

Measurement of Rheological Properties of Biogas Digester Content Using a Rotational Rheometer with a Ball Measuring System

Lieselotte Van Looveren, Nils Engler

The rheological characterization of digester content from biogas plants presents challenges due to its heterogeneous composition, particularly in systems processing high-fiber substrates. Understanding these properties is crucial for optimizing key operational processes such as pumping, stirring, and heat exchange, thereby improving overall plant efficiency.

This manuscript presents a novel method for measuring the rheological properties of digester content without the need to first separate solid fractions. The method uses a rotational rheometer with a ball measuring system (BMS). Compared to conventional rheometric methods, this approach allows a more representative analysis by preserving its actual composition.

Furthermore, the applicability of the proposed method was demonstrated using digester contents from a full-scale agricultural biogas plant. Samples were collected from the main digester (MF), post-digester (PF), and the digestate storage (DS). Results revealed a significant reduction in apparent viscosity (consistency factor, K) from approximately $250 \text{ Pa} \times \text{s}$ in the MF to $10 \text{ Pa} \times \text{s}$ in the DS, attributed to microbial degradation and ongoing disintegration processes. In contrast, the flow index (n) remained relatively stable, exhibiting only a slight increase from the MF to the FPS, with average values ranging between 0.250 and 0.300. The slight increase in the flow index suggests that the digestate exhibits reduced viscosity at higher shear rates, such as during mixing.

Despite its advantages, the BMS method has some limitations, including potential sedimentation effects, particle-sphere interactions, and temperature sensitivity. However, averaging multiple flow curves improves measurement consistency and provides accurate data on the consistency factor (K) and flow index (n) of analyzed digestate.

Overall, these findings affirm the BMS-based rheometer as a reliable tool for routine rheological assessments in biogas research.

Keywords

Rheology, Digestate, Rheometer, Ball Measuring System (BMS), Biogas

Measuring the rheological properties of digester contents from biogas plants is a major challenge. For clarity, this manuscript uses the term “digester content” to refer to the contents of all stages of the fermentation process, from the main digester, the post-digester and the digestate storage.

Characterization of the rheological properties can help to improve overall process efficiency of biogas plants. Additionally, rheological monitoring of digester content offers valuable insights into the impact of additives and disintegration techniques on process performance (GIENAU et al. 2018). Precise knowledge of the rheological properties is essential for the optimization of stirring systems

and agitation processes in anaerobic digestion. Agitators account for a significant portion of the total power consumption in biogas plants. A study conducted at a typical agricultural biogas plant (NAEGELE et al. 2012) revealed that the stirring technology accounts for 51.6% of the plant's total electricity consumption. Even though this study only considers a single case, it demonstrates the importance of rheological properties for the energy optimization of biogas plants. Non-Newtonian properties also play a role here, as the start-up power of the agitators is usually significantly higher than the power consumption in stationary agitation mode due to the shear-thinning properties of the digester content (SCHNEIDER and GERBER 2020). Effective mixing is essential for ensuring uniform distribution of heat and nutrients within the digester, as well as facilitating microbial access to degradable substrate, even distribution of microorganisms and overall reduction of gradients in the fluid phase. Additionally, proper mixing helps to prevent sedimentation and floating layer formation, while also promoting the release of biogas from the digester content. Furthermore, stirring inhibits foam formation, thereby reducing operational problems such as clogged gas pipes and reduced biogas production (SINGH et al. 2019).

At the German Biomass Research Centre (DBFZ), a new method for measuring the rheological properties of digester contents has been developed which (i) takes non-Newtonian properties into account, (ii) provides quantitative measurement results, and (iii) does not require the prior separation of particles or fibers. The method was applied in a biogas plant that mainly processes cow and horse manure, substrates that are characterized by high particle loading due to their high straw content.

State of the Art

Digester content from anaerobic digestion is a complex three-phase mixture of liquid, granular or fibrous solids, and entrapped gas bubbles. These materials typically exhibit non-Newtonian, shear-thinning flow behavior. In such fluids, viscosity is dependent on the shear rate (and temperature). The rheological properties can be described by a flow curve, describing the apparent viscosity as a function of shear rate. Simple methods that only allow a qualitative description of the flow behavior cannot depict these complex relationships.

A widely used rheometer to determine flow behavior of fluids is the rotational rheometer. It consists of two main components: a stator and a rotor. The test sample is placed in the narrow gap (typically ≈ 2 mm) between these two components, and the viscosity is determined by applying a controlled shear rate via rotational motion. Due to the narrow measuring gap, it is necessary to separate larger particles or fibers before measurement, which is the case with the vast majority of digester samples.

