

Review

Utility Aspects of Sugarcane Bagasse as a Feedstock for Bioethanol Production: Leading Role of Steam Explosion as a Pretreatment Technique

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Abstract: Lignocellulosic biomass is a powerful approach to produce sustainable biofuels and the further achievement of the goal of biomass conversion into a second-generation clean energy that can cope with the depletion of fossil reserves and rising energy requirements. In the conversion process, a pretreatment is essential to overcome the recalcitrance of the lignocellulosic biomass; accelerate its disintegration into cellulose, hemicellulose, and lignin; and, in turn, obtain an optimal yield of fermentable sugars in the enzymatic hydrolysis. In addition to this, it should be industrially scalable and capable of enhancing fuel properties and feedstock processability. Here, steam explosion technology has stood out due to its results and advantages, such as wide applicability, high efficiency in the short term, or lack of contamination despite its conventionality. This gentle and fast pretreatment incorporates high temperature autohydrolysis and structural alteration by explosive decompression. The steam explosion method has been one of the most effectual, especially for the hydrolysis of cellulose from agricultural wastes due to the lower quantity of acetyl groups in the composition of hemicellulose. In this aspect, sugarcane bagasse is a promising feedstock for bioethanol production due to its high cellulosic content and elevated availability. The objective of this review has been to compile the latest information on steam explosion pretreatment, stages, equipment, variables involved, by-products generated, as well as the advantages and disadvantages of the technique. At the same time, its feasibility and viability using sugarcane bagasse as feedstock has been discussed. Finally, the effectiveness of the technique with different feedstocks has been evaluated.

Keywords: steam explosion pretreatment; sugarcane bagasse; lignocellulosic biomass; bioethanol; feedstock



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1. Introduction

The necessity of finding alternative pathways for power supplementation in a more sustainable way has been promoted because of the current carbon emissions that contribute to climate change [1]. Thus, the energy-from-waste (EfW) concept has been introduced as an alternative pathway where the use of refuse-derived flues, household waste and non-hazardous industrial by-products are considered as potential sources for energy production [1]. This productive model fits with the sustainable model of circular economy in which the concept ‘end-of-life’ is replaced by reutilization, recycling and recovering. Hence, by-products or wastes from one industry become raw material for another. In addition, another productive model can be easily combined with bio-economy, which promotes the use renewables based on biomass by-products [2]. Indeed, in the past years, the production of renewable fuels using lignocellulosic waste from agricultural activities has been considered as an alternative to traditional fuels [3]. Agricultural residues are defined as unusable and unstable materials derived from agricultural production which are directly linked to the cultivation of crops, and these materials are characterized

by their biodegradability and solid and lignocellulosic composition [3,4]. In this way, the lignocellulosic non-edible biomass discarded by agriculture feedstock can be used as raw material to obtain biofuel, being considered second-generation (2G) biofuel [1,5]. Lignocellulosic biomass is mainly composed by cellulose, hemicellulose, and lignin, which are low energy-density compounds, so a pretreatment step is necessary so plant-specific enzymes can release sugars for biofuel production [5,6]. Moreover, 2G biofuels present several advantages compared to first generation biofuels, such as the cheaper non-edible matter used since it is the waste obtained as a result of an industrial activity; no fuel–food competition since non-edible matrices are used; and the reuse of by-products obtained after pretreatment of raw materials to obtain animal fodder to be used, for example, in livestock feed, thus reinforcing the circular economy. In this way, sugarcane bagasse (SCB), wheat, barley, rice and corn straw, and sorghum can be used for bioethanol production [7]. In the specific case of sugarcane production, it has an associated production of residues that may reach up to 30% of the production [8], which may represent more than 100 MT of residues. This huge volume of residues has several drawbacks. From an economic point of view, they require being effectively managed, which has an associated cost for the industry. From an environmental point of view, they represent a potential source of CO₂ since they are likely to be burned or, if not, accumulated in landfills, which threatens the quality of environmental (i.e., air and water pollution and noise, among others, and their potential consequences) and public health. Therefore, the reutilization of these by-products as part of a circular economy model is key to reduce management costs of residues to sugarcane industries and their negative environmental impact. In fact, their reutilization as raw material for bioethanol production has double benefits. It would avoid the release of this biomass to the environment, and it would promote the use of bioethanol produced using SCB, which is less carbon intensive compared with fossil fuel, so air pollution can be reduced [9]. Therefore, these two approaches have the potential of reducing the footprint of sugarcane industries and fuel utilization.

