

## High-rate biogas production from waste textiles using a two-stage process

Azam Jeihanipour<sup>a</sup>, Solmaz Aslanzadeh<sup>b</sup>, Karthik Rajendran<sup>b,\*</sup>, Gopinath Balasubramanian<sup>b</sup>,  
 Mohammad J. Taherzadeh<sup>b</sup>

<sup>a</sup> Department of Biotechnology, Faculty of Advanced Sciences and Technologies, University of Isfahan, Isfahan 81746-73441, Iran

<sup>b</sup> School of Engineering, University of Borås, 50190 Borås, Sweden

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### ABSTRACT

The efficacy of a two-stage Continuously Stirred Tank Reactor (CSTR), modified as Stirred Batch Reactor (SBR), and Upflow Anaerobic Sludge Blanket Bed (UASB) process in producing biogas from waste textiles was investigated under batch and semi-continuous conditions. Single-stage and two-stage digestions were compared in batch reactors, where 20 g/L cellulose loading, as either viscose/polyester or cotton/polyester textiles, was used. The results disclosed that the total gas production from viscose/polyester in a two-stage process was comparable to the production in a single-stage SBR, and in less than two weeks, more than 80% of the theoretical yield of methane was acquired. However, for cotton/polyester, the two-stage batch process was significantly superior to the single-stage; the maximum rate of methane production was increased to 80%, and the lag phase decreased from 15 days to 4 days. In the two-stage semi-continuous process, where the substrate consisted of jeans textiles, the effect of N-methylmorpholine-N-oxide (NMMO) pretreatment was studied. In this experiment, digestion of untreated and NMMO-treated jeans textiles resulted in 200 and 400 ml (respectively) methane/g volatile solids/day (ml/g VS/day), with an organic loading rate (OLR) of 2 g VS/L reactor volume/day (g VS/L/day); under these conditions, the NMMO pretreatment doubled the biogas yield, a significant improvement. The OLR could successfully be increased to 2.7 g VS/L/day, but at a loading rate of 4 g VS/L/day, the rate of methane production declined. By arranging a serial interconnection of the two reactors and their liquids in the two-stage process, a closed system was obtained that converted waste textiles into biogas.

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### 1. Introduction

The annual global production of end-of-life waste textiles is steadily increasing, causing an increasing concern regarding the impact of the disposal of this enormous amount of waste on the environment. In spite of textile waste in fact being a potentially rich source of energy and materials, the current normal routine to dispose of this waste is by incineration or as landfilling. Interestingly, of the world's total textile production, around 40% of the fiber consumption comprises cellulose [1], the same percentage as the average content of cellulose in lignocellulosic materials.

Waste textiles are mainly composed of cotton and viscose fibers, and holds, thanks to their cellulose content, a significant potential for

production of different biofuels, such as biogas [2]. For instance, in 2008, the influx of clothing and textiles to Sweden was 131,800 tons [3]. Assuming that the total amount of waste textiles in Sweden nearly equals the amount of imported clothing and textiles, and that 40% of the textile fibers consists of cellulose, approximately 53,000 tons of cellulose is wasted every year. A yield of 415 ml methane (at STP) per g cellulose implies that the amount of waste textiles produced in Sweden would suffice as substrate for producing more than 20 million Nm<sup>3</sup> of methane, equaling in the region of 4 TWh power per year; to be compared with 11 TWh estimated to be the biogas potential of ley crops, straw, potato, and sugar beet tops in Sweden [4].

Fossil fuels are currently dominating the global energy market. However, the growing world population along with diminishing fossil fuel reserves have resulted in a global interest in gradually shifting the energy source from fossil to alternative fuels [5,6]. In addition, environmental pollution caused by e.g. the dumping of waste materials in the environment, is one of the most important issues the world is facing today. Biogas, produced by means of anaerobic digestion of biological waste, is a renewable bioenergy and a potential alternative to petroleum-based fuels [7]. In addition

Abbreviations: CSTR, continuously stirred tank reactor; SBR, stirred batch reactor; UASB, upflow anaerobic sludge blanket bed; GC, gas chromatography; IC, ion-exchange chromatography; HPLC, high performance liquid chromatography; VFA, volatile fatty acid; AMPTS, automatic methane potential testing system.

\* Corresponding author. Tel.: +46 33 435 4855; fax: +46 33 435 4008.

E-mail address: [Karthik.Rajendran@hb.se](mailto:Karthik.Rajendran@hb.se) (K. Rajendran).

to the methane itself, biogas production holds the potential to minimize the waste pollution, thus protecting the environment [8]. From the perspective of resource efficiency, biogas production has a higher output–input energy ratio compared to, for example, current ethanol production systems [9]. Furthermore, in terms of emissions, biogas production might be better for the environment than incineration of waste [10,11].

Methane-rich biogas has different applications. It may serve as a source for heat, steam, and electricity, and can be further upgraded to vehicle fuel, or for production of chemicals. It may also be used as a household fuel for cooking and lighting, or in fuel cells. Taking all these aspects into account, being a well-established technology for generating bioenergy, biogas production is one of the most environmentally beneficial processes for replacing fossil fuels [8,12]. Furthermore, development of new technologies has facilitated biogas production for combined heat and power (CHP) systems in small scale ( $\leq 100$  KWe) [13]. Thanks to biogas production being an uncomplicated process, which is a significant advantage, it can be located near the place where waste is produced, and the waste producers can be the end-users of the biogas, hence evading problems related to transport of both wastes and biogas. Such systems (so-called on-farm biogas plants), have in Germany been commercially installed in thousands [14], mainly using biomass from agriculture. Apart for the conventional waste streams such as municipal solid wastes (MSW) and manure, the recent trend includes the pretreated lignocellulosic biomass, wooden fractions and agricultural residues are used for biogas production [15].

