Anaerobic digestion of jatropha curcas press cake

Responsibility

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Contents

Appendices

Figures

Tables

1 Introduction

For FACT Fuels Foundation Ingenia investigated the anaerobic digestability of four different samples of jatropha curcas press cake. These samples were obtained by cold pressing of jatropha curcas seeds. The digestion experiments were executed in the Ingenia laboratory on a 1 litre scale during 60 days.

1.1 Situation

M.Sc. student Peter Beerens from TU/e is doing research on the cold pressing of jatropha curcas seeds. He works with a Danish BT50 expeller press. This press can be fitted with different nozzle aperture sizes. Part of Peter's research is to investigate the oil quantity and quality.
Ingenia has been commissioned by FACT Foundation to do digestion research on the press cake that falls free from Peter's research.

1.2 Feedstock

Four samples of press cake have been received and their digestion characteristics have been determined in Ingenia's laboratory facility. This involves regular measurement of the biogas production under controlled circumstances. To be able to draw conclusions from the measurements, some chemical characteristics of the samples need to be known. The feedstock samples are as follows:

1. Cake pressed with a nozzle aperture size of 9 mm. Press feedstock were complete jatropha seeds, none of them peeled (i.e. 100% hull content).
2. Cake pressed with a nozzle aperture size of 7 mm. Press feedstock were complete jatropha seeds, none of them peeled (i.e. 100% hull content).
3. Cake pressed with a nozzle aperture size of 7 mm. Press feedstock were complete and peeled jatropha seeds in a ratio of 2 to 1 (i.e. 67% hull content).
4. Cake pressed with a nozzle aperture size of 7 mm. Press feedstock were complete and peeled jatropha seeds in a ratio of 1 to 2 (i.e. 33% hull content).

The samples were analysed for some chemical properties at Analytisches Labor Gelsenkirchen (ALGE). This was done for a good understanding of the digestion behaviour of the different samples. The determinations that have been done on the press cake samples include:

- Dry matter content (wt%) (this number equals '100% – water content')
- Ash content (wt%) (this number equals '100% – organic matter content')
- C, H, N, O content (wt%)
- Kjeldahl-N content (mg/kg) (this number expresses the amount of nitrogen that is bound in ammonium and organic components)

The results from the laboratory analysis have been summarized in the table below. It can be seen that the dry matter content (expressed as water content) is virtually the same for all samples. The water content is not a characteristic property of the feedstock alone, it depends a lot on storage and processing conditions. Hence it is not used further.
Comparing the results from the 7 mm experiments it can be seen that the content of C and O have a positive tendency for increasing hull content. The contents of hydrogen, nitrogen and ash, on the other hand, have a decreasing tendency for increasing hull content.
It can be seen that the sum of C, H, N and O content is not 100%. The remainder (7 wt% on average) consists of other elements, like P, Na, K, etc.

Table 1-1 Chemical properties of jatropha press cake samples

Nozzle aperture size		9 mm	7 mm	7 mm	7 mm
Amount of hulls included		100%	100%	67%	33%
Water content	wt%	7,3	7,3	7,3	7,9
Ash content	wt%	5,96	5,28	5,88	6,57
Carbon content	wt%	48,5	48,4	47,6	48,3
Hydrogen content	wt%	6,6	6,7	6,9	7,4
Nitrogen content	wt%	2,9	3,8	5,4	6,5
Oxygen content	wt%	32,8	34,9	33,4	31,1
Kjeldahl-N content	wt%	2,9	3,8	5,0	6,2

1.3 Objectives

The goal of this investigation is the determination, for 4 different samples of jatropha press cake, of:

- the biogas yield per tonne dry matter;
- the biogas yield per tonne organic matter;
- the time-integrated* concentrations of CO_2 and H_2S and
- the concentration of methane and the heating value of the biogas*;
- the ratio of C:N in the samples;

- the pH value of the digestate after completion of the digestion;
- characteristic components for digestion in the biomass

These parameters are essential starting values in the design of a digester for this feedstock.

* It must be remarked that at this scale and this batch wise way of operation it is not possible to determine the instantaneous biogas composition, as the total gas production rate is too low for this. These results serve as an indication to see if the digestion process runs well and to see if harmful H_2S is being produced. The methane content, and therewith the heating value, is calculated from the concentrations of CO_2 and H_2S.

2 Approach and methodology

Digestate from the pilot digestion installation of a large Dutch sugar factory was applied as a starting material for the reaction. Apart from active sludge (methanogenic bacteria) this digestate contained undigested biomass (sugar beet pulp). In order to determine the gas production from the jatropha press cake the total gas production per reactor was corrected for the gas production of the undigested beet pulp at the start of the experiments.