As previously mentioned, pretreatment is an essential technological step for the conversion of lignocellulose into fuels and biochemicals [10]. The aim of this preliminary step is to reduce the lignin and/or hemicellulose content by modifying the cell wall structure of the biomass. This processing increases the surface area and accessibility to carbohydrates such as cellulose, thereby increasing the yield of fermentable sugars [11,12]. However, it is worthy to note that the pretreatment stage has a relevant economic impact since it may represent over 40% of the overall cost of a cellulosic ethanol process [11]. Therefore, the selection of a suitable pretreatment will significantly increase the efficiency of the hydrolysis [13]. Comparative studies are relevant for the optimization of each pretreatment technology on an industrial scale [14]. Some of the most commonly applied pretreatments are bleaching mixtures (acetate buffer and sodium chlorite aqueous solution at 100 °C) and alkaline (sodium hydroxide at 120 °C) and/or acid (acetic acid and nitric acid at 105 °C) methods. For instance, these methods have been used for the isolation of cellulose from agro-industrial residues such as corn cobs, corn husks, grape wood, pomegranate peel, strawberry pomace or fava pod. The highest cellulose extraction yield (26%) was obtained for corn cobs. Nevertheless, the presence of lignin and hemicellulose was not confirmed [15]. Cellulose was also isolated from wheat straw using acidified sodium chlorite and alkaline hydrogen peroxide at temperatures below 80 °C. Along with this work and after the analysis of the pre-treated residues, it was found that both methods were successful in isolating cellulose from wheat straw and increasing its content, achieving yields of 81.4% and 79%, respectively [16]. Regarding the effectiveness of pretreatments for the revaluation of SCB, numerous approaches have also been described, for example, in the optimal application of Na₂CO₃ and ionic liquids (ILs) to extract cellulose microfibrils and, afterwards, cellulose nanofibrils (CNFs) from

SCB. This approach provided a maximum yield (obtained as the percentage of the weight of the residue after pretreatment divided by the initial weight of the washed bagasse used) of 83.77% and a crystallinity index of 62.80% [17]. However, steam explosion (SE) pretreatment is becoming more attractive than other pretreatment techniques because it is a hassle-free method with low capital investment, high scalability, and reduced chemical hazards in the process, among other advantages [18]. To illustrate, a recent study found that sugarcane pretreatment conditions by SE lead to highest hydrolysis conversion with 80% at 200 °C with an SF of 4.12. Before finding the optimal condition for pretreating SCB, the authors assessed various temperature conditions ranging from 170 °C to 200 °C. Yet, in order to ascertain the actual productivity of SE, economic analyses are essential to elucidate the real potential of the process [19]. In addition, SE has been assessed with several raw materials. Considering the increasing interest in obtaining new alternative pathways for the fuel production generated by both climate change and circular economy perspective, different studies have highlighted the advantages presented by SE pretreatment [20,21]. This physicochemical pretreatment seems to be suitable for lignocellulosic biomass bioethanol production. In this way, SCB has been also studied as a potential raw material for this biofuel production [13,21]. Thus, this review aims to compile current information regarding 2G biofuel production focusing on two parameters: the pretreatment technique applied (steam explosion) and the feedstock used (sugarcane bagasse).

2. Sugarcane Bagasse as a Potential Raw Material for Bioethanol Production

Sugarcane (*Saccharum officinarum* L.) is a tropical grass that belongs to the Gramineae family and the *Saccharum* spp. genus [22] and is characterized for being large and perennial. The sugarcane cultivation requirements are 6–12 months to grow with 60–100 cm³ of water [23]. Brazil, China and India are the main producers of SCB, with almost 500 MT generated every year from the sugarcane industry, which provides an important contribution to economic development [9,22,24]. SCB yielded the highest crop straw production between 2012 and 2022 according to the Food and Agricultural Organization Corporate Statistical Database (FAOSTAT) [25]. Sucrose is the main product of sugarcane, which accumulates in the internodes of the stalk [9]. However, the percentage of waste generated during sugarcane production varies between 25 and 30% [8], which may represent 125–150 MT of residues. The residues produced by the sugarcane industry are mainly two types, and they can be classified as straw, which is the harvest residue, and as bagasse, which is the fibrous fraction after the extraction of the sugarcane stem juice [22]. These two by-products are characterized by their lignocellulosic composition, with cellulose, lignin and hemicellulose being the major components and having also extractants and ashes in their composition [26]. SCB is composed of approximately 45–50% cellulose, 25–30% hemicellulose, 25% lignin and 2.4–9% ash [9]. Nevertheless, this may vary depending on different factors such as chemical composition of the soil, climatic conditions and variety of the crop, among others [9]. Table 1 shows variations in the composition of SCB, sugarcane fiber and sugarcane straw. The relative abundances of component units are usually calculated on the basis of the volume integration of the raw material and are expressed in percentage of dried weight [27]. The chemical characterization of vegetal biomass is highly relevant for their consideration as potential sources of carbon to produce bioethanol. Indeed, for bioethanol production, it is important to evaluate the cellulose, hemicellulose and lignin composition of the raw material [9]. Owing to its high yield of sugar and lignocellulosic biomass, SCB is regarded as an excellent alternative energy source to substitute fossil fuels [28,29].

