medium
Sokoflok 58	**S 58**	medium	medium	Sokoflok53:Sokoflok54 in 4:1 ratio	**S 53 : S 54**	low	medium

Dewatering assessment method

We quantitatively evaluated the dewatering capacity of digestate using the method CST (capillary suction time) in line with Standard ČSN EN 14701-1 (75 8061). For measurements, we used a Whatman 17 chromatography paper, thickness 0.92 mm, with the flow rate of 190 mm/30min. The surface area of the CST cell bottom was 6.36 cm^2. Considering the fact that it was not possible to obtain the data of the time of capillary suction of the input digestate sample (even after 7200 s there was no capture of the sample on the filter paper for the first closing contact), the method was replaced by a dewatering screener test, which is also used under the plant conditions. The principle of the test lies in measuring the mass variations in the filtrate of the sample in time intervals ranged 5-60 s during free passage through a uniform screen under constant ambient conditions (sample mass, screen surface area/mesh size, atmospheric pressure, temperature). Such data resulted in a dewatering curve, which is subsequently compared with the input and flocculated samples. This way, we may assess which flocculant appears as the most efficient for the given suspension in case that the standard CST test cannot be carried out. During the screener test, we used a Santorius 510 analytical high-speed balance (d=0.1 g), a timer, a 500-ml-measuring cylinder, 500-ml-beakers, a screen (150mm in diameter and 0.2mm mesh size), and a funnel.

Optimum technological procedure

The working volume of digestate for the experiments was 100 ml. Assessing the results of the course of flocculation, we subjectively evaluated the formation/disintegration of the floccules. First, the flocculation was implemented so that it simulated the plant conditions, i.e. the flocculant (0.1% solution) was batched and the whole volume was decanted to fix the flocculation (from one beaker to another and back). The suspension was decanted 30 times.

With regard to their specific properties and to minimise the consumption of the polymer, we simulated various conditions for the tested flocculants, under which the polymer was mixed into the mixture. Considering the speed of the undergoing adsorption reactions during the initial experiment ($t_{flocc.}$=<60s), it was vital to determine the optimum mixing rate and the number of dose stages (to ensure the most suitable conditions for flocculation). We experimentally carried out a modified flocculation Jar test of the rapid dispersion of polymer organic flocculant (POF) solution in the suspension at high revolutions. We tested diverse mixing rates in dependence on time – 500, 600, 700, 800, 900 and 1000 rev/min and mixing time of 10, 20, 30 and 40 s in all the rates. This resulted in the determination of the optimum time variation in the mixing rate gradient to sustain the reflocculation capacities of the formed floccules, and we recalculated the flocculants doses required for the point.

Finally, we selected the most suitable flocculants in terms of their consumption and floccule stability. These were used for the verification experiments, while we assessed the dewatering capacity of the suspension using the above described dewatering tests as well.

Based on the final dewatering curves, we used the most efficient flocculant for the mechanical dewatering of 7 litres of digestate using an AKMR 300/4-OO-20 laboratory filter press at working pressure of 0.7 MPa. To be able to clearly compare the obtained results, the consumption of flocculants was recalculated as kg of total solids (TS).

Analysis of the separation products

The products obtained from the separation of digestate solid phase were subjected to analyses. The filter cake was used to determine the values of total solids and combustibles in accordance with EN 12879, the values of total nitrogen, phosphorus as P_2O_5, potassium as K_2O in g/kg of TS, and the C:N ratio in line with Czech Standard Operating Procedures of ÚKZÚZ (Central Control and Testing Institute of Agriculture), which were used to carry out the analyses of samples in an accredited laboratory, Laboratory Moravia, Ltd.

In the separated liquid phase (filtrate), we determined the indicators of chemical oxygen demand using the potassium dichromate method (COD_{Cr}) according to ISO 6060, biochemical oxygen demand (BOD_5) according to EN 1899-1, ammonia nitrogen according to ISO 7150-1, total phosphorus by Czech Standard Operating Procedures of ÚKZÚZ, as well as the elements Cl, Mn, Zn, Fe and the values, such as pH, suspended solids ($SS_{105°C}$), dissolved substances ($DS_{105°C}$), dissolved inorganic salts ($DIS_{550°C}$). The analysis of the liquid phase was carried out in the laboratory of the Water Technology and Water Management Department of the Faculty of Mining and Geology at the VSB - Technical University of Ostrava.

