



Effect of power ultrasound and Fenton reagents on the biomethane potential from steam-exploded birchwood

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ABSTRACT

The global demand for non-fossil energy sources is increasing rapidly. As a result, biogas presents a suitable alternative; however, first generation biofuels (e.g., sugar cane) potentially impact food crops globally. Second generation biofuels based on lignocellulose-based biomass are being used more frequently as they do not impact food crops. Furthermore, in Northern Europe, there is a significant interest in utilizing birchwood and paper mill waste for biogas production due to its high availability. The utilization of birchwood for biogas has significantly improved in recent years with the improvement of required pretreatment processes. To date, the most effective and economically feasible pretreatment in an industrial context is the steam explosion of lignocellulose-based biomass. Despite this, there is potential for releasing more digestible components from this biomass by efficiently degrading the lignocellulose components. This research presents another pretreatment that can be applied to steam-exploded wood based on ultrasonication and Fenton reagents. It was observed that by treating the steam exploded birchwood with ultrasonication and mild concentrations of Fenton reagents, an increase in the rate of biogas production was achievable. This would allow the increase in biogas yield of a continuously feed industrial anaerobic digester without increasing the size of the reactor.

1. Introduction

Biofuel production has been shown as a real alternative to fossil fuels with an estimated potential generation of up to 25% of the global energy requirements by 2035 [1], including the anaerobic digestion of biomass to produce biogas. Currently, 1st generation biofuels are the most prolific globally, produced mainly in the US and Brazil from corn or sugar cane [2]. Although these substrates account for a large portion of the biofuels generated globally, these sources are also potential food sources, meaning there is a requirement for the move to non-food stock biomass sources [3,4]. As a consequence, the 2nd generation of biofuels is under development, and is based on using lignocellulosic biomasses such as forestry and papermill residues. These feedstocks are abundant, do not compete with food sources and are cheap [5]. This suggests that the production of biogas from lignocellulosic material could be a significant substitute for natural gas.

The constituents of lignocellulose are the three polymeric components: cellulose, hemicellulose and lignin. The lignocellulose structure is rigid and obstinate, meaning that the biochemical conversion into usable sugars is rather demanding [3,6–9]. Due to this, pretreatment of lignocellulosic biomass is necessary for hydrolysis of the compound

[3,10]. This is essential for allowing access of the required microbes and enzymes to the biomass material for efficient saccharification. Several methods of pretreatment have been developed to improve hydrolysis and saccharification of biomass. These include acid and alkali treatment, ball milling, hot water treatment, ammonia fiber expansion, microwave irradiation, organosolv treatment and steam explosion [11–13]. Steam explosion is a thermal-based pretreatment. It has evolved into a process that is one of the simplest, efficient and environmentally friendly methods for lignocellulose pretreatment [3,10,14–16]. Steam explosion is achieved by applying a high temperature and pressure steam to the biomass, then rapidly dropping the pressure to cause a vapor explosion. The pretreatment works well on hardwoods [3,17,18], including willow [19] and birch [20]. Moreover, it has been shown with willow and birch wood that steam explosion not only improves hydrolysis of the biomass, but also the biogas production yield through anaerobic digestion [3,19,20].

Birch wood is of major interest for industrial biomethane production in Northern Europe as it is widespread and readily available [20]. On unused agricultural land in Northern Europe, the typical productivity of birch wood is 3 tons dry weight per ha per year [3], with a suggested rotation period of 15–20 years when coupled with biofuel production [21]. The increase of the

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microbiological and enzymatic access to the biomass after steam explosion allows higher levels of methane production to be achieved from hardwoods like birch [19,20]. However, there is still a significant amount of organic components in the steam-exploded hard wood that are not easily accessible for utilization by the microbes. Previous attempts for increasing the potential for biomethane production from steam exploded birch have assessed the potential of an enzymatic step after steam explosion [11]. Cellulase has been observed to increase the sugar content of the sample significantly, allowing a higher biomethane potential to be achieved from birch wood [20]. Despite this, the amount of enzyme required for implementation in the industry is substantial, potentially resulting in a high and undesired economic cost.

