INVESTIGATION OF BIOGAS DIGESTATE AS FIBER MATERIALS FOR COMPOSITES

*Marion Gebhardt**
Researcher
German Institutes of Textile and Fibre Research
Koerschtalsstraße 26, 73770 Denkendorf, Germany
E-mail: marion.luetz@uni-hohenheim.de

Andreas Lemmer
Researcher, Deputy Director
State Institute of Agricultural Engineering and Bioenergy
University of Hohenheim
Garbenstraße 9, 70599 Stuttgart, Germany
E-mail: andreas.lemmer@uni-hohenheim.de

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Abstract. Fiber reinforced plastics with synthetic fibers are widely used. Plant fibers are also known to produce more sustainable composites. However, there is a great interest in finding alternatives to classical natural fibers. The digestate of biogas plants seems to be such an alternative. Biogas plants are fed with plant-based substrates and during the digestion, the biomass is degraded. In this study, the fiber quality of digestates from four biogas plants with different initial substrates is investigated. Therefore, typical fiber properties, such as slenderness ratio, cell wall components, and the potential fiber performance, are measured. According to the general definition, the solid part of the digestate is a fiber material. The slenderness ratio is 5 or higher and the density is 1.5 gcm^{-3}, which is typical for natural fibers. Fibers with similar properties are already used in composite materials.

Keywords: Bio composites, waste material, natural fibers, biorefinery.

INTRODUCTION

Fiber-based composites with different reinforcing fibers are used in various industries. Fiber-reinforced composites typically consist of a reinforcing fiber component, which absorbs the forces, and a matrix, which gives the shape and protects the fibers from environmental influences. In general, fibers are defined as thin filamentary structures (Crowther 1995) that have a slenderness ratio (length/diameter) of at least 3:1 (Schenek 2001). It is possible to mix individual short (1-10 mm), long (smaller than 25 mm), or continuous fibers with the polymer matrix. Fibers are often processed into reinforcing textiles (eg nonwovens, woven, or tailored fabrics) (Schürmann 2007). Fibers for textile applications have a very large slenderness ratio of 1000:1 and more (Schenek 2001). The requirements for nonwoven technology are fibers with a length of up to 5-30 mm, which must not be too slender. Very short fibers are often processed as fillers in thermoplastics, for example, in wood plastic composites (WPC), where wood flour and wood fibers are common (Vogt 2006; Schürmann 2007). The scarcity of resources and the goal of reducing the high energy consumption in the production of many composites are forcing the industry to use alternatives. Natural fibers, especially plant fibers such as flax, are often used to produce sustainable composites. The density of plant fibers is typically approximately 1.5 gcm^{-3} (Schenek 2001) and their specific (density-related) properties are comparable to those of glass fibers (AVK 2013; Salit et al 2015). Plant fibers must be extracted from plants by biological, chemical, or sometimes, mechanical methods. This causes additional process steps and energy consumption (Gessner 1955; Ahmed and Akhter 2001). Plant fibers are grown on agricultural land that cannot be used for feed or food production. Various fiber

* Corresponding author

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crops are grown around the world. For example, 264152ha of flax and 82265ha of hemp were harvested in 2019 (FAO 2021). Fiber yield varies due to weather conditions and crop varieties (Riedel and Nickel 2000). In 2019, the global flax fiber yield was 4.2tha−1, whereas yield of hemp and sisal fibers was both 2.5tha−1 (FAO 2021). As a lignocellulosic material, plant fibers are mainly composed of the cell wall materials cellulose, lignin, and hemicellulose. To save land and energy, it is interesting to find other fiber sources. Fibrous residues are sometimes used in short fiber-reinforced plastics or filled plastics. An example of residue use is fibers from coconut production in nonwovens for composites (Sergion et al 2005; Bradley and Conroy 2019; Obeng et al 2020). Another example is fibers or wood flour from sawmill by-products of the wood industry in thermoplastic WPC and derived timber products (Sörgel et al 2006; Vogt 2006). Cellulose and lignin content and length of selected plant fibers and agricultural waste materials are shown in Table 1.

