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Lignocellulosic Biomass Valorisation by Coupling Steam Explosion Treatment and Anaerobic Digestion

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Abstract: Lignocellulosic biomass valorisation presents a promising avenue for sustainable and renewable energy production. In this study, the synergistic potential of coupling steam explosion (SE) treatment with anaerobic digestion (AD) was explored to maximize the efficient conversion of lignocellulosic biomass into valuable biogas. The SE process, a cost-effective technique for biomass fractionation, plays a pivotal role in breaking down complex biomass components, rendering them more amenable to subsequent biological treatments. In the present work, we investigated the impact of various SE conditions, including temperature, time, and acid concentration, on the breakdown of lignocellulosic residues. Through the quantification and analysis of sugars and their degradation products, the optimization of steam explosion conditions at lower temperatures and shorter time periods, along with the presence of a lower concentration of acid catalysts, efficiently releases sugars. Maintaining these conditions helps prevent byproducts. The evaluation of the $(S/I)_{vs}$ ratio during anaerobic digestion reveals an optimal 1/2 ratio, maximizing biogas production. This innovative approach demonstrates significant potential for the valorisation of lignocellulosic biomass, contributing to a more sustainable and efficient utilization of renewable resources in the pursuit of clean energy solutions.

Keywords: lignocellulosic biomass; steam explosion treatment; anaerobic fermentation; biogas; biomethane

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1. Introduction

Renewable energy sources have become imperative due to climate change and the subsequent need to reduce greenhouse gas emissions. Biomethanation, as a prominent renewable energy technology, presents a promising solution by utilizing organic waste to generate clean and sustainable energy. Through the process of biomethanation, the anaerobic digestion converts organic matter, including agricultural residues, food waste, and sewage sludge, into biogas that is rich in methane (CH_4). This renewable energy source offers multiple environmental benefits, including a significant reduction in greenhouse gas emissions and the diversion of organic waste from landfills, thereby mitigating environmental pollution. Exploring the potential of biomethanation not only contributes to the renewable energy sector, but also addresses the pressing issues of waste management and sustainable resource utilization [1]. In this context, exploring the potential of the biomethanation of lignocellulosic biomass as a renewable energy solution holds an immense promise for a more sustainable future [2].

To promote the growth of anaerobic microorganisms and maximize biogas production, it is essential to maintain optimal conditions, particularly regarding temperature and pH, which have a significant influence on the performance of the conversion [3]. The optimal pH range for anaerobic digestion typically lies between 6.5 and 8.5. Temperature, on the other hand, plays a critical role in determining the performance and efficiency of anaerobic digestion. Different temperature ranges correspond to specific types of microorganisms and biochemical reactions, consequently affecting the overall process, and they can be categorized into three main temperature ranges: Mesophilic temperature range, which is between 25 °C and 40 °C (77 °F and 104 °F). This is considered the standard temperature range for most applications due to its balanced performance in terms of biogas production, process stability, and nutrient retention [4]; Thermophilic temperature range, which is between 50 °C and 60 °C (122 °F and 140 °F). A high temperature accelerates the biological reactions, leading to a faster degradation of organic matter and increased biogas production rates [4]; and Psychrophilic temperature range, which is below 28 °C (82.4 °F), typically in the range of 10 °C to 28 °C (50 °F to 82.4 °F). This range is effective in conserving heat energy while achieving CH4 values that are comparable to those observed under mesophilic/thermophilic conditions. This concept is suitable for specific applications in colder climates or for low strength wastewaters [5–7].

The choice of temperature range depends on factors such as the feedstock characteristics, available infrastructure, energy requirements, and the desired objectives of the anaerobic digestion process. It is crucial to assess the specific requirements of each feedstock and consider the local climatic conditions when selecting the appropriate temperature range for anaerobic digestion [5].

Various toxic substances can act as inhibitors in anaerobic digestion, affecting the performance and efficiency of the process, thus resulting in reduced biogas production and process instability, e.g., toxic substances (heavy metals, pesticides, organic solvents, phenolic compounds), excessive levels of ammonia NH_3 and ammonium NH_4^+ (it can inhibit the activity of methanogenic microorganisms), low pH (acidic conditions can inhibit the methanogenic microorganisms), and insufficient levels of essential nutrients, such as nitrogen, phosphorus, and trace elements, which can limit microbial growth and activity [8,9].