Table 1. Composition of the main sugarcane chemical compounds expressed in percentage of dried weight.

| RM | Pretreatment before Measurement | Cellulose | Hemicellulose | Lignin | Xylan | Sucrose | Ash | Ref. |
|-----|--------------------------------------|-----------|---------------|--------|-------|---------|------|------|
| SCB | Not specified | 57.68 | 12.41 | 7.89 | - | - | 2.20 | [30] |
| SCB | Dried in an oven at 65 °C | 29.19 | - | - | 16.51 | 25.75 | - | [31] |
| SCB | Air-dried at 50 °C | 36.4 | 20.1 | 29.9 | - | - | 5.4 | [27] |
| SCF | Dehydration in hot air oven at 60 °C | 39.70 | 36.39 | 7.37 | - | - | 5.63 | [32] |
| SCB | Dried at 105 °C | 38.8 | 26 | 32.4 | - | - | 2.8 | [33] |
| SCS | Air-dried until a 10% final humidity | 33.5 | 27.1 | 25.8 | - | - | 2.5 | [34] |
| SCS | Not specified | 44.5 | 30.4 | 12.3 | - | - | 7.5 | [35] |
| SCB | Non-treated | 40.1 | 23.8 | 23.6 | - | - | 3.5 | [10] |
| SCB | Non-treated | 38.7 | 23 | 16.9 | - | - | - | [36] |
| SCB | Air-dried at NST | 43.1 | 31.1 | 11.4 | - | - | 5.5 | [37] |
| SCB | Not specified | 57.68 | 12.41 | 7.89 | - | - | 2.20 | [30] |

Abbreviations: RM: raw material; SCB: sugarcane bagasse; SCF: sugarcane fibers; SCS: sugarcane straw; NST: no specified temperature.

As stated above, biomass characterization is a noteworthy step for establishing optimal process conditions. Hence, delineating the specificities of SCB as a lignocellulosic feedstock is key to its close scalability. Concerning these aspects, several authors [34] conducted a study in which 60 bagasse samples were characterized. The results provided the following average of the structural compounds of SCB: 42.2% of cellulose, 27.6% of hemicelluloses, 21.6% of lignin, 5.63% of extractives and 2.84% of ashes. As can be seen in Table 1, the composition of the samples did not differ substantially between them. This research supports the vantage of using SBC for obtaining bioethanol since the bioethanol yield is closely related to the biomass composition.

Moreover, the composition of feedstocks tends to vary depending on a large number of factors, so the stability of SCB may be considered an important advantage over other raw materials [38]. Another work serves as example of the potential of SCB to obtain bioethanol [39], where the authors considered the biorefinery route to convert SCB into various products, such as nanocrystalline cellulose, lignin and biohydrogen. They further outlined the rationale for selecting SCB as feedstock, arguing the availability of surplus bagasse, elimination of logistics, lower pretreatment costs, and additional revenue for the industry in the off-season. The findings revealed how over 80% of the SCB biomass was biorefined to yield the target products with a zero-liquid discharge strategy [39]. Another different point from SCB is the fact that it usually provides a high organic content (>90% on a total solids basis) which results in a high theoretical biofuel yield [40]. It is emphasized that SGB is in a niche as it is being used on a large scale for bioenergy and biorefinery, and therefore provides a training ground for new innovative technologies [41]. To move further away from the chemical composition of SCB, the world's annual SCB has a lignocellulose potential of 243 million tons, or 4.3 EJ on an energy basis, equal to 6.8% of the world's current bioenergy supply. It completes to give prominence to the potential of SCB lignocellulose compared to other feedstocks and its value as a renewable resource [40,41]. Since cellulose, hemicellulose and lignin have a high association, it is necessary to apply a pretreatment method that disrupts the plant cell wall organization so the polysaccharides can be more accessible to enzymes [7]. As a suitable application of SE as pretreatment for bioethanol production using lignocellulosic biomass, SCB has been studied as a potential feedstock by different authors [27,42,43].