Digestate from biogas plants is mostly used as fertilizer. In Germany, industry is urged to use more sustainable raw materials to achieve the goal of a bioeconomy (BMBF 2020). Digestate can be a solution for fiber and composite production. Due to the lignocellulosic components, digestate can be an interesting raw material for industrial purposes. Essel et al (2015) mixed small amounts of purified and treated digestate into the production process of medium-density fiberboard. An addition of 20% digestate does not influence the mechanical properties, a higher addition leads to a decrease. In another study, digestate was added to various plastic products, such as films, to reduce weight and increase biodegradability (König and Fudel 2005).

Biogas technology is used worldwide to generate renewable energy through anaerobic microbial conversion of substrates fed to the digesters of these plants. Approximately, 132,000 technical-scale plants are currently in operation worldwide. In addition, the number of small-scale household biogas plants installed is estimated to be in the millions (Jain 2019). In Europe, there are currently approximately 17,000 biogas plants, of which 10,000 are located in Germany (Königsberger et al 2019). In Germany, the most commonly used substrates for biogas plants are manure from livestock with 12% of the substrates (dry mass 12%) and energy crops with 79% (dry mass 17-32%), with corn silage accounting for the largest share (Foreest 2012; Torrijos 2016; Daniel-Gromke et al 2017; Scarlat et al 2018). The anaerobic degradability of substrates and their conversion rate into biogas is mainly influenced by the proportion of cell wall components and their water content. In contrast to the cell contents, the lignocellulose complexes of the cell walls can only be degraded to a small extent under the anaerobic conditions of the biogas process and are found as fibrous materials in the aqueous suspension of the fermentation residues (digestate) together with the minerals

Table 1. Cellulose and lignin content of different plants related to dry mass from Fortea-Verdejo et al (2017), Garrote et al (1999), Ververis et al (2003), Obeng et al (2020), Bradley and Conroy (2019) Nielsen (2005), Tiefenthaller (2006).

Fibrous part of the plant or waste material	Cellulose (%)	Lignin (%)	Fiber length (mm)	Dry mass (%)	Dry mass after digestion (%)
Cotton	90	NN	12-64	—	—
Flax	70	2	3-4	—	—
Softwood	42	29	4	—	—
Hardwood	41	19	1	—	—
Coconut husk	—	41-46	—	—	—
Grass silage	34	9	—	—	—
Corn silage	21	7	—	32	11
Cattle slurry	15-25	7-9	—	8	6
Pig Slurry	10-23	4-10	—	6	4
Solid manure	13	17	—	25	15

(Schimpf 2014). Undegraded fiber materials from the digestate of biogas plants are potentially suitable for the production of composite materials. During anaerobic digestion, the biomass is partially degraded and the fibers are released, which corresponds to the typical biological extraction processes for fiber production. If the solids of the digestate are used for the production of fiber composites, the added value of biogas production can be increased. The biogas plant can be a kind of biorefinery because energy and industrial raw materials are produced. The fibers of the digestate are potentially very interesting raw materials for industrial use, as they can be produced in large quantities at low cost and with low energy input. So far, little is known about the quantity and quality of extractable fibers from digestate. The aim of this work is, therefore, to evaluate the influence of different initial substrates on fiber quantity and quality.

MATERIALS AND METHODS

Samples are taken from four economically operating biogas plants in southern Germany (hereafter referred to as A, B, C, and D). The samples were taken from the three process steps: the fermenter (F1), the secondary fermenter (F2), and the storage tank (ST). A questionnaire was used to obtain further information about the biogas plant and the quantities of substrates processed. For initial characterization, the extracted fibrous materials were analysed using the van Soest analysis (Van Soest and Robertson 1970) based on DIN EN ISO 13906 and 16472 (DIN 2006, 2008). Before analysis, the digestate was ground to powder with a cutting mill. The different cell wall components were determined in three steps. To obtain neutral detergent fiber (NDF), all components that do not belong to the cell wall were washed out with a so-called neutral detergent solution. For the so-called acid detergent fiber (ADF), the cell wall components, except lignin and cellulose, were removed by boiling for 1 h in acid detergent solution. To obtain the so-called acid detergent lignin (ADL), cellulose was removed by exposure to 72% sulfuric acid for 3 h. After all three steps, the samples were washed with hot water to remove the solutes and dried at 105°C. After performing the dissolution steps, the remaining material was burned at 500°C to obtain the ash content. The proportions of the solution fractions (NDF/ADF/ADL) were calculated using the mass of the dried material m_{dry}, the mass of the fresh material m_{fresh}, and the mass of the ash after burning mash with Eq (1).