However, the potential of other available biological wastes, such as cellulosic waste textiles, as substrate in small-scale biogas plants, has not been adequately investigated. From the literature, there was very little work that has been focused on the textile waste as a substrate for anaerobic digestion. Previous work includes pre-treating waste textile containing cellulosic blend fibers and high crystalline cellulose fibers in a batch assay [16,17] in order to increase the biogas production. Additionally, textile wastes were never tested in a two-stage process.

The present study is focused on investigating the feasibility of using waste cellulosic textile fibers for production of biogas, employing different processes and comparing their efficacy.

## 2. Materials and methods

The first step was to examine a one-stage batch process (i.e. in an SBR) and a two-stage (i.e. in an SBR and a UASB) anaerobic digestion, using two different substrates (viscose and cotton fibers, both blended with polyester fibers), without separating the cellulosic fibers. In the two-stage process, textile was converted into biogas in a closed system, which was obtained by arranging a serial inter-connection of the liquids of both reactors. However, previous attempts have been made to separate cellulose from mixed fibers [18] by e.g. dissolution of cellulose [19] and subsequently regenerating it. Jeihanipour et al. [16] recently developed a process for separating the cellulosic part from waste textiles, using an environmental friendly cellulose solvent, i.e. N-methylmorpholine-N-oxide (NMMO), in order to facilitate the production of biogas or bioethanol from waste textiles [17]. Hence, the second step of the present study comprised a semi-continuous two-stage anaerobic digestion process, comparing biogas yield from NMMO-treated and untreated cotton-based waste textiles, at different organic loading rates.

### 2.1. Materials and inoculums

The three waste textiles used in the present study were woven textiles: orange (50% polyester, 50% cotton), blue (40% polyester, 60% viscose), both provided by local shops in Borås (Sweden), and also

used blue jeans textiles (100% cotton). Prior to the experiments, the first two textiles were cut into small pieces (approximately  $2.5 \times 2.5$  cm<sup>2</sup>), while the jeans textiles were ground into fine materials. NMMO was provided by BASF (Ludwigshafen, Germany) as a 50% water solution.

The inoculum used in the CSTRs and SBRs was obtained from a 3000-m<sup>3</sup> municipal solid waste digester, operating under thermophilic (55 °C) conditions (Borås Energy & Environment AB, Sweden). The granulated anaerobic sludge used as seed in the UASB reactors was provided from a UASB reactor treating municipal wastewater in Hammarby Sjöstad (Stockholm, Sweden).

### 2.2. Pretreatment procedure

For pretreatment of the ground jeans textiles, the NMMO solution was concentrated to 85% in a rotary vacuum evaporator (Laborota 20, Heidolph, Schwabach, Germany), equipped with a vacuum pump (PC 3004 VARIO, Vacuubrand, Wertheim, Germany). The concentrate was mixed with ground jeans textiles (6% w/w dry matter) in an oil bath at 120 °C for 3 h under atmospheric conditions, using a mixer for continuous blending in order to dissolve the cellulose [17]. The resulting cellulose–NMMO solution was then added to boiling water while mixing continuously, thereby regenerating the dissolved cellulose. Using a vacuum filter, the regenerated cellulose was separated from the NMMO–water solution, and washed with hot water. The washed cellulose was stored wet at 4 °C until used for anaerobic digestion.