During the first eight days the gas production from only the starting material was monitored per digester. This reveals the gas production of the different reactors compared to each other. It turned out that:

- the biogas yield of the starting material in digester 1 is 1,07 times the biogas yield of digester 5;
- the biogas yield of digester 2 is 1,05 times the biogas yield of digester 5;
- the biogas yield of digester 3 is 1,06 times the biogas yield of digester 5 and
- the biogas yield of digester 4 is 1,02 times the biogas yield of digester 5.

The first four digesters were fed with the jatropha press cake samples and number 5 was used as a blank. In this way an accurate correction was possible. The blank was fed with cellulose.

Throughout the report cubic meters (m^3) are used to measure the gas volume. Unless otherwise specified, normal conditions are meant (ambient temperature and atmospheric pressure, T = 293 K and p = 1 bar).

3 Results

3.1 Results

The amount of biogas that was produced over time was recorded per reactor. The biogas yield curves are printed in the figures A-1 to E-1 in the appendices. The total results of the digestion experiments, both measured and calculated, are summarized in the table below.

Table 3-1 Total results of digestion experiments (= calculated)*

	cumulative biogas yield [m3/kg dm]	cumulative biogas yield [m3/kg om]	biogas CO2 [vol%]	biogas H2S [mg/m3]	biogas CH4 [vol%]*	cumulative CH4 yield* [m3/kg dm]	carbon conversion [wt%]*	pH digestate	C:N ratio
sample 1 (9 mm 100%)	0,84	0,89	16%	<0,18	84%	0,70	85%	7,58	17
sample 2 (7 mm 100%)	0,89	0,94	18%	<0,18	82%	0,73	90%	7,58	13
sample 3 (7 mm 67%)	0,84	0,89	17%	<0,18	83%	0,70	86%	7,58	9
sample 4 (7 mm 33%)	0,95	1,01	17%	<0,18	83%	0,78	96%	7,54	7

3.2 Discussion

The total (cumulative) biogas yield was measured in litres and then expressed in m^3 biogas per kilogram of dry matter and per kilogram of organic matter. The concentration of CO_2 and H_2S was measured at several intermediate stages (see Table 3-2) as the biogas had to be vented away from time to time. From these measurements the average CO_2 concentration was calculated. This average CO_2 concentration was used to calculate the CH_4 content of the biogas. The carbon conversion was calculated by dividing the mass of carbon atoms in the biogas by the mass of carbon in the press cake that was supplied. The carbon mass in the press cake was taken from the ALGE measurements (Table 1-1) as wt% d.m.; the carbon mass in the biogas follows by multiplying the amount of biogas (expressed in mol) with the molar mass of carbon (12 g/mol) under the assumption that every molecule in the biogas contains 1 carbon atom, i.e. the biogas consists fully of CH_4 and CO_2.

It must be emphasised that the CO_2 concentration in the table above is only relevant for the small-scale reaction that was performed in this research. Key numbers from Ingenia's database of digestion research show that digestion of these samples at pilot scale and industrial scale would have given a CO_2 concentration in the biogas of about 40-50%. Hence the CH_4 content would be 50-60% and the LHV about 18-22 MJ/kg. It is this value that must be calculated with in dimensioning a full scale biogas

installation on jatropha press cake. The value of about 30 MJ/kg that would be calculated from the composition above is not realistic for large-scale installations.

For the sake of completeness the intermediate measurements of the CO_2 concentration are included in table 3-2 below.

Table 3-2 measurements of gas concentrations at intermediate stages

sample	[CO_2] after 71 h	[CO_2] after 245 h	[CO_2] after 841 h	[CO_2] after 1466 h	[H_2S] *
1	19%	15%	16%	12%	< 0.18 mg/m^3
2	20%	17%	18%	13%	< 0.18 mg/m^3
3	20%	17%	15%	11%	< 0.18 mg/m^3
4	20%	17%	16%	11%	< 0.18 mg/m^3

In all cases the H_2S concentration was below the odour threshold of 0,18 mg/m^3 and below the detection limit of the measuring devices used. The H_2S concentration in the produced biogas is so low that it won't pose a problem for any known application.

4 Conclusions and recommendations

The most important conclusion that is allowed, based on the experiments done, is that the samples can be digested. Some important remarks must be made:

- The rate of decomposition and hence the biogas production rate of the 4 samples is low (it took 60 days residence time to get this biogas yield);
- The total biogas production per gram of biomass “as received” is fairly high (as expected) because of the high content of organic matter;
- The concentration of CO_2 that is measured, does not correspond to larger installations. Hence the CH_4 yield mentioned in this report (about 0,7 m^3/kg dm for all four samples) is not realistic in industrial practice. A CH_4 content of 50-60% (CH_4 yield about 0,5-0,6 m^3/kg) and an LHV for the biogas of 18-22 MJ/kg must be expected.
- A 60 days residence time is too short to convert the digestable fraction completely into biogas (the cumulative gas yield curves did not tend towards an asymptote);
- The difference in cumulative biogas yields between the 4 samples is significant.