$$NDF/ADL/ADL\% = ((m_{dry} – m_{ash})/m_{dryfresh}) * 100. \tag{1}$$

The exact content of cellulose, hemicellulose, and lignin were determined by subtracting the individual percentages (see Eqs 2-5). The amount of each component is given as a ratio of dry mass (Schuldt and Dinse 2010).

$$\text{soluble}\% = 100\% - \text{NDF}\%. \tag{2}$$

$$\text{hemicellulose}\% = \text{NDF}\% - \text{ADF}\%. \tag{3}$$

$$\text{cellulose}\% = \text{ADF}\% - \text{ADL}\%. \tag{4}$$

$$\text{lignin}\% = \text{ADL}\% - \text{ash}\%. \tag{5}$$

The dry mass content was determined according to DIN 12880 and DIN 12879. For the determination, a quantity of the respective fermentation residue was weighed and dried at 100°C until no further mass loss was detected and then weighed again (Pfeiffer and Thrän 2015). Eq (6) was used to calculate the dry mass content.

$$DM\% = \frac{m_{dry}}{m_{fresh}} * 100. \tag{6}$$

To avoid errors related to cavities in the fiber material, the dried digestate was compacted and formed into flat tablets of 10-mm diameter at a pressure of approximately 10 bar. The density measurement was carried out according to EN ISO 1183-2. Instead of a density column, a row of density mixtures with *n*-heptane (0.68 gcm^{-3}), carbon tetrachloride (1.59 gcm^{-3}), and 1,3 dibromopropane (1.99 gcm^{-3}) was prepared in different beakers. The fermentation residue pills were immersed in the solvent mixtures one after the other until a floating state was reached. At this point, both materials have the same density (DIN 2004).

Fiber length was determined for all digestates according to DIN 53808-1 (Saville 1999). First, a sample of each of the digestates was placed in a glass dish and scanned. On the resulting image, all the individual fibers of each sample were traced and measured using an image processing program (Rueden et al 2017). This way, approximately 1000 individual measurements were taken from each sample. In addition to the fiber length, the slenderness ratio is also an important parameter. To determine this ratio, the fiber diameter must first be determined. The shapes of the digestate are very irregular and do not have a uniform round cross section. To obtain an approximate value for the slenderness ratio, the width of the fibers was measured directly from the scans. This determination was carried out on 100 individual fibers of each sample. Using wet sieve analysis according to DIN 66165, the samples were allocated and classified according to their size (Fritsch, analysette3, Idar-Oberstein, Germany). In this way, the proportion of potentially processable fibers was determined. Sieving was performed with water and vibration (amplitude 2 mm) at cycles of 10 min.

The sieves had a mesh size of 2, 1, 0.5, 0.25, and 0.125 mm, and were stacked in descending order. Each sample was placed on the coarsest sieve and then sprinkled with water. The sample material remaining on the sieves was rinsed, filtered, and dried. To determine the proportions of each size class, the filtration residues are weighed (Schmidt et al 2003).

RESULTS AND DISCUSSION

Description of Biogas Plants and Used Substrates

All four biogas plants investigated are located in southern Germany. In biogas plant A, the gas is purified and fed into the public gas grid. In plants B, C, and D, the gas is used in a combined heat and power plant (CHP) to generate electricity. In all four selected biogas plants, mainly agricultural materials are used as substrates. The input substrates with the corresponding quantity shares of the investigated biogas plants are shown in Table 2. The collected samples and data represent for only 1 mo of the year. Plant A uses only plant substrates, with chopped hop vines making up the largest portion. Plant B uses cereal whole-plant silage and manure as main substrate. Plant C is also fed mainly with animal excrement and the second main component is grass silage. Plant D is mainly fed with animal excrement (liquid and solid manure) and maize silage. Plants B to D are representatives of typical German biogas plants as described (see Section 1) because around 70% of the biogas plants are fed with up to 50% excrements (Daniel-Gromke et al 2020). Plant A is an exception as it does not use animal excrement and only a small amount of energy crops. In addition to the feedstocks, data were also collected on the average gas production in the biogas plants. Taking into account the density of the biogas produced, the average amount of digestate formed was calculated using the difference between the feedstock and biogas produced. The monthly digestate formation varied between 5709 tm^{-3} (plant A) and 240 tm^{-3} (plant C). Biogas plants A, B, and D separated the materials from the ST into a solid and a liquid fraction. The investigated material was the solid fraction.