As previously explained, SE pretreatment achieves hemicellulose hydrolysis, lignin transformation and cellulose crystallization by applying high temperatures (160–270 °C) and pressurized steam (20–50 bar) for a time that varies between seconds and minutes [44]. Therefore, when SE is applied, there are different parameters affecting the sugar release off the feedstock. The most relevant ones include particle size, temperature, residence time and the combination of temperature and residence time, also named SF [44]. In this way, the different applications of SE using SCB have been optimized by several authors. Their achievements and conclusions are presented below. For instance, Espirito Santo et al. [45] studied SE applied in SCB at different conditions, including the combinatorial use of SE

with H₂SO₄ and SE with H₃PO₄. A better cellulose yield was achieved when SE was simply applied since the combined used with H₃PO₄ led to higher lignin yield, whereas the incorporation of H₂SO₄ led to a higher hemicellulose yield, as it is shown in Table 2. The optimized time, pressure and temperature conditions where the cellulose released was the highest for each pretreatment were 200 °C, 10.5 min, 14.2 atm (SE); 180 °C, 4 min, 10 atm (SE + H₂SO₄); and 195 °C, 7.5 min, 14.2 atm (SE + H₃PO₄) [45]. Moreover, results obtained in this study showed that SE pretreatments using high temperature and short residence time lead to better yields than the combination of low temperature and long residence time. This phenomenon is explained because of the accumulation of fermentation inhibitory by-products, such as organic acids, furan compounds and phenolic acids, that ultimately lead to yield losses [45]. Other authors also studied the SCB pretreatment with SE. Results showed how short residence time pretreatments lead to better hemicellulose removal, in agreement with the outcomes of other previously published studies [46]. The results indicated that the higher removal of hemicellulose and lignin was obtained when operational conditions of SE were 210 °C, 15 min and 1% of H₂O₂, achieving 92.4 and 29.7% removal, respectively [46]. Researchers also compared SE and acid hydrolysis (AH) applied in SCB to obtain bioethanol [47]. Their results showed a six-times higher carbohydrate yield when SE was applied when compared to AH. Moreover, the negative impact of long residence time was confirmed since results showed that pretreatment over 30 min leads to lower total amount of carbohydrates [47]. In fact, the higher yields of carbohydrates obtained in this study were when SE was applied with 160 °C, 30 min and 6.805 atm, as it is shown in Table 2 [47]. Autohydrolysis (AHS) of SCB was also studied as an environmentally friendly pretreatment to obtain bioethanol. In fact, the authors ran a study where AHS pretreatment was applied in SCB matrix to obtain xylooligosaccharides [48]. The yields achieved after this pretreatment ranged from 51.88 to 66.67% of bioethanol. The differences obtained were related to the use of a buffer solution that the stabilized pH and led to a maintenance of both cellulose and yeast activity [48]. A novel pretreatment methodology applied to SCB was run by Duy The Pan and Chung-Sung Tan by using supercritical CO₂ [49]. The authors compared the glucose recovery obtained after 72 h of enzymatic hydrolysis with three pretreatments: single supercritical CO₂, supercritical CO₂ followed by H₂O₂ and supercritical CO₂ followed by ultrasound. The higher glucose recovery, with a yield of 97.8%, was obtained when supercritical CO₂ was combined with H₂O₂. Moreover, this pretreatment was the only capable of increasing the glucose recovery after 48 h of enzymatic hydrolysis [49]. Considering that one shortcoming of obtaining bioethanol from SCB is the inhibition produced by different compounds, including lignin, the authors ran a study where sequential NaOH and hydroxymethylation pretreatment was applied [50]. The authors compared this sequential process with the single alkaline pretreatment, achieving an increment of 13% of bioethanol, which was linked to the lower lignin content found in the sequential NaOH pretreatment followed by a hydroxymethylation process [50].

Table 2. Comparison of bioethanol and other compounds recovery considering different sugarcane bagasse (SCB) pretreatments.

| Pretreatment | Operational Conditions | | | Recovery (%) | Ref. |
|-------------------------------------|------------------------|------------|----------------|--------------------------------|------|
| | Temp (°C) | Time (min) | Pressure (atm) | | |
| SE+AHS | 195 | 7.5 | 18 | 73.8 bEtOH, 0.58 g/L/h EtOH | [7] |
| AHS | 200 | 10 | - | 51.88–66.67 bEtOH (11.96 g/L) | [48] |
| K ₃ PO ₄ 6.4% | 144 | 60 | - | 53.04 bEtOH | [51] |
| SE | 160 | 30 | 6.8 | >150 mg/g TC, 87.16 mg/mL EtOH | [47] |
| H ₂ SO ₄ 10% | 100 | 60 | - | 251.1 mg/g TC, 58.7 mg/mL EtOH | [47] |
| SE+H ₂ O ₂ | 210 | 15 | - | 86.9 C; 92.4 HM; 29.7 Lig | [46] |