Advancements in the understanding of ultrasonication in recent years may provide an alternative to enzymatic treatments of birch. Ultrasound is an acoustic wave that has a frequency above 20 kHz, whereas a power input above 10 W signifies the ultrasonication range [22]. The ultrasonication acoustic waves can be directly or indirectly transmitted into the medium through a transducer. The major effects that are induced by the propagation of the acoustic wave into the medium are acoustic cavitation and acoustic streaming. Cavitation results in the formation of small gas bubbles that oscillate in size due to the acoustic wave. Eventually these gas bubbles violently collapse, generating local temperatures of up to 5000 °C and pressures of up to 2000 atms [23]. This collapse transmits both chemical and mechanical effects into the medium, resulting in sonolysis forming a variety of radical and reactive species (i.e., HO·, O₃, H₂O₂ and O), from H₂O [24]. These can diffuse into the surrounding medium and can react with the nearby solutes in the media.

The Fenton reaction is based on the oxidation and reduction of Iron by H₂O₂ and also produces a variety of reactive and radical species (i.e., HO·, OH⁻, HOO· and H⁺). During the Fenton reaction, ferric ions react with H₂O₂ forming ferrous ions, HOO· and H⁺. Then the ferrous ions can react with the H₂O₂ to produce ferric ions, HO· and OH⁻. This cycle can proceed continuously until the H₂O₂ is depleted, meaning the amount of radical species produced by the Fenton reaction is relative to the amount of H₂O₂ added [25]. Therefore, the efficiency of the Fenton reaction is dependent on mainly the H₂O₂ concentration; however, the Fe²⁺: H₂O₂ ratio, the pH and the duration of the reaction are also important [26].

The hydrolysis process of the steam-exploded birch wood could be improved with the increase in HO· radicals [27–32]. Furthermore, recent observations show that by using ultrasonication in combination with Fenton reagents leads to an increase in the saccharification of biomass containing lignocellulose [27,33,34]. Despite this, it has not yet been shown whether this increase in saccharification will result in an increase of the biogas production by anaerobic digestion of biomass containing lignocellulose. Therefore, purpose of this research is to assess whether the use of ultrasonication, a Fenton-like reaction or a combination of both has a positive impact on the biomethane production of steam-exploded birchwood.

2. Materials & methods

2.1. Raw material

Birch wood (*Betula pubescens*) originated from a tree harvested in Norway. The wood was shredded into chips of around 30 mm. This was then dried at room temperature.

2.2. Steam explosion pre-treatment

The steam explosion pre-treatment was performed as described previously [3,14], using a steam explosion instrument designed by Cambi AS (Asker, Norway), at the Norwegian University of Life Sciences in Ås, Norway. The milled birch was added to the pressure vessel and subjected to 210 °C with a residence time of 10 min. Prior to the treatment, the pressure vessel was preheated for 10 min at 210 °C. The

steam-exploded birch wood was stored at –20 °C before further use. The dry matter content of the steam exploded sample was 34%.

2.3. Inoculum

The microbial inoculum for the biomethane potential experiments was obtained from the Biokraft biogas plant (Skogn, Norway), from a large-scale continuous mesophilic anaerobic digester. Prior to the biomethane potential test, the inoculum was incubated at 39 °C to reduce endogenous biogas production. The inoculum was diluted to 9 g volatile solids (VS) per liter. The diluted inoculum had a pH of 7.5.

2.4. Ultrasonication

The ultrasonication experiments were conducted at a pH of 4 in a volume of 200 mL. The steam-exploded birch was dissolved in water at 5% wet weight (i.e., 10 g of steam exploded birch per 200 mL of water). FeCl₃ was added to water-birch mixture where indicated. Ultrasonication was performed at a frequency of 24 kHz for 2 h, at a power level of 30 W using a UP200S ultrasonic transducer (Hielscher, Germany). Samples were maintained at –20 °C before further use.

2.5. Fenton-like reaction

The samples had H₂O₂ added to them at concentrations indicated. The reaction was maintained for 2 h.

2.6. Biomethane potential

The processed samples were digested anaerobically in sealed batch bottles. These were performed in triplicate with inoculum and the processed samples, and one batch with only inoculum alone as a negative control batch. The bottles were flushed with nitrogen gas and closed with a rubber septum and sealed as described by Ekstrand et al. [35]. These were incubated at 39 °C indefinitely with regular agitation.

2.7. Analysis

2.7.1. Composition analysis

Analysis of the dry matter (DM) and volatile solids (VS) was performed on the steam exploded birch wood and inoculum using standard methods [36]. The dry matter was determined by incubation of the birch wood and inoculum at 105 °C for 24 h. The mass of the samples after the incubation was compared to the mass pre-incubation to acquire a percentage of DM. Samples were then incubated at 550 °C for 4 h in a muffle furnace. The weight of the samples after incubation in the muffle furnace were compared to the weight of the sample before incubation to acquire a percentage of VS.