Dry Mass Content

Figure 1 shows the dry mass content of the samples studied. The dry mass content is in the same range for the three separated ST-digestates (ST-S) for plants A, B, and D. As in the material from plant C (ST-L), the liquid fraction is not separated, dry mass content is lower. The digestate from the plants with a separation of liquid and solid fractions has a dry mass content of more than 15%, whereas the material from plant C (without separation) has a dry mass content of 6% only. However, similar dry mass contents were found in all four plants in the process steps F1 and F2, as substrate as well as digestate were treated in the same way in all plants. SD is highest in the samples obtained from plant A. The dry mass content of the solid fraction of the digestate is in the same range as the initial substrates. This is caused by the separation because in general, the fermentation decreases the dry mass content (see Table 1)

Table 2. Relative shares of the input substrates, produced gas, and digestate in the month of sampling at the investigated biogas plants.

—	—	—	Biogas plants			
—	—	—	A	B	C	D
Substrate fresh mass [t/mo.]	—	—	7377	499	290	805
Substrates [%]	Plant silage	Maize silage	23	7	0,06	48
		Grass silage	5	19	43	4
		Whole plant silage	—	29	—	—
		hop vines silage	60	—	—	—
	Solid manure	Horse manure	—	—	19	13
		cattle manure (solid)	—	35	0,1	10
	—	Liquid manure	—	—	36	23
	Other plant material	Sugar beet	—	2	—	—
		Digestate	—	3	—	—
		Corn cob grist	11	—	—	—
		Grain	—	5	2	—
		Grain dust	—	—	—	0,7
Produced gas and digestate	—	Biogas [m^3/mo.]	1,263,322	53,465	37,657	137,962
	—	Digestate fresh mass [t/mo.]	5709	381	240	623

(Zethner et al 2002). The comparison between the separated and unseparated digestate shows that the separation increases the dry mass. For the yield of potential fibers, separation is a helpful process step. If only dry mass content is considered, fiber recovery from the initial substrate would be preferable. Under these circumstances, however, a combination of energy extraction and industrial raw material is not possible.

Cell Wall Components

Figure 2 shows the content of the cell wall components cellulose and lignin, related to the dry

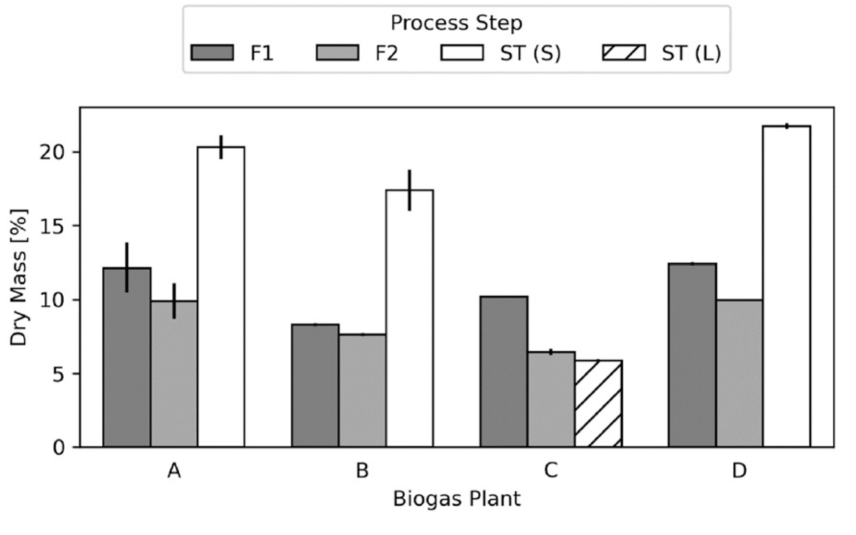

Figure 1. Dry mass content in % of the digestate samples from the investigated biogas plants A-D in the different process steps primary fermenter (F1), secondary fermenter (F2), and storage tank (ST), separated (S) and not separated (L).