Lignocellulosic biomass, derived from agricultural and industrial waste, represents a vast and abundant feedstock for biomass-to-biogas conversion. However, because of its complex structure, which is mainly composed of a linear, semi-crystalline, homopolysaccharides, highly polymerized "Cellulose", an amorphous heteropolysaccharide "Hemicellulose", and a heterogeneous phenylpropanoid macromolecule "lignin", which connects the first two components together, the biomass requires pretreatment methods to enhance its hydrolysis, thus enabling the breakdown of cellulose and hemicellulose into fermentable sugars. Subsequently, anaerobic digestion processes can efficiently convert these sugars into biogas, predominantly consisting of methane and carbon dioxide. Various pretreatment approaches have been used such as acid, alkali hydrothermal pretreatment, ionic liquid pretreatment, etc. Hydrothermal methods are the most used for the conversion into biofuels products, and their efficiency depends on the process's severity, looking at factors such as temperature, the time of the reaction, and the concentration of a catalyst, if any [10]. In general, the hydrothermal pretreatment of cellulose leads to the production of fermentable monomer sugars (cellobiose, glucose, and xylose), and the treated hemicellulose fraction essentially consists of glucose and pentoses [11]. Depending on the treatment temperature range (150–375 °C), various hydrothermal pretreatment methods can be employed for lignocellulosic biomass. The selection of the method depends on the intended application of the pretreated biomass [12]. When the temperature is between 150 °C and 250 °C, the resulting products primarily consist of a solid fraction comprising lignin and cellulose, along with a liquid fraction containing hemicellulose, sugars, acids, and degradation products [13]. Conversely, at temperatures above 250 °C, the predominant product is an organic liquid known as bio-oil, accompanied by a solid residue, an aqueous phase containing a high organic carbon content (such as oligomers, monomers, furfural, and 5-HMF), and a light gas phase (including CO_2, CO, H_2, and CH_4). These variations in product composition highlight the impact of hydrothermal pretreatment conditions on the resulting fractions and offer opportunities for the target utilization of lignocellulosic biomass in different applications.

Steam explosion (SE) is a widely employed hydrothermal technique for lignocellulosic materials hydrolysis. It consists of two distinct phases: steam cracking and explosive decompression. During the process, biomass is subjected to high temperatures, typically around 200 °C, and high-pressure steam for a short duration (on the order of minutes). The level of treatment in steam explosion (SE) is quantified using the concept of severity, which is a function of the operating temperature, treatment time, and the use of a catalyst. Higher temperatures and longer treatment times result in higher severity levels, leading to increased biomass disruption and the depolymerization of lignocellulosic components. Controlling the severity level allows for the optimization of steam explosion conditions to achieve desired outcomes, such as maximizing the release of fermentable sugars or altering the physicochemical properties of the biomass; this is because an excessive severity can lead to the excessive degradation of biomass components and the formation of inhibitory compounds at high concentrations, e.g., formic acid, levulinic acid, furfural, hydroxymethyl furfural, etc. [3,14,15].

SE can be performed without catalysts. This is commonly known as 'autohydrolysis', where the treatment is due to the catalytic action of water and acetic acids released from sugars as xylans, which catalyse the hemicellulose hydrolysis reaction. Uncatalyzed SE has been studied with different lignocellulosic biomass such as sugarcane bagasse [16], wheat straw [17], and sunflower stalks [18]. In the case of a catalysed treatment, the utilization of a catalyst becomes essential for its ability to facilitate a reduction in the reaction temperature and enhance the solubilization of organic components [17,19,20].