2.7.2. Gas production volume

Measurement of the produced gas volume was achieved using an optimized liquid displacement method. The volume of gas was then adjusted for temperature using Charles's law.

2.7.3. Gas composition and calculation

Regular 10 mL samples of gas were extracted from each experiment for compositional analysis. The composition was analyzed by a gas chromatograph (SRI 8610C, SRI Instruments, USA), equipped with a thermal conductivity detector using hydrogen as the carrier gas. A standard mixture of CO₂, CH₄, H₂ and N₂ was used as a calibrating gas. Using the volume of gas calculated with the liquid displacement method, and the percentage of methane present in the samples, methane production was calculated for the experiments. All methane production levels are the average of 3 separate experiments and have been corrected by subtracting the endogenous methane production levels for the inoculum only negative control experiments.

2.7.4. Volatile fatty acid content

The volatile fatty acids (VFAs) were measured by extracting regular liquid samples of 2 mL from each experiment. These samples were then centrifuged at 10000 rpm using a Microfuge 20 (Beckman Coulter) for 10 min. The clear supernatant was taken from the top of the centrifuge tube after centrifugation and passed through a 0.45 μm membrane syringe filter (514-0071, VWR). The samples were then placed in GC vials and setup on the AutoSystem XL (Perkin Elmer) GC machine with autosampler.

3. Results

3.1. Steam exploded birch

Birch wood chips were subjected to steam explosion conditions with a temperature of 210 °C for 10 min. To quantify the intensity of the steam explosion, these two variables can be combined into a single value known as a reaction ordinate (R_o) [37]. The R_o can be calculated into what is commonly coined as the severity factor ($\log R_o$) [20]. Vivekanand et al. (2011) observed that a $\log R_o$ between 4.2 and 4.8 obtains the highest methane yield during anaerobic digestion [20]. For this research, the severity factor of the steam explosion treatment was 4.24. As a general guideline, the higher the severity, the darker and stickier the product. The product also has a less obvious fiber structure (Fig. 1). These effects are typical from steam explosion of birch wood [38].

3.2. Ultrasonication and Fenton-like reactions

The sonication experiments were undertaken on the steam exploded birch wood as described previously. The ultrasonication was applied at a power of 30 W and frequency of 24 kHz. The steam exploded birch was dissolved in 200 mL at a mass of 10 g w/w for each experiment. FeCl_3 was added to concentrations outlined in Table 1 prior to the ultrasonication experiments. The pH was adjusted to 4 before commencing with the ultrasonication treatment as this has been observed to be the optimum pH for lignin-based biomass degradation by means of the Fenton-like reaction [34], which may occur during ultrasonication due to the production of H_2O_2 . The duration of the ultrasonication treatment was 2 h.

After the ultrasonication experiment was completed, H_2O_2 was added to the samples at the concentrations outlined in Table 1. This allowed the main Fenton-like reaction to take place within the sample. The samples were incubated for 2 h to undergo the Fenton-like reaction. BMP tests were then performed using these samples as the substrate. Fig. 2 display the amount of biogas obtained from each sample over time, while Table 2 displays the total biogas and methane produced after 35 days. The VFA content observed in these experiments is shown in Table 3.



Fig. 1. Steam explosion treatment of birchwood. Birchwood chips of around 30 mm prior to steam explosion (A), compared to the dark and sticky birchwood with a less obvious fiber structure after steam explosion (B).

Table 1

Ultrasonication and Fenton reagent reaction specifications.

Experiment	$[\text{FeCl}_3]$ (M)	$[\text{H}_2\text{O}_2]$ (M)	Ultrasonication duration (h)
Control 1	0	0	0
1	0	0	2
2	0.05	0	2
3	0.001	0.01	0
4	0.001	0.001	2
5	0.001	0.01	2
6	0.005	0.05	2
7	0.01	0.1	2

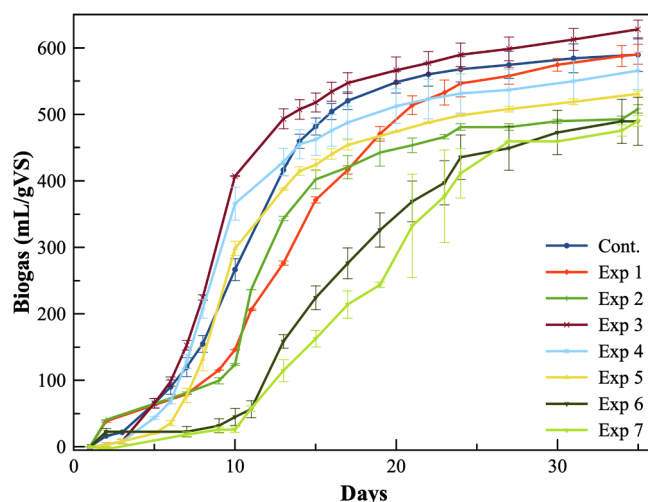


Fig. 2. Biogas production over 35 days. The biogas production of the 7 experimental samples and the control 1 sample (Cont.) is shown over a 35-day period in mL per gVS. The error bars are indicative of the standard deviation of the mean of three independent triplicates.