Verification of the optimised procedure on several samples

We also carried out and verified the effectiveness of the flocculant dosing procedure when changing the rate gradient as well as we used a digestate sample from a different biogas plant, i.e. a sample with different feed material parameters as pH value, total solids, loss on ignition, or the amount of organic matter.

RESULTS AND DISCUSSION

The digestate sample used for the experiments (locality of Stonava) had the following values: pH value – 8.1; total solids – 3.3%, loss on ignition – 73%. First, we tested decanting for flocculation. To form larger and more compact floccules under constant conditions of polymer dispersion in the suspension (decanting), the doses of all used cationic flocculants ranged from 51 to 66 g/kg of digestate TS (see Fig. 1). It may be stated that for such batching and mixing of the suspension to ensure flocculation, the doses of all tested flocculants were enormously high and unrealistic for the plant conditions in terms of economy. However, it was useful to help to identify suitable flocculants. Using flocculants from medium to high charge density (optimally 60-80% in mole) of predominantly medium molecular weight, large floccules formed, which were not stable or resistant to the action of shear forces. During the experiment (decanting), the floccules destabilised and disintegrated. On the contrary, applying medium molecular weight flocculants with low charge density (10-35% in mole), rather smaller and more frequent floccules formed, which were stable throughout the experiment, which was more advantageous for the subsequent mechanical dewatering.