A specific variant of rotational rheometers is the mixing rheometer, composed of a rotating stirrer (rotor) and a stationary vessel (stator). Among these, the rotating vane rheometer is widely adopted due to its ability to minimize wall-slip effects (BARNES and NGUYEN 2001). However, its applicability is limited for samples containing fibrous solids, which can wrap around the stirrer and disrupt measurements. These devices require measurement times of 10 minutes or more to measure complete flow curves over a wide shear rate range. This can be problematic for digester samples with a strong tendency to sediment.

The systems described above are frequently used in practice and have proven their suitability in many applications. Nevertheless, there are some limitations, such as in the case of very uneven grain size distribution or when long fibers are present in the fermenter broth.

Pipe viscometers are also frequently employed in agricultural biogas plants to determine the rheological behavior of digester content (MÖNCH-TEGEDER et al. 2015). A pump drives the fluid through a pipe with known dimensions. By maintaining laminar, steady-state flow, the pressure differential across the pipe is measured as the sample fluid passes through. If the pipe diameter is large enough, it is not necessary to separate particles or fibers beforehand. However, a significant disadvantage of this method is that it requires large quantities of sample volume (approximately 100 Liters or more), as well as ample physical space, making it unsuitable for lab-scale experiments (SCHNEIDER and GERBER 2020).

Rotational viscosimeters with a ball measurement system (BMS) have been shown to be effective for characterizing the rheological properties of construction materials such as plaster and cement (BEITZEL 2014). However, several factors need to be considered, including the potential sedimentation of particles during the measurement process, which may affect the accuracy of the results. Additionally, the measuring sphere can interact with particles and fibers, potentially dragging them and causing fluctuations or deviations in the measured data. Due to these limitations some studies advice against using the BMS for digester content flow behavior (SCHNEIDER and GERBER 2020).

Despite these objections, the BMS has a number of advantages. The advantages of this system are particularly evident when measuring digester samples with very high viscosities and high particle loads. In addition, BMS devices require comparatively small sample volumes (approx. 0.5 liters), making them particularly suitable for routine measurements from laboratory-scale biogas reactors. Finally, the measurement time is comparatively short (usually less than 3 minutes), which reduces the effects of sedimentation in many types of samples. Using a BMS and the method presented in this manuscript, the influence of particles and fibers on the measurement result can be minimized, and the measurement of real digester samples is possible without any sample pretreatment.

Material and methods

Measurement principle and equipment

A modular compact rheometer type MCR 92 (Anton Paar, Austria), equipped with a ball measurement system type BM 15 was used in this study.

The principle of using a ball measurement system is shown in Figure 1. A sample volume of approximately 0.5 L is required, which is more than is usual for rotational rheometers, but far less than for pipe viscometers. Due to the comparatively small sample volume, the method is particularly suitable for examining samples from laboratory digesters, from which larger sample volumes cannot usually be taken. The sample is kept at a controlled temperature using a double-walled heating jacket. The samples are measured at their respective operating temperatures. For digester samples, this is typically around 39 °C. The temperature significantly affects the flow behavior of fluids and therefore is closely monitored by a temperature sensor.

A sphere ("ball") is drawn through the sample via a rotating arm with a radius r . The angular velocity, and therefore the shear rate, is increased logarithmically in 40 steps as the sphere makes a full rotation of 2π or 360 °. Limiting the measurement to a single rotation is important to prevent the ball from entering areas of the liquid that it has already passed through. This process forms a characteristic trail in the fluid, particularly with highly viscous samples.

The measurement is performed in controlled shear rate (CSR) mode i.e. the rheometer controls the shear rate as a function of the angular velocity of the sphere, and measures the torque to maintain the respective velocity. Ten data points are measured by the rheometer during each of the 40 single shear rates and the mean is calculated. The output is a flow curve of 40 datasets showing the shear rate and the corresponding torque. Assuming a creeping (stokes) flow with a Reynolds number $Re < 1$, the apparent viscosity can be calculated from the torque and the geometry of the BMS for each shear rate.

