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D3.3 FINAL REPORT ON THE TRIALS - TECHNOLOGIES FOR NUTRIENT RECOVERY FROM DIGESTATE

DIGESMART

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European Commission | Executive Agency for Small and Medium-sized Enterprises

Project website: www.digesmart.eu

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INTRODUCTION

This report is the deliverable 3 in work package 3 of the DIGESMART-project. It gives an overview of the different trials performed and results achieved with the DIGESMART installation. It consists of a chapter on the stripping and scrubbing and a chapter on the solar drying testing. The design and redesign of the stripping-scrubbing system during the project is outlined including relevant items from an economical and environmental point of view, such us materials to be used, configuration of the system, dimensioning or consumables to be used in the process. In addition, key operational parameters of the process are discussed and the main outcomes attained by their evaluation during preliminary trials and continuous mode operation of the installation are presented.

STRIPPING AND SCRUBBING START-UP TRIALS

Detailed information regarding the installation design as well as the evaluation of operational conditions and results from the different trials performed are stated in the following sections.

INSTALLATION SETUP

A test rig system for the DIGESMART solution was built in Ypres (Belgium) at the construction facilities of Vermeulen NV, the in-house company with DETRICON.

Figure 1. Test rig system for stripping & scrubbing at the DETRICON company.

The first system had a designed capacity of 0.5 ton per hour. The installation is isolated and the used material is stainless steal 304 in order to work in low pressure and high temperature regions. The pilot has a stripping volume of 1-3 m^3, a ventilation flow of 1000 - 1800 m^3/h, with an air speed of 0.2 - 0.8 m/s. It can be seen in Figure 1, from left to right, a mixing tank to add CaO to the liquid fraction of the digestate, a filter, a vacuum pump, the scrubber and 3 stripping tanks. All the elements are connected and the air can circulate from stripper to scrubber and back to the first scrubber without any exhaust.

As manure and digestate production and the related nutrient excesses in streams and groundwater is a constant growing problem. The market demand developed from small scale installations with the advantage of being mobile to large scale installations without the possibility of being mobile.

In order to ensure a more positive pay-back and reach the market more satisfactorily, the eventual pilot system needed a minimum capacity of 20,000 tonnes/year, which implies a big stripping volume. As big stainless steel 304 tanks are quite expensive, the system was adapted for the use of polyester tanks with a special coating to endure high temperatures and a corrosive medium.

After the start-up trials, a number of different adaptations of the design of the system were undertaken during DIGESMART project (especially at the end of 2014) in order to improve the market replication possibilities in Belgium and involved EU-countries, being the most noteworthy to mention the following ones:

a) A single concentric reactor is used in the final design of the demo unit (pilot plant installation located in Gistel). The incorporation of the stripping and scrubbing process in a single tank facilitated the energy recovery making the system more efficient. Also, this integrated recovery solution would imply reduced investments and handling in comparison with other technologies in the market.

b) Change to a bigger stripping and scrubbing volume and ventilation flow in order to maximize the recovery of NH_3 gas from the liquid digestate fraction.

c) Specific design of packed stripping tower which maximise the stripping capacity per cubic metre of liquid digestate fraction. This is a key aspect in order to minimise the pay-back of the installation making affordable the stripping-scrubbing solution for installations that were too small in the past and made not feasible the implementation of the treatment.

d) Nitric acid in the scrubbing step was decided to be used as acid instead of other acids due to higher value of ammonium nitrate in comparison to other ammonium fertilizers. The acid storage tank was modified according to the new acid to be used.

e) Several final adjustments of the properties of the green fertilizer were developed. The goal was to develop a technical pure product with exactly the same concentration as the chemically produced equivalent material.

This resulted in the eventual DIGESMART pilot plant installation.