### 2.3. Experimental setup

#### 2.3.1. Reactors

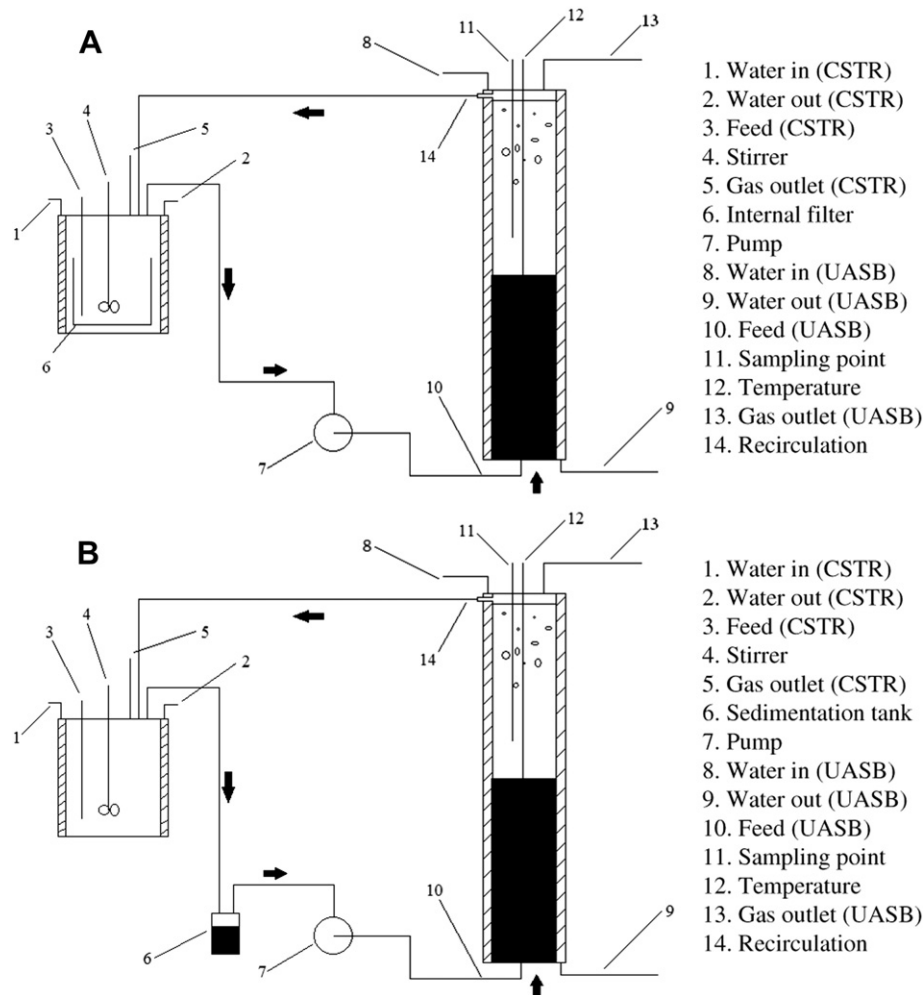
Two types of reactors, a continuous flow stirred tank reactor (CSTR), modified for batch process as stirred batch reactor (SBR) and an upflow anaerobic sludge blanket bed (UASB) reactor, both made of polymethylmethacrylate (PMMA), were used in different configurations. The CSTR had a working volume of 3 L (an internal diameter of 18.5 cm and a height of 18.5 cm), while the working volume of the UASB was 2.25 L (an internal diameter of 6.4 cm and a height of 70 cm). Temperature was set at 55 °C for the CSTR and SBR, and at 34 °C for the UASB, using a temperature-controlled water-bath with water recirculation through the reactor's double jacket. Both types of reactors were equipped with a feed inlet, a liquid sampling point, an outlet, and a gas line to the gas measuring system, which had a gas sampling port. The CSTR and SBR were equipped with an impeller for continuous mixing of the contents. The inlet of the UASB reactor had a mesh to avoid large particles entering the system (Fig. 1B).

#### 2.3.2. Reactor seeding and start up

The UASB reactors were seeded with 1.28 L of granular anaerobic sludge. The remaining volume of the reactor was filled with water. Upon receipt, the inoculum for the CSTR was stored in an incubator at 55 °C for three days, to degrade easily degradable organic matter still present in the inoculum, and to remove dissolved methane. The CSTR and SBR were filled with 2.5 L of inoculum and 0.5 L of nutrient solution to set the C:N:P:S ratio to 500:20:5:3, in accordance with the cellulose concentration in the beginning of the experiment. The final nutrient concentrations for the basic medium (1 g cellulose/L, containing inorganic macronutrients) were (in mg/L): NH<sub>4</sub>Cl (76.4), KH<sub>2</sub>PO<sub>4</sub> (5.18), MgSO<sub>4</sub>·7H<sub>2</sub>O (0.27), CaCl<sub>2</sub>·2H<sub>2</sub>O, (10.00), and trace nutrients, 1 ml/L [20].

#### 2.3.3. Reactor configurations

In the present study, the efficacy of single-stage and two-stage batch processes as well as a two-stage continuous process for



**Fig. 1.** Schematic diagram of the CSTR–UASB combined system with internal recirculation. (A) Batch process equipped with internal filter in the SBR and (B) semi-continuous process equipped with sedimentation tank.

anaerobic digestion of waste textiles was examined. The arrangements of the reactor facilities are schematically illustrated in Fig. 1. In the one-stage batch process, an SBR was used as a digester. In the two-stage batch process, the SBR was serially connected with a UASB reactor. Liquid effluent from the SBR was continuously pumped to the UASB reactor at a rate of 3 L/day. At this flow rate, the hydraulic retention times (HRT) in the SBR and the UASB reactors were ca. 24 and 18 h, respectively. A peristaltic pump with a tube diameter of 1.02 mm was used. The effluent of the UASB reactor was continuously fed back to the SBR. The SBR of the two-stage batch process was equipped with a cylindrical filter around the impeller, and the textile wastes were placed inside the filter. The liquid outlet of the SBR passed through the filter, while the textiles were retained within the SBR. With this filter, the polyester part of the textile could be recovered after the process was completed.

The configuration of the reactors in the two-stage continuous process was quite similar to that in the two-stage batch process. The difference was the removal of the internal filter in the SBR, placing a sedimentation tank (with a volume of 100 ml) in liquid line to the UASB, before the pump, to settle the large particles (Fig. 1).

#### 2.4. Experimental operations

The batch processes were conducted by feeding the SBRs with cotton/polyester (50/50) and viscose/polyester (60/40) textiles, to

establish a cellulose concentration of 20 g/L. After 25 days, the process was interrupted, and the remaining textiles were separated, washed, and studied in a stereomicroscope. In the semi-continuous processes, 2 two-stage systems were used to digest ground jeans textiles and NMMO-treated jeans textiles. The OLR of the process was increased stepwise from 2 up to 4 g VS/L/day. Once a day, a certain amount of substrate was fed into the CSTR, in accordance with the desired OLR. The HRT of UASB was controlled by changing the speed of the pump in the beginning of each step. Each OLR was continued for more than three HRTs in the CSTR, when a steady state condition was attained. Table 1 describes the conditions of the different steps during the process, including the OLRs and their respective HRTs, flow rates, and durations.

