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## Industrial and Classical Pre-treatment for Enhanced Biomethane Production from Cellulosic Materials in Absorbent Hygiene Products

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### Abstract

The introduction of absorbent hygiene products (AHPs) has helped improve the quality of life for people, but AHPs also cause environmental problems; diaper manufacturing, distribution, use, and waste management produce 2.7 Mt of greenhouse gases (GHG) per year. This study measures the biochemical methane potential (BMP) of cellulose recycled from AHPs and aims to evaluate the impact of pre-treatments applied to AHP-derived cellulose on biogas production. The cellulose from AHPs has been divided into four categories with different pre-treatment or no pre-treatment: new cellulose (UC), treated post-consumer cellulose (TC), treated unused cellulose (TU), and cellulose soaked in NaOH, using microcrystalline cellulose (MC) as a benchmark. The measure of biogas production shows that TC resulted in the highest biogas production yield (426.9 mL/g volatile solid (VS)), while cellulose treated with NaOH resulted in the highest methane production yield (309.7 mL/g VS). The results of TC were mostly in line with microcrystalline cellulose.

**Keywords:** Alkaline, Biogas production, Diaper, Microcrystalline cellulose, Waste management.

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### 1. Introduction

Absorbent hygiene product (AHP) is a generic term for a broad group of products such as diapers and personal care wipes. They are generally composed of a plastic fraction (mainly polyethylene and polypropylene), a so-called super-absorbing polymer (generally sodium polyacrylate), and cellulosic material [1]. Global AHP production, especially for baby diapers (which make up 95% of the market), is challenging to quantify. In Europe alone, approximately 21 billion units are used annually, totalling nearly 700 million kilograms of raw materials. Disposed AHPs amount to 2-7% of total municipal solid waste in Europe and in the USA respectively, and even reaching 15% in other countries; most of them (between 68% and 80%) are landfilled [2] and the rest is generally incinerated. Moreover, the global market of AHPs is soaring, driven by high birth rates, increased affordability and the awareness of health issues for infants in developing countries [3], and a continuous increase in life expectancy in industrialized countries.

Landfilling and incineration harm the environment, emitting pollutants like particulate matter, greenhouse gases (methane, carbon dioxide, etc.), and contaminants such as metals and chemicals, leading to surface and groundwater pollution, acidification, and toxicity for humans and animals. In addition, landfilling or incineration

requires vast spaces [4]. Recovering and reusing polyethylene and polypropylene from AHPs can significantly benefit the environment [9]. Some technologies have already been developed for the recovery of plastics from AHP waste streams, mostly involving shredding, sterilization, and separation [3,6].

The cellulose portion, apart from plastics, could be used for paper production or making lower-grade AHPs like bed or animal absorbent sheets. However, this has limited market potential due to abundant alternative paper waste [7]. Another option for this latter portion of AHPs is the exploitation of its energy potential by anaerobic digestion (AD). Biogas, a gaseous biofuel, mainly composed of methane and carbon dioxide, is produced by microorganisms breaking down organic molecules in anaerobic conditions. By processing cellulose from AHPs in anaerobic digesters, these emissions would be prevented and biogas would be employed as a versatile energy carrier, before or after upgrading to biomethane. Compared to the incineration of AHPs, such energy exploitation would have several environmental advantages, including the preliminary recovery of plastic polymers and the reduced environmental impact of biogas, as an alternative to combustion [8].

Generally, biopolymers such as cellulose, hemicellulose, and lignin are non-optimal substrates as they are difficult for most anaerobic microorganisms to digest basically due to the insolubility of lignocellulosic materials [7]. Moreover, the specific composition of the biomass to be digested is also a factor, as lignin may have an inhibitory effect on AD, while crystalline cellulose is too hydrophobic to be readily hydrolyzed. Cellulose is indeed a linear polymer of  $\beta(1\rightarrow4)$  linked D-glucose where hydrogen bonds may form among several hydroxylic groups on the same chain or close-by chains. Depending on the orientation of cellulose chains there may be high crystallinity or low crystallinity (amorphous) regions; the higher the crystallinity is, the more the cellulose is recalcitrant to AD. To overcome these barriers, several pre-treatments have been investigated to reduce the recalcitrance of lignocellulose to biological processes, which are summarized in Table 1.