OPTIMAL STRIPPING OPERATIONAL CONDITIONS

As stripping and scrubbing is a well-known chemical process, the trials could be initiated by considering the reported theoretical knowledge on the technology and from some practical examples. Mainly, it is relevant for the process to mention that the parameters: temperature, pressure and pH, are correlated with the equilibrium of ammonia in the gas and liquid phases. Anyhow, it has been also reported that relatively low recovery yields could be attained regardless of the selection of proper pH and temperature values, due to mass transfer constraints. For this, the use of packed stripping towers was considered, as indicated above, as it provides large surface area for mass transfer mechanisms.

With regard to pH and temperature impacts on the process, the corresponding tendencies concerning ammonia and ammonium ion equilibrium are shown in the Figure 2.

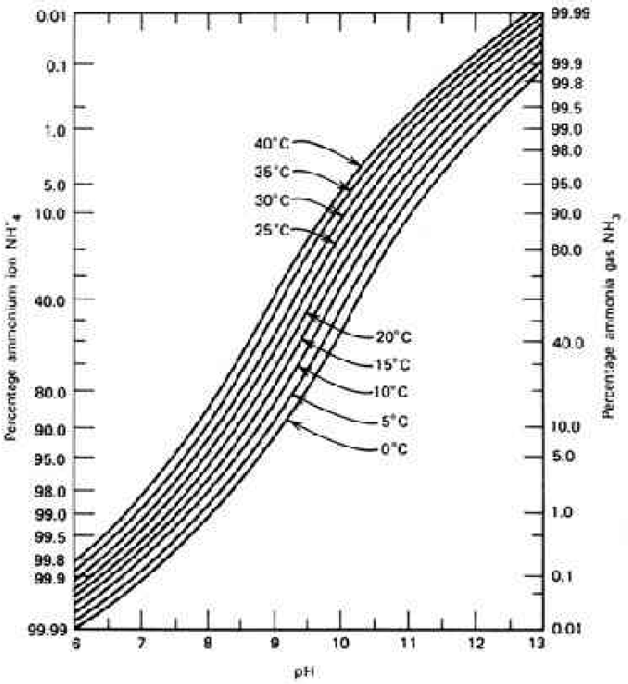

Figure 2. Effects of pH and temperature on the distribution of ammonia and ammonium in water (from Liao et al. 1972).

As observed, the pH value is a crucial factor which directly impacts on the conversion of ammonium to ammonia gas, and accordingly high values of pH would provide greater recovery rates of nitrogen from the liquid by stripping/scrubbing. In fact, it is generalised in ammonia air stripping processes the addition of alkali (e.g. use of lime or caustic soda) to increase pH above 10-11, or to degas the influent in order to remove CO_2 and thus lowering the buffer capacity. These actions would result in the conversion of ammonium to dissolved ammonia and thus the transfer to ammonia gas to the gas stripping stream. During the trials, high doses of lime, lowers the lifetime of the packing material as it blocks over time. It is an important issue of this technology.

Alternatively, the volatility and recovery of ammonia can be enhanced by increasing the temperature, without the aforementioned disadvantages of lime dosage. As it can be seen in Figure 2, certain augmentations in the process temperature could mean fairly higher ammonia recovery yields. Hence, a feasible strategy would be to maintain certain process temperatures in order to reduce the use of chemicals and consequently minimising costs, and even the use of heat excess in the process for the heating up the digestate so that the technology would be potentially more competitive.

In the light of this reported information, the stripping/scrubbing technology was designed and evaluated under a variety of operational conditions to assess their effects on the recovery efficiency. Specifically, the evaluation of conditions was carried out to optimize the stripping capacity in terms of nitrogen recovered from the liquid fraction of digestate per hour and volume of package in the column; furthermore, the operation under conditions of minimal use of electrical and thermal energy and chemical demand was attempted.

Minimal use of electrical energy

The electrical consumption of the recovery system was optimised by searching the minimum of the ratio of the power consumption (recirculation flow, ventilation speed and pressure drop) and recovery rate of nitrogen (see Figure 3).