Table 2. Cont.

| Pretreatment | Operational Conditions | | | Recovery (%) | Ref. |
|---|------------------------|------------|----------------|--|------|
| | Temp (°C) | Time (min) | Pressure (atm) | | |
| SF-CO ₂ +H ₂ O ₂ | | 60 | | 97.8 Glu | |
| SF-CO ₂ +Ultrasound | 187 | 240 | 154 | 65.8 Glu | [49] |
| SF-CO ₂ | | - | | >55 Glu | |
| NaOH 0.7% | 70 | 360 | - | 53.3–68.8 Glu; 67.8–74.7 xylose → 10.67 g/L | [50] |
| NH ₄ -OH-H ₂ O ₂ +IL | 100 | 360 | - | 87.4 Glu; 55.5 glucan; 19.8 xylan 0.42 g EtOH/g G, 14.1 g/L EtOH | [36] |
| Imidazole | 160 | 60 | - | 55.7 solid | |
| HOAc | 107 | 30–90 | - | 80 bEtOH | [52] |
| Na ₂ CO ₃ | 195 | 15 | - | 69.1 C; 4.1 HM; 9.5 lignin → 16.1 g EtOH/100 g biomass | [37] |
| SE | 200 | 10.5 | 14.2 | 52 C; 3.9 HM; 33.1 Lig | |
| SE+H ₂ SO ₄ | 180 | 4 | 10 | 50.5 C; 6.9 HM; 30.8 Lig | [45] |
| SE+H ₃ PO ₄ | 195 | 7.5 | 14.2 | 50.2 C; 2.7 HM; 35.2 Lig | |

Abbreviations: SE: steam explosion; AHS: autohydrolysis; SW: subcritical water; EtOH: ethanol; bEtOH: bioethanol; TC: total carbohydrates; IL: ionic liquid; HOAc: acetic acid; Glu: glucose; C: cellulose; HM: hemicellulose; Lig: lignin.

Overall, and as it is shown in Table 2, there are several works that have studied the suitability of SE and other pretreatments in SCB as biomass. In this way, SE has shown successful results that strongly support the application of this pretreatment to produce bioethanol through the reutilization of SCB.

3. Steam Explosion as Lignocellulosic Biomass Pretreatment

SE was pioneered and patented as a biomass pretreatment process in 1926 by Mason [53]. SE pretreatment is a physicochemical modification technology that couples autohydrolysis and biomass alteration through high temperature and explosive decompression with application in food raw materials [43]. SE processes can be operated in continuous or batch mode. Batch reactors are usually used for laboratory-scale pretreatment, while continuous systems are typically used for large-scale industrial processes [43,53]. The lignocellulosic materials that can be treated with SE are extensively diverse [54]. Indeed, its competence has been successfully demonstrated in the fractionation of a broad range of lignocellulosic raw materials, such as wheat straw, hay, SCB, corn stover, birch wood and numerous other chemical platforms from a large range of lignocellulosic feedstocks [55,56]. Figure 1A shows a schematic diagram of the continuous operation process of SE using SCB as biomass where the main three parts of the equipment are represented: steam generator, steam explosion chamber, and material receiving container [57].

The SE process is usually divided into two independent stages. An initial one where the vapor boiling and explosion phase takes place, hence along this stage thermochemical reactions operate. For this, SE presses steam at high pressure (1–3.5 MPa) and temperature (180–240 °C) into cell walls and plant tissues for an abbreviated period (30 s) to several minutes (20 min). The second phase, in which physical tearing occurs, is a process of adiabatic expansion and conversion of thermal energy into mechanical energy [58,59]. Thus, its high throughput relies on combination of the thermochemical action of high-temperature boiling coupled with the physical tearing action of instantaneous blasting [43,57,60]. Temperature and residence time are known as the combined pretreatment severity factor (SF). In this regard, the recalcitrance of the biomass (e.g., lignin content) to the hydrolysis process is one of the conditions that most affects this factor [55,61]. Generally, SF is utilized in the analysis of reaction kinetics with solid and liquid phases involved. In this way, the aim of the SF calculation is to come up with a pretreatment strategy approach that fulfills the expected requirements for the product and the process [62]. Specifically, the SF was designed to enable both process monitoring and prediction of cellulose, hemicellulose and lignin after pretreatment. The effects of pretreatment are assumed to follow first-order

kinetics and to fit to the Arrhenius equation [63,64]. Hence, SF is an influential parameter that defines the relationship between hydrothermal severity (operating conditions and physicochemical changes) and lignocellulosic biomass fractionation [65], and provides an estimation of the intensity of the SE treatment. Indeed, to reach the maximal efficiency of SE pretreatment, it is required to optimize the factors that modulate the toughness of the pretreatment conditions. The inter-dependence of these factors (SF) is calculated through R_0 , a parameter that may be considered as a scaling strategy for a batch operation of the SE reactor (Equation (1)).