Agudelo et al., 2016, studied the impact of the severity factor on sugar production from an agriculture residue 'tripticale' at 200 °C for 15 min; they concluded that at higher severity conditions, the sugars initially produced may be further degraded into inhibitors components at high concentration, which can affect a further enzymatic treatment, e.g., furanic acids, formic acid, levulinic acid, etc. [21]. While Pielhop et al., 2016, studied the impact the explosion had (rapid decompression) on the enzymatic digestion of spruce wood chips (SE 235 °C for 10 min), they found an increase of 30% in the digestibility yield with an explosion compared to the process without rapid decompression. An exceptional yield was achieved without a chemical treatment [22,23]. Other researchers were focused on the effect of steam explosions on the surface morphology. For that, Meng et al., 2013, found that the increase of the factor of the severity of steam explosion increases the pore size distribution of a lignocellulosic substrate (*Populus trichocarpa x deltoides*), which may facilitate enzymic access in the case of enzymatic hydrolysis. The results agree with Muzamal et al., 2015, and Satari et al., 2018 [24–26]. The size distribution of lignocellulosic biomass particles is a crucial factor in both steam explosion (SE) and the subsequent anaerobic digestion processes [22,25]. The efficiency of these processes is influenced by the size, dimensions, and structure of the biomass particles [27]. More specifically, the size distribution of the lignocellulosic biomass directly affects the breakdown of the structures and the subsequent bioconversion of the material. Its optimization is imperative for achieving enhanced accessibility to enzymes, thereby maximizing the overall effectiveness of bioconversion processes [28,29].

Many proposed applications require the use of the steam explosion process in sequence with other pretreatment methods in order to pre-treat lignocellulosic biomass, e.g., the production of sugars and antioxidants [30,31], the production of xylo-oligosaccharides [31], the production of ethanol [32], saccharification [33], and the production of composite [34], etc.

The main objective of the present research is to investigate the synergistic integration of steam explosion as a pretreatment step with anaerobic digestion for lignocellulosic biomass (Figure 1). The study aims to examine the impact of different steam explosion treatment conditions on the defibration process of lignocellulosic biomass and its subsequent digestion by methanogenic microorganisms. The evaluation will involve the quantification of sugar production, the assessment of sugar degradation levels, and the determination of the biogas production potential. By comprehensively exploring these aspects, a deeper understanding of the combined steam explosion and anaerobic digestion approach can be achieved, contributing to the knowledge and the potential optimization of lignocellulosic biomass utilization for sustainable energy generation.

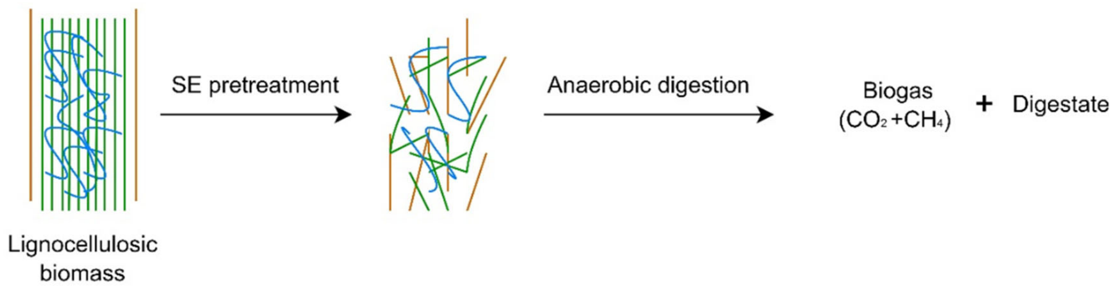

Figure 1. Lignocellulosic biomass valorisation by technological coupling of steam explosion and anaerobic digestion.

2. Materials and Methods

2.1. Feedstock and Inoculum

The lignocellulosic biomass used in the present work was softwood (Spruce) chips from the Eastern Townships region, Quebec, Canada, reduced to ¼″ (6.35 mm) in size. Two types of inoculums were used in the present work. The mesophilic inoculum was procured from the Biomethanation Center of SEMECS, Quebec, CANADA, from an organic food waste digester operating at mesophilic conditions of 37 °C. The psychrophilic inoculum was procured at the Sherbrooke Research and Development Center of Canada in Lennoxville from a manure waste digester operating at 25 °C.