No solids were withdrawn from the reactors during the experimental period in neither the batch nor the continuous processes, except when sampling for the analyses. Liquid and gas were sampled twice a week throughout the running process, and the

**Table 1**

Organic loading rates (OLR), hydraulic retention times (HRT) in the CSTR and the UASB reactor, and phase durations, determined at different stages.

Stage	OLR (g VS/L/day)	HRT in CSTR (day)	HRT in UASB (day)	Duration (day)
1	2.0	10.0	7.50	30.0
2	2.7	7.5	5.62	30.0
3	4.0	5.0	3.75	15.0

volumes of produced gas were recorded. The gas samples were analyzed directly by gas chromatography (GC), while the liquid samples were stored in the freezer at  $-20\text{ }^{\circ}\text{C}$  for later analyses.

2.5. Analytical methods

The cellulose content of the substrates was determined according to the method provided by the National Renewable Energy Laboratory in the USA [21]. The gas production was recorded by using the Automatic Methane Potential Testing System (AMPTS, Bioprocess control AB, Lund, Sweden), whose function is based on water displacement and buoyancy, with a measuring resolution of 13 ml. The instrument was equipped with a laptop computer and the volumes of produced gas vs. time were recorded for each reactor. The composition of the biogas produced during anaerobic digestion was measured using a gas chromatograph (Auto System Perkin Elmer, Waltham, MA) equipped with a packed column (Perkin Elmer,  $6' \times 1.8''\text{OD}$ , 80/100, mesh) and a thermal conductivity detector (Perkin Elmer) set to  $200\text{ }^{\circ}\text{C}$ . The inject temperature was set to

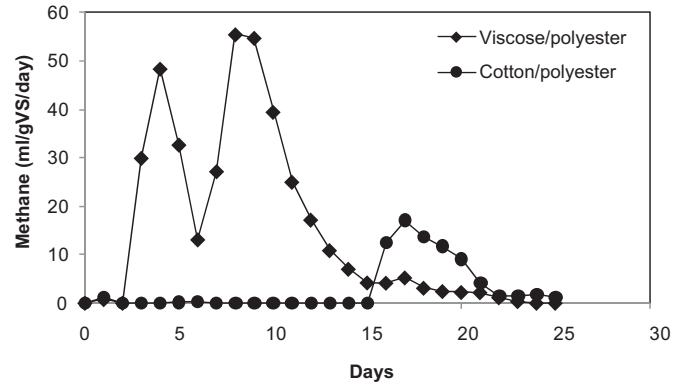


Fig. 2. Rate of methane production from (◆) viscose/polyester and (●) cotton/polyester in the single-stage batch process.