If the intention is to erect a large-scale digester for jatropha press cake, it is recommended to repeat this experiment at a pilot scale in a (semi) continuous way of processing to determine an optimum in biogas production and to make a better comparison with current reality (industrial digestion). As the gas production rate is rather low, it can be recommended in this stage already to choose a large digester size for a long residence time in any practical scale digestion of jatropha press cake that may be considered, or alternatively to be confident with a lower biogas yield at a shorter residence time.

APPENDIX A Data digester 1

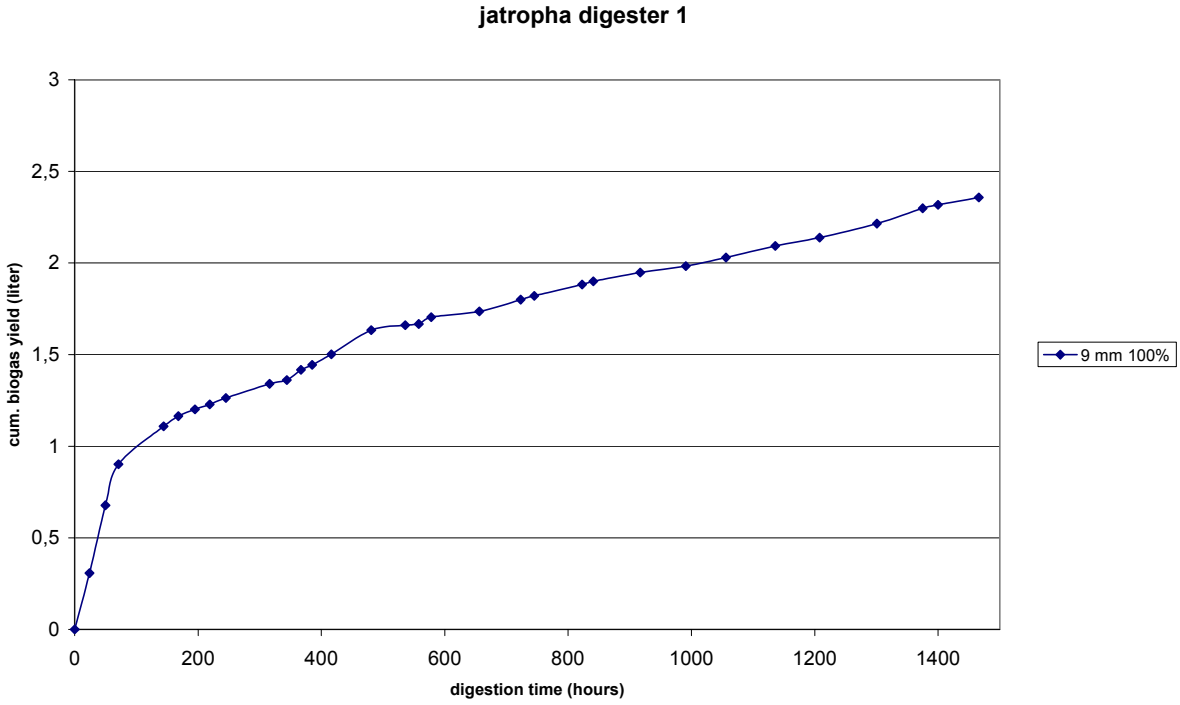

Figure 4-1 Biogas production curve digester 1 (sample 1)