Another notable feature of this biomass is the inclusion of super-absorbent polymers (SAP) alongside lignocellulosic material. SAP typically consists of granular sodium polyacrylate ( $C_3H_3NaO_2$ ) and is crucial in AHP for enhanced comfort, dryness, and preventing leakage of excreta. It has the capacity to retain water at levels ranging from 100-1000% of its own weight, surpassing the liquid-holding capability of cellulose alone [27–29], which may inhibit anaerobic digestion in wet conditions by sequestering most of the water and immobilizing nutrients and microorganisms. While plastic components and lignocellulosic materials can be mechanically separated, SAP first needs to be dehydrated via a chemical process to reduce its volume before it can be removed. Nonetheless, a portion of SAP, up to 40% in weight, may still be found in the cellulosic fraction of recovered materials [5,6].

In 2018 a novel, integrated method for recovering lignocellulosic materials from AHP was patented [6]. The described apparatus combines sterilization through autoclave treatment, shredding, drying, and separating lignocellulosic material from plastic, producing cellulose with a purity of 85-95%. This process, coupled with a subsequent, thorough decontamination and sterilization step, ensures the safety of the material for further use. As shown in the introduction, shredding, oxidative, and liquid hot water pre-treatments, which have been already applied using the patented methodology, have a positive effect on the methane yield of AD of lignocellulosic materials, reducing crystallinity and increasing availability by also cleaving a portion of glycosidic bonds. Sodium hydroxide is one of the most studied, inexpensive, and effective pre-treatments for lignocellulosic biomasses. Strong bases can cleave lignin crosslinks by reducing crystallinity and polymerization, increasing porosity and surface area, and freeing cellulose and hemicellulose from lignin.

This study aims to measure the biomethane potential (BMP) of cellulose recycled from AHPs and evaluate pre-treatment effects on biomethane yield. Three biomass samples from treated AHPs, new ones, and unused diapers were examined. Alkaline treatments were tested alongside existing sterilization and separation methods to gauge their impact on biomethane production. Specifically, the study compares the biomethane yield of industrially treated cellulose with alkaline-treated cellulose, a widely recognized effective pre-treatment for anaerobic digestion substrates, revealing similar biogas yields.

**Table 1** Pretreatment method and parameters for reducing the recalcitrance of lignocellulose.

Methods	Parameters	Results	Reference
Physical	Liquid hot water	100-230 °C, up to a few hours	7-220% increase [10]
	Explosive	160-260 °C, 0.7-4.8 MPa, up to a few minutes	40% increase [10-13]
	Shredding	Particle size 0.003 - 30 mm	30% increase [10,13,14]
	Extrusion	0.45-3.5 MPa, a few minutes, 60-90 °C	8-70% increase [13,15-17]
Chemicals	Alkaline	NaOH, Ca(OH) <sub>2</sub> , CaO, KOH, loading 1-10% w/w, 170 °C, up to 10 days	3-230% increase [10,13,18,19]
	Acid	H <sub>2</sub> SO <sub>4</sub> , HCl, HNO <sub>3</sub> , H <sub>3</sub> PO <sub>4</sub> , acetic acid, loading 1-4% w/w, 170° C, up to 30 days	20-200% increase [10,13]
	Oxidative	H <sub>2</sub> O <sub>2</sub> , loading 1-4% w/w	33-120% increase [41,20,21]
	Wet oxidation	180-220 °C, oxygen pressure up to 1.2 MPa, few minutes	34-136% increase [21-23]
Biological	Fungal	White, brown, soft rot fungi, basidiomycetes, aerobic conditions, up to 8 weeks at 28-37 °C	500% increase [24]
	Microbial	Yeast, cellulolytic bacteria, aerobic conditions, up to 20 days at 20-55 °C	25-96% increase [24,25]
	Enzymatic	Laccase, pectinase, cellulase, hemicellulases, up to 24 hours at 37 °C	35% increase [10,13,21,26]
	Ensilaging	Mixture of fermentative bacteria, yeast, fungi, anaerobic conditions for 7 weeks	15% increase [13]