$$\left(R_0 = e^{T_{exp}-100/14.75} \right) \quad (1)$$

It is calculated by combining the lignocellulose pretreatment reaction time and temperature related to the boiling point of pure water into one single parameter, where, T_{exp} refers to the experimental temperature. Therefore, 100 is the reference temperature and 14.75 is the arbitrary constant ω being the activation energy of the first order kinetics [66].

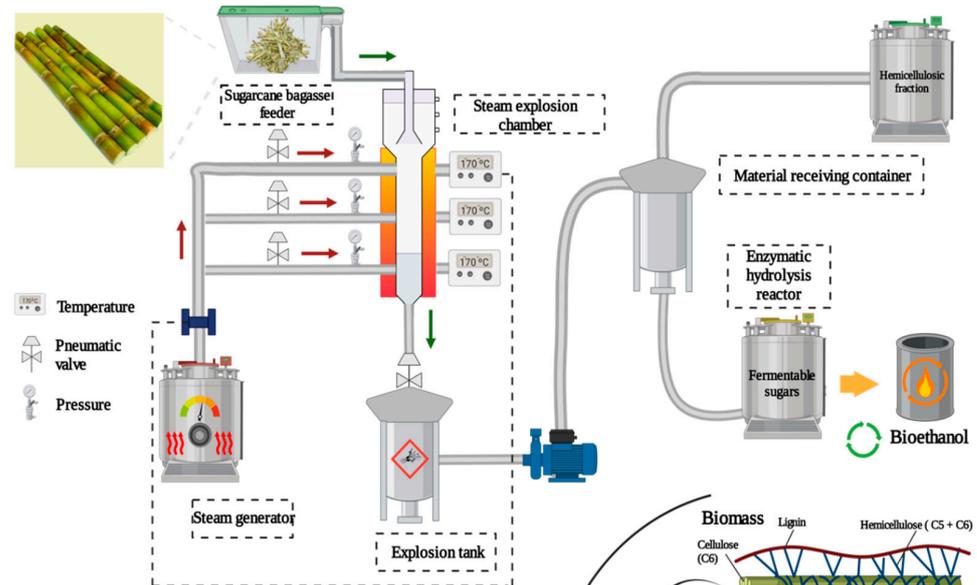
Other influencing factors are biomass particle size, moisture content, the rate of diffusion of vapor and liquid through the particle, the ratio of solid to liquid loaded in the SE container, the presence of chemical solvents involved in previous steps, or the addition of a chemical catalyst prior to steam pretreatment [55,61,67]. To determine the optimal combinations of these factors, it would be required to carry out an endless series of experiments which are strongly minimized through the application of statistical designs of the experiments [68].

To propose the development of a factorial experiment that will cover all possible combinations of the selected levels, considering the impact of all factors and the interactions between them, would serve as a powerful tool that brings the most comprehensive insight into the behavior of the system [69]. For example, in a work assessing the influence of pretreatment SF on the fractionation of softwood using a protic acidic ionic liquid, the statistical analysis consisted of the design of a three-level business requirement document (BRD)/response surface method (RSM) involving three key pretreatment variables. Thus, the respective levels of each variable were 20, 30 and 40 min (time); 160, 170 and 180 °C (temperature); and 70, 80 and 90 wt%, while the response was the extraction of lignin. The key conditions of the process made it possible to achieve a quick pretreatment, which yielded a pulp rich in highly digestible cellulose (>90% glucose yield) [63].