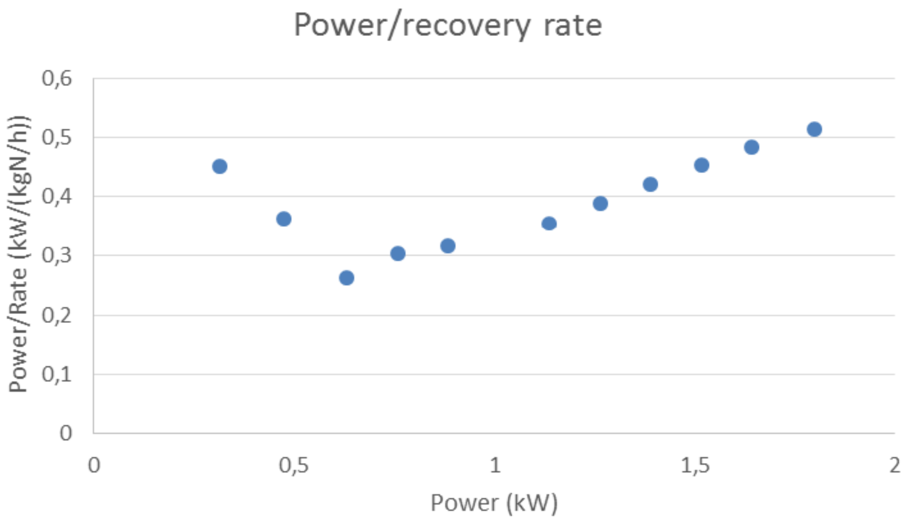

Figure 3. Correlation between the power consumption and the recovery rate of nitrogen.

As observed, a tendency shift takes place at a power of about 0.6 kW: when operating below that value the ratio power/nitrogen recovery decreases with the power as greater recovery yields are attained, whereas higher power values would imply lower efficiencies (higher ratio power/recovery rate) since the enhancements in the nitrogen recovery yields would not balance the corresponding energy required. According to the optimal flows of digestate and air, the design of the new pilot plant was established.

Minimal use of thermal energy and chemical demand

The pilot plant was isolated with 6 cm of PUR, reducing the heat losses with 65%. The Detricon heat recovery system makes it possible to recover 95% of the heat. The reduction of external tubes, isolation, heat recovery, internal heat losses of the pumps and ventilator and the heat of the exothermic reaction between nitric acid and ammonia, allowed working at temperatures around 45°C without an external heat source. In order to increase the temperature, an external heat source would be needed, using up till 1 kWh/m³ (see Table 3 – D4.2). An optimum was found at

60°C at atmospheric pressure because it opens the possibility to work with polyester. This temperature serves as preliminary goal temperature for the full scale pilot plant.

On the other hand, the chemical demand (CaO) is reduced as temperature increases. For that reason, the design and operational conditions of the installation makes it possible to have an efficiency of nitrogen recovery up to 95% with a minimal chemical demand of CaO (1-4 kg/t of liquid digestate fraction) when operating under proper temperature conditions. It was observed during the operation that temperature increases until reaching equilibrium at 40-42ºC. As higher temperatures require more energy input and increases the risk of foam formation and free fatty acids increase in the recirculating air, besides lowering the quality of the biobased ammonium nitrate adding colour and odours, the working temperature of the DIGESMART installation was established until a maximum of 50ºC.

REACHING CONTINUOUS MODE FULL SCALE PILOT

During the first trials the pilot plant was able to process 2-3 m^3/h of digestate so it will be able to process the liquid fraction of the biogas plant that produces about 30,000 m^3/year of digestate. Thus, the targeted throughput is achieved.