2.2. Steam Explosion Pretreatment

The thermochemical pretreatment used in this paper is a steam explosion. It is dependent on the temperature (at saturation pressure) and the catalyst concentration. The hydrolysis products include oligomers, monomers, and furfural compounds produced by the breakup of intramolecular and intermolecular hydrogen bonds, depending on the process's conditions. The process consists of several steps:

1. Impregnation: The biomass was loaded into a beaker and hydrated with water. If necessary, the catalyst should be added at this step according to its required concentration ($\%w/w$).
2. Filtration and explosion reaction: the melange biomass/solvent was then filtered to remove the excess water and loaded into the steam explosion reactor. The temperature is maintained between 200–210 °C at the saturation vapor pressure. As described in the previous part, the SE is composed of two phases: steam cracking and explosive decompression. In the first phase, steam cracking consists of diffusing then condensing the steam at a high pressure inside the structure of the biomass. The water condensed at high temperatures will initiate the hydrolysis of the acetyl groups and induce the formation of organic acids. Depending on process's conditions, the acids formed catalyse the hydrolysis of the hemicellulose fractions, modify the degree of crystallinity of the cellulose fraction and the structure of lignin. During the second phase, the explosive decompression causes a sudden drop in pressure, which will cause shear forces that modify the physical properties of the biomass. The treatment (two phases) takes between 3 and 6 min. Figure 2 illustrates the operating system (SE) process.
3. At the end of the reaction, the valve of the reactor is opened to collect the biomass, which is then conserved at temperatures of 4 °C in order to avoid fungus formation at the top of the biomass.

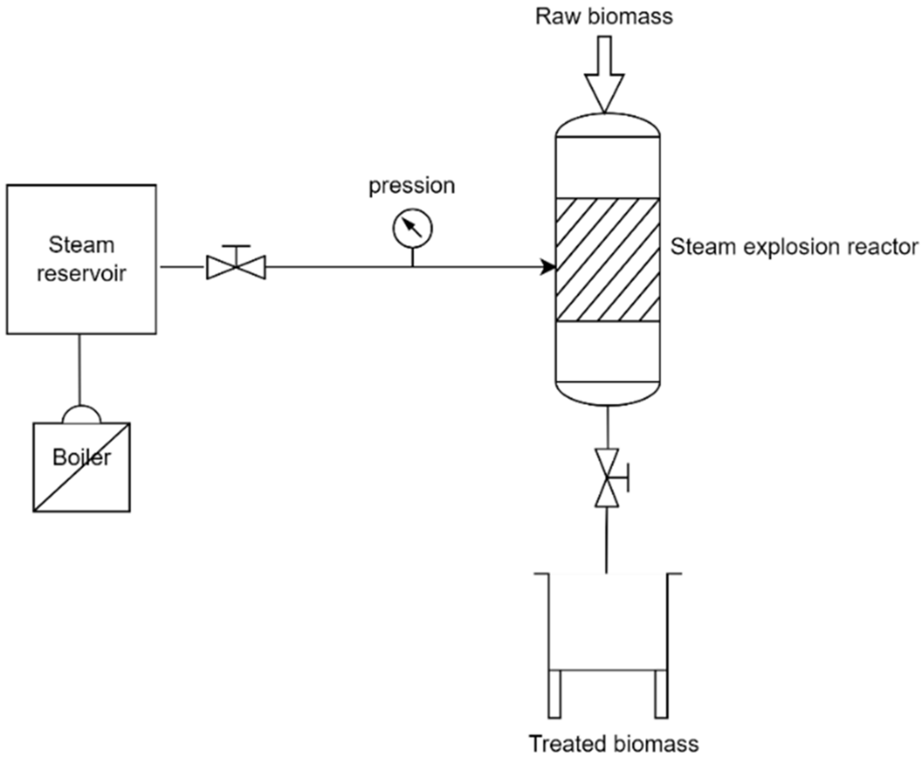

Figure 2. Schematic diagram of steam explosion process.

The steam explosion experiments detailed in Table 1 were conducted in accordance with the specified parameters. It is crucial to emphasize that the intricate details associated with the steam explosion (SE) step are exceptionally sensitive and are exclusively owned by CRB Inc. Substrates were retrieved from a mixture of different steam explosions with controlled severities (triplicates).

Table 1. Conditions of steam explosion pretreatment.