Table 2

Biogas and methane total production.

Experiment	Biogas After 35 days (mL/gVS)	Methane After 35 days (mL/gVS)	Maximum rate (mL/gVS/day)
Control 1	598.80 \pm 32.49	341.32	72.11
1	590.44 \pm 6.40	335.37	41.32
2	508.18 \pm 36.45	289.66	54.87
3	490.44 \pm 8.19	279.55	43.37
4	627.92 \pm 17.68	357.92	77.26
5	565.79 \pm 4.24	322.50	74.40
6	530.90 \pm 30.41	302.61	65.68
7	489.54 \pm 15.05	279.04	27.24

Table 3

Concentration of volatile fatty acids.

Experiment	Initial pH	After 40 days (mg/L)		
		Acetic Acid	Propionic Acid	Butyric Acid
Inoculum	7.51	52.95 \pm 1.37	n.d.	n.d.
Control 1	7.53	380.92 \pm 9.88	39.00 \pm 0.93	n.d.
1	7.53	321.33 \pm 10.81	30.65 \pm 1.36	n.d.
2	7.50	291.48 \pm 8.75	25.64 \pm 1.53	n.d.
3	7.51	243.33 \pm 10.02	23.89 \pm 1.55	n.d.
4	7.48	338.73 \pm 7.60	32.45 \pm 1.21	n.d.
5	7.49	316.17 \pm 8.53	27.99 \pm 0.89	n.d.
6	7.51	328.10 \pm 9.15	33.03 \pm 0.86	n.d.
7	7.52	254.47 \pm 8.89	24.74 \pm 1.76	n.d.

n.d. = Not detected.

To compare the effect that the ultrasonication and Fenton-like treatment has on the BMP from steam-exploded birchwood, a baseline experiment was performed (Control 1, Table 1). The substrate for this baseline experiment was steam exploded birchwood without any further pre-treatment. By performing a BMP test using the steam exploded birch wood as the substrate, 598.80 ± 32.49 mL per gVS of biogas was obtained. This gas was made up of 57% CH₄, meaning 341.32 mL per gVS of CH₄ was produced (Table 2). These production levels are similar to those previously reported [20].

The effect of the ultrasonication without added FeCl₃ or H₂O₂ (Experiment 1), was assessed to determine if the ultrasonication would alone increase the BMP. The BMP after 35 days was decreased by 1.4%, but the margin of error suggests there is no significant change in biogas production. Experiment 2 assessed the impact of the ultrasonication treatment on the BMP when FeCl₃ is present. Here a significant decrease of 15.13% in the BMP is observed. To assess the potential impact of the Fenton-like reaction alone without the ultrasonication treatment, experiment 3 was designed; however, although a lower concentration of Fenton reagents were used in this experiment, a significant reduction of 18.1% in the BMP was observed relative to control 1.

Experiments 4–7 focused on the treatment of the steam exploded birchwood by both ultrasonication and the Fenton-like reaction. For this, varied concentrations of FeCl₃ and H₂O₂ reagents were added to the samples during the pre-treatment process (Table 1). The recorded BMPs of these experiments seem to display an inverse relationship to the concentrations of FeCl₃ and H₂O₂, i.e., as the concentrations of these reagents increased, the BMP decreased. This suggests that the reagents used for the Fenton-like reaction may have a negative impact on the microbiology of the inoculum, resulting in a reduction in the BMP. Despite this, as seen in experiment 4, the lowest concentration of the Fenton reagents in combination with the ultrasonication treatment resulted in a slight increase of 4.9% in the BMP compared to control 1; however, the margin for error suggests that this is not a significant increase in BMP.

3.3. Two-stage anaerobic digestion

An assessment of whether the birchwood digestate could be treated with ultrasonication and Fenton reagents to get further biogas was also analyzed. The digestate from the control 1 from the previous section (Section 4.2) was used as the substrate, and was exposed to ultrasonication and Fenton reagent treatments as outlined in Table 4.

