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Biogas Production from *Jatropha curcas* Press-Cake

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ABSTRACT

Seeds of the tropical plant *Jatropha curcas* (purge nut, physic nut) are used for the production of oil. Several methods for oil extraction have been developed. In all processes, about 50% of the weight of the seeds remain as a press cake containing mainly protein and carbohydrates. Investigations have shown that this residue contains toxic compounds and cannot be used as animal feed without further processing. Preliminary experiments have shown that the residue is a good substrate for biogas production. Biogas formation was studied using a semicontinous upflow anaerobic sludge blanket (UASB) reactor; a contact-process and an anaerobic filter each reactor having a total volume of 110 L. A maximum production rate of 3.5 m^3 m^{-3} d^{-1} was obtained in the anaerobic filter with a loading rate of 13 kg COD m^{-3} d^{-1}. However, the UASB reactor and the contact-process were not suitable for using this substrate. When using an anaerobic filter with *Jatropha curcas* seed cake as a substrate, 76% of the COD was degraded and 1 kg degraded COD yielded 355 L of biogas containing 70% methane.

Index Entries: Biogas; anaerobic filter; press cake; *Jatropha curcas*; methane production; renewable energy.

INTRODUCTION

Jatropha curcas (purge nut, physic nut) from the family of Euphorbiaceae is a common shrub of 3–6 m height. It originates in West India and is common in most arid areas of South America, Africa, and

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Asia. Because of its extraordinary drought resistance, *Jatropha curcas* has attained economical importance in areas with extreme climates and soil conditions.

Different parts of the plant are used in nature medicine. The oil obtained from the seeds is used in the soap industry and also as source of energy *(1)*. After transesterification of the oil it can be used as substitute for diesel oil, which is of interest for developing countries. This process is currently being carried out in Nicaragua as a development aid project.

The press cake of most oil seeds can be used as animal feed. Experiments have shown that the direct use of *Jatropha curcas* press cake for animal feed purposes is not possible because of the presence of toxic compounds such as curcin, a toxalbumin, and other equally negative substances such as phorbolic esters *(2)*.

Another possibility of utilizing organic wastes is to convert these to biogas by means of an anaerobic fermentation. In this microbiological process, organic matter is converted to biogas with a high content of methane as a utilizable energy source. Biogas technology is very suitable for the partial treatment of all organic wastes *(3)*. For example, in many food production processes, by-products cannot be used and recycled since they often contain organic and inorganic components. If directly discharged, the environment would be seriously polluted. In tropical countries, the anaerobic methane fermentation can be carried out at an ambient temperature, without any additional costs for heating. This relatively simple technology has made the biogas process interesting for developing countries.

The aim of this study was to find out the suitability of the press cake for anaerobic fermentation. The advantages of converting the press cake to biogas are a profitable removal of the press cake and the possibility to use the biogas directly within the process.

Environmental protection, improvements of hygiene and energy production are the relevant aspects of the biogas process. Biogas plants offering benefits for agriculture and the environment are used in Europe *(4–6)* as well as in Africa and Asia *(7,8)*. The usefulness of decentralized family size biogas plants has been investigated in many different countries. Whereas studies of biogas plants in India showed very good results *(9,10)*, other experiments in South Africa have pointed out some difficulties, such as the effects of shortages of water and manure, and the sceptisism of the users *(11)*.

MATERIALS AND METHODS

Substrate

An aqueous suspension of press cake residue from *Jatropha curcas* without any additonal chemicals was used as substrate. The composition of press residues of *Jatropha curcas* seeds is given in Table 1. The actual

Table 1
Composition (%) of Seed Press Cake from *Jatropha curcas*

dry weight	a.	90.86	b.	91.40
ash		6.03		6.55
org. dry weight		84.83		84.92
proteine		24.54		53.11
fat		6.40		6.32
fibre		32.26		5.60
starch		0.63		0.68
sugars		0.71		9.36
hemicellulose		5.55		1.94
cellulose		20.3		6.43
lignin		19.46		0.53

a. with Shells, b.without Shells

substrate suspension (30 g dry weight/L) was obtained after crushing the seed cake residue and seperation of the shells by sedimentation. Removal of the shells was necessary since in preliminary experiments the shells caused clogging in the pipes and were hardly fermentable. A composition of the substrate suspension is given in Table 2.

A COD:N:P ratio of 17.7:1:1according to R. Braun *(12)* showed that a well-balanced nutrient composition was present and inhibition of methane fermentation because of ammonia formation was not expected.