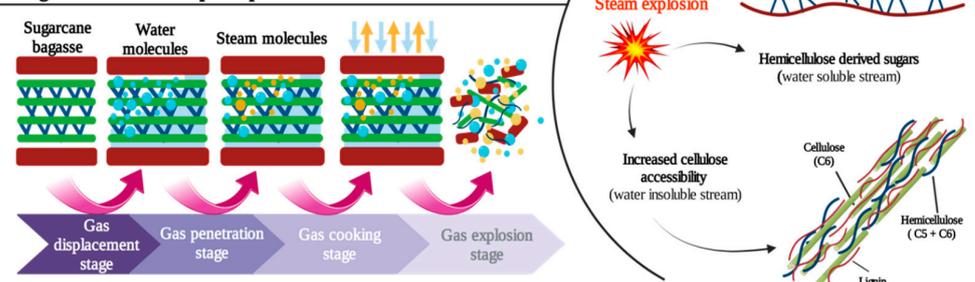
In a deeper way, in the above two vapor explosion phases, the following processes are principally involved: acid hydrolysis, thermal degradation, mechanical fracture, hydrogen bond breakdown and structural rearrangement [57]. Subsequently, these stages will be developed focusing on a specific feedstock. Figure 1B shows the structural changes of the three main components of lignocellulosic biomass during SE pretreatment. This hydrolysis leads to the decomposition of the lignocellulosic raw material by the alteration of the chemical structure of lignin. Lignin depolymerizes by cleavage of the β -O-4 bonds, and the fragments condense, giving rise to a more stable polymer [70]. This depolymerization may eventually trigger a partial removal and/or redistribution of lignin [12]. The alteration of the native lignin structure and its redeposition in the pretreated biomass are complicated interactions. They are dependent on the source of the biomass and the detailed heat and mass transfer reactions occurring inside the specific SE reactor and still require intensive investigation [71]. Therefore, the removal of biomass components such as hemicelluloses and lignin will lead to a significant increase in glucose yield after enzymatic hydrolysis [20]. This increase in yield can range from 20% to 85%, depending in many cases on the severity conditions used during SE [72]. Thus, the benefits of hydrolysis, apart from enhancing the extractability of lignin polymer, result in the enhancement of the biodegradability of the raw material. More specifically it involves the release of mono- and oligosaccharides, the improvement of cellulose accessibility and the reduction in the crystallinity index of the holocellulosic content [73,74]. In the case of cellulose, it suffers nearly no structural changes, it is mostly retained in its original form, and only mild depolymerization occurs under soft reaction conditions. However, apart from the solubilization of carbohydrate

polymers into soluble sugars (mainly glucose, xylose and arabinose), the pretreatment also results in the formation of lignocellulosic by-products, as illustrated by Figure 1C [75].

A. Standard set-up used for the pretreatment of sugarcane biomass by steam explosion



B. Lignocellulosic disruption process and molecules released



C. Overview of by-products derived from steam explosion (SE) biomass pretreatment

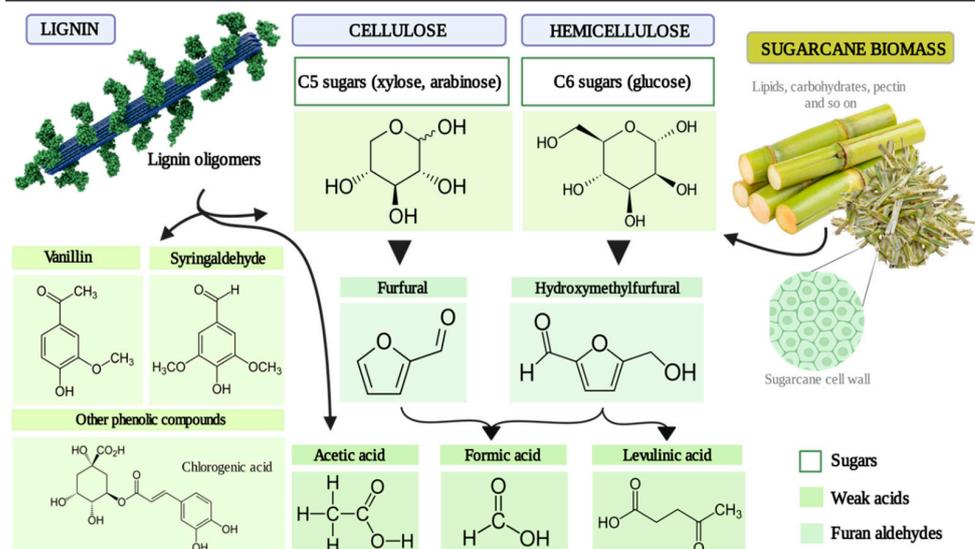


Figure 1. An insight into steam explosion (SE) pretreatment for bioethanol production using sugarcane bagasse (SCB) as biomass. (A). Diagram of industrial SE pretreatment of SCB for bioethanol production. (B). Structural changes of SCB during SE pretreatment. (C). By-products obtained after applying the steam explosion pretreatment in sugarcane biomass.