## 2. Materials and methods

### 2.1 Cellulose pretreatments

Untreated, unused cellulose from AHPs, in this study referred to as “untreated cellulose” (UC), was selected from a combination of baby diapers from several producers readily found on the market. To confirm the substrate for AD process was using cellulose in original state, diapers were manually opened, and cellulose was shredded by hand. At the same time, granular SAPs, were removed from samples until SAP content was down to 35±5% of cellulose weight in samples.

Recycled, post-consumer, and post-industrial cellulose from AHPs was provided by the manufacturer Fater S.p.A. The material had been previously pre-treated by the manufacturer, undergoing mechanical shredding up to about 0.5 cm flakes, autoclave sterilization, and chemical treatments with a strong oxidant for fiber degradation [6]. Two distinct samples of recycled cellulose were considered in this study, that is treated, post-consumer cellulose (TC), and treated, unused cellulose (TU). The same treatments were given for both biomass types.

To identify the best procedure to enhance the BMP of cellulose from AHPs, some UC samples underwent additional pre-treatments: for this purpose, NaOH was used, being the most common and consolidated methodology, as discussed in the introduction. Several alkaline pre-treatment tests were carried out to identify the optimal operational conditions, by varying concentration, temperature, and time. According to these preliminary tests and previous studies samples were kept soaking for 120 hours at 60±2 °C after the addition of NaOH in an amount equal to 10% by weight of the sample. After these further pre-treatments, biomasses were also washed with distilled water to remove the NaOH residue in the biomass and dried out at 105 °C for 6 hours.

### 2.2 Biomass characterization and Inoculum

All samples were characterized in terms of total solids (TS) and volatile solids (VS). TS and VS were determined according to EPA (1684) method: total solids were measured by weight loss in an oven by heating the sample up to 105 °C for 12 hours, and as such evaporating all water content; similarly, volatile solids were measured by weight loss in a furnace by heating the sample up to 550 °C for 2 hours, leaving only incombustible ashes. To evaluate cellulose content, fluff samples were also characterized by thermogravimetric analysis (TGA).

By measuring the weight loss of the sample as a function of temperature, it is possible to deduce the cellulose content of the fluff samples. TGA was performed on 7 mg samples in an N<sub>2</sub>-saturated controlled atmosphere starting at 25 °C, and gradually rising to 1,000 °C. The inoculum was wet cattle manure which was collected from the biogas plant of Leonessa Società Agricola s.r.l., located in Leonessa (Rieti, Italy). Batch vials were kept in constant conditions until no more biogas production could be measured (40 days), when digestate was collected, characterized, and immediately used for the experiments described.

### 2.3 Biomethanation potential test

BMP tests were carried out in wet conditions in 500 mL lab-scale batch reactors, loaded with 100 g of samples and 100 g of distilled water. Each test was carried out in triplicate together with a blank test containing only the inoculum; microcrystalline cellulose (MC) (Sigma-Aldrich®) was also tested in triplicate to check for the effect of the experimental setup on results. Batch reactors were kept at a constant temperature of  $39.0 \pm 0.5$  °C and the anaerobic digestion process was prolonged until no more biogas was produced, or for at least 21 days. Inoculum to biomass ratio was equal to 3 considering VS for each trial. Biogas production was measured volumetrically by water displacement, and biogas composition was measured by gas chromatography with an INFICON 3000 (SRA Instruments, Cernusco sul Naviglio, Italy). Full details are described in [30]. Accuracy and limit of quantitation are 0.01% v/v and 0.1% v/v, respectively.

Six reactor triplicates, five for each type of biomass (UC, TC, TU, NaOH-TC, and MC) and another one for blanks were readied, as shown in Table . The inoculum to biomass ratio was set “3:1” considering Volatile Solids (VS) content, as previous batch trials with similar biomass composition yielded the maximum result with this proportion.