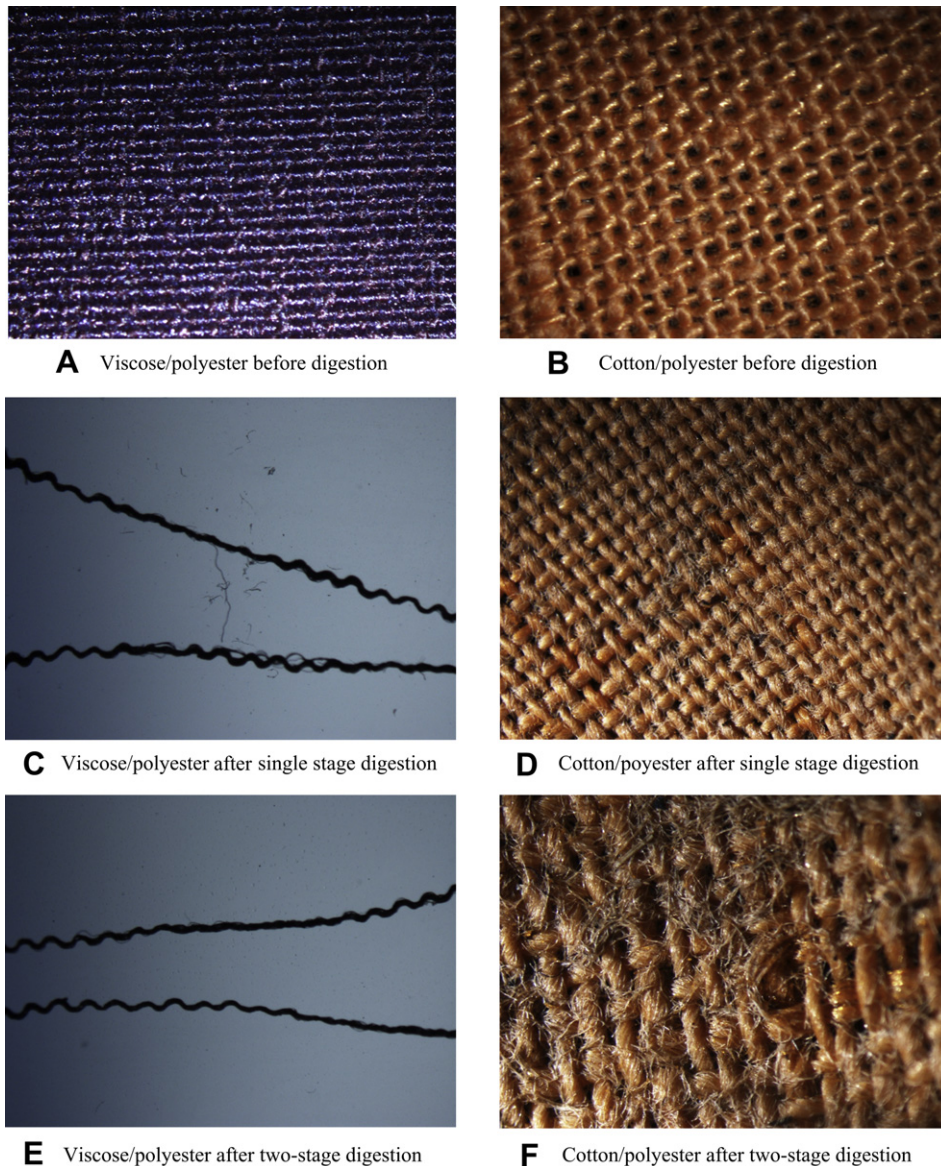


Fig. 3. Stereomicroscopic pictures of viscose/polyester and cotton/polyester before and after single-stage and two-stage digestions.

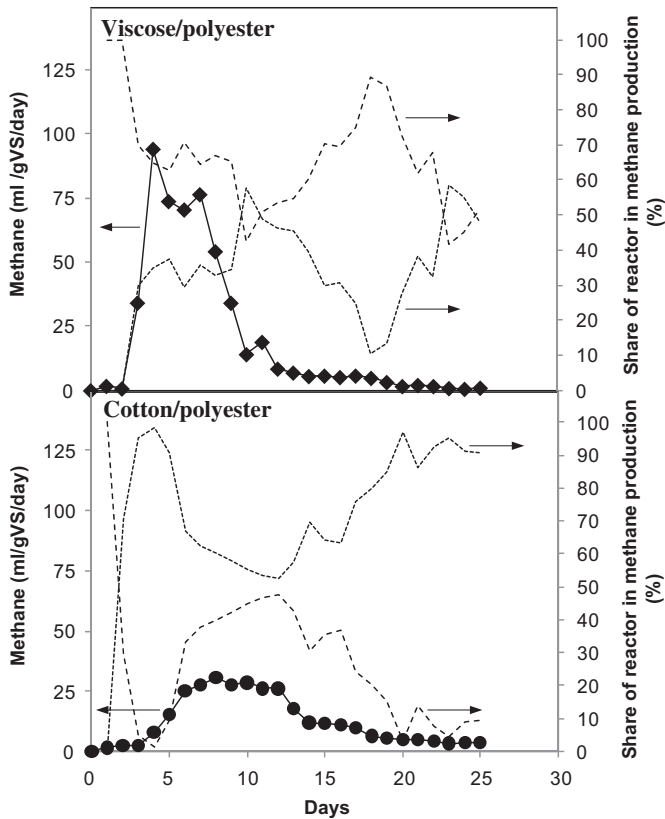


Fig. 4. Rate of methane production from (◆) viscose/polyester and (●) cotton/polyester in the two-stage batch process. The amount of methane, produced in each reactor, presented as (---) % share of the CSTR and (- - -) % share of the UASB.

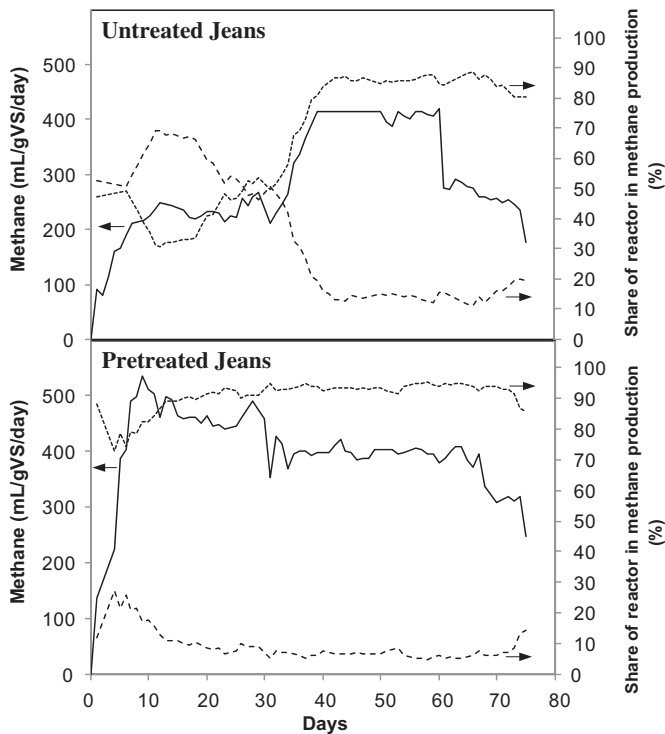


Fig. 5. Rate of methane production from untreated and pretreated jeans textiles in the semi-continuous process. The different line styles represent: (—) methane volume, (---) % share of methane produced in the CSTR and (- - -) % share of methane produced in the UASB.