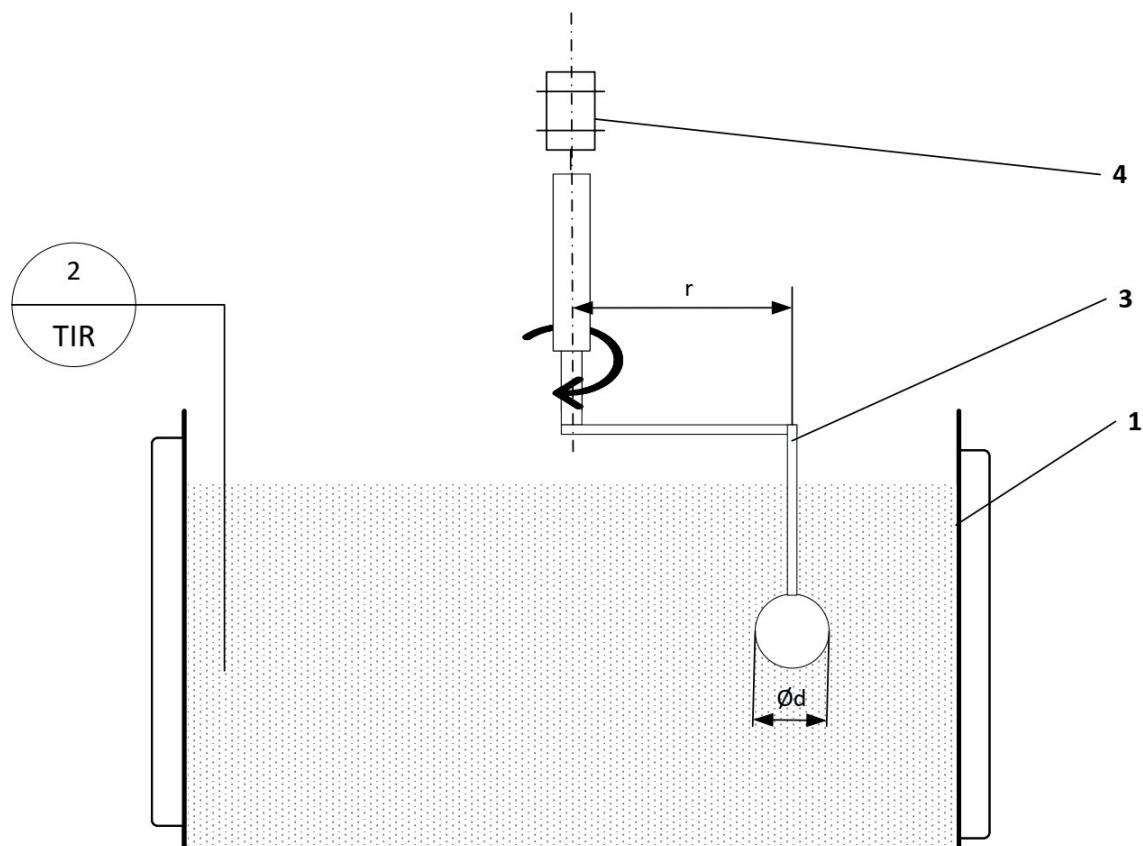


Figure 1: Scheme of the Rheometer MCR 92 with ball measuring system; 1. Sample cup with double jacket, 2. Temperature sensor, 3. Rotating arm with ball measuring system BM 15, 4. Motor

The viscosity of Newtonian fluids remains constant at different shear rates, which is defined in the linear relationship characterized by a flow index of one ($n = 1$) (Eq. 1):

$$\tau = \eta \cdot \dot{\gamma} \text{ [Pa]} \quad (\text{Eq. 1})$$

$$\eta = K \text{ [Pa} \times \text{s]} \quad (\text{Eq. 2})$$

Digester fluids are known to be non-Newtonian fluids that exhibit shear-thinning behavior (BAUDEZ et al. 2011), their rheological properties can be described by a flow function. The Ostwald de Waele model is a commonly used flow function, its advantage is its simplicity as it describes the non-Newtonian

nian behavior using merely two parameters (Eq. 3) and has proven its applicability for biogas digester fluids (BAUDEZ et al. 2011, 2013).

$$\eta(\dot{\gamma}) = K \cdot \dot{\gamma}^{n-1} \quad (\text{Eq. 3})$$

With:

η' = Apparent dynamic viscosity in Pa \times s

K = Consistency factor in Pa \times s

n = Flow index

$\dot{\gamma}$ = Shear rate in s^{-1}

The Consistency factor K characterizes the dynamic viscosity η at a shear rate of 1 s^{-1} , thus having the same dimensional units of $\text{Pa} \times \text{s}$. The non-Newtonian behavior of the fluid is quantified by the flow index n .

It is important to emphasize again that the system is limited to measurements within the creeping flow regime around the sphere, as defined by Stokes' law of friction. Specifically, this means the Reynolds Number must remain below one ($\text{Re} < 1$) (STOKES 2010).

Measurement procedure

Using the equipment described above, the following measurement procedure was developed to measure real digester samples.