**Table 2** Summary of volatile solids (VS) content in biomass and inoculum.

Biomass Type	Biomass [g VS]	Inoculum [g VS]	VS in biomass [%]	VS in inoculum [%]	Initial Substrate concentration w/w (%)
UC	7.21	21.64	64.4	24.4	14.4
TC	7.34	22.01	75.8	24.4	14.6
TU	7.24	21.71	66.7	24.4	14.4
NaOH-TC	7.47	22.40	93.0	24.4	14.9
MC	7.51	22.53	100.0	24.4	15.0
Blank	0.00	24.40	-	24.4	12.2

## 3. Results and Discussion

### 3.1 Biomass characterization

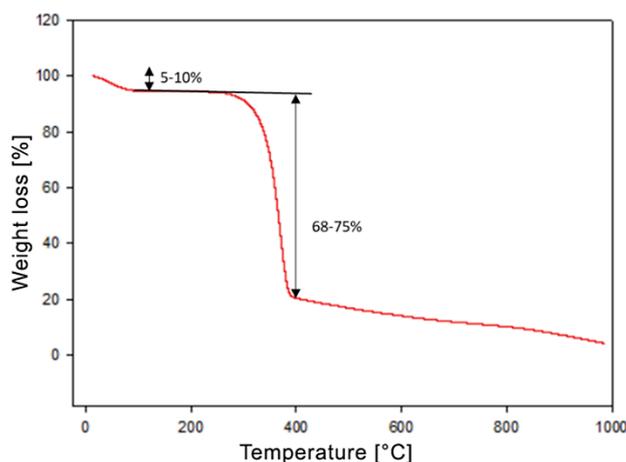
Table summarizes the total and volatile solids characterization results for each biomass. Volatile solids on total solids represent the percentage of VS in the sample once water content is removed. As expected, VS content for TC and TU is very similar, as these two biomasses were supplied by the same provider and were subjected to identical treatments. Nonetheless, visually TC and TU are distinctly different, as highlighted in Figure 1. Differences in SAP content may be responsible for the higher dustiness of TU: while both biomasses have an SAP content below 40%, as per the supplier specification, the content may vary. Moreover, mechanical and chemical treatments may have affected TU and TC differently considering conditions on entry: being soaked in excreta, SAPs in TC were probably completely hydrated before autoclave treatment, whereas dry SAP could still be present in TU even after autoclave treatment since the water absorption rate of the SAPs was generally too slow for the brief treatment to completely hydrate the polymer. These values can be ascribed to water and the presence of urine, which has a high pH value due to urea and urate breakdown. In general, the water absorption process of SAPs is based on the dissociation of metal ions when water molecules enter the SAPs, which ionizes and negatively charges the hydrophilic groups, resulting in the establishment of a concentration chain between the solution in SAP and the exterior solution [31]. In deionized water, the water absorption rate of SAPs rises 200 to 1500 times, whereas it decreases in high ionic strength and high pH solutions [32,33]. The higher content of volatile solids in NaOH-treated samples is to be ascribed to the additional drying step they were subjected to, nearly removing all water content, especially compared to UC samples.

**Table 3** Characterisation of biomasses for total solids and volatile solids.

Biomass	Total solids [%]	Water content [%]	Volatile solids [%]	Volatile solids on total solids
UC	85.0	15.0	64.4	0.76
TC	94.0	6.0	75.8	0.81
TU	94.6	5.4	66.7	0.71
NaOH-TC	97.6	2.4	93.0	0.95
MC	100.0	0.0	100.0	1.00
Blank	27.2	72.8	24.4	0.90

