Recirculation of Process Water in a Wet Fermentation of Organic Fraction of Municipal Solid Waste

A. Zentner*, C. Dornack

1 Institute of Waste Management and Circular Economy, Technische Universität Dresden, – Pratzezwitzer Straße 15, 01796 Pirna, Germany

**INTRODUCTION**

The mechanical biological treatment plant (MBT) of Freienhufen is used to stabilize residual waste. Since the rural districts Elbe - Elster and Oberspreewald - Lausitz match their waste management with federal law, organic fraction of municipal solid waste (OFMSW) will be collected separately in future. Hence the anaerobic digestion process has to be converted. The accomplishment has to refer to the existing operating regime to reduce investment costs. This contains a wet fermentation. In order to facilitate the conversion of the operating process, suitable particle sizes and volumetric loads have to be examined. In addition, the liquid phase of the digestate shall be recirculated maximal to save both fresh water and waste water disposal costs. The one year lasting investigations were performed in lab-scale with a various number of reactors. Before feeding the bio-waste was pre-treated. In order to do that, the bio-waste was milled to particle sizes of 2, 4, 8, and 10 mm. In addition, the digestate was dewatered to gain process water. While using the process water fresh water was substituted in varying proportions. The feeding of the reactors was adjusted to the standards of the operating plant. For that reason, the dry matter content in the reactor was adjusted at 10.5 %. Depending on the delivered raw material, this restriction led both to unsteady water requirements and volumetric loading. As a result of investigations an optimal particle size as well as optimal proportion of recirculated process water were defined. For that reason, comprehensive analyses were conducted weekly to characterize the delivered raw material as well as the liquid and solid phase of the digestate in order to determine critical moments due to recirculation of process water. In conclusion, liquid and solid phase of the digestate should be evaluated with regards to application as fertilizer.

**ABSTRACT**

The mechanical biological treatment plant of Freienhufen is used to stabilize residual waste. Since the rural districts Elbe - Elster and Oberspreewald - Lausitz match their waste management with federal law, organic fraction of municipal solid waste (OFMSW) will be collected separately in future. Hence the anaerobic digestion (AD) process of the MBT has to be converted. The accomplishment has to refer to the existing operating regime to reduce investment costs. This contains a wet fermentation. In order to facilitate the conversion of the operating process, suitable particle sizes and volumetric loads have to be examined. In addition, the liquid phase of the digestate shall be recirculated maximal to save both fresh water and waste water disposal costs. In the framework of one year lasting experiment the focus was segmented into five parts:

- Description of quality and quantity of separately collected OFMSW in the course of the seasons
- Pretreatment of separately collected OFMSW with respect to an efficient operating regime and with due regard to laboratory requirements
- Elaboration of best practice in AD conforming to specifications of plant operator
- Development of a process water recirculation management taking enrichment of nutrients and accumulation of contaminants into account
- Assessment of residuals (digestate) of AD regarding application as fertilizer

**CHARACTERISATION OF OFMSW IN COURSE OF SEASON**

The OFMSW in two-week cycle in two different territories was separately collected. Following the technical rules of LAGA PN 98 the sampling and pretreatment was realized by the plant operator. For that purpose, the heterogeneous...
OFMSW was homogenized and primary grind ed to a particle size of approximately 20 mm (Figure 1).

Subsequently, the sampling material was delivered to the Institute of Waste Management and Circular Economy (IAK). The material was sampled on site again following the technical rules of LAGA PN 98 (Figure 2). Approximately 6 kg FM were taken and pretreated for AD. Another 6 kg FM conducted as retained samples and for tests in terms of elution.

**Impact by course of seasons and settlement structure**

The samples from OFMSW were taken both for fresh matter analytics and dry matter analytics. The analytical data of the substrates provide an overview about typical properties of OFMSW in the course of seasons as well as about the composition of waste in dependence of the settlement structure.