Inoculum

For seeding of both the batch reactor and the semicontinuous reactors, an active slurry from a biogas plant running on pig manure was used. In case of the batch reactors, an inoculum concentration of 20% (v/v) and in case of the semicontinously operated reactors an inoculum concentration of 30% (v/v) was used.

Batch Fermentation

Preliminary batch experiments were carried out in 3-L glass fermenters to investigate the fermentability of the substrate. Seed cake residues were used at different concentrations. Fermentation experiments with the shells of the seeds that consist of about 90% lignin should indicate whether they are degradable or not. In the course of these experiments, volcanic stones that were used as a support in the anaerobic filter were tested for their toxicity with overall gas production as indication. All experiments were carried out at 37°C. Gas production and the pH value of the substrate were measured daily; solids, total nitrogen, and ammonium were measured at the beginning and at the end of each series of experiments.

Table 2
Composition of the actual suspension of the seed press cake from *Jatropha curcas*

total nitrogen [g L^{-1}]	1.65
dry weight [g L^{-1}]	28.9
chemical oxygen demand (COD) [g L^{-1}]	29.2
organic dry material [g L^{-1}]	22.5
total phosphate [g L^{-1}]	1.73

Semicontinuous Fermentation

The formation of methane during the fermentation of seed cake was investigated in three different types of semicontinuously operated reactors utilizing the experimental data from the initial batch fermentation. All reactors were constructed from stainless steel tubing having a length of 2.87 m and a diameter of 0.25 m. The total volume of each bioreactor was 110 L. A schematic presentation of the reactor is given in Fig. 1. One reactor was operated as an anaerobic filter with a working volume of 73.8 L caused by partially filling with volcanic stones as a support. The second bioreactor was used as an UASB (upflow anaerobic sludge blanket)-reactor and the third as a contact-process type of bioreactor. The principal aim in all three reactor types was to increase the active biomass concentration in the fermenter either through growth of bacteria on the volcanic stones or by the formation of granular sludge or recirculation of biomass. In case of all three reactors, the substrate was added every 12 h.

Anaerobic Filter

The anaerobic filter was inoculated with 30% of its volume using digested sludge obtained from a biogas plant running on pig manure *(13)*. The homogenized substrate was added at a concentration of 16 kg COD m^{-3}. During the first 5 d period, the high content of shells not being significantly degraded caused trouble in the tubes. Subsequently, shells in the substrate were first removed by sedimentation.

During the start-up period, substrate was added during 17 d at a concentration of 0.20 and for 6 d at a concentration of 0.30 kg COD $m^{-3}d^{-1}$. From twentyfifth day on, depending on process stability, the loading rate was increased daily by 10% so that after 61 d a loading rate of 10.57 kg COD $m^{-3}d^{-1}$ was attained. In the following period, the loading rate was kept constant for 40 d since the pH value decreased and gas containing sludge came out of the gas outlet. This was probably caused by an increase of the solid concentration as a consequence of the short retention time. The loading was once again then slowly increased until a final value

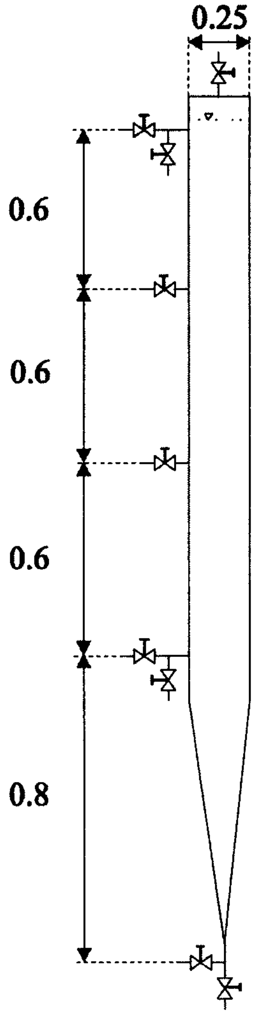

Fig. 1. Schematic presentation of the reactor (all dimensions in meters).

of 28 kg COD $m^{-3}d^{-1}$ was reached after 172 d. Because of the formation of gas containing sludge that could not be prevented and the decreasing pH the fermentation process broke down.

UASB-Reactor and the Contact Process

The start-up procedure for the UASB-reactor was the same as described for the anaerobic filter. After 37 d a loading rate of 1.42 kg COD $m^{-3}d^{-1}$ was attained, but a decrease of the pH value and a formation of gas containing sludge, which was blown out of the gas outlet, made a reduction of the loading rate to 0.73 kg COD $m^{-3}d^{-1}$ necessary. After a period of 60 d, a maximal loading rate of 2.93 kg COD $m^{-3}d^{-1}$ was achieved. A further increase of the loading rate failed because of the instability of the process.