uren totaal:	uren jatropha	Datum	vergister 1 Gasopbrengst	Gasopbrengst cumulatief	gecorrigeerde cumulatieve gasopbrengst
0		18-09-06 14:42	0	0	
20		19-09-06 10:50	0,33	0,33	
42		20-09-06 8:50	0,41	0,74	
66		21-09-06 8:50	0,45	1,19	
90		22-09-06 8:50	0,36	1,55	
162		25-09-06 9:00	0,71	2,26	
186	0	26-09-06 8:50	0,19	2,45	0
210	24	27-09-06 9:00	0,50	2,95	0,31
236	50	28-09-06 11:10	0,50	3,45	0,68
257	71	29-09-06 8:15	0,31	3,76	0,90
330	144	2-10-06 9:30	0,40	4,16	1,11
354	168	3-10-06 9:45	0,12	4,28	1,16
381	195	4-10-06 13:05	0,08	4,36	1,20
405	219	5-10-06 12:15	0,07	4,43	1,23
431	245	6-10-06 14:10	0,10	4,53	1,26
502	316	9-10-06 13:15	0,26	4,79	1,34
530	344	10-10-06 17:00	0,16	4,95	1,36
553	367	11-10-06 16:05	0,12	5,07	1,42
571	385	12-10-06 9:40	0,06	5,13	1,44
602	416	13-10-06 16:05	0,1	5,23	1,50
667	481	16-10-06 9:05	0,25	5,48	1,63
722	536	18-10-06 16:05	0,2	5,68	1,66
744	558	19-10-06 14:30	0,07	5,75	1,67
764	578	20-10-06 10:45	0,08	5,83	1,70
842	656	23-10-06 17:00	0,14	5,97	1,74
909	723	26-10-06 12:05	0,15	6,12	1,80
931	745	27-10-06 9:50	0,02	6,14	1,82
1009	823	30-10-06 15:30	0,17	6,31	1,88
1027	841	31-10-06 9:25	0,05	6,36	1,90
1103	917	3-11-06 13:55	0,09	6,45	1,95
1177	991	6-11-06 15:30	0,1	6,55	1,98
1242	1056	9-11-06 9:00	0,1	6,65	2,03
1322	1136	13-11-06 16:55	0,16	6,81	2,09
1394	1208	16-11-06 16:20	0,1	6,91	2,14
1487	1301	20-11-06 14:00	0,14	7,05	2,21
1561	1375	23-11-06 15:45	0,17	7,22	2,30
1586	1400	24-11-06 17:00	0,03	7,25	2,32
1652	1466	27-11-06 10:55	0,05	7,3	2,36

APPENDIX B Data digester 2

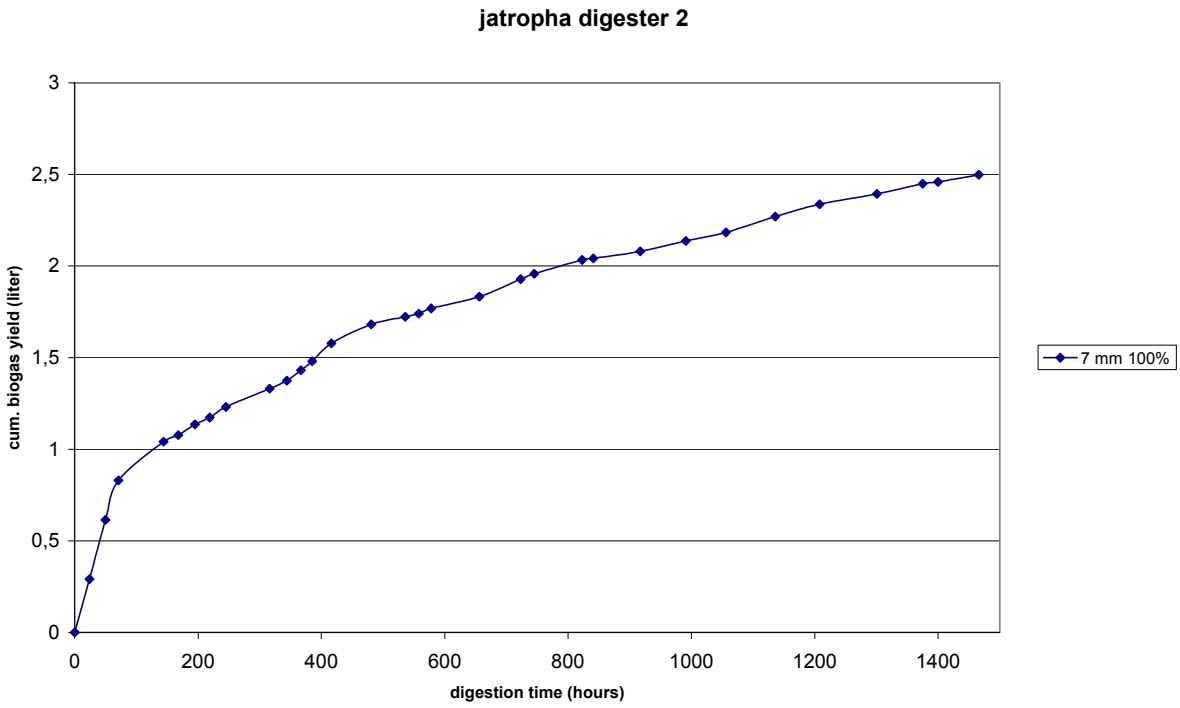

Figure 4-2 Biogas production curve digester 2 (sample 2)