All samples of even sampling numbers have their origin in rural area with one bigger core of settlement. The samples with odd sampling numbers have their origin in more rural areas. The one year lasting experiment includes the handling of 52 samples in the period of April 2017 to March 2018. For better understanding an overview about the samples is given in Table 1.

Since the existing operating regime at MBT Freienhufen is adjusted to a dry matter content of 6% the water content of OFMSW is granted high priority. Additionally, the water content provides data about the composition of the waste and is related to the organic dry matter (Figure 3).

The content of impurities (e.g. metal, batteries, plastics) was below 0.5 wt%. One can obtain that the water content of OFMSW has a huge variety in the range of 48 - 70 %. The minimum water content appears in spring and the maximum appears in winter. The organoleptic investigation showed that the composition of OFMSW is dominated by green waste prior in spring and autumn. Thus, both rain events and ratio of green waste/ kitchen waste had a measurable impact to the water content. The impact of settlement structure negligible since there cannot be evaluated any tendencies.

Beyond that, the content of green waste is related to the organic dry matter content. Green waste is scratched and raked from the surface of the ground. Due to this circumstances dirt and soil is collected by coincidence. Figure 4 will provide an overview about that relations.

The OFMSW was dried and ground to a particle size of 10 mm. The distribution of particle sizes after sieving leads to the perception that most of the organic matter occurs in particle sizes between 2 - 4 mm. The evaluation of relative concentration reveals that the content of organic matter increases from 27 % at a particle sizes of less than 0.63 mm to 60 % at a particle sizes of 4 mm. Thus, the thesis above can be proved. While scratching and raking green waste from the surface of the ground inert material such as sand, stones, and soil will be collected as well.

**Figure 1.** Sampling and pretreatment of OFMSW on-site at MBT Freienhufen

**Figure 2.** Sampling of OFMSW at IAK

**TABLE 1.** Allocation of sampling numbers and course of seasons

| No. | Month     | Sampling | No. | Month     | Sampling |
|-----|-----------|----------|-----|-----------|----------|
| 1   | 04/2017   | BA 1-4   | 5   | 08/2017   | BA 18-22 |
| 2   | 05/2017   | BA 5-9   | 6   | 09/2017   | BA 23-26 |
| 3   | 06/2017   | BA 10-13 | 7   | 10/2017   | BA 27-30 |
| 4   | 07/2017   | BA 14-17 | 8   | 11/2017   | BA 31-35 |
|     |           |          |     |           | 9         | 12/2017   | BA 36-39 |
|     |           |          |     |           | 10        | 01/2018   | BA 40-44 |
|     |           |          |     |           | 11        | 02/2018   | BA 45-48 |
|     |           |          |     |           | 12        | 03/2018   | BA 48-52 |
Five times a week. Five incremental samples of one week were unified to one mixed sample and prepared for analytics. The focus of process variation was on:
- Particle size of OFMSW for feeding
- Ratio of recirculated process water and fresh water in feeding
- Accumulation of inhibiting contaminants

For that reason, comprehensive analyses were conducted weekly to characterize both solid and liquid phase of the digestate. These include important parameters for AD and composting as for instance TOC, DOC, TKN, ammonium, COD, phosphate, organic acids, FOS/TAC, nutrients and carbon balance.

**Chemical properties of OFMSW**
In addition, the bio-waste was analyzed in the laboratory in regard to parameters being relevant for AD. The focus was on nutrients (e.g. ammonia, TKN, sulphur, phosphorous, TOC) and heavy metals (e.g. Pb, Cd, Zn) (see Table 2).

Within evaluation of analytical data of OFMSW inhibiting effects can be excluded. The obtained data are comparable both with literature values and from one’s own experience [1-9].

Most of the carbon, approximately 98 %, is present as organic matter. The carbon content increases in November 2017 (BA 32). From this point until the end of the experiment it is 30 – 55 % higher than in spring and summer 2017 crucial influenced by less content of green waste and major acceptance of separately collection of OFMSW at households.