It is assumed that sampling from full-scale digesters is carried out in accordance with good professional practice and in compliance with the applicable standards. A total sample volume of approximately 2.5 L is required to perform a complete measurement procedure. The time span between sampling and measurement should be as short as possible. Samples should be measured at the operating temperature of the digester from which they were taken. The temperature controller of the heated sample cup (Figure 1) has to be adjusted accordingly. If necessary, the sample should be tempered in advance. Then, the sample (approx. 500 mL) is filled into the sample cup up to a specified level. The fill level is the same for all samples and is checked using a device specifically designed for this purpose. Once the correct amount of sample has been filled in, the temperature sensor (pos. 2 in Figure 1) is mounted. The measurement starts when the specified temperature has been reached. The rheometer moves the measuring ball to the preset position and performs a logarithmically accelerated rotation of 360° as described above. Angular velocity and acceleration are adjusted for the method in such a way that a shear rate range of 0.05 to 100 s^{-1} , i.e. four decades, is covered during a full rotation. The required torque is measured and the shear stress and apparent viscosity are calculated as a function of the shear rate. Due to the collision of the sphere with particles or fibers, the flow curve obtained in this way shows more or less strong random fluctuations. During the development of the method, it became apparent that these random fluctuations can already be largely eliminated when calculating the mean value from four individual measurements. When calculating the mean value from more than four individual measurements, the variance did not become significantly smaller. Therefore, the procedure of measuring is repeated four times with each sample. For each measurement, new sample material is filled into the sample cup and the sphere is cleaned with water.

Figure 2 shows four flow curves for a sample collected from the main digester of an agricultural biogas plant. The varying intensity of the fluctuations in the individual curves reflects the sample's inhomogeneity due to its high fibre content.

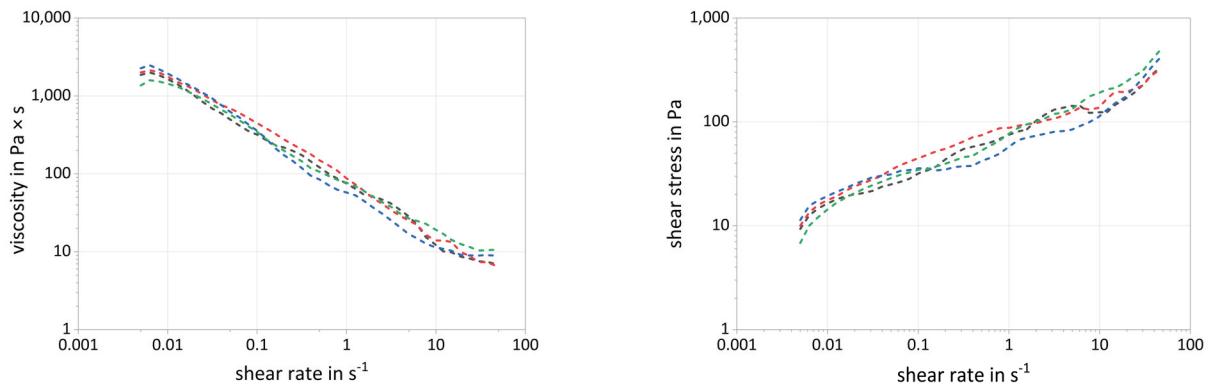


Figure 2: four individual flow curves measured from the same digester sample. Apparent viscosity vs. shear rate (left) and shear stress vs. shear rate (right). Random fluctuations due to particle disturbances

The averaged flow curves, calculated from the individual curves in Figure 2 are shown in Figure 3. It can be seen that the random fluctuations are largely eliminated by averaging.

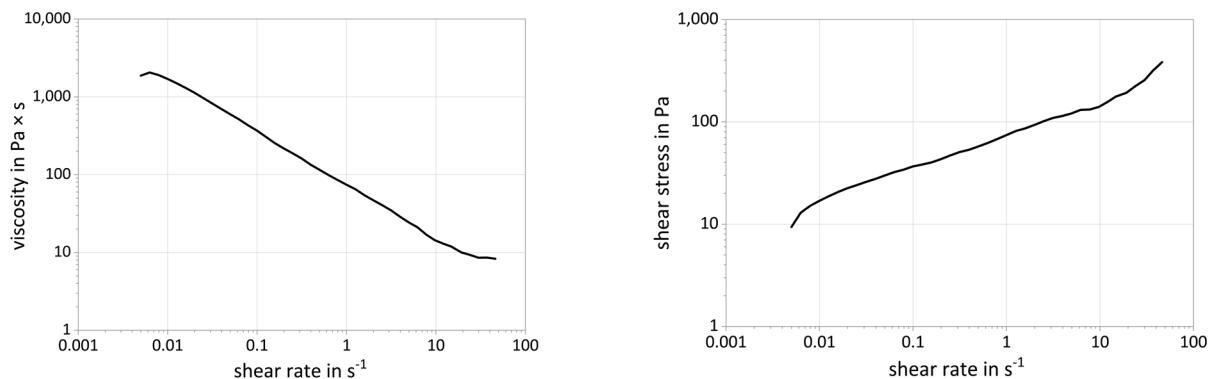


Figure 3: averaged flow curve from the same four single measurements shown in figure 2. Random fluctuations are mostly eliminated

Data processing

The subsequent data processing is based on the average flow curves determined as described above. In a first step, it is checked whether the boundary condition of a creeping flow is fulfilled over the entire measured shear rate range. For this purpose, the Reynolds number Re is calculated iteratively using the apparent viscosity calculated for each shear rate. Especially for samples with low viscosity, it can happen that this boundary condition is not fulfilled at higher shear rates. Data points where $Re > 1$ are excluded from further evaluation. The parameters K and n of the Ostwald de Waele flow function (showed in equation 3) are calculated from the flow curve generated in this way using linear regression. As part of quality management, it was defined that the measurement is considered valid if the evaluable shear velocity range still covers at least two decades after eliminating invalid data points and the coefficient of determination is higher than 0.97.