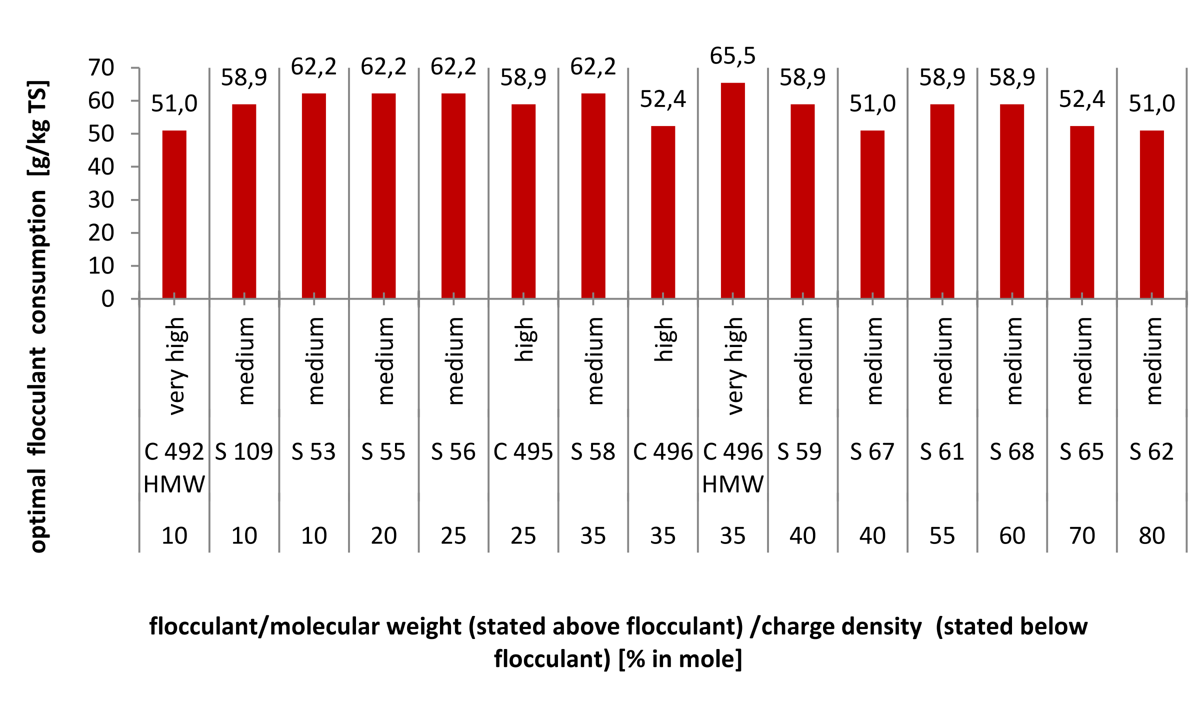

Fig.1: Superfloc/Sokoflok flocculants – optimal consumption to form compact floccules; single-stage dosing of flocculant; mixing via decanting

Testing the combination of fast-slow mixing and two-stage dosing of flocculant

Based on the results from Fig. 1, the further experiments focused on flocculants of a lower charge density because of the apparent positive results concerning the floccule formation. Therefore, we used 2 already tested flocculants (C492 HMW and S 53) and added 6 more flocculants of low charge density (C 492, S 53 CN, S 53 GP, S 54, S 54 CN, S 54 GP). Out of interest, we also produced a mixture of two selected flocculants, namely S 53 and S 54 in 4:1 ratio. The obtained results of flocculant consumption are in Fig. 2.

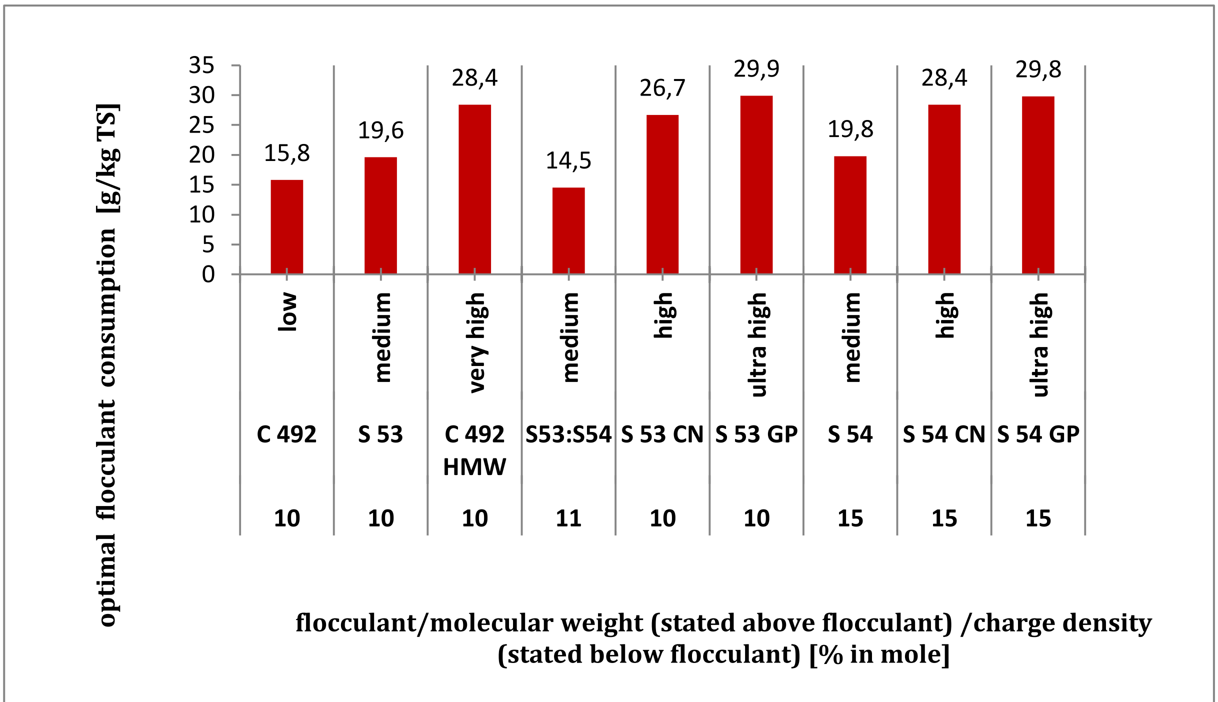

Fig. 2: Superfloc/Sokoflok flocculants - optimal consumption to form compact floccules; two-stage dosing of flocculant at different mixing time and intensity