However, in comparison to the UASB-reactor, the formation of gas-containing sludge in the contact process could be reduced by mixing the reactor from top to bottom. Washed out micro-organisms were recirculated daily. Within a period of 47 d, the loading rate could be increased to 2.65 kg COD $m^{-3}d^{-1}$ and until the sixty-eighth day to 6.80 kg COD $m^{-3}d^{-1}$. The further increase of the loading rate was not possible because of formation of gas-containing sludge and the instability of the process.

Analytical Methods

In order to enhance the performance of a biogas fermentation process and to avoid process failure, certain operating parameters must be controlled *(14)*. Therefore, the pH value and gas production were measured daily, Kjeldahl nitrogen, ammonia, total solids, and the organic dry material was measured weekly in samples taken from different heights of the reactor. Sampling was done at the bottom of the reactor and at a height of 0.80 m, 1.4 m, 2.0 m, and 2.6 m. The pH value was also measured daily with a pH meter at different heights of the fermenter in order to determine the pH distribution over the length of the reactor. The COD was analyzed according to Deutsche Einheitsverfahren (DEV) *(15)*. Removal efficiency was calculated from COD difference between the inflow and the effluent of the reactors. Kjeldahl nitrogen was determined after dissolving all samples with sulfuric acid and determining ammonium nitrogen using the distillation method *(15)*. Total and volatile solids, fixed and suspended solids were analyzed according to DEV *(15)*. Total phosphate was analyzed photometrically as vanadate-molybdate-complex according to DEV *(15)*. Total volatile acids were determined with gaschromatography (HP 4890 II) using a FID. Daily gas production was measured by a volumetric gas meter. The gas volume was corrected to standard temperature and pressure. The methane and carbon dioxide concentrations were analyzed by displacement of NaOH in a measuring pipe and absorption of the carbon dioxide by NaOH with an accuracy of 0.1%. H_2S was determined by the reaction of H_2S and lead acetate (colorless) to lead sulfide (yellow), in test tubes (Fa. Dräger, Lübeck,Germany).

RESULTS AND DISCUSSION

Batch Fermentation

Preliminary batch experiments showed the following results: The volcanic stones showed no inhibition on methane formation. One kilogram seed cake residue (organic dry material) gave 446 L of biogas. The theoretical total yield of gas as calculated from different substrate components would be 649 L kg^{-1} organic dry material. The nut shells from the seeds are only slightly degradable and gave only 37 L biogas per kg dry weight. This might be a result of their high lignin content *(16)*. Eighty percent of the substrate was converted during the initial 4 d 90% within 7 d.

Semicontinuous Fermentation

The UASB-reactor showed a high instability during the process as judged by the formation of gas-containing sludge. Variation of the loading rate always caused a decrease of the pH-value and an increase of the amount of volatile acids. The maximal loading was only 2.4 kg COD $m^{-3}d^{-1}$, the biogas yield was 0.31 m^3kg^{-1} $CODd^{-1}$, and the maximal production rate 1.4 m^3 per m^3 reactor volume and day. One reason for the instability in this case could be that the formation of granular sludge that is necessary for the successfull operation of this type of reactor, could not be observed during the whole experimental period. The presumable reason for this is the high content of solids in the substrate. In comparison with anaerobic sewage sludge as an inoculum, an UASB-reactor showed a 71% removal efficiency and produced 2.94 m^3 biogas per m^3 wastewater with an organic loading rate of 4.5 kg COD $m^{-3}d^{-1}$ *(17)*.

The determining factor for the fermentability of a substrate according to the UASB-principle is also the COD concentration. Highly loaded waste water, i.e., from either pectin or potato starch production, from baker's yeast production or from cellulose pulp plants having a COD higher than 8 kg m^{-3} is normally treated in fixed-bed reactors *(18)*. In accordance with these results it was not surprising that the anaerobic filter was more highly suitable for the treatment of the investigated substrate with a COD of about 29 kg m^{-3} than a sludge-bed reactor.

Bioreactors with a biomass recycle can be operated at higher reactor volume loading rates than UASB-reactors *(19)*. In the contact process used here a production rate of 1.9 $m^3m^{-3}d^{-1}$ and a biogas yield of 0.25 m^3kg^{-1} COD d^{-1} was obtained at a maximum loading rate of 6.8 kg COD $m^{-3}d^{-1}$.

The anaerobic filter allowed the highest loading rates to be attained in comparison to the other two types of bioreactors. The anaerobic filter system was most tolerant to variations of the loading rates, and the formation of gas-containing sludge could be reduced with recirculation of the rising solids to the bottom of the reactor. The influence of the loading rate on biogas yield and production rate of the anaerobic filter is shown in Fig. 2. Many different support materials have been described in the literature such as plastic tubes and organic supports *(20)*. Volcanic stones were chosen because of their availability in Nicaragua and the very low costs. They turned out be highly suitable for the retention of biomass in the reactor.