**Figure 1** Visual comparison of TU (above) and TC (below) biomasses.

TGA analysis (Figure 2) shows a first weight loss between 5% and 10% due to water loss below 100° C. The greatest weight loss, meanwhile, can be observed between 270° C and 380° C and, in accordance with previous scientific literature, can be ascribed to the degradation of fibrous compounds, and to the breaking of glycosidic bonds. According to Adibi et al., after the first weight loss relative to water, in the temperature range between 225° C and 425° C a loss due to the decomposition of non-cellulosic compounds and cellulose is observed, between 425° C and 600° C, a further final weight reduction related to the loss of decomposition products between 150° C and 450° C [34]. In various studies, the percentage differences in weight loss and the different trends in the thermogravimetric curve detected depend on the type of cellulose, i.e., on the maturity of the fibers and any treatments, and, therefore, on the ratio between cellulose and non-cellulosic components. Base on the major degradation range of UC has been observed between 270°C - 380°C which is due to the degradation of structural sugar components. This shows that proper pre-treatment can effectively improve biogas performance, which the result was in line with sample TC, TU and NaOH-TC.



**Figure 2** TGA analysis results for an UC sample.

The optimization procedure used in this study is called a metaheuristic (MH). Five recent metaheuristic algorithms were utilized to construct the surrogate-assisted optimization approach for the TOT heat exchanger. The setup of optimization parameters is outlined in Table 2. The total number of function evaluations (FEs) is set to 20,000 FEs, and the population size is 50. The performance of the metaheuristic algorithms was evaluated using the hypervolume indicator [50] with the statistical tests including average (Mean), standard deviation (Std), maximum (Max) and minimum values (Min).

### 3.2. Biogas production

While experimental conditions were identical for each biomass, each sample type behaved differently. Results for batch reactors belonging to the same triplicate trial were consistent. As such, results are shown for the most productive sample. Table 4 **Error! Reference source not found.** summarizes biogas and methane yield for each biomass type. The experimental error resulting from the experimental setup is much larger than the standard deviation for each mean and is equal to 12% as described below.

**Table 4** Biogas and methane productions of anaerobic digestion trials.

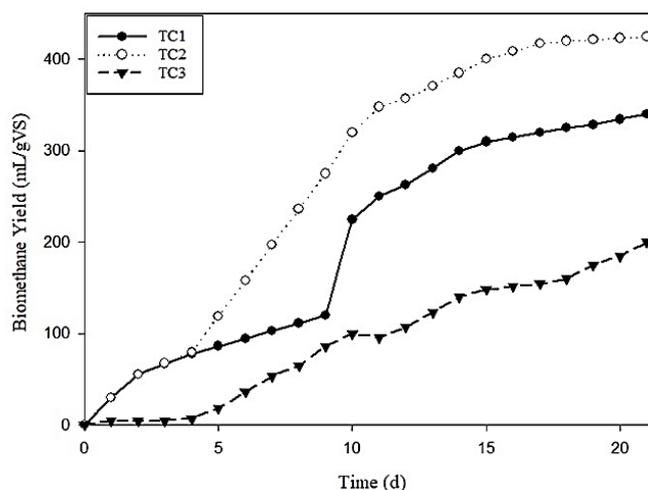
Biomass Type	Biogas production yield [mL/g VS]	Methane content [%]	Methane production yield [mL/g VS]
UC	286.4	72	206.2
TC	426.9	72	307.4
TU	385.6	70	269.9
NaOH-TC	418.5	74	309.7
MC	497.4	74	368.1
Blank	2.2	-	-

As expected, blank trials, consisting only of digestate, were only weakly active and stopped production altogether after 3 days. Average biogas production was 2.2 mL/g VS. This value was subtracted from any other measure to correct for inoculum production. The theoretical methane production yield of pure cellulose from anaerobic digestion, based on its chemical composition, is 415 mL/g VS. The maximum methane production yield measured during microcrystalline cellulose trials (MC) was 368 mL/g VS, highlighting a production efficiency of 88%. It is also worth pointing out that as the 21-day mark production curves still did not reach a plateau, and thus the production efficiency is bound to be higher. As pure microcrystalline cellulose is a known, accessible digestion substrate, the production efficiency depends on the experimental setup and on the specific bacterial community present in the inoculum.