Experiment	Conditions
1	Lower conditions of T° and time
2	Medium conditions of T° and time
3	Higher conditions of T° and time
4	Medium conditions, lower concentration of an acid catalyst
5	Medium conditions, higher concentration of an acid catalyst

2.3. Anaerobic Digestion Conditions

The set-up of the anaerobic digestion of the pretreated biomass occurs in 1 L glass bottles; nitrogen was injected inside the bottles to ensure an anaerobic medium. The bottles were placed on stirrer plates to ensure uniform distribution of microorganisms, substrates, and nutriments throughout the digestion vessel. The specific conditions for the digestion process are influenced by two factors:

1. The temperature of the process: The anaerobic digestion was performed either at a mesophilic temperature of 38 °C or at a psychrophilic temperature of 25 °C.
2. The ratio of substrate to inoculum volatile solid: This parameter, denoted as SV (solids or volatile solids), determines the relative proportions of the organic biomass feedstock (substrate) and the microbial organic biomass (inoculum) used in the digestion process.

In the experimental setup, a biogas collection system was utilized to capture and measure the biogas produced during the anaerobic digestion process. Gasbags with a capacity of 250 mL were employed for this purpose.

Regular sampling is conducted at various time intervals to monitor the progress of the anaerobic digestion process. During each sampling event, biogas samples are collected from the gasbags and analysed to determine their composition. The composition analysis may involve measuring the concentrations of methane (CH_4), carbon dioxide (CO_2), and nitrogen (N_2), which are the primary components of biogas.

2.4. Analysis Methods

The analysed biomass following pretreatment underwent high-performance liquid chromatography (HPLC) to quantify the levels of organic acids, sugars, and the resulting degraded byproducts released during the steam explosion process. The analyses were carried out utilising an Agilent 1260 Infinity 2 HPLC system, featuring a variable wavelength detector (VWD), an isocratic pump, a column oven set at 60.02 °C, and a mobile phase comprising sulfuric acid (H_2SO_4).

Samples subjected to SE pretreatment under varying conditions were analysed using inductively coupled plasma (ICP) techniques PerkinElmer Avio 500. The concentrations of cations and heavy metals were determined and compared to the inhibitory IC50 value reported in the literature. The components analysed included aluminium (Al), calcium (Ca), chromium (Cr), cobalt (Co), copper (Cu), iron (Fe), molybdenum (Mo), magnesium (Mg), manganese (Mn), nickel (Ni), potassium (K), sodium (Na), boron (B), and cadmium (Cd).

The measurement of the organic loading rate in anaerobic digestion (AD) bioreactors was conducted by calculating the volatile solids (VS) ratio between the biomass and the inoculum $(S/I)_{VS}$. Volatile solids are the organic components that can be converted into biogas during the AD process; these are measured according to the standard methods at 550 °C.

The composition of the biogas generated by anaerobic digestion (AD) was analysed using gas chromatography GC (456-GC Scion) to quantify the relative concentrations of methane (CH_4), carbon dioxide (CO_2), and nitrogen (N_2). By analysing the biogas composition, the efficiency of the anaerobic digestion process and the quality of the biogas produced can be evaluated.

3. Results and Discussion

Images were taken on the solid part of the pretreated biomass and illustrated in Figure 3. According to this figure, the SE process causes significant changes to the physical state of the biomass. The high-pressure steam rapidly penetrates the biomass, resulting in the disruption of the lignocellulosic structure; this leads to the fragmentation and separation of the fibres, breaking down the lignin and hemicellulose bonds. As a result, the biomass undergoes physical and structural modifications in function of treatment severity. Under lower temperatures and pressure conditions during the steam explosion (SE), inadequate biomass destruction occurred, leading to a substantial portion of the wood remaining unaffected. As a result, this specific sample, which is not represented in the figure, has been excluded from the subsequent analyses and did not undergo anaerobic digestion treatment.

Figure 3. SE effect on wood chips morphology. (**a**) Untreated biomass, (**b**) SE with medium conditions of T and P, (**c**) SE with high conditions of T and P, (**d**) SE with a low concentration of an acid catalyst, (**e**) SE with a high concentration of an acid catalyst.