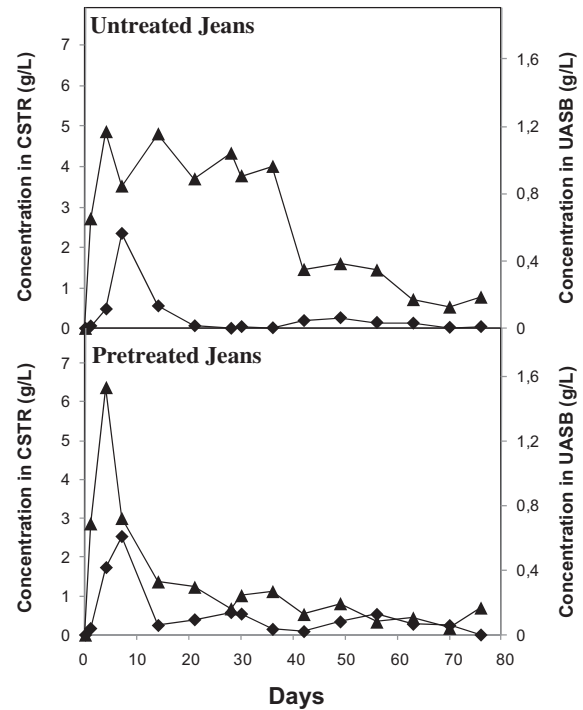


Fig. 6. Variation of VFA concentrations in (▲) CSTR and (◆) UASB during digestion of untreated and pretreated jeans in the semi-continuous process.

150 °C, and the oven temperature to 75 °C. The carrier gas used was nitrogen, kept at a maintained pressure of 0.70 bar and a flow rate of 40 ml/min at 60 °C. A 250- $\mu$ l pressure-tight gas syringe (VICI, Precision Sampling Inc., LA) was used for the gas sampling.

Liquid samples were centrifuged at 10,000 rpm for 10 min, and solid particles were removed by filtration through a 0.2- $\mu$ m filter prior to analyses for pH, soluble chemical oxygen demand (COD), and volatile fatty acid (VFA) concentrations. The COD was measured using an HACH apparatus equipped with a UV-vis Spectrophotometer (HACH, Germany), using Digestion Solution COD vials (operating range 0–15,000 mg COD/L). The VFA concentrations, comprising acetic acid, propionic acid, butyric acid, isobutyric acid, valeric acid, and isovaleric acid, were analyzed by HPLC (Waters 2695, Waters Corporation, Milford, MA, USA) with a UV detector (Waters 2414), utilizing an ion-exchange column (Aminex HPX-87H Bio-Rad, Hercules, CA) working at 60 °C, and using 5 mM sulfuric acid as eluent, with a flow of 0.6 ml/min. The macronutrients, ammonium and potassium, were analyzed with an Ion Chromatograph (Metrohm, Herisau, Switzerland), using a cation column holding an eluent flow rate of 1 ml/min; the pressure was set at 7–9 MPa, and the temperature was 35–40 °C. The eluent solution consisted of 4 mmol tartaric acid and 0.75 mmol dipicolinic acid per L water. Samples were diluted with the eluent, and pH was adjusted to 2–3. They were then centrifuged at 10,000 rpm for 4 min, and filtered through a 0.45  $\mu$ m filter prior to injection. The texture of the textiles before and after batch digestion was studied, using a NIKON stereomicroscope (SMZ800, Tokyo, Japan) fitted with a C-DSD230 camera.

### 3. Results and discussion

#### 3.1. Batch digestion

##### 3.1.1. Single-stage anaerobic digestion in the SBR

The cumulative methane produced during 25 days of single-stage anaerobic digestion in the SBR is presented in Fig. 2. The

**Table 2**

The COD, the ratio of methane to carbon dioxide in the CSTR and the UASB reactor, and the COD removal efficiency of the UASB reactor, during digestion of untreated and pretreated jeans at different stages. The efficacy of the UASB-digesters (expressed as COD removal efficiency in percent) was calculated by dividing the difference between COD inlet and outlet with the COD inlet.

Substrate	OLR (g VS/L/day)	COD (mg/L)		COD removal efficiency (%)	Ratio of methane to carbon dioxide	
		CSTR	UASB		CSTR	UASB
Untreated jeans	2.0	6169 ± 1348	2027 ± 626	66.8 ± 9.7	1.46 ± 0.09	5.33 ± 0.55
	2.7	4395 ± 1234	1198 ± 235	72.0 ± 3.3	1.97 ± 0.26	5.40 ± 0.46
	4.0	2873 ± 562	1276 ± 185	56.6 ± 10.5	2.17 ± 0.04	5.03 ± 0.20
Pretreated jeans	2.0	4377 ± 652	2019 ± 638	53.4 ± 14.4	1.92 ± 0.29	4.72 ± 0.53
	2.7	3212 ± 416	1822 ± 239	42.2 ± 11.5	2.14 ± 0.13	4.18 ± 0.15
	4.0	2833 ± 267	2220 ± 305	22.9 ± 14.5	2.18 ± 0.06	3.63 ± 0.31

reactor fed with viscose/polyester textiles had a 2-day-long lag phase, whereas in the reactor fed with cotton/polyester, the corresponding lag phase before gas production was triggered was about 15 days long. The longer lag phase may be due to the different texture of cotton/polyester, providing a smaller contact surface area for cellulolytic microorganisms to work on. Once the biogas production started, the production rate from cotton/polyester was slower than from viscose/polyester. The theoretical methane yield was calculated according to Buswell formula [22], which is 415 ml/g VS for cellulose. Within 12 days of gas production from viscose/polyester, more than 80% of the theoretical yield of methane was acquired, to be compared with the 17% yield from cotton/polyester, gained during the 10 days following the lag period (Fig. 2). Differences in contact surface area, molecular structure of cellulose, and chemistry of the dyes and reagents covering the cotton and viscose fibers, are possible reasons for this huge difference in digestion outcome between the viscose/polyester and cotton/polyester waste textiles utilized in these experiments. The maximum rate of biogas production from viscose/polyester reached 55 ml/g VS/day after 8 days.