As for the reduction of ammonia during the stripping process, values from 4 kg NH_4-N/t to 0.5 kg NH_4-N/ton were achieved, which means 87.5% of reduction; therefore, the recovery yield is certainly within the expected range of 50-95%. The installation could be pushed to recover even more ammonium, but then this would result in an efficiency decrease. As shown in Figure 2, the recovery rate goes down as the recovery yield goes up as the ammonia concentration goes down. A higher residence time corresponds with a lower yearly capacity and higher operational costs.

The first trials provided positive results, with an ammonium nitrate product reaching a constant concentration. To assess the system ability for internal heat use, a continuous trial of 36 hours was run (March 2016) and temperature was measured (figure below).

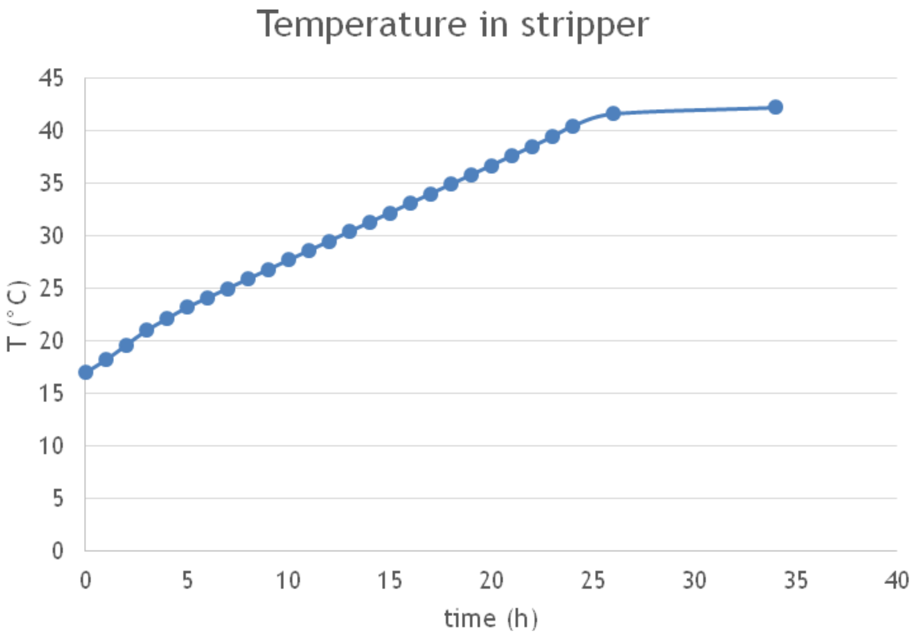

Figure 4. Temperatures build up in the installation

The system attained a stable self-generated temperature between 40–45 °C, whereas it would be advised to use external heat coming from the biogas-CHP unit to achieve the optimum of 60°C. Anyhow, this sub-optimum at 45 °C is still deemed to give sufficient ammonium recovery, and also the detrimental effects indicated above related to the operation under higher temperatures would be avoided.

Figure 5. Continuous trials in the installation set up in Gistel (Belgium).

Adding an extra buffer tank made it possible to work continuously and take samples more easily, see Figure 6.

Figure 6. Adding an extra buffer tank to work in a continuous way. The liquid fraction of the digestate is continuously refreshed while the installation continues with the stripping and scrubbing process.

The recovery yield of the installation (50-95%) was measured according to the capacity. In the figure below, the performance of the pilot installation in Gistel (BE) is shown. The operational costs are:

Chemicals: 2 kg CaO/ton

Electricity: 2,5 kWh/ton

Heat: 0 kWh/ton

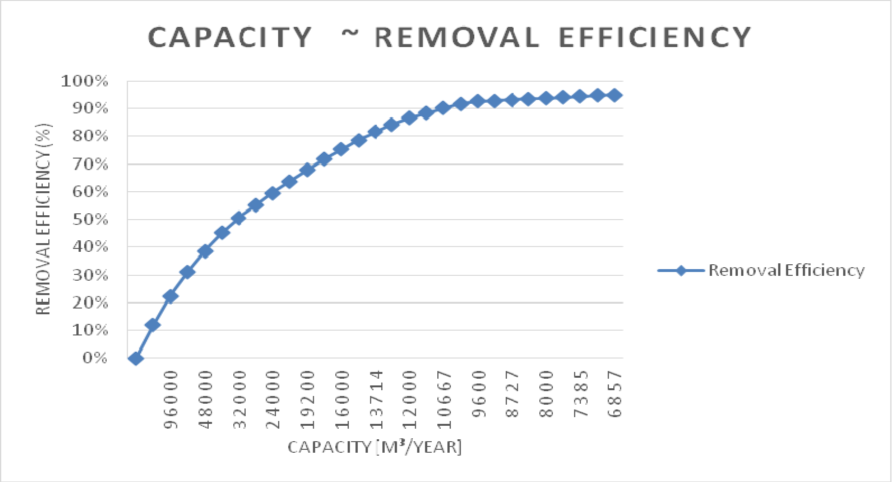

Figure 7. Graph showing the removal efficiency in function of the capacity

The data of the figure above is given in the table below. Removing 60% of the N, the pilot has a capacity of 24,000 m³/year.

Table 1. Capacity and removal efficiency of the stripping&scrubbing system

Capacity (m³/year)	Removal Efficiency
	0%
192000	12%
96000	22%
64000	31%
48000	39%
38400	45%
32000	51%
27429	55%
24000	60%
21333	64%
19200	68%
17455	72%
16000	75%
14769	79%
13714	82%
12800	84%
12000	87%
11294	88%
10667	90%
10105	92%
9600	93%
9143	93%
8727	93%
8348	93%
8000	94%
7680	94%
7385	94%
7111	95%
6857	95%

As a result of these trial and calculations the DIGESMART solution consists of a tank-in-tank stripping and scrubbing technique:

1. Lowering the air speed and thus lowering the pressure drop over the ventilator and resulting in a lower electrical demand.
2. No external tubes are used for the air transport, lowering the heat loses.

3. With heat exchange between the influent and effluent, recovering 95% of the thermal heat. As heat losses are minimal, the thermal heat from pumps, ventilator and exothermic reaction of ammonium nitrate formation can compensate it (see Figure 3)

4. With a purifying step on the recovered ammonium resulting in a pure end product with a stable concentration.

5. A compact system with a small footprint. For example, the pilot plant in Gistel, treating 24.000 tons/year, has a footprint of 120 m².

SOLAR DRYING TRIALS

In total 4 trials were conducted during the project DIGESMART on the solar drying module developed. The first test was carried out on 12 July, loading the evaporator with 60 kg of digestate. This was the first test with digestate and was intended mainly to verify the general behaviour of the installation. The weather conditions were favourable.

The second test on 16 August was carried out loading the evaporator with 60 litres of digestate, and taking samples each hour. In this second test too the weather conditions were fine, with good solar radiation and an ambient temperature between 19 and 24 degrees °C. The third and fourth tests have been carried out on 27 and 28 August 2016.

In the third test on 27 August the configuration of the plant was modified. The liquid solar collector was excluded, in order to test the behaviour of the installation with only the air solar collector and the evaporator, to see if under favourable conditions the system could work without the high efficiency under vacuum collector, working with liquid as a means to transfer the heat.

In the 28 August test the solar collector under vacuum was added again to the system. Those differences in configuration were meant to assess the contribution of the liquid solar collector to the behaviour of the whole installation.

For solar installations, generally speaking, a good solar radiation can be considered to be around or more than 6 kWh/m^2-day, a situation that is met in clear sky conditions in summer. Solar radiations under 4 kWh/m^2-day can be considered low, and under 3 kWh/m^2-day very low. The conditions during the test are given in Table 1.