The highest yield of biogas could be obtained in the anaerobic filter. The yield of gas decreased with increasing loading rates to 0.35 m^3 $kg^{-1}COD$ d^{-1} at a loading rate of 13 kg COD $m^{-3}d^{-1}$. This value is equivalent to 71% of the calculated theoretical yield of gas. These results are in agreement with the values obtained for the COD removal. At a loading of 13 kg COD $m^{-3}d^{-1}$ a conversion of about 75% was measured. A comparison of the three reactor types is given in Table 3.

Compared to the results obtained by other authors *(21)*, treating cotton waste water (COD 5000 mg l^{-1}), the specific biogas yield was observed

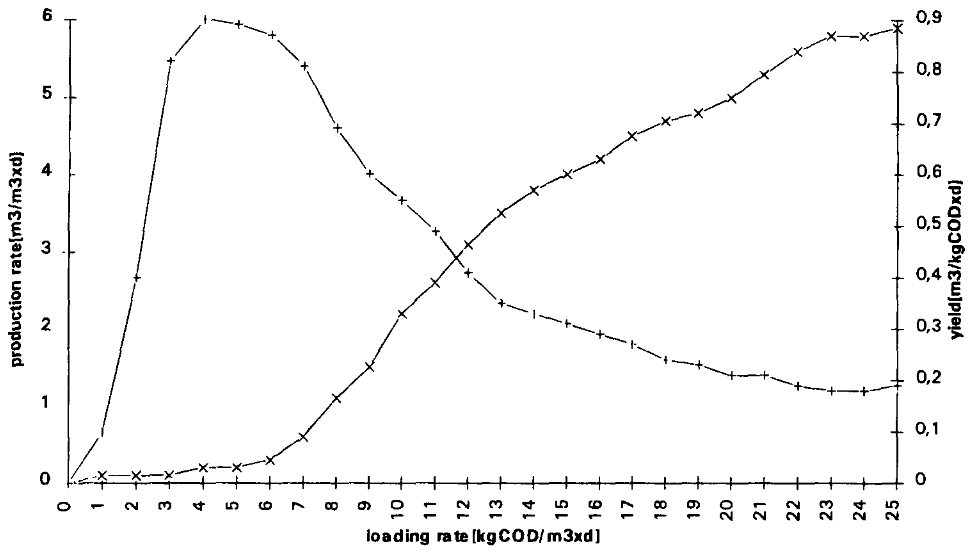

Fig. 2. Biogasproduction in an anaerobic filter with press cake residue as substrate: + biogas yield, x production rate.

Table 3
Comparison of the Results of an UASB-Reactor, a Contact Process and an Anaerobic Filter Using *Jatropha curcas* Seed Cake as a Substrate

	UASB-reactor	Contact process	Anaerobic filter
max. stable loading rate [kgCODm^{-3}d^{-1}]	2.4	6.8	13
biogas yield [m^{3}kg^{-1}CODd^{-1}]	0.31	0.25	0.35
production rate [m^{3}m^{-3}d^{-1}]	1.4	1.9	3 5
removal efficiency [%]	-	-	75

to be 0.44 to 0.48 m3 per kg COD removed for 2.0 d and 1.5 d retention time, respectively. Similary an upflow anaerobic reactor used for the treatment of palm oil mill effluent was operated with organic loads ranging from 1.2 to 11.4 kg COD m^{-3}d^{-1} and hydraulic retention times from 15 to 6 d. The overall substrate removal efficiency was up to 90%. Daily gas production varied in the range 0.69 to 0.79 m^3 kg^{-1} COD d^{-1} *(22).*

Although the active volume of the anaerobic filter was one-third less than that of the UASB-reactor and the contact system because of the volume of the volcanic stones, production rate was higher than the production rate in the UASB-reactor with a loading rate of 0.9 kg COD m^{-3} d^{-1} and higher than that of the contact process with a loading rate of 2.4 kg COD m^{-3} d^{-1}. A comparison of the maximum production rates between the three reactor types is given in Fig. 3.