The optimal C:N:P:S – ratio of > 600:15:5:1 cannot be adjusted by using OFMSW for AD exclusively [2]. This leads to the assumption of less biogas production due to missing carbon source and ammonification because of surplus of nitrogen.

**METHODOLOGY**

The investigations were performed in lab-scale. The reactors were driven with a specific volume of three liters. The AD was realized by quasi-continuous feeding using cow manure as inoculum. In order to that, feeding and sampling took place five times a week. Five incremental samples of one week were unified to one mixed sample and prepared for analytics. The focus of process variation was on:
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**TABLE 2. Chemical properties of OFMSW**

| Parameter | Arithmetic average [mg/g] |
|-----------|--------------------------|
| Ammonia   | 1.75 ± 0.56              |
| TKN       | 14.51 ± 2.29             |
| Sulphur   | 2.39 ± 0.49              |
| Phosphorous| 2.74 ± 0.54              |
| TC        | 317.81 ± 49.59           |
| TOC       | 311.62 ± 56.14           |
| Pb        | 0.017 ± 0.11             |
| Cd        | < LOD                    |
| Zn        | 0.128 ± 0.35             |
heavy metals. Additionally, the biogas composition was measured daily to evaluate the effect of varying volumetric loads and determine critical moments due to recirculation of process water. In conclusion, liquid and solid phase of the digestate should be evaluated with regards to application as fertilizer.

Pretreatment of OFMSW for experimental investigations
As shown in section 2 sampling of separately collected OFMSW took place at plant site as well as at IAK. The particle size amounted to 20 mm. Larger wooden components were separated since they are not biodegradable in AD. These wooden components will serve as structure material in subsequently conducted composting facility. By drying the bio-waste at a temperature of 105 °C for 24 hours to different effects were achieved. On the one hand the bio-waste was stabilized and a stable quality was guaranteed. On the other hand, the substrate could be grinded to various particle size in the range of 2 – 10 mm (Figure 5).

AD in experimental scale
The experimental investigations were realized in two stages. The first stage represented a preliminary set up consisting of three reactors with a specific volume of three liters. Two reactors were fed with dried and grinded bio-waste using cow manure as inoculum. The third reactor was driven with cow manure as blank value. The first set up pursued the following goals:
- Adaptation of microorganisms
- Adjust stable process conditions
- Adjust dry matter content of 10.5 %
- Preliminary investigations in dewatering of digestate to gain process water

The second stage constituted the major experiment with six reactors (Figure 6). Two reactors were driven exclusively with cow manure. Four reactors represented two varying scenarios in duplicate. The variations of the process were geared to diversity in particle size and enhancement of fresh water substitution by process water recirculation.

The process is based on operating AD at MBT Freienhufen. Thus, the retention time was adjusted at 25 days in mesophilic conditions of 35 °C. The dry matter content was raised from 6 to 10.5 % since grinded OFMSW has a lower resistance in stirring than residual waste. The feeding of the reactors took place quasi-continuously from Monday till Friday. For that reason, dried and grinded was mixed with fresh water according to the retention time within the reactor. Following adjustments were realized:
- Different particle sizes were fed: 10/8/4/2 mm
- Fresh water was gradually substituted by recirculated process water: 50/65/75/100 %

Process water recirculation
The sampling took place simultaneously to feeding. Five incremental samples of one week were unified to one mixed sample and prepared for analytics. According to the retention time five times 168 g (840 g) of digestate were removed of each AD reactor every week. Subsequently the gathered digestate was separated into solid and liquid phase (Figure 7).