For better distribution of flocculants in the volume of the suspension, the most suitable option concerning the 9 flocculants (Fig. 2) was identified as 30-second mixing at 1 000 rev/min for the first partial application of POF. This concerned the dose required for the first flocculation phase, when we perceived the formation of first floccules; at the same time, the effect of bridging among the floccules was still minimal. This dose of used flocculants ranged from 6 to 10 g/kg TS and the mixing time of 30 s at 1 000 rev/min permitted optimal dispersion of the polymer in the suspension. For the second dose of POF, the lower mixing rate of 500 rev/min was more suitable. The time needed for visible flocculation was $t < 5$ s. To minimise shear forces after circa10 s of mixing at 500 rev/min, we had to reduce the revolutions to minimum, otherwise partial destabilisation/ disintegration of floccules occurred.

The total specific dose of polymer in the two-stage dosing ranged from 14.5 to 29.9 g/kg TS of digestate and we arrived at the conclusion that flocculants of lower molecular weight manifested better results under optimal conditions of the modified Jar test. The effect of high molecular weight of flocculants C 492 HMW, S53 CN, S53 GP, S54 CN and S54GP manifested in higher doses and observable higher viscosity of the separated liquid phase when compared with the flocculants of low to medium molecular weight. In flocculants C 492, S53 and S54, we also tested diverse mutual proportions. We had positive results as for the consumption of flocculants in applying the mixture S53 and S54 in 4:1 ratio. In this case the minimum required specific dose in the first stage (1000 rev/min, 30 s) was approximately 6.1 g/kg and later in the second stage (500 rev/min, 5-10 s) it was 8.44 g/kg TS of digestate. In C 492, the minimum required dosing was 7 g/kg (first stage) and 8.8 g/kg TS of digestate (second stage).

The subsequently carried out test of dewatering capacity of the digestate sample using the screener test method (Fig.3) shows that the mixture of flocculants Sokoflok 53 and Sokoflok 54 (4:1) renders a higher amount of filtrate for the dose of 14.5 g/kg TS of digestate than the Superfloc C 492 dose of 15 g/kg TS of digestate. Therefore, the first mentioned dose was selected to dewater 7 l of digestate using a AKMR 300/4-OO-20 laboratory filtration press while adhering to the identical technological procedure. In this case the minimum effective dose was even reduced to 13.2 g/kg TS of digestate. The final products of separation were subjected to chemical analyses.

In the separated liquid phase (filtrate from the sludge press), we determined the following indicators: COD_{Cr} 8 400 mg/l, BOD_5 932 mg/l, $SS_{105°C}$ 1 200 mg/l, $DS_{105°C}$ 7 520 mg/l, $DIS_{550°C}$ 3 070 mg/l, pH 8.32, $N\text{-}NH_4$ 1 140 mg/l, P_{total} 46 mg/l, Cl^- 540 mg/l, Mn 2.05 mg/l, Zn 4.2 mg/l, and Fe_{total}15.1 mg/l.

In the solid phase (filter cake), we determined total solids – 15.6%, combustibles – 76.7 % in TS, potassium as K_2O 21.1 g/kg in TS, phosphorus as P_2O_5 37.6 g/kg in TS, C:N ratio – 8, and N_{total} – 4.76 g/kg in TS.