The liquid portion of the pretreated biomass was subjected to high-performance liquid chromatography (HPLC) analysis. Figure 4 presents the results of quantifying organic acids, sugars, and the resulting byproducts released during the steam explosion process:

- The concentration of Xyl-Man-Gal and glucose is highly sensitive to all three factors.
- Depending on the acid concentration, glucose is the most sensitive sugar, and it reaches its maximum at a higher acid concentration of 1%.
- 5-HMF, being a dehydration product of glucose (hexoses), increases in production with higher temperature, time, and acid concentration.
- The concentration of levulinic acid and formic acid is higher at 1% acid concentration, indicating the degradation of 5-HMF at this concentration.
- Furfural, as a degradation component of pentoses such as xylose, increases with increasing temperatures and reaction times. However, at a higher 1% acid concentration, its concentration decreases simultaneously with that of xylose.
- At a lower explosion temperature of 170 °C and a reaction time of three minutes, the conditions are not sufficient to degrade the lignocellulose, and the wood chips remained intact without any size reduction at the reactor outlet.

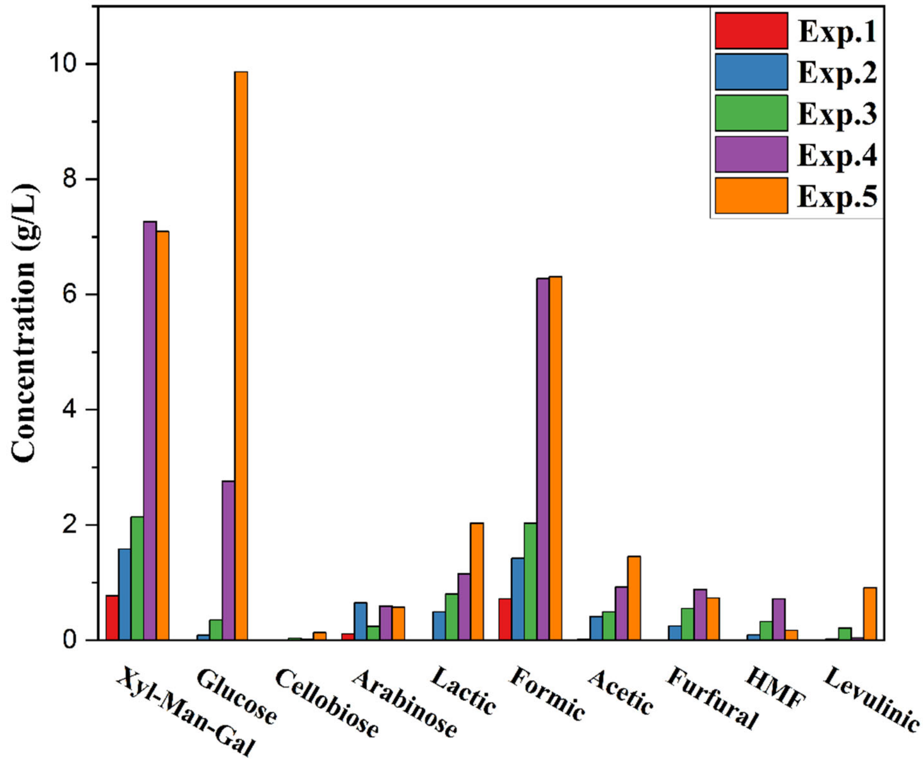

Figure 4. The HPLC Quantification of Sugars and Degradation Compounds under Steam Explosion (SE) Conditions.

The hydrolysis of lignocellulosic residues to release sugars and their derivatives is strongly influenced by the conditions of the steam explosion (SE) pretreatment. These conditions impact the breakdown of the complex lignocellulosic structure, making the carbohydrates more accessible for subsequent enzymatic hydrolysis. Optimizing the SE pretreatment conditions is essential to achieve a high sugar yield and maximize the release of sugar derivatives, such as glucose, xylose, arabinose, and other monosaccharides. Simultaneously, minimizing the production of digestion inhibitors is crucial for maintaining process stability and enhancing the overall performance of anaerobic digestion.

Table 2 presents the results of cation and heavy metal concentrations in pretreated samples under different conditions; it indicates that the concentrations of cations and heavy metals in the steam-exploded samples did not exceed the IC50 value (1 mg/L = 1000 ppb). This finding suggests that the SE pretreatment conditions employed in this study did not lead to the release of inhibitory levels of these elements, as reported by Guo et al., 2019 [35,36].

Table 2. ICP Quantification of cations and heavy metals of pretreated biomass (ppb).