Table 2. Solar radiation conditions during the trials

Day	Digestate load (kg)	Horizontal average incident radiation (W/m^2)	Inclined average incident radiation (W/m^2)
12 July	60	no available data from dataloggers	no available data from dataloggers
16 August	60	707	785
27 August	40	579	773
28 August	40	353	455

Taking into consideration the results and experience build-up of the 12th of July and 16th of August test, the protocol for the 27 and 28 August tests was concluded as follows:

- The system was charged with 40 kg of digestate, just enough to cover all the surface of the evaporator.
- Data logging was set to register data every 5 minutes.
- The beginning of the test, after the charging of the evaporator with digestate, was set at 9h40.
- Samples of digestate and condensate were taken every two hours, at 11h40, 13h40 and 15h40.
- Samples of digestate were taken also before the beginning of the test, while filling the evaporator.
- End of test was set at 15h40, as the sun passed behind the solar collectors at about 16h00.

Figure 8. The samples taken during the 27 and 28 August tests

To assess the performance of each component of the solar evaporation system, the tests have been carried out monitoring temperature and relative humidity inside the air circuit, as well as the solar radiation.

The measured parameters have been:

- solar radiation on the horizontal plan
- solar radiation on the solar collectors
- ambient temperature
- process air temperature and humidity in different points of the air circuit
- process air flow
- digestate condensation mass after evaporation.

Samples taken were analysed on the relevant parameters such as dry matter content, total nitrogen, ammonium content, nitrate content, potassium content, phosphorus content, pH and conductivity.

RESULTS

The results on the technical performance will only be highlighted for the most successful trial date and are represented in Figure 8 to 10. During the test of 27 August the weather was favourable and this proved to be the best trial date.

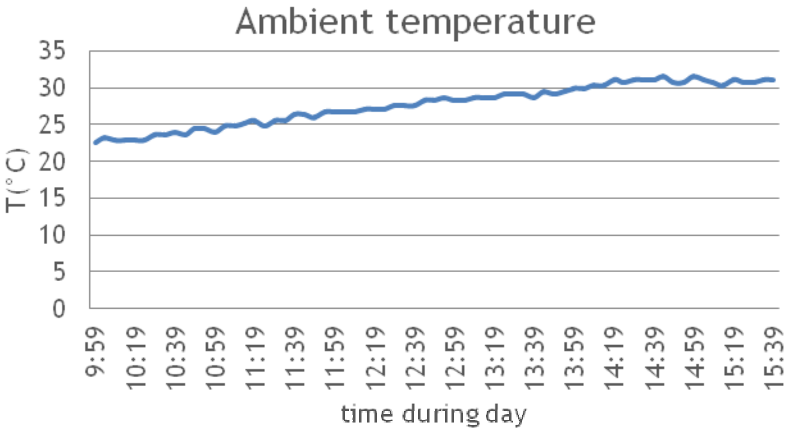

Figure 9. The ambient temperature during the 27th of August

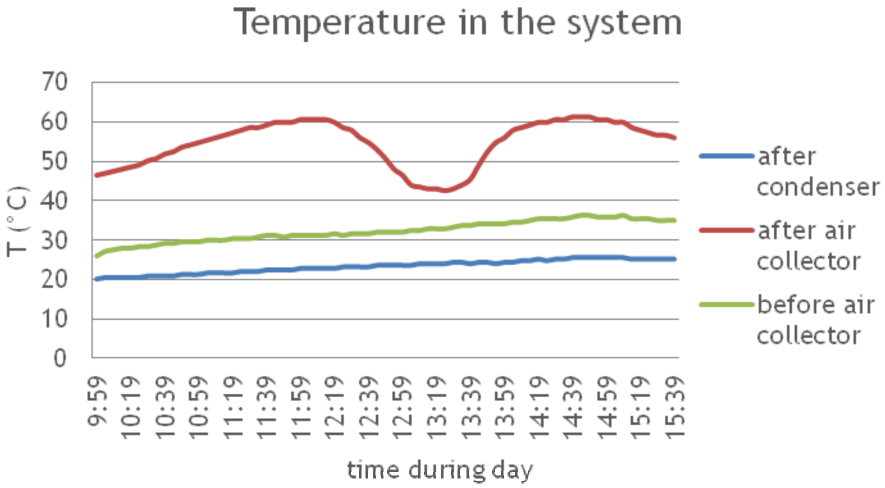

Figure 10. The temperatures after the condenser, before the solar air collector, and after the solar air collector during the 27 August test