uren totaal:	uren jatropha	Datum	vergister 2 Gasopbrengst	Gasopbrengst cumulatief	gecorrigeerde cumulatieve gasopbrengst
0		18-09-06 14:42	0	0	
20		19-09-06 10:50	0,36	0,36	
42		20-09-06 8:50	0,4	0,76	
66		21-09-06 8:50	0,41	1,17	
90		22-09-06 8:50	0,36	1,53	
162		25-09-06 9:00	0,69	2,22	
186	0	26-09-06 8:50	0,18	2,4	0
210	24	27-09-06 9:00	0,48	2,88	0,29
236	50	28-09-06 11:10	0,45	3,33	0,61
257	71	29-09-06 8:15	0,3	3,63	0,83
330	144	2-10-06 9:30	0,4	4,03	1,04
354	168	3-10-06 9:45	0,1	4,13	1,08
381	195	4-10-06 13:05	0,1	4,23	1,14
405	219	5-10-06 12:15	0,08	4,31	1,17
431	245	6-10-06 14:10	0,12	4,43	1,23
502	316	9-10-06 13:15	0,28	4,71	1,33
530	344	10-10-06 17:00	0,18	4,89	1,37
553	367	11-10-06 16:05	0,12	5,01	1,43
571	385	12-10-06 9:40	0,08	5,09	1,48
602	416	13-10-06 16:05	0,14	5,23	1,58
667	481	16-10-06 9:05	0,22	5,45	1,68
722	536	18-10-06 16:05	0,21	5,66	1,72
744	558	19-10-06 14:30	0,08	5,74	1,74
764	578	20-10-06 10:45	0,07	5,81	1,77
842	656	23-10-06 17:00	0,17	5,98	1,83
909	723	26-10-06 12:05	0,18	6,16	1,93
931	745	27-10-06 9:50	0,03	6,19	1,96
1009	823	30-10-06 15:30	0,18	6,37	2,03
1027	841	31-10-06 9:25	0,04	6,41	2,04
1103	917	3-11-06 13:55	0,08	6,49	2,08
1177	991	6-11-06 15:30	0,12	6,61	2,14
1242	1056	9-11-06 9:00	0,1	6,71	2,18
1322	1136	13-11-06 16:55	0,18	6,89	2,27
1394	1208	16-11-06 16:20	0,12	7,01	2,34
1487	1301	20-11-06 14:00	0,12	7,13	2,39
1561	1375	23-11-06 15:45	0,14	7,27	2,45
1586	1400	24-11-06 17:00	0,02	7,29	2,46
1652	1466	27-11-06 10:55	0,05	7,34	2,50