The data processing described above is carried out in an automated process based on an MS Excel[®] worksheet. A results report is automatically generated from this worksheet. This report contains, besides details about the sample and the date of measurement, the calculated parameters K and n , the coefficient of determination of linear regression, the standard deviation of K and n across the four

measured flow curves and the evaluable shear rate range. In addition, the report displays the measured data points and the obtained regression in graphical form. An example of a standard report is shown in Figure 4.

Results Report

Rheometer Paar MCR 92 BM 15

1. General Measurement Data

Date:	2024-09-19
Sample-ID	BK-24-1843
Sample Description:	Main digester sample, taken 2024-09-16
Measuring temperature:	39.0 °C
Operator:	LVL

2. Results

evaluable shear rate range

from	0.0315 s^{-1}
to	23.5 s^{-1}

Results of Regression (Flow function according to Ostwald-de Waele)

Consistency factor K	215.4 $\text{Pa}\times\text{s}$
Flow index n	0.245

Coefficient of determination R^2	0.987
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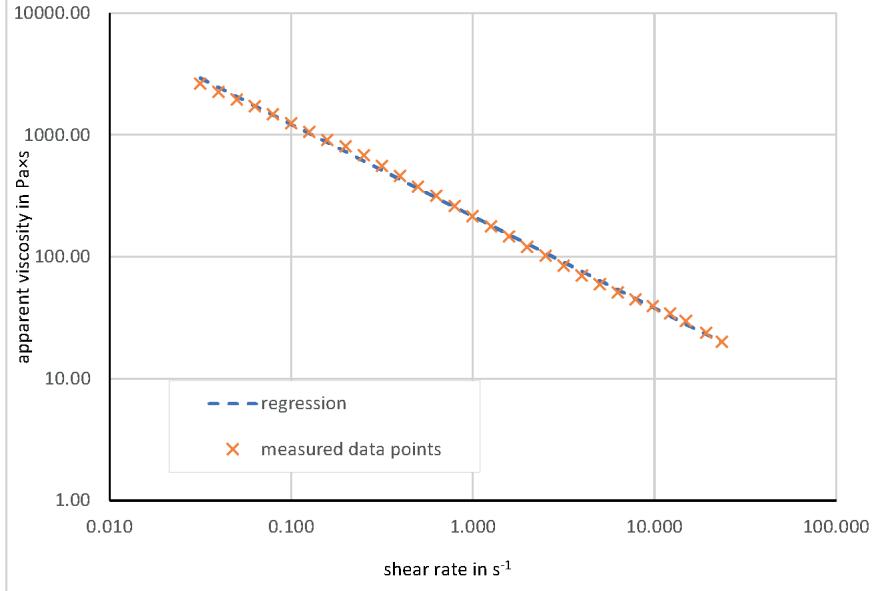


Figure 4: Report - Result sheet of rheological characterization of digester content from the Main Digester

Method Validation

Due to the unavailability of standard references for non-Newtonian fluids, certified Newtonian viscosity reference standards are used in order to validate the method and to calibrate the Rheometer. Dynamic viscosity η and density ρ of the fluids are certified as a function of temperature. When applying the Ostwald de Waele model to Newtonian fluids, the flow index is one ($n = 1$), and thus the consistency factor K corresponds to the dynamic viscosity η (Eq. 2). A calibration of the measuring system is therefore possible even with Newtonian calibration fluids.

The calibration measurements with five different standards were conducted at temperatures ranging from 25 to 40 °C. The standard method for measuring and data analysis was applied as described above and the consistency factor K was calculated. The results from 18 measurements are presented in Figure 5. There is a clear linear correlation between the measured K and the certified viscosity η . The calibration factor was determined by linear regression from the data points of the 18 individual measurements.