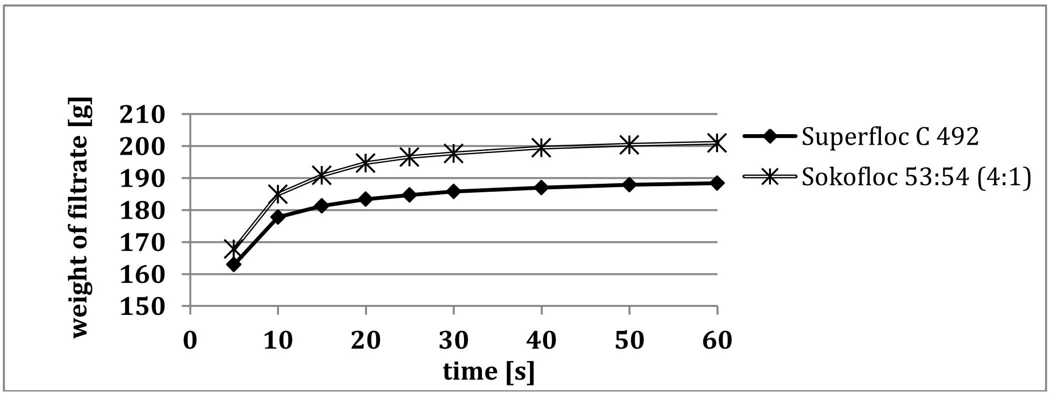

Fig. 3: Flocculation dewatering tests using Superfloc C 492/Sokoflok 53 and Sokoflok 54 mixture in 4:1 ratio

On the grounds of the obtained experimental results with the BGP Stonava digestate sample, we applied the most effective flocculation procedure on a digestate sample from the biogas plant Vrahovice, which manifested different input parameters. We used a mixture of flocculants Sokoflok 53:54 in 4:1 ratio, two-stage dosing, in the first stage for mixing at 1000 rev/min for 30 s, and in the second-stage the mixing rate was 500 rev/min for 5 s, later reduced to minimum, i.e. 50 rev/min for 30 minutes. The input parameters of the digestate sample were as follows: pH value – 8.5, TS – 6.9 % and LOI (or the amount of organic matter) – 64%. In this case, the flocculation was successful even at lower consumption of flocculant related to TS of digestate than in the case of digestate from BGP Stonava, i.e. in the first stage, it was approximately 2.4 g/kg, and later in the second stage, it was 9.4 g/kg TS of digestate. Based on the analyses of the separated components of the dewatered digestate, we may conclude that already for the discussed dosing, the final mixtures of flocculants rendered changes in the representation of some indicators in the filtrate after sample dewatering when compared to digestate dewatering without the use of flocculant (Heviánková et.al., 2014), when the liquid phase remains more polluted when related to COD.

Limitations

The stated results were obtained using digestate samples from two different biogas plants processing agricultural wastes, namely cattle's liquid manure, silage maize and haylage, and operating in the mesophilic regime. We expect that in biogas plants which process different products, e.g. slaughterhouse wastes, bone meal or household waste, the resulting digestate may differ reflecting the differences in the feedstock composition. This, however, leaves space for future research.

CONCLUSIONS

When testing the flocculation of digestate using only organic flocculants, we obtained valuable results in flocculation during two-stage flocculant dosing at different mixing time and intensity. In total, we tested 21 flocculants as well as 1 mixture of two selected flocculants which appeared most suitable for their consumption and efficiency during the flocculation. The obtained results imply that it is practicable to use flocculants of low charge density and medium to high molecular weight. The best results were reached using the mixture of flocculants Sokoflok 53: Sokoflok 54 in 4:1 ratio. These cationic polyacrylamid flocculants belong to a single range as for the length of their molecular chain or molecular weight (approximately $3.5\text{-}4.5\ .10^6$) and charge density. Both were prepared separately as 0.1% solutions, and mixed subsequently. In the digestate from BGP Stonava, the dose required for effective flocculation and mechanical dewatering was 14.54 g/kg TS and in the digestate from BGP Vrahovice it was 11.8 g/kg TS. Using the dose of 14.54 g/kg TS of the S53:S54 mixture, the level of the pollution during digestate dewatering dropped by 66 %; in case of comparing $N\text{-}NH_4$ and P_{total} in the liquid phase, there is a substantial decrease of 55 % ($N\text{-}NH_4$) and 77 % (P_{total}) when using flocculants in the digestate dewatering. Considering the fact that the best results were obtained using a mixture of two analogous flocculants, there is a possible path for future research in testing various mixtures of flocculants.

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