When doubling the hydraulic retention time to 3.6 d using a stable loading rate, the anaerobic filter had a 20% higher production rate than before. The maximal production rate of 3.5 m^{3}m^{-3}d^{-1} was obtained at a maximum loading rate for a stable process of 13 kg COD m^{-3}d^{-1}. A further increase of

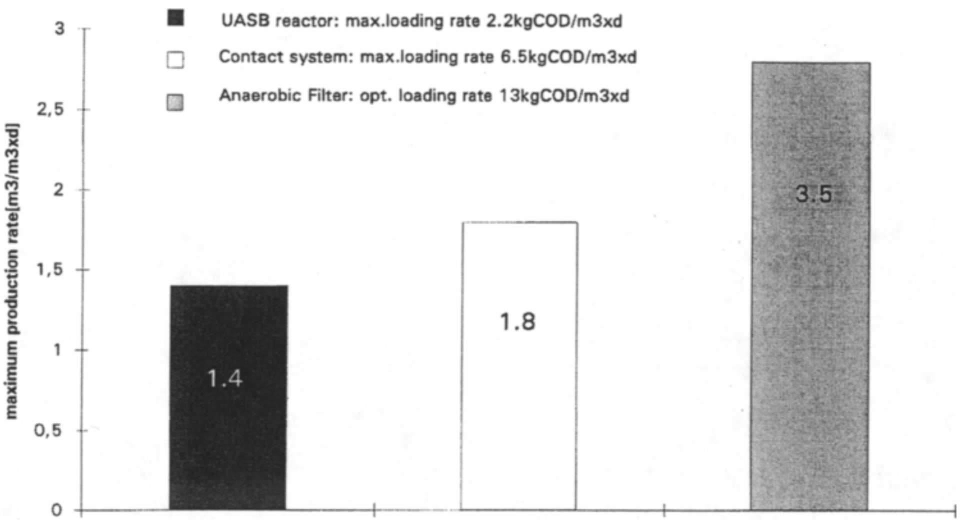

Fig. 3. Biogasproduction in three different bioreactor types: UASB, contact system, anaerobic filter. Comparison of production rates with press cake residue from *Jatropha curcas* as substrate.

production rate should be possible with a higher hydraulic retention time. The substrate concentration could not be increased in order to keep the sedimentability of the shells for their removal from the suspension. Appropriate removal of the shells before fermentation would allow higher substrate concentrations and this would lead to larger hydraulic retention times.

The highest concentration of ammonia measured in the anaerobic filter was 1630 mg L^{-1} at a loading rate of 13 kg COD m^{-3} d^{-1}. The major part of the nitrogen present in the substrate was converted to ammonia, but a concentration till 3000 mg L^{-1} shows no inhibition on methane fermentation according to Henze *(23)*. Micro-organisms can be adapted after a certain time to concentrations higher than 8000 mg^{-1} NH_4-N.

In judging the quality of biogas, the content of methane and H_2S is the determining factor. The content of methane will depend on the composition of the substrate and on the fermentation conditions. The percentage of CO_2 in biogas increases with decreasing pH values and with excessive activity of the hydrolytic and acetogenic micro-organisms since CO_2 cannot be completely used by methanogenic bacteria. In case of the anaerobic filter, the methane content varied between 54.2 and 72.4 V% during a 24 h period. A high correlation between pH-values during this interval and a certain effect of the time of substrate addition is observed. The methane content was always measured before substrate addition and was on an average 70.0 V%. In comparison, in the biogas obtained from cotton waste water a methane content of 67% was measured *(21)*.

The average content of H_2S in biogas was about 0.39% (v/v). Because of the corrosive effect of H_2S a reduction to 0.2% (v/v) before combustion

Table 4
Results of the Anaerobic Filter at a Loading Rate of 13 kg $CODm^{-3}d^{-1}$ Using *Jatropha curas* Seed Cake as a Substrate

Production rate $[m^3m^{-3}d^{-1}]$	3.5
Biogas yield $[m^3kg^{-1}CODd^{-1}]$	0.35
COD Removal efficiency [%]	75
methane content [%]	70
H_2S content [%]	0.39

would be necessary *(24)*. A content of 0.39% (v/v) H_2S in biogas corresponds to a concentration of less than 20 mg L^{-1} H_2S in solution according to Braun *(12)*. A toxic effect of H_2S on is not assumed since the methane fermentation is only inhibited by concentrations of H_2S above 50 mg L^{-1} *(25)*. A summary of all the results from the anaerobic filter is given in Table 4.

In many developing countries high population growth rates, the low economic growth, environmental issues such as deforestation, atmospheric pollution, and water depletion cause serious problems *(26,27)*. The provision of adequate and decentralized produced energy is crucial for the overall development prospects. A number of options for moving towards sustainable development are put forward. These options encompass, among others, biomass and biogas. Biogenic fuels supply at the moment approx 15% of the world's energy *(28)*, but in the future energy scenario these alternative gaseous fuels will most likely be the most important fuels.

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