APPENDIX C Data digester 3

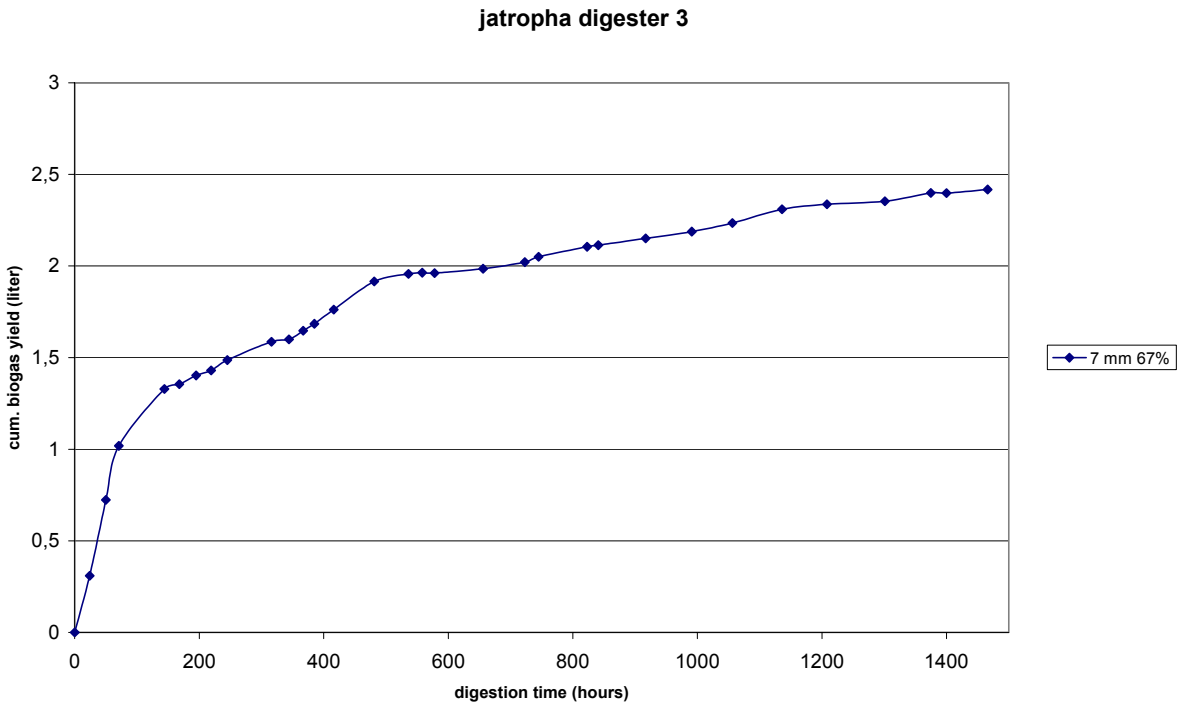

Figure 4-3 Biogas production curve digester 3 (sample 3)

uren totaal:	uren jatropha	Datum	vergister 3 Gasopbrengst	Gasopbrengst cumulatief	gecorrigeerde cumulatieve gasopbrengst
0		18-09-06 14:42	0	0	
20		19-09-06 10:50	0,36	0,36	
42		20-09-06 8:50	0,43	0,79	
66		21-09-06 8:50	0,43	1,22	
90		22-09-06 8:50	0,35	1,57	
162		25-09-06 9:00	0,67	2,24	
186	0	26-09-06 8:50	0,17	2,41	0
210	24	27-09-06 9:00	0,5	2,91	0,31
236	50	28-09-06 11:10	0,54	3,45	0,72
257	71	29-09-06 8:15	0,38	3,83	1,02
330	144	2-10-06 9:30	0,5	4,33	1,33
354	168	3-10-06 9:45	0,09	4,42	1,35
381	195	4-10-06 13:05	0,09	4,51	1,40
405	219	5-10-06 12:15	0,07	4,58	1,43
431	245	6-10-06 14:10	0,12	4,7	1,49
502	316	9-10-06 13:15	0,28	4,98	1,59
530	344	10-10-06 17:00	0,15	5,13	1,60
553	367	11-10-06 16:05	0,11	5,24	1,65
571	385	12-10-06 9:40	0,07	5,31	1,68
602	416	13-10-06 16:05	0,12	5,43	1,76
667	481	16-10-06 9:05	0,27	5,7	1,92
722	536	18-10-06 16:05	0,21	5,91	1,96
744	558	19-10-06 14:30	0,07	5,98	1,96
764	578	20-10-06 10:45	0,04	6,02	1,96
842	656	23-10-06 17:00	0,13	6,15	1,99
909	723	26-10-06 12:05	0,12	6,27	2,02
931	745	27-10-06 9:50	0,03	6,3	2,05
1009	823	30-10-06 15:30	0,16	6,46	2,11
1027	841	31-10-06 9:25	0,04	6,5	2,11
1103	917	3-11-06 13:55	0,08	6,58	2,15
1177	991	6-11-06 15:30	0,1	6,68	2,19
1242	1056	9-11-06 9:00	0,1	6,78	2,23
1322	1136	13-11-06 16:55	0,17	6,95	2,31
1394	1208	16-11-06 16:20	0,08	7,03	2,34
1487	1301	20-11-06 14:00	0,08	7,11	2,35
1561	1375	23-11-06 15:45	0,13	7,24	2,40
1586	1400	24-11-06 17:00	0,01	7,25	2,40
1652	1466	27-11-06 10:55	0,03	7,28	2,42