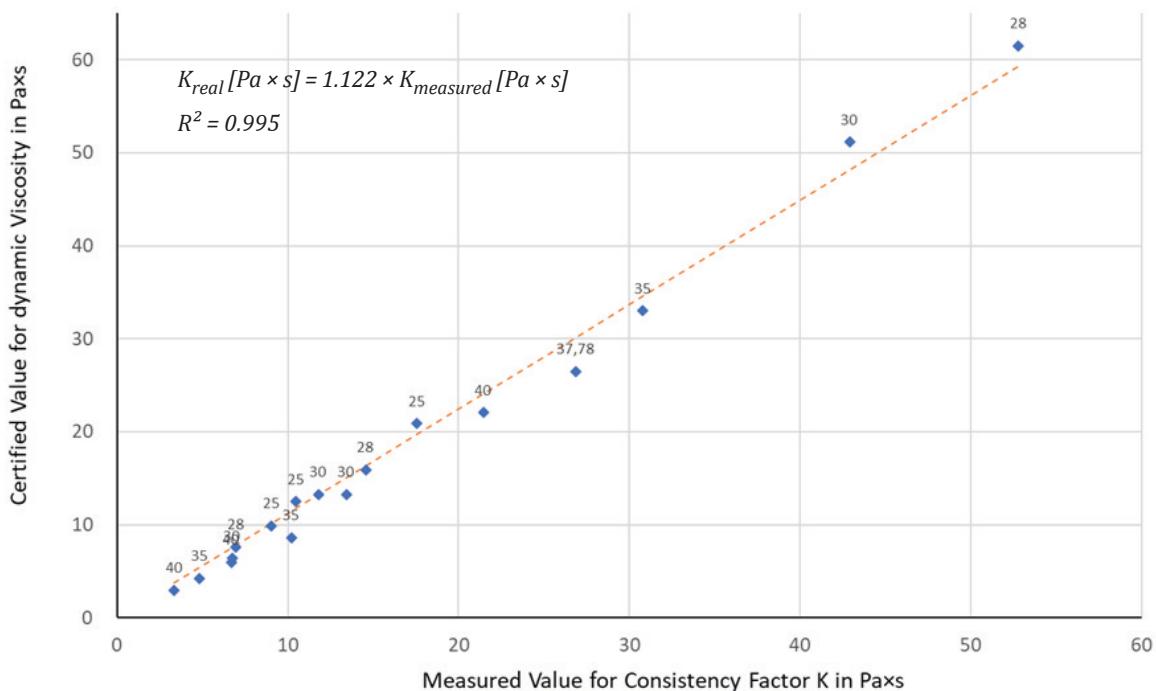


Figure 5: Calibration curve for Rheometer MCR 92 with BM 15: measured with five different viscosity reference standards at different temperatures ranging from 25 to 40 °C; Temperatures are indicated above each measure point

The calibration factor is taken into account in the data processing procedure described above. As part of quality management, it has been decided that validation must be repeated every six months, at the latest after 100 measurements, with at least five calibration points. The calibration factor must be redetermined each time and stored in the data evaluation.

Application of the method

The developed method was applied to study changes in the rheological properties of the digester contents of an agricultural biogas plant. The plant consists of a main digester (MD), a post digester (PD), and a digestate storage (DS), each with a capacity of approximately 2,000 m³. The biogas plant primarily processes agricultural biomass, with a daily input ranging from 50 to 100 tons, approximately 80% of which consists of solid manure from cattle and horses. In order to cope with the high dry matter content of the input, the operator regularly exchanges digester content between the PD and the MD.

To assess the rheological properties of the individual stages of fermentation, samples from all three stages (MD, PD and DS) were collected regularly. Figure 6 shows the results obtained for the consistency factor K of 18 measurements at samples from each of the three stages (MD, PD and DS, taken over a period of 36 weeks. A significant decrease in viscosity was observed during the anaerobic digestion/disintegration processes from approximately $250 \text{ Pa} \times \text{s}$ in the MD to $10 \text{ Pa} \times \text{s}$ in the DS. The viscosity in the MD and PD were sometimes similar, particularly during periods when the digester content was transferred over in the other digester to provide a better consistency, e.g. for stirring, as mentioned above.