Sample	Ca	Fe	K	Mg	Na	Al	Cd	Co	B
SE without a catalyst	726.7	196.9	132.8	115.7	120.3	71.1	1.7	26.4	14.6
SE with a lower concentration of the catalyst	446.6	194	125.4	84.9	177.1	131.5	1.6	26.5	16.4
SE with a higher concentration of the catalyst	462.4	318.9	122.9	62.3	268.9	159.1	1.9	26.3	31.4

Cr, Cu, Mn, Mo, Ni, Pb are absent in all samples.

Once all the analytical tests have been carried out, pretreated samples were subjected to anaerobic digestion using mesophilic and psychrophilic inoculum. The volatile solid ratio, defined as the ratio of volatile solids in the substrate to volatile solids in the inoculum, was varied and monitored during the digestion process; $(S/I)_{Vs} = 1/1$ and $(S/I)_{Vs} = 1/2$. The methane yield of each bioreactor was evaluated using GC technic. The results illustrated in Figure 5 demonstrate that the methane yield is higher in Bioreactor 1, where steam explosion occurs in the presence of lower concentrations of the acid catalyst and a substrate/inoculum (S/I) ratio of 1/2. However, a higher acid concentration (Bioreactor 3) leads to a decrease in the methane yield. On the other hand, increasing the pretreatment time (Bioreactor 2 and Bioreactor 5) improves the methane yield, while an increase in the substrate/inoculum ratio in the bioreactor (Bioreactor 2 and Bioreactor 4) negatively impacts the methane yield. These results confirm that the pretreatment of lignocellulosic waste using steam treatment/explosion in the presence of a low concentration of an acid catalyst has been proven to enhance the biomethane yield.

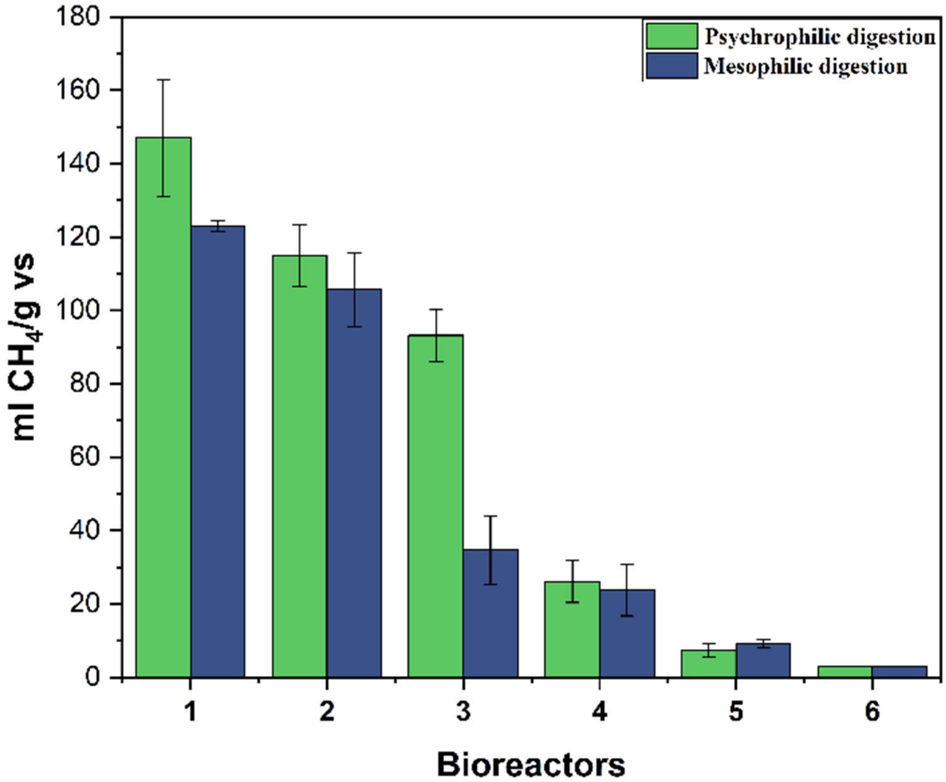

Figure 5. Cumulative methane production (mL/g vs). Bioreactor 1: SE with a lower concentration of the acid catalyst, S/I = 1/2; Reactor 2: SE with higher conditions, SF #3, S/I = 1/2; Bioreactor 3: SE a higher concentration of the acid catalyst, S/I = 1/2; Bioreactor 4: SE with higher conditions, S/I = 1/1; Bioreactor 5: SE with lower conditions, S/I = 1/2; Bioreactor 6: Raw biomass (without SE treatment).