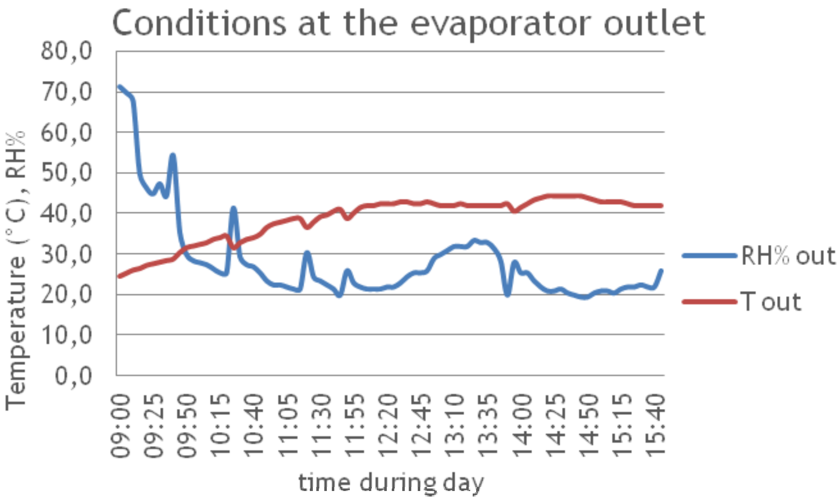

Figure 11. Temperature and relative humidity at the evaporator outlet. The low relative humidity indicates that the air-vapour mix is far from saturation

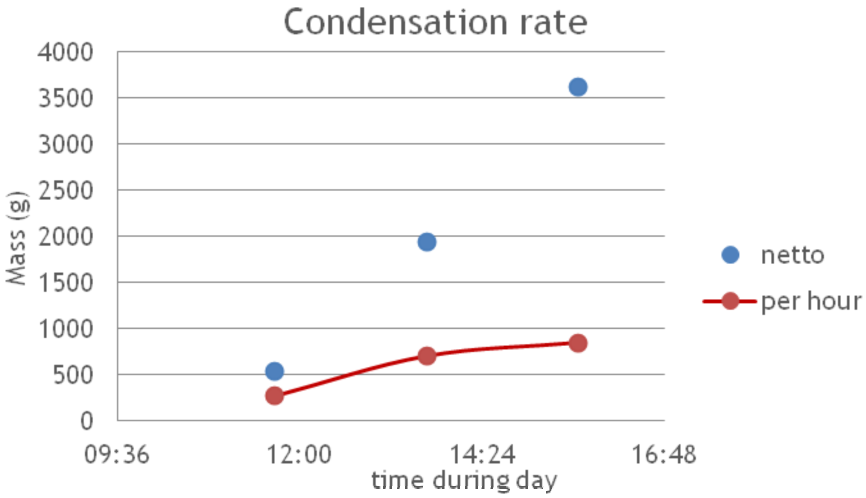

Figure 12. The total and the hourly condensation rate on 27 August

The quantity of obtained condensate, slightly less than 4 litres, was on the other hand not satisfactory as it was very far from the goal that was forecasted, 26 litres in a day. The problems derive from the balance in the different elements of the plant. The main problem consisted in the fact that as the process air does not evaporate enough vapours from the digestate, and enters the condenser too far from saturation conditions. The condenser worked with water at a temperature of 18 °C, and was able to lower the temperature of the air-vapour mix to a level between 19 and 24 degrees, depending on the moment. The result of these unfavourable working conditions involved a poor evaporation/condensation rate. The solar air collector performed the heating of air as expected, increasing the temperature of air of 20 - 35 degrees, depending on the moment.