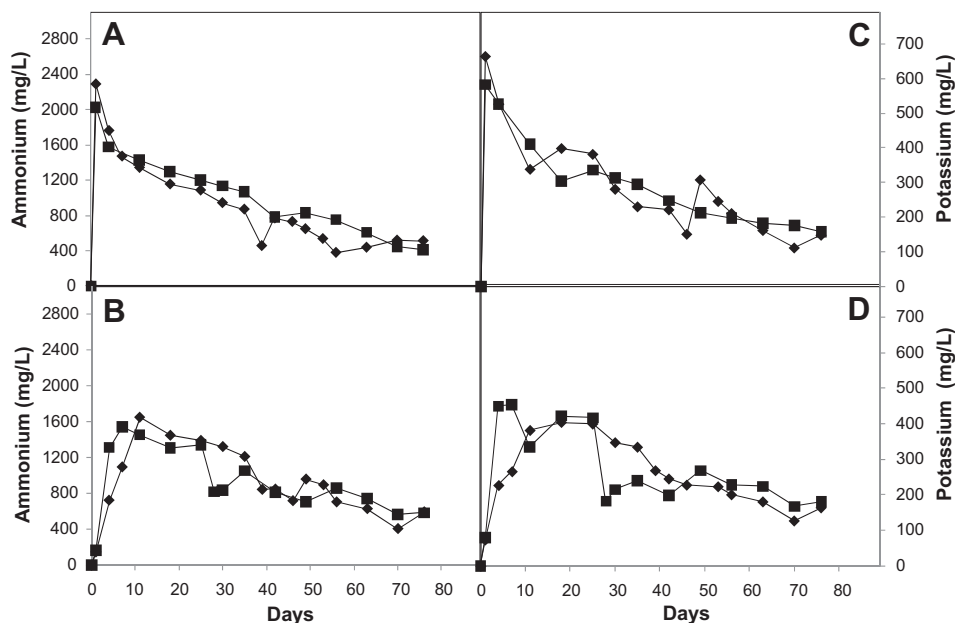
The appearances of the textiles used in the batch process, as shown in stereomicroscopy before and after digestion, are presented in Fig. 3. The microscopy revealed that viscose/polyester had disintegrated fibers compared to cotton/polyester, consequently facilitating the process of degradation of viscose/polyester by

microorganisms. Single-stage and two-stage digestion of viscose/polyester (Fig. 3C and E) did not differ much, while degradation of cotton/polyester was more successful in the two-stage process than in the single-stage digestion (Fig. 3D and F).

### 3.1.2. Two-stage anaerobic digestion

The cumulative methane production acquired over 25 days, and its share (in percentage) of the SBR and the UASB reactor, is presented in Fig. 4. Though the gas production from both textiles (cotton/polyester and viscose/polyester) started after three days, the initial rate of biogas production from viscose/polyester was superior compared to the initial production rate of cotton/polyester, where it was low in the single-stage digestion as well. The total gas production from viscose/polyester did not differ between the single-stage process and the two-stage process.

Jeihanipour et al. [16] reported that under batch conditions, methane yield from untreated cotton/polyester was lower than from viscose/polyester; after six days of digestion, only about 4.95% of the theoretical yield was acquired from cotton/polyester, while 36.28% was produced from viscose/polyester. In the present study, however, biogas production from cotton/polyester reached 40% of the theoretical yield after 10 days of digestion, while 80% of the theoretical yield was attained from viscose/polyester after 12 days of digestion. The maximum rate of methane production from



**Fig. 7.** Variation of ammonium and potassium concentrations in the CSTR (A and C) and the UASB (B and D) during digestion of (◆) untreated and (■) pretreated jeans in the semi-continuous process.

cotton/polyester in the two-stage process reached 30.6 ml/g VS/day on day 8, and in the single-stage process, this textile produced a maximum methane volume of merely 17 ml/g VS/day, which was achieved only on day 17. This implies an 80% yield increase when using the two-stage process rather than the single-stage SBR (during this time period). This efficacy increase may be due to a more efficient conversion of VFA into methane in the UASB process than in a single-stage process. The SBR produced the major share of gas from both textiles, as compared to the UASB reactor. Since the textiles were neither milled nor pretreated, the contact surface area available for the microorganisms' degradation of the textiles was probably low.