APPENDIX D Data digester 4

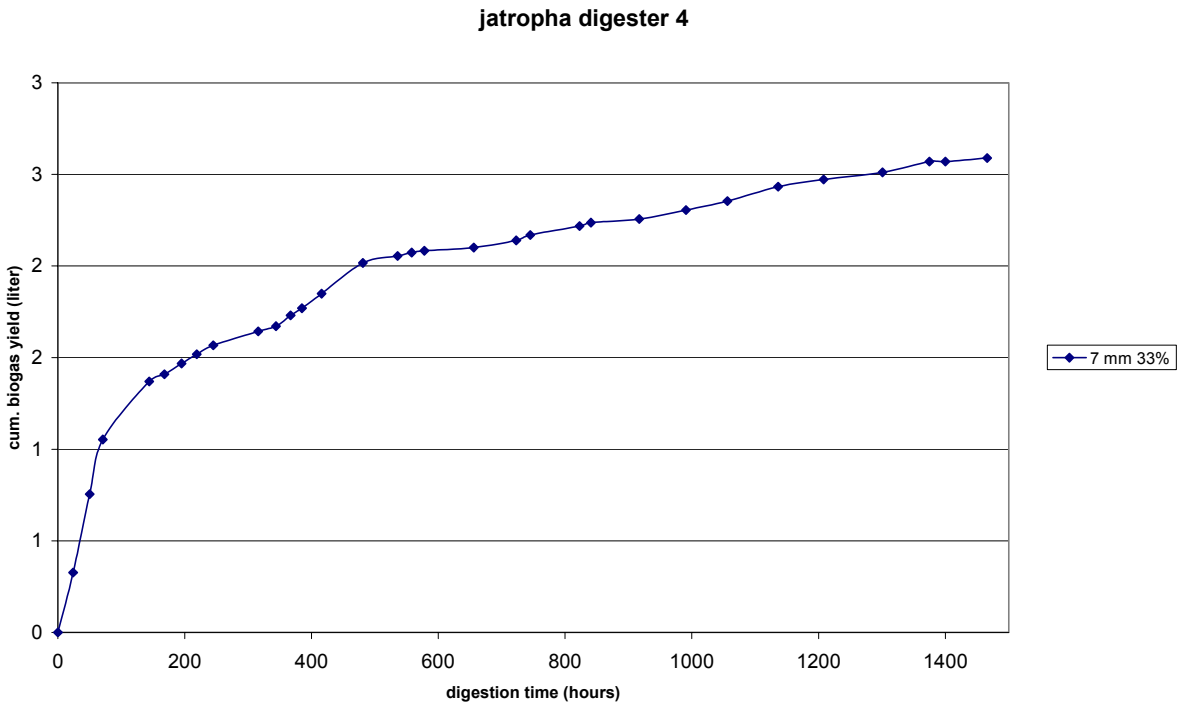

Figure 4-4 Biogas production curve digester 4 (sample 4)

uren totaal:	uren jatropha	Datum	vergister 4 Gasopbrengst	Gasopbrengst cumulatief	gecorrigeerde cumulatieve gasopbrengst
0		18-09-06 14:42	0	0	
20		19-09-06 10:50	0,37	0,37	
42		20-09-06 8:50	0,42	0,79	
66		21-09-06 8:50	0,40	1,19	
90		22-09-06 8:50	0,33	1,52	
162		25-09-06 9:00	0,64	2,16	
186	0	26-09-06 8:50	0,16	2,32	0,00
210	24	27-09-06 9:00	0,51	2,83	0,33
236	50	28-09-06 11:10	0,55	3,38	0,75
257	71	29-09-06 8:15	0,38	3,76	1,05
330	144	2-10-06 9:30	0,50	4,26	1,37
354	168	3-10-06 9:45	0,10	4,36	1,41
381	195	4-10-06 13:05	0,10	4,46	1,47
405	219	5-10-06 12:15	0,09	4,55	1,52
431	245	6-10-06 14:10	0,11	4,66	1,57
502	316	9-10-06 13:15	0,25	4,91	1,64
530	344	10-10-06 17:00	0,16	5,07	1,67
553	367	11-10-06 16:05	0,12	5,19	1,73
571	385	12-10-06 9:40	0,07	5,26	1,77
602	416	13-10-06 16:05	0,12	5,38	1,85
667	481	16-10-06 9:05	0,28	5,66	2,02
722	536	18-10-06 16:05	0,20	5,86	2,05
744	558	19-10-06 14:30	0,08	5,94	2,07
764	578	20-10-06 10:45	0,05	5,99	2,08
842	656	23-10-06 17:00	0,12	6,11	2,10
909	723	26-10-06 12:05	0,12	6,23	2,14
931	745	27-10-06 9:50	0,03	6,26	2,17
1009	823	30-10-06 15:30	0,15	6,41	2,22
1027	841	31-10-06 9:25	0,05	6,46	2,24
1103	917	3-11-06 13:55	0,06	6,52	2,26
1177	991	6-11-06 15:30	0,11	6,63	2,31
1242	1056	9-11-06 9:00	0,10	6,73	2,35
1322	1136	13-11-06 16:55	0,17	6,90	2,43
1394	1208	16-11-06 16:20	0,09	6,99	2,47
1487	1301	20-11-06 14:00	0,10	7,09	2,51
1561	1375	23-11-06 15:45	0,14	7,23	2,57
1586	1400	24-11-06 17:00	0,01	7,24	2,57
1652	1466	27-11-06 10:55	0,03	7,27	2,59