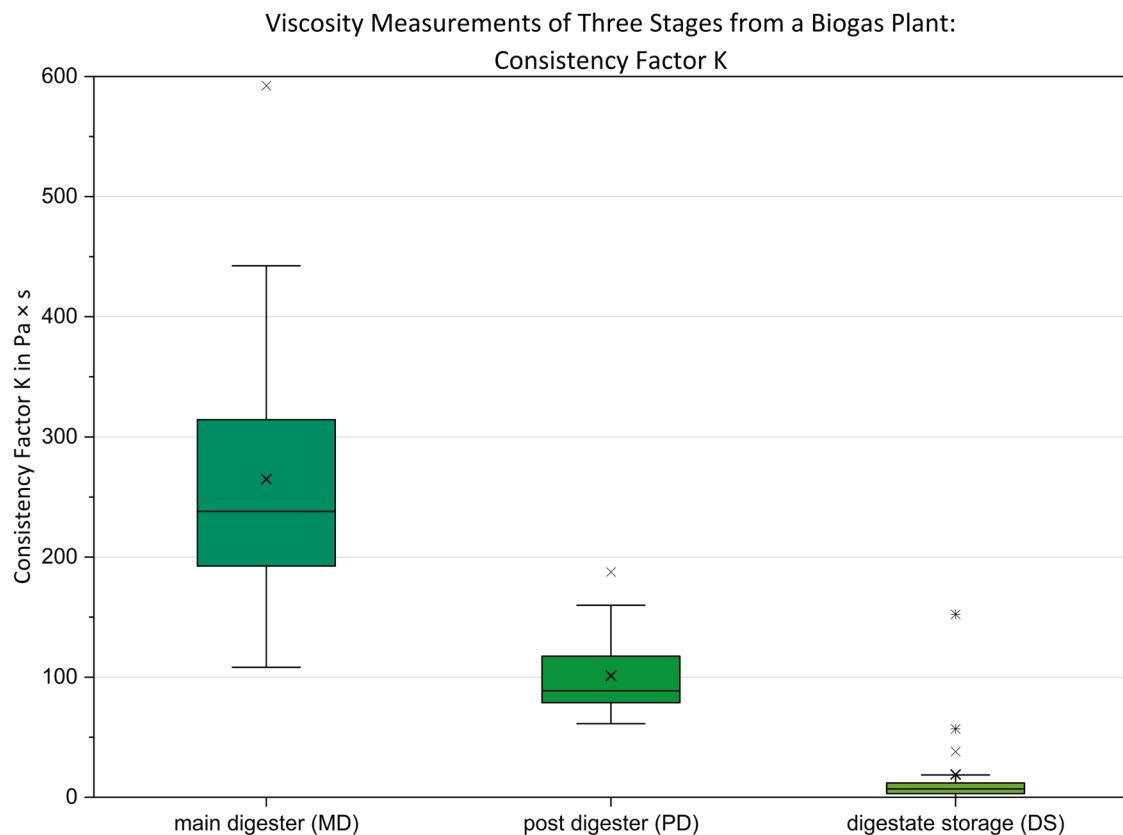


Figure 6: Boxplot of the Consistency Factor (K) measured on samples collected every two weeks across 18 sampling dates from each of the three stages (MD, PD, and DS)

The flow index (n) remained relatively constant throughout the process. Average values ranged between 0.250 and 0.300 which are characteristic for shear-thinning fluids. A slight increase was observed throughout the process from the MD to the DS, as shown in Figure 7. This indicated that the viscosity decreases less significantly at higher shear rates.