### 3.2. Semi-continuous two-stage anaerobic digestion

#### 3.2.1. Gas production

The accumulated volume of methane produced per gram VS per day from untreated jeans and NMMO-pretreated jeans are presented in Fig. 5, which also illustrates the share of methane production in the CSTR and the UASB reactor, expressed as percentage. The volume of methane produced per gram VS per day increased with an increased OLR. Comparing biogas production from untreated and treated jeans revealed that an OLR of 2 g cellulose/L/day (stage 1) produced 200 ml/g VS/day from untreated jeans, but more than 400 ml/g VS/day from treated jeans, i.e. pretreatment increased methane production with 100%. Furthermore, an accumulation of VFA in the CSTR with untreated jeans evidently resulted in a lower methane production during the experimental period (Fig. 6). When increasing the OLR from 2.0 to 2.7 g VS/L/day, the microorganisms adapted to the conditions, resulting in acquiring 91% of the theoretical methane yield from untreated jeans and 96% from treated jeans. However, increasing the OLR to 4.0 g VS/L/day did not improve the methane production any further. The CSTR was responsible for the largest share of the total methane production (~90%) from treated jeans, most likely as a result of the enzymatic degradation of the cellulosic part of the textiles being facilitated by the pretreatment.

The only comparable information found was the application of rumen microorganisms in combination with a high-rate UASB using filter paper cellulose as substrate produced 438 ml/g VS/day, which is equivalent to 98% of the theoretical yield. This slightly higher yield compared to present study could be explained by presence of ruminant bacteria which have high efficiency to hydrolyze even the cellulose based material with high-crystallinity [23].

#### 3.2.2. COD and COD removal efficiency

The chemical oxygen demand (COD) during the operation, measured from the influent and effluent of the UASB reactor, is illustrated in Table 2. The efficacy of the UASB digestion of untreated jeans textiles increased from 66.8% to 72.3% when increasing the OLR from 2.0 to 2.7 g VS/L/day and decreasing the HRT from 10 to 7.5 days. A further increase in the OLR to 4.0 g VS/L/day decreased, however, the COD removal efficiency to 56.6% when processing untreated jeans in the UASB reactor. When treated jeans textiles were used, a decrease trend (from 53.4% to 22.9%) in the COD removal efficiency was observed when the OLR was increased from 2.0 to 4.0 g VS/L/day. The COD in the CSTR decreased with increasing OLR and decreasing HRT, when processing untreated as well as pretreated jeans textiles. Furthermore, during the entire process, the COD in the UASB reactor processing untreated jeans decreased from 2027 mg/L to 1276 mg/L while a more stable COD around 2000 mg/L was established when digesting pretreated jeans. An increase in the OLR decreased the COD in the CSTR, regardless of textiles having undergone pretreatment or not. However, an increase in OLR resulted in a decreasing efficiency of the COD removal in the UASB reactor.

Mahmoud et al. [24] studied the COD removal efficiency of a single-stage UASB reactor and a combined UASB-digester system, and found that the COD removal efficiency was higher in the combined system (30%) compared to the single-stage system (about 5%). In the present study, for the complete process, the average COD removal efficiency was 65.1% for untreated jeans and 39.5% for treated jeans.

#### 3.2.3. Effect of nutrients

The concentrations of the macronutrients (ammonium and potassium) during the process, are illustrated in Fig. 7. The nutrient concentration decreased with time in both digesters for both textiles which was due to activity of the cells to remove COD, produce biogas and of course some biomass. The final ammonium concentrations (Fig. 7A and B) in the CSTR and the UASB were in the range of 600–800 mg/L, while the potassium concentration (Fig. 7C and D) decreased to around 150 and 200 mg/L in the CSTR and the UASB, respectively. After decreasing the nutrients to a minimum level, in spite of no nutrients or water being added to or removed from the system, the two-stage process was still able to produce biogas with a good yield. This observation may be because of endogenous metabolism which causes autohydrolysis of some of the biomass present in the reactor.

#### 3.2.4. Ratio of methane to carbon dioxide

The ratio of methane to carbon dioxide in each stage is illustrated in Table 2. By increasing the OLR from 2.0 to 4.0 g VS/L/day, the ratio in the CSTR increased from 1.46 to 2.17 and from 1.92 to 2.18 for treated jeans and untreated jeans, respectively. However, the ratio for untreated jeans in the UASB reactor was stable at around 5 throughout the experimental period, while the ratio for treated jeans during the same period, decreased from 4.72 until 3.63. Accumulation of VFA in the CSTR with untreated jeans, increased the ratio of methane to carbon dioxide in the UASB reactor. Pretreatment with NMMO decreased the crystalline structure, and increased the digestibility of the material. Consequently, during digestion of treated jeans, the ratio of methane to carbon dioxide increased in the CSTR but decreased in the UASB reactor. The treated jeans textiles were easily degraded to methane in the CSTR with no accumulation of VFA.

## 4. Conclusions

The comparison of single-stage and two-stage batch digestion processes for producing biogas from cotton/polyester and viscose/polyester with no pretreatment or milling revealed that gas production efficacy is highly affected by the molecular structure of the textile. In the semi-continuous process, pretreatment of textiles had a significant effect on the biogas production, due to a more accessible surface area for the degradation of cellulose fibers. Despite the complex structure of cotton/polyester, the initial rate of biogas production was higher and the lag phase shorter in the two-stage batch process, in comparison with the single-stage CSTR. It was furthermore concluded that when digesting treated or untreated jeans textiles, the semi-continuous two-stage process was able to handle a high OLR with a shorter HRT, in the CSTR as well as in the UASB reactor.

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