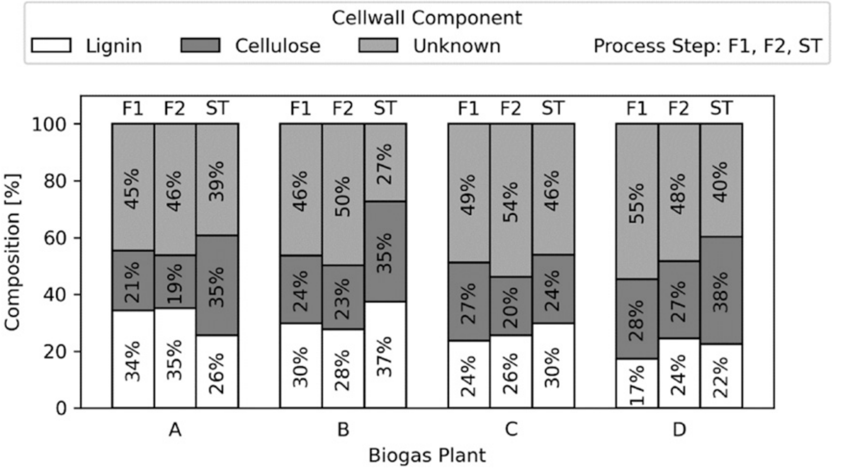

Figure 2. Cell wall composition of the investigated samples of the biogas plants A-D in the process steps primary fermenter (F1), secondary fermenter (F2), and storage tank (ST) related to dry mass.

mass content. To evaluate the fiber quality, shares of cellulose and lignin are most important. In all plants, cell wall content increased during the process, with a higher cell wall content in ST in comparison to F1 samples. The lowest cell wall content was found in D-F1 with 45%.The cellulose content of all samples examined decreases from F1 to F2 and increases again in ST. However, the trend was less clear for the lignin content. In the material from ST, highest lignin content was found in plant B with 37%. Compared with the values of the initial substrates (literature data, see Table 1), the lignin content of the 170 digestates is higher at all process stages and for all plants. The cellulose content of the digestates is similar compared with that of the silages, but clearly higher than that of the animal excrement. This fact is also due to the degradation in the biogas plant. The high proportion of cell wall components is related to their impeded degradation in the fermentation. The remaining biomass is degraded, therefore, the proportion of cell wall substance increases. The reason is that cell wall components are hard to degrade by the microorganisms in the fermentation (Schimpf 2014). Since the content of cellulose and lignin is an important characteristic for natural fibers, the use of digestate is preferable to the use of the initial substrates. The proportions of cellulose and lignin are in the range of wood fibers (see Table 1) in all the samples from process step ST. However, compared with wood fibers, their cellulose content is lower and their lignin content is higher. For a possible application of the digestate fibers in composites, it is positive that the digestate fibers have a similar composition to wood fibers, which are already used (Vogt 2006).

Wet Sieve Analysis

Figure 3 shows the amount of material in the sieves after wet sieving. The percentage of digestate over 1 mm (1and 2 mm mesh size) accounts for more than half of the total fermentation residues in the plants A, B, and D. This result was the same for all process steps and for all three plants. The materials from biogas plant C have a more uniform distribution across all sieves. Only for C-F1 the share of particles on the 2-mm sieve is slightly higher. The proportion of 2-mm fibers decreased from F1 to ST. While more than 80% coarse particles were found in ST, F1, and F2 contained 70% and 60%, respectively. In the case of C, approximately 50% of coarse particles were found for F1, whereas for the other two process stages, only approximately 20% were found. For B, the share of coarse particles varied between 76% (F1) and 47% (ST) depending on process