APPENDIX E Data digester 5

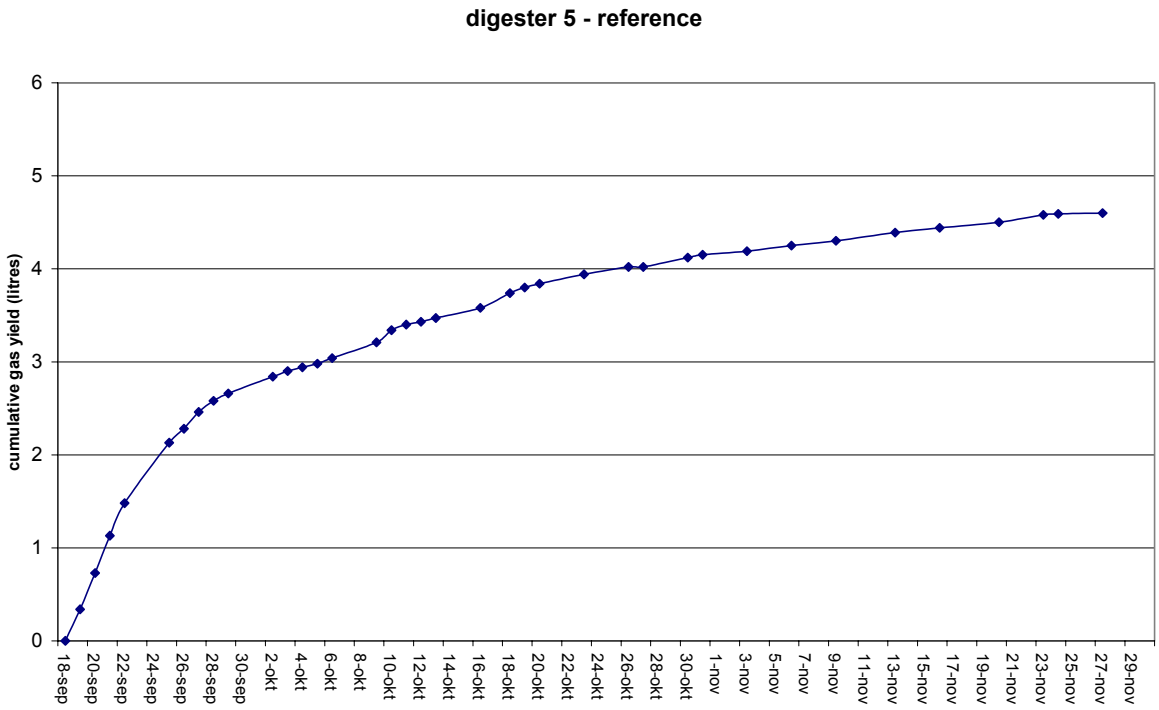

Figure 4-5 Biogas production curve digester 5 (blank)

uren totaal:	uren jatropha	Datum	vergister 5 Gasopbrengst	Gasopbrengst cumulatief
0		18-09-06 14:42	0,00	0
20		19-09-06 10:50	0,34	0,34
42		20-09-06 8:50	0,39	0,73
66		21-09-06 8:50	0,40	1,13
90		22-09-06 8:50	0,35	1,48
162		25-09-06 9:00	0,65	2,13
186	0	26-09-06 8:50	0,15	2,28
210	24	27-09-06 9:00	0,18	2,46
236	50	28-09-06 11:10	0,12	2,58
257	71	29-09-06 8:15	0,08	2,66
330	144	2-10-06 9:30	0,18	2,84
354	168	3-10-06 9:45	0,06	2,90
381	195	4-10-06 13:05	0,04	2,94
405	219	5-10-06 12:15	0,04	2,98
431	245	6-10-06 14:10	0,06	3,04
502	316	9-10-06 13:15	0,17	3,21
530	344	10-10-06 17:00	0,13	3,34
553	367	11-10-06 16:05	0,06	3,40
571	385	12-10-06 9:40	0,03	3,43
602	416	13-10-06 16:05	0,04	3,47
667	481	16-10-06 9:05	0,11	3,58
722	536	18-10-06 16:05	0,16	3,74
744	558	19-10-06 14:30	0,06	3,80
764	578	20-10-06 10:45	0,04	3,84
842	656	23-10-06 17:00	0,10	3,94
909	723	26-10-06 12:05	0,08	4,02
931	745	27-10-06 9:50	0,00	4,02
1009	823	30-10-06 15:30	0,10	4,12
1027	841	31-10-06 9:25	0,03	4,15
1103	917	3-11-06 13:55	0,04	4,19
1177	991	6-11-06 15:30	0,06	4,25
1242	1056	9-11-06 9:00	0,05	4,30
1322	1136	13-11-06 16:55	0,09	4,39
1394	1208	16-11-06 16:20	0,05	4,44
1487	1301	20-11-06 14:00	0,06	4,50
1561	1375	23-11-06 15:45	0,08	4,58
1586	1400	24-11-06 17:00	0,01	4,59
1652	1466	27-11-06 10:55	0,01	4,60