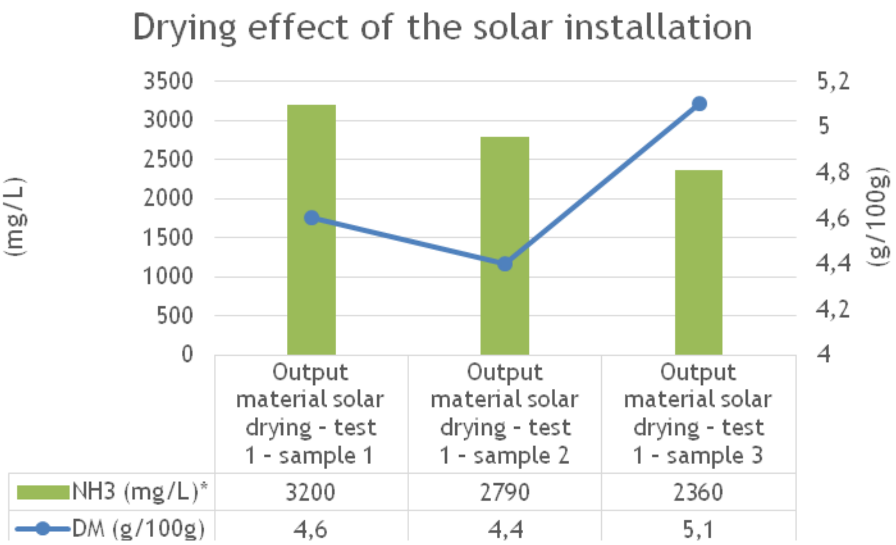

Figure 13. Solar drying effect on the input material

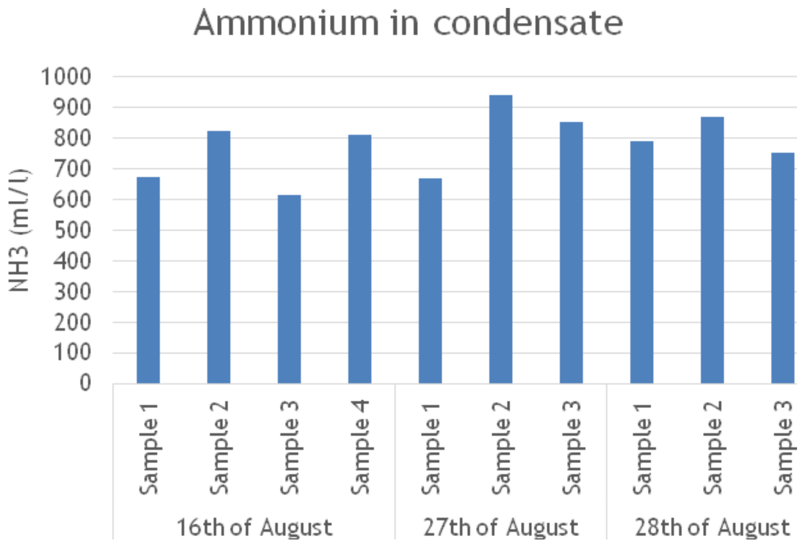

Figure 14. Ammonium concentration of the condensate after solar drying

The drying effect of the solar installation is demonstrated by the results given in Figure 11. It is clear that the material is drying going from a dry matter percentage of 4,6 to 5,1. In the other trial this effect was also demonstrated. Furthermore, the condensate produced contains ammonium as is shown in Figure 12. This means that clear water is not produced as the input material still contains much ammonium. This volatile substance evaporates with the moisture and is thus found in the condensate. Starting with an input material with lower ammonium content would be advisable. The solar system showed some results, but it far from optimised and needs further investigation.