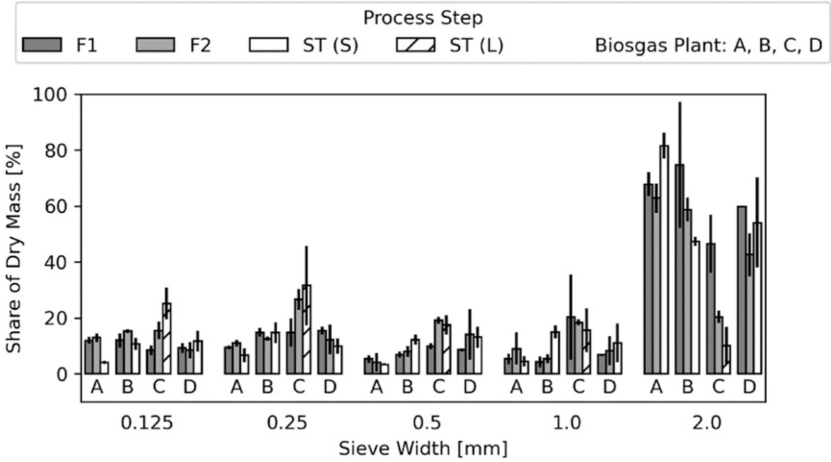

Figure 3. Share of the digestate samples in sieves with different sizes after wet sieving. Biogas plants A-D in the process steps primary fermenter (F1), secondary fermenter (F2), and storage tank (ST), separated (S) and not separated (L) related to dry mass.

step. D did not show such a clear decrease, as more fibers were found in ST with 55% than in F2 with 47%. The lower content of coarse particles in process step ST compared with F1 is caused by the duration of the digestion. The longer the materials remain in the digestion process, the more the plant materials are degraded.

The differences among the biogas plants are caused by the substrates used. Plants B and D were fed with a high proportion of maize silage and plant A shows a high proportion of ensiled hop vines. Both are high-fiber feedstocks, as indicated by literature data in Table 1. Plant C contains a high proportion of 205 manure (liquid and solid) and has fewer coarse particles.

Fiber Geometry

Results of the fiber measurement are presented in Fig 4. Classified by process stage, the fiber length distribution and the degree of slenderness are shown for all studied fermentation residues. Before data analysis, outliers were identified based on the interquartile range and excluded (Frigge et al 1989; Shevlyakov et al 2013). In the figures, only the density function of the length and the slenderness ratio are shown for readability. For fiber length, the functions of the samples from F2 and ST show clear peaks and a small distribution. The material from F1 has a wider fiber length distribution. For the F1 samples from all plants, no clear differences in length distribution were found. The mean fiber length of all samples is approximately 3 mm, and the upper limit is 8-9 mm. In the F2 samples, larger differences in length distribution were found, with a mean fiber length of 4 mm for plant A and a mean fiber length of 2-3 mm for the other plants. The same applies to the upper limit. For plant C it is 6 mm and for plant A 12 mm. The ST-digestates have a similar distribution, except for A-ST. The mean value of C-ST is the lowest with 1.6 mm, whereas the others are in the range of 2-3 mm. In addition to the mean value, the upper limit is also striking. The lowest maximum fiber length was found for C-ST with 4.2 mm, the highest is found for A-ST with 9.5 mm. The mean and maximum values of the slenderness ratio of all samples increased from F1 to F2, but decreased in the last process step ST. D-F1 had the lowest mean slenderness ratio, which is 5, whereas the highest value of 10 was found for B-F1. The fibers of A-F1 showed a wider distribution of the slenderness ratio compared with the other samples. Unlike the others, the function has no peak at one slenderness ratio. The samples from process step ST showed a