Correlating the HPLC results (Figure 4) with methane production (Figure 5) reveals interesting trends. Bioreactor 1, characterized by SE under lower acid concentrations and a 1/2 substrate/inoculum ratio, exhibits higher a methane yield. This suggests a positive correlation between the efficient sugar release (Figure 4) and the enhanced biomethane production.

The efficiency of the digestion process was evaluated by analysing the conversion of organic matter by methanogenic microorganisms. This analysis was conducted based on the measurement of volatile solids at the beginning and the end of the anaerobic digestion process in Bioreactors 1, 2, and 3, which demonstrated substantial biomethane production. The results are summarized in Table 3. The data clearly indicate that the conditions employed in Bioreactor 1 (presence of a lower concentration of the acid catalyst) result in the conversion of over 70% of the lignocellulose into biomethane.

Table 3. The efficiency of the anaerobic digestion process in function of volatile solid conversion.

Bioreactor	Conditions	Conversion (%)
Bioreactor 1	SE with a lower concentration of the acid catalyst, S/I = 1/2	72.64 ± 0.59
Bioreactor 2	SE with higher SE conditions, S/I = 1/2	36.71 ± 0.82
Bioreactor 3	SE with a higher concentration of the acid catalyst, S/I = 1/2	1.22 ± 0.06

As known, in the context of a steam explosion, the catalyst plays a vital role in promoting the breakdown of complex biomass components into simpler, more digestible compounds. As a result, the optimised catalysed steam explosion process leads to higher conversion rates of organic matter into biomethane.

4. Conclusions

The utilization of lignocellulosic biomass for biomethanation presents numerous advantages, such as the use of non-food resources, the potential valorisation of waste, and a reduction in greenhouse gas emissions. By implementing advanced process optimization and bioreactor design, the potential of biomethanation from lignocellulosic biomass can be further explored to unlock its full potential as a sustainable and renewable energy solution.

The novelty of the present work lies in the development of an innovative methodology that maximizes the valorisation and utilization of lignocellulosic waste by coupling the steam explosion process at critical conditions with anaerobic digestion. The optimization of steam explosion pretreatment conditions is crucial for the efficient hydrolysis of lignocellulosic residues, releasing sugars and their derivatives, which serve as valuable feedstocks for downstream processes. Through the careful consideration of steam explosion conditions, such as temperature at saturation pression, time (3–6 min), and utilising the acid catalyst at lower concentrations (<1%), the desired level of sugar release can be achieved while preserving the quality of the released compounds and preventing the formation of undesirable byproducts, such as degradation compounds or inhibitory substances. Additionally, the analysis of the $(S/I)_{vs.}$ ratio provides valuable insights into determining the optimal substrate/inoculum ratio for efficient organic degradation and biogas production. A value of (1/2) has been identified as the optimal ratio for this purpose, contributing to the maximization of the biogas yield from the anaerobic digestion process. In conclusion, the optimization of steam explosion parameters and the $(S/I)_{vs.}$ ratio are crucial steps in evaluating the feasibility of the combined treatment approach and advancing the utilization of lignocellulosic biomass for renewable energy production. Statistical and economic assessment will be essential to comprehensively evaluate the sustainability of the process coupling for converting lignocellulose waste into biogas This study will provide valuable insights into the feasibility, cost-effectiveness, and potential returns on investment associated with this sustainable process.

Author Contributions: Conceptualization, I.E.A. and N.A.; methodology, O.C.; software, O.C.; validation, O.C., I.E.A. and N.A.; formal analysis, O.C.; investigation, O.C., I.E.A. and N.A.; resources, I.E.A. and O.C.; data curation, O.C.; writing—original draft preparation, O.C.; writing—review and editing, O.C., I.E.A. and N.A.; visualization, O.C., I.E.A. and N.A.; supervision, N.A. and I.E.A.; project administration, N.A. and I.E.A.; funding acquisition, I.E.A. All authors have read and agreed to the published version of the manuscript.

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