Figure 5. Dried and grinded bio-waste in different particle sizes

Figure 6. AD reactors in lab-scale at IAK
RESULTS OF MAIN AD EXPERIMENTS

Varying scenarios were performed during the long-term experiment. Following Table 3 will provide an overview about adjusted parameters. The sampling was conducted five times a week from Monday till Friday. Just in time and on-site the conductivity, pH and redox potential were measured to evaluate the process stability.

pH, conductivity and redox potential

The pH value decreased from 7.8 to a stable range between 7.2 to 7.35 independently from process variation. The high starting point is caused by cow manure which is used as inoculum. Since one stage reactors performed AD the pH is in stable state. Acetogenic and methanogenic microorganisms will find best circumstances for their performance [2, 7].

The conductivity decreased from 17.8 mS/cm a stable range between 10 to 12 mS/cm independently from process variation. The high starting point is caused by cow manure as well. The non-appearance of higher conductivity values is presumably caused by high concentrations of free ions. Since conductivity is below 30 mS/cm inhibiting effects to methanogenic microorganisms excepted [5].

The redox potential is stable within the first six month of the major experiment at -400 to -300 mV independently from process variation. Thus acetogenic and methanogenic microorganisms will find best circumstances for their performance. At the end of the experiment the redox potential raised to -200 mV. In comparison with simultaneously conducted experiments one can conclude that the measuring device was non-conforming.

Production and composition of biogas

The specific biogas yields were approximately 180 NL/(kg org. DM). One could monitor that the yields increased over time (Figure 8) until ca. 240 NL/(kg org. DM). The yields were higher in A-reactors than in B-reactors even when process was changed in particle size and ratio of recirculated process water. In December an obvious change took place and the yields achieved peaks of approximately 500 NL/(kg org. DM). The results were obviously comparable to literature values [9]. This leads to the assumption that biogas production is not influenced by process variation in particle size and ratio of recirculated process water. Moreover, feeding and volumetric loading have an impact to biogas production. According to Figure 9 the biogas production is related to volumetric loading. The volumetric loading increased over time since organic content of OFMSW increased measurable and successively more organic matter is recirculated by process water. Thus, the volumetric loading increased from approximately 2.2 - 3.6 kg org. DM/(m³*d).

Another effect occurred in relation to feeding procedure. At Christmas break the feeding was converted from quasi-continuous to discontinuous feeding. Simultaneously the specific biogas yield increased to a large extent. After Christmas break the feeding was reconverted to quasi-continuous feeding and the biogas yield decreased again.

The composition of biogas was measured five times a week according to the sampling. As shown in the above figures the development of the biogas composition followed the same tendencies. Methane concentration increased from 45 % to 52 % over time and within increase of volumetric loading (Figure 10). Thus the maximum of volumetric loading is not reached. In other circumstances the biogas yield and the methane concentration would decrease [8].

### TABLE 3. Description of varying scenarios

| Timeframe/substrate | Reactor A2/A3 | Reactor B2/B3 | Process water | Feeding |
|---------------------|--------------|--------------|---------------|---------|
| BA 14-15            | 10 mm        | 8 mm         | 0 %           | Quasi-continuous |
| BA 16-19            | 10 mm        | 8 mm         | 50 %          | Quasi-continuous |
| BA 20-24            | 10 mm        | 4 mm         | 50 %          | Quasi-continuous |
| BA 25               | 10 mm        | 4 mm         | 65 %          | Quasi-continuous |
| BA 26-30            | 2 mm         | 10 mm        | 65 %          | Quasi-continuous |
| BA 31-37            | 2 mm         | 10 mm        | 75 %          | Quasi-continuous |
| BA 38-39            | 2 mm         | 10 mm        | 100 %         | Quasi-continuous |
| BA 40-41            | 2 mm         | 2 mm         | 100 %         | Quasi-continuous |
| BA 42-44            | 2 mm         | 2 mm         | 75 %          | Quasi-continuous |
| BA 45-46            | 4 mm         | 4 mm         | 75 %          | discontinuous |
| BA 47-52            | 4 mm         | 4 mm         | 65 %          | discontinuous |