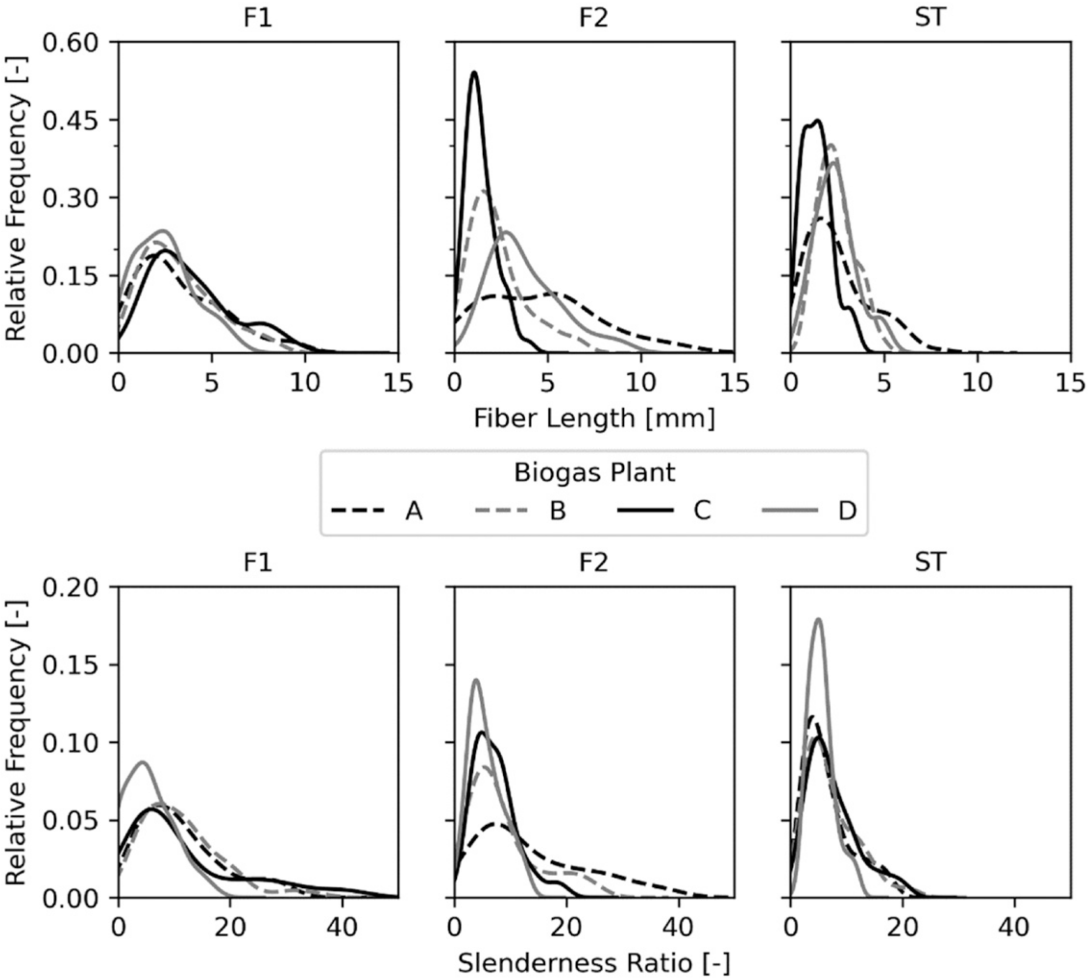

Figure 4. Slenderness–Slenderness and Length deviation of the samples from the four biogas plants in the process steps primary fermenter (F1), secondary fermenter (F2), and storage tank (ST). The three upper diagrams show the length deviation, and the lower diagrams show the slenderness–slenderness deviation.

more equal distribution compared with the previous process steps. The median of all the plants is 5-6, but the maximum values show larger differences. The highest slenderness ratio of the samples D-ST is 13 and not approximately 20 as compared with the other plants. D and C are the plants with the highest proportions of liquid manure, which could potentially explain the difference in the slenderness ratio. The fact that D is the only plant using cereal dust is also an indication of this. In addition, a higher proportion of maize silage is used compared with the other plants. This is also likely a reason for the different geometry of the fibers.

A is the only plant that contains ensiled hop vines, and in a comparatively very high proportion. The presence of the vines also explains the fact that the length and the slenderness ratio in the F2 differ from those of the other plants. The hop vines are the only lignified biomass among the substrates. Due to the different composition, the degradation also differs from that of the less lignified substrates. As with the slenderness ratio, the

Table 3. Density of the dried digestates from the four biogas plants A-D and the three process steps primary fermenter (F1), secondary fermenter (F2), and storage tank (ST).