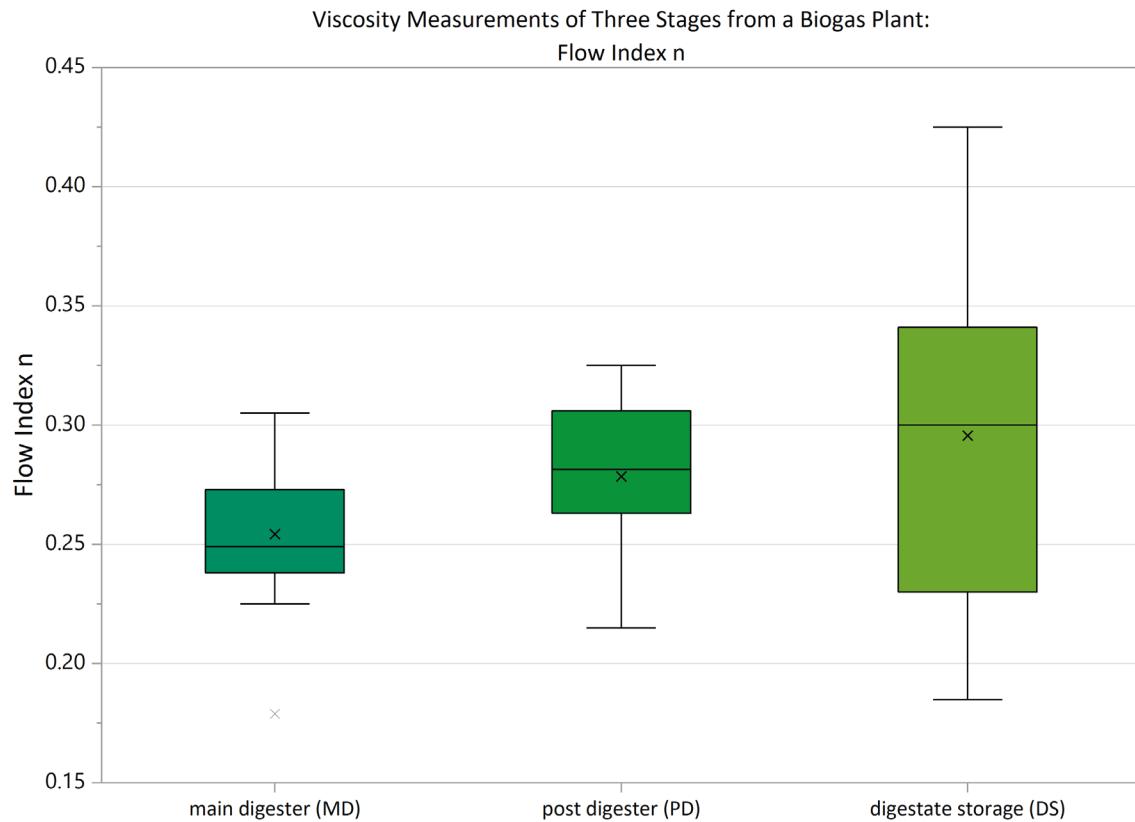


Figure 7: Boxplot of the Flow Index based on samples collected every two weeks across 18 sampling dates from each of the three stages (MD, PD, and DS)

The statistical significance of the differences of K was confirmed by a t-test, as shown in Table 1. As the p-value was smaller than 0.05, a significant difference between the two groups is to be assumed (KIM 2015). The consistency factor differs clearly for each digestion step in the biogas plant, whereas the differences were noticeably smaller for the flow index. In fact, no significant difference in flow index was observed between the post digester and the digestate storage.

Table 1: t-test results for K and n comparing the main digester (MD), post digester (PD), and digestate storage (DS). Values of $p < 0.05$ indicate significantly different groups

t-Test	p-value K in Pa \times s	p-value n
MD - PD	3.50E-05	0.02
PD - DS	4.60E-08	0.36
MD - DS	1.30E-07	0.03

The figures A1, A2 and A3 in the appendix show, based on measurements taken at three different sampling times, that the viscosities change during the three stages of fermentation, but that the overall difference remains the same. Furthermore, the figures demonstrate that the average of four individual curves results in a curve that is suitable for evaluation, even if there may be considerable fluctuations in individual measurements.

Future work/outlook

Despite the advantages of the BMS method, several limitations must be considered:

Particle-sphere interactions lead to fluctuations in recorded data. As shown above, these random fluctuations can be compensated for by taking multiple measurements and calculating the average value, but this is not the case for all samples. If the coefficient of determination of the regression falls below the specified threshold value of 0.97, a reliable measurement result cannot be given.

Another drawback is that the BMS is less suitable for low-viscosity samples, as the boundary conditions of a Stokes flow with $Re < 1$ cannot be maintained at low shear rates. The measurements are moreover temperature sensitive; therefore, proper temperature control of the sample is required before measuring.

The method is currently being applied in several research projects. In one study, the influence of enzyme addition on the viscosity of digester contents is investigated in laboratory-scale reactors. Another research project is studying the effect of ultrasonic treatment on the flow behavior of digester contents in a full-scale biogas plant. The results of these studies are planned to be published.

An important measure that still needs to be taken is an inter-laboratory comparison test, preferably with real fermenter samples and different measurement methods, in order to check the comparability and validity of the measurement results. Such a comparison would enable reliable conclusions to be drawn from the data obtained using this method.

Conclusions

The assessment of rheological properties in digester content from biogas plants presents significant challenges due to its complex composition, particularly when samples contain larger particles and/or fibers. This study demonstrated the applicability of a ball measuring system (BMS) used with a rotational rheometer to evaluate the rheological properties of digester content without requiring prior separation of fibers and larger particles. Compared to other rheometric methods, this approach allows for a more representative analysis of digester content, which inherently contains solid fractions that influence its flow behavior. The ability to measure the unaltered sample provides a more accurate reflection of the actual operational conditions within biogas plants. Moreover, due to the small sample volume required, the method (unlike, for example, tube rheometers) is suitable for measuring samples from laboratory reactors.

Regardless of digester content samples exhibiting non-Newtonian behavior, the system can be calibrated using commercially available Newtonian calibration fluids. Although this method does not allow for direct calibration of the flow index, it still provides valuable insights into the rheological properties of the samples.

The results of a first application of the developed method revealed a significant decrease in viscosity (Consistency Factor, K) throughout the anaerobic digestion process, with values dropping from approximately $250 \text{ Pa} \times \text{s}$ in the MF to $10 \text{ Pa} \times \text{s}$ in the DS. This reduction is attributed to the ongoing disintegration and degradation processes within the biogas plant. In contrast, the flow index (n) remains relatively constant throughout the process, with only a slight increase observed from the MD to the DS, with values ranging from 0.250 to 0.300. This suggests that while viscosity decreases, the shear-thinning behavior of the digester content remains stable with a slight increase detected, meaning the viscosity decreases less significantly with higher shear rates.

Overall, these findings enhance the understanding of digester content flow behavior and demonstrate the potential of the BMS-based rheometer as a reliable tool for routine rheological assessments in biogas research, both in industrial plants and laboratory-scale bioreactors.

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