### TABLE 4. Process stability in terms of pH, conductivity and redox potential

| Parameter          | Reactor A2/A3 | Reactor B2/B3 |
|--------------------|--------------|--------------|
| pH                 | Stabilization at pH of 7.2 to 7.35 |
| Conductivity       | Stabilization at 10 to 12 mS/cm |
| Redox potential    | Varying redox potential between -400 to -200 mV |
The measured concentrations for hydrogen and hydrogen sulphide are negligible. Since they were below 200 ppm they were within the related measurement tolerance (Figure 11). There is no tendency to be extrapolated. At the end of the experiment the concentration of hydrogen sulphide raised. This effect is related to process water recirculation and should be investigated in future. Gaps in Figures 10 and 11 can be explained with technical malfunctions.
Solid and liquid phase of digestate
The digestate was evaluated for separate utilization of solid and liquid phase. Thus both phases were analyzed for inhibiting effects in AD and composting. Parameters of investigation are presented in the following Table 5.

**Liquid phase** The liquid phase gained by dewatering of digestate was foreseen to recirculate as process water. Thus, fresh water should be substituted and waste water should be avoided. For this reason, the quality and quantity of the liquid phase was evaluated. Following descriptions constitute an excerpt.

The dry matter content of the liquid phase increased from approximately 3 to 7.7 % within the major AD experiment. Since we know about the particle size distribution of section 2.1 OFMSW contained a large amount of particle sizes ≤ 1mm. These particles passed the filter in dewatering partly and accumulate in the process water. The organic content of dry matter in liquid phase was about the whole time 70 ± 3 %. As a consequence, the volumetric loading increased by raising the ratio of recirculated process water.

In terms of recirculation of process water nitrogen occupies an important role. Especially ammonia which is soluble in water and accessible to plant. At the beginning of the major experiment the ammonia concentration was about 2 g/l, caused by cow manure. Over time the concentration decreased rapidly to 0.75 – 0.85 g/l independent of process variation. After Christmas break the concentration raised again to 1.05 g/l independent of process variation. This leads to the assumption that will accumulate under conditions like high volumetric loading. Additionally, the activity of microorganisms is affected by conversion from quasi-continuous to discontinuous [4]. The inhibiting effect of ammonia is in dependence of temperature and pH-value. The adjusted conditions in major AD experiment should allow concentrations until 3.5 g/l [3]. As a consequence, the liquid phase is suitable for recirculation but should be monitored over time to avoid inhibiting and toxic effects to microorganisms.

Additionally, there was an evaluation of organic acids. The sum of organic acids in all reactors and process variations was below 4 g/l. So the process stability was not affected by toxic concentrations. The ratio of acetic acid and propionic acid was > 2. Thus the efficiency in degradation and metabolism is in good circumstances. Furthermore, the FOS/TAC was analyzed. The ratio was below 0.3 and represented stable conditions. Sufficient buffer capacities in order to organic acids are delivered by cow manure as inoculum [6].

**Solid phase** The solid phase of digestate contained approximately 73 % water. While using smaller particle sizes the water content tended to 80 % due to large surface area and stronger water binding capacities. While using AD approximately 40 % of organic matter could be degraded. The solid phase of digestate has to be composted after AD since approximately 40 % of organic matter could be degraded. The solid phase of digestate has to be composted after AD since approximately 40 % of organic matter could be degraded. The solid phase of digestate has to be composted after AD since approximately 40 % of organic matter could be degraded. The solid phase of digestate has to be composted after AD since approximately 40 % of organic matter could be degraded. The solid phase of digestate has to be composted after AD since approximately 40 % of organic matter could be degraded. The solid phase of digestate has to be composted after AD since approximately 40 % of organic matter could be degraded. The solid phase of digestate has to be composted after AD since approximately 40 % of organic matter could be degraded. The solid phase of digestate has to be composted after AD since approximately 40 % of organic matter could be degraded.