Biogas plant	A			B			C			D		
Process step	F1	F2	ST	F1	F2	ST	F1	F2	ST	F1	F2	ST
Density (g/cm^3)	1.51	1.53	1.53	1.55	1.54	1.47	1.54	1.54	1.57	1.54	1.54	1.51

different substrate composition may cause different degradation and, thus, also affect the length distribution during the biogas process. However, compared with the common natural fibers, the fibers (see Table 1) are rather short and the length is more similar to the fibers of softwood. For all four plants, most of the fibers have a mean slenderness ratio of at least 5. According to the common definitions for fibers, a large part of the fermentation residue particles, therefore, counts as fibers. On the other hand, they cannot be called textile fibers suitable for textile production, as their slenderness ratio is well below 1000:1. Thicker and short fibers can be used for the nonwoven production (Russell 2006; Fuchs and Albrecht 2012). The fermentation residues meet all the requirements of a fiber and a suitable fiber for nonwoven production. Nonwovens are a common reinforcement for composites (Schürmann 2007). Short fibers and particles like sawmill by-products are commonly used for composites (Vogt 2006). For these reasons, the use of digestate should also be possible.

Density

The density of natural fibers is usually given as 1.5 gcm^{-3}. Table 3 shows the density values of all studied digestate samples. All fermentation residues studied are close to 1.5 gcm^{-3} and are, thus, comparable to other natural fibers. The density is also in the same range independent of plant and process stage. The variations observed are caused by the substrate composition; no trend was observed with respect to the process stage.

Output of Usable Fibers from the Digestate

Using the information obtained from the digestate, listed in Table 1 and the fractions of the sieves with 1- and 2-mm mesh size, the number of usable fibers is estimated. The data of process step ST is used for the calculations because this material is eligible for production. The biogas plants are fed with plant-based biomass and excrements, so the influence of these substrate classes on the fiber output is of interest. Figure 5

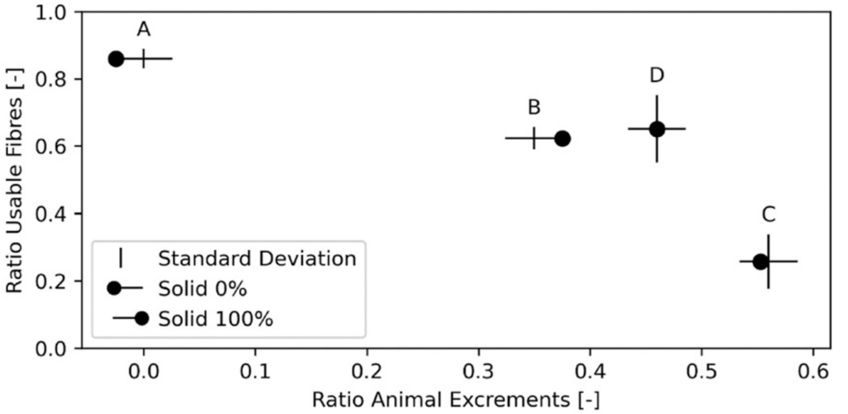

Figure 5. Relationships between quantity of excrements in the substrate and the potential fiber output in the dry mass for the storage tank (ST) samples of the four biogas plants A-D.

shows the relationship between the amount of animal excrement and the ratio of potentially usable fibers in the dry mass of the digestate. With 86%, plant A, the plant without animal excrements, has the highest ratio of potentially usable fibers in the dry mass. The lowest ratio of usable fibers (25%) was found for plant C. The difference between plant B and D is small (62% and 65%). A higher ratio of excrements leads to a higher deviation of the fiber output, compared with plant-based substrates. The more excrement, especially liquid excrement, is included in the substrate mix, the lower is the ratio of usable fibers. Since animal excrement is one of the main initial substrates in German biogas plants (Daniel-Gromke et al 2017), the number of plants with a high output of fibers seems to be low.

CONCLUSIONS

The digestate, according to the general definition, is a fiber with properties similar to those of wood fibers. The initial biogas substrate does not affect the fiber quality, but the quantity of potential fibers. A high proportion of (liquid) animal excrement reduces the quantity of fibers, however, excrement is commonly used. A long time in the biogas plant results in shorter fibers, but the quality (cell wall content) increases. The degradation during the anaerobic digestion cannot fully replace fiber extraction. Because of the high output and the similarity to wood fibers, digestate is an option for composites.

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