The BMP tests on the pre-digested birch wood (experiments B-1–B-5) did not display a significant increase in biogas production over control 2 (Fig. 3, Table 5). Because there was carryover of inoculum with the pre-digested birch wood from the first AD stage, and no pre-treatment was applied to the control, majority of the biogas produced in the second stage was probably not due to further hydrolysis of components in the substrate.

By adding the total biogas volume produced by control 1 from the previous section (Section 4.2), the total biogas production volume from the two-stage anaerobic digestion can be calculated (Table 5). Experiment B-4 was observed to produce the highest total biogas of 733.50 mL per gVS, with a total methane production of 418 mL per gVS; however,

Table 4

Ultrasonication and Fenton reagent reaction specifications for pre-digested steam exploded birch wood.

Experiment	[FeCl ₃] (M)	[H ₂ O ₂] (M)	Ultrasonication duration (h)
Control 2	0	0	0
B-1	0	0	2
B-2	0.001	0.001	2
B-3	0.001	0.01	2
B-4	0.005	0.05	2
B-5	0.01	0.1	2

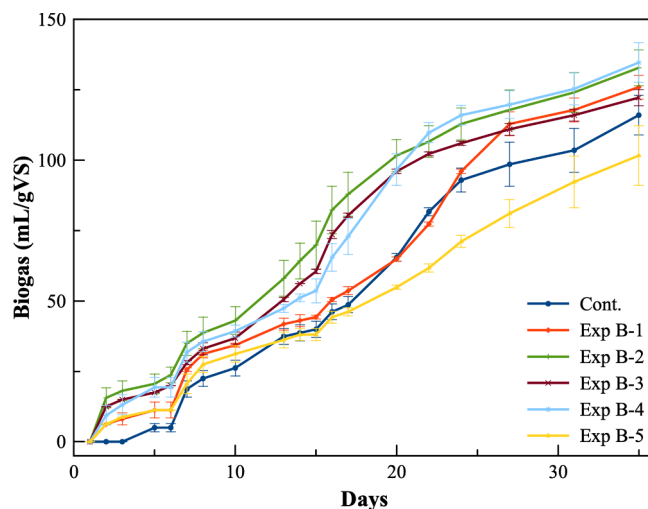


Fig. 3. Second-stage biogas production over 35 days. The biogas production of the 5 experimental samples and the control 2 sample (Cont.) is shown over a 35-day period in mL per gVS. The error bars are indicative of the standard deviation of the mean of three independent triplicates.

Table 5

Two-stage biogas production.

Experiment	Total Biogas (mL/gVS)	Total Methane (mL/gVS)
Control 2	714.79	407.43
B-1	724.77	413.12
B-2	731.63	417.03
B-3	721.03	410.99
B-4	733.50	418.10
B-5	700.45	399.26

there is no significant increase above that of the control of this second stage experiment, suggesting the second stage of treatment did not significantly increase the accessibility of lignin, and effectively only increased the batch test from 35 days to 70 days.

3.4. Biogas production rate

The rate of biogas production is an important variable to consider for industrial biogas production as it can decrease the required retention time, allowing for an increase in the overall capacity of the plant. The biogas production rate maximum was calculated by determining the slope of the exponential phase of the gas production over time. Table 6 shows the calculated biogas production rate during the most productive period for each sample. The biogas production rates were reduced in Experiments 1–3, 6 and 7 due to the treatment severity. However, the biogas production rates in experiments 4 and 5 were increased compared to control 1. This suggests that the harsher sonication and fenton-like treatments had a negative impact on the biogas production rate, whereas the milder treatments resulted in an increase in

Table 6

Biogas production rate.

Experiment	Maximum rate (mL/gVS/day)
Control 1	72.11
1	41.32
2	54.87
3	43.37
4	77.26
5	74.40
6	65.68
7	27.24

the biogas production rate. This resulted in a shorter retention time to attain a near maximum BMP, which in a continuously fed industrial biogas process plant will result in an overall increase in biogas capacity without increasing the size of the reactor.

4. Conclusions

This research has displayed the biogas potential for using ultrasonication and Fenton reagents of steam-exploded birchwood. It has been observed that the use of both of these treatments allows significant increases in the rate of biogas production during anaerobic digestion. Furthermore, by using low concentrations of the Fenton reagents, the effect seems to be improved. This would allow for a significant improvement in biogas yield in an industrial plant where steam exploded birch is the primary biomass source, without having to increase the size of the anaerobic digester.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.ultsonch.2019.104675>.

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