Untreated biomass (UC) showed the lowest methane production yield, equal to 206.2 mL/g VS after 21 days of anaerobic digestion. AHP fluff is formed of lignocellulose, which primarily comprises three types of polymers (i.e., cellulose, hemicellulose, and lignin) [35]. Due to lignocellulose being bound by lignin, untreated lignocellulose impairs the accessibility of cellulose and hemicellulose by enzymes and microorganisms. Therefore, the higher the lignin content is, the lower the biogas production will be.

TU reactors showed a low biogas production yield (385.6 mL/g VS). This value is nonetheless higher than the yield of UC samples, confirming that the industrial treatment these samples underwent has a positive effect on biogas production. Moreover, the value is only slightly lower than that of unused, NaOH-treated samples, further substantiating that the effect on biogas yield by the industrial treatment is in line with the alkaline treatment for this biomass. TC results were mostly in line with those of microcrystalline cellulose, as shown in Figure 3. In particular, TC2 had a production yield of 426.9 mL/g VS after 21 days of production, which is only slightly below

the average production for MC, while TC1 and TC3 had a lower production yield, respectively 341.5 and 173.2 mL/g VS.



**Figure 3** Evolution of biomethane production yield from TC trials.

The remarkable difference in results between TC trials can be ascribed to substantial inhomogeneity in the tested biomass: as shown in Figure 1, cellulosic matter varies between fluffy and compact forms, while plastic residues and residual excreta can be found in non-negligible quantities. No selection was performed on the biomass to be primed for anaerobic digestion to ensure sample representativity; consequently, samples may differ in VS content and in the availability of carbon sources. These differences in performance cannot solely result from the experimental setup, which was identical for each sample run. The unequal trend in biogas production for TC3 between days 8 and day 9 is due to foam forming on the surface of the sample, trapping gas until the growing pressure reaches the level where most biogas is freed. Except for this specific event, the curve representing biogas yield for TC3 closely mirrors the ones for TC1 and TC2; the lower final yield may also depend on the pressure build-up caused by foaming [36].

Furthermore, a high SAP content may effectively reduce the available water by sequestering it, together with nutrients and microorganisms in solution, and indirectly increase the total solids content of the reactor which, together with the lack of mixing in the experimental setup, could inhibit the anaerobic digestion process. Moreover, the increase in the maximum biogas and methane production seen in TC compared to TU samples can be ascribed to the addition of human excreta to the lignocellulosic fluff, which can be found in the samples even after industrial pre-treatments. As such, a portion of volatile solids in TC samples is more easily accessed by bacteria and contributes to an increase in the maximum production.

Pre-treated AHP fluff (NaOH-TC) showed an increase in methane production yield of 50% (from 206.2 to 309.7 mL/g VS) compared with the UC in AD. The saponification and breakage of lignin-carbohydrate linkages are thought to be the function of NaOH pre-treatments, enabling delignification of lignocellulosic biomass and leading to more porosity, surface area, and reduced degree of polymerization [21]. NaOH-treated samples have also shown the maximum yield which is in line with that obtained from TC samples, and a higher yield than TU samples. This is especially interesting when choosing pre-treatments for lignocellulosic biomasses, as the alkaline treatment has been shown to increase biogas production more than the industrial treatment alone, by turning recalcitrant lignocellulosic biomasses into a more available substrate for anaerobic digestion.

#### 4. Conclusion

The rising demand for AHPs in emerging nations has spurred global interest in proper recycling methods. This study assesses the BMP of cellulose from AHPs and evaluates pre-treatment impacts. The industrial treatment combining sterilization and oxidative chemicals was shown to a biomethane yield of 307.4 mL/g VS and an alkaline treatment of biomethane yield of 309.7 mL/g VS, respectively. Industrial treatments yield biomethane efficiently, suggesting cost-effective, eco-friendly AHP waste management. Anaerobic digestion proves promising for AHP fluff treatment. Future research should focus on separating SAP and plastics from AHPs for reuse or energy recovery, aiding landfill reduction, and promoting renewable energy.

#### 5. Acknowledgements

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