### TABLE 5. Investigated parameters in solid and liquid phase of digestate

| Parameter                  | Solid phase | Liquid phase |
|----------------------------|-------------|--------------|
| Water content              | X           | X            |
| Org. DM                    | X           | X            |
| Total Kjeldahl Nitrogen    | X           | X            |
| Ammonia                    |             | X            |
| Total Carbon               | X           |              |
| Total Organic Carbon       | X           |              |
| Total Phosphorous          | X           |              |
| Total Sulphur              | X           |              |
| Dissolved Organic Carbon   |             | X            |
| Chemical Oxygen Demand     | X           | X            |
| Ortho-phosphate            | X           |              |
| Sulphate                   | X           |              |
| Organic acids              | X           |              |
| FOS/TAC                    | X           |              |
| Heavy metals               | X           | X            |

**Figure 11. Occurrence of hydrogen and hydrogen sulphide**

SELECTED TOPIC: Biogas composition

| Biogas Composition | O A H2 | O B H2 | O A H2S | O B H2S |
|--------------------|--------|--------|---------|---------|
| CO2                |        |        |         |         |
| CH4                |        |        |         |         |
| C2H6               |        |        |         |         |
| N2                 |        |        |         |         |
| H2                 |        |        |         |         |
| H2S                |        |        |         |         |
Five incremental samples of one week were unified to one mixed sample. The liquid phase after dewatering was used partly for recirculation. Scenario 1 represents the process without process water recirculation. After dewatering 11.3% of the digestate was separated in form of solid digestate. 88.5% of the resulting process water respectively 83.7% of the entire digestate has to be treated in a waste water treatment plant. The loss in performance and transferring amounts to 5.6% (Figure 12).

Scenario 2 represents the recirculation of process water of approximately 73%. After dewatering 17.2% of the digestate was separated in form of solid digestate. 82.9% of the resulting process water respectively 60.6% of the entire digestate were recirculated to the AD process. The loss in performance and transferring amounts to 1.8% (Figure 13). Figure 14 represents the normalization of scenario to an input of 1 ton of bio-waste.

**Figure 12.** Water balance without process water recirculation

**Figure 13.** Water balance with process water recirculation. Substitution rate 82%

**Figure 14.** Normalized water balance
CONCLUSIONS

The project focused on the conversion of input material in AD of MBT Freienhufen. Residual waste shall be substituted by separately collected OFMSW. The experiments in AD, dewatering and recirculation of process water revealed reliable results in anaerobic degradation while implementing different process variations. The success and quality of anaerobic degradation and biogas production are mainly influenced by volumetric loading. Thus the course of seasons as well as quality and ratio of recirculated process water have had the major impact.

Since there was no measurable and reproducible impact of particle sizes it is recommended to avoid maximum shredding. The input of energy will not be in appropriate proportion to higher biogas yields.

If liquid phase of digestate will be used as recirculated process water the MBT Freienhufen is able to substitute large amounts of fresh water. It is recommended to substitute in a ratio of 65 to 70 % to avoid toxic risks (e.g. ammonification) for microorganisms. If the recirculation of process water will be implemented in future to MBT Freienhufen a storage tank with aeration has to be added to the technical concept. The aeration will inhibit AD processes in the storage tank and will prevent settlement of dry matter content from process water.

In order to use solid and liquid phase as fertilizer a post treatment is necessary. The properties of solid and liquid phase did not match the requirements by law. Thus, composting with adequate co-substrate as well as mixing with structure material from pretreatment are possible pathways to ensure a good composition for composting.

The course of seasons revealed a huge variety in composition of separately collected OFMSW. The MBT Freienhufen has to manage the balancing act between municipal waste disposal and gain in energy. Kitchen waste is suitable for AD processes because of high water contents and high content of organic dry matter. By contrast, green waste is more suitable for composting. The implementation of MBT Freienhufen is able to substitute large proportion to higher biogas yields.

The implementation of MBT Freienhufen is able to substitute large proportion to higher biogas yields.

fermentation requires large amounts of water which can be covered partly by kitchen waste with high moisture.

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