Nowadays, SE is considered to be the only physical pretreatment method that can be applied alone or in combination with other chemical pretreatments to efficiently delignify biomass [76]. This has often been combined, for example, with wet oxidation intended to treat larger particle sizes and to operate with higher substrate loadings [77]. Further outstanding benefits of SE pretreatment are the extensive hydrolysis of hemicellulose polymers and the reduction in biomass particle size [25,71]. Smaller particles have more available surface area and the lignin droplets act as a binder, which improves particle-to-particle contact and binding capacity [43]. Also, SE has a high potential for energy efficiency, low capital investment, and lower environmental impact compared to other pretreatment technologies. [78]. Nonetheless, a significant contribution of studies is still needed to propose an economically valuable utilization of biomasses such as SCB and to explore the different limitations that may arise when selecting a pretreatment as SE.

4. Steam Explosion Bioethanol Production Applications in Diverse Matrices

The highly efficient applicability of the SE as pretreatment process for lignocellulosic biomass may trigger its utilization with a wide variety of raw materials. Indeed, this thermomechanical technique has already been applied to different biomass types. Therefore, a brief analysis is presented below to provide an overview of the potential expansion of this pretreatment technique to other agro-industrial residues. Thus, this section compiles published results from different studies using coffee husks, olive tree prunings, reeds, hornbeam wood, sorghum, and corn. These studies have been synthesized in Table 3.

Table 3. Synthesis of results obtained for different feedstocks using the SE technique.

| Biomass Raw Material | Temp (°C) | Time (min) | P (bar) | SF (S ₀) | Results | Highlights | Ref. |
|----------------------|-----------|------------|---------|----------------------|---|--|------|
| Coffee husks | 210 | 15 | - | 4.41 | %RM: 62.2 C; 54.1 HM; 43.3 Lig; 3.49 (C+HM)/Lig (g/g) | 48.6% EH | [42] |
| Coffee husks | 120 | 60 | 2 | 2.37 | %RM: 28.9 C; 16 HM; 38.9 Lig; electricity production 0.59 kWh kg/CH | methane: 144.96 NmL CH ₄ g/COD (yield); 144.1 g bEtOH/kg dry raw material | [79] |
| OTPs * | 210 | 15 | 20 | 4.41 | 4.23 Glu; 3.72 xmg; 0.55 AR; 0.67 HCOOH; 1.87 HOAc (g/100 g RM) | bEtOH/kg dry raw material | [80] |
| Reeds | 200 | 15 | - | 4.12 | %DM: 21.5 (non-pretreated); 93.8 DM (pretreated) 43.4 C; 0.1 HM; 15.2 Lig | 89% methane (yield) | [81] |
| Hornbeam wood * | 190 | - | 28 | 4.08 | %DM: 32.1 glucan; 16 xmg; 25.4 Lig; 7 TS; 13 EL; 98.4 Glu and 64.6 FS | 251 L bEtOH/ton of DM | [82] |
| Sorghum | - | 5 | 15 | - | bEtOH yield: 20.5 g/100 g; reducing sugar yield: 49.6 mg/g | 43 g residues | [78] |
| Wheat straw * | 50 | 3 | 12 | 3 | The highest soluble xylose extraction was 284 mg/L | Yield over 94%. | [66] |
| Corn | 200 | 2 | - | - | 90.3% bEtOH; %RM: 60 HM | 0.5% of H ₂ SO ₄ was used | [83] |
| Corncoobs | - | 5 | 10 | - | %Conversion: 83.4 sugars; 90 glucan; 41 xylan | Interactions of recalcitrant factors | [84] |

Abbreviations: Temp: temperature; P: pressure; SF: severity factor; RM: removal material; C: cellulose; HM; hemicellulose; EH: enzymatic hydrolysis yield; OTPs: olive tree prunings; xmg: xylan-mannan-galactan; AR: arabinose; HCOOH: formic acid; HOAc: acetic acid; bEtOH: bioethanol; DM: dry matter; EL: enzyme loading; FS: fermentable sugars; Glu; glucose; Lig: lignin; Glu: glucosa; TS: total solids. * Response surface methodology.

The authors separated the solid and liquid fractions of coffee husks after SE pretreatment for saccharification and biogas generation [42]. Results evidenced that the pretreatment was an essential step to minimize the biomass recalcitrance and to promote the contact between lignocellulosic substrate and enzyme. In this way, the mass transfer from the solid to the liquid phase was enhanced and the hydrolysis stage was boosted. In addition, the increased biodegradability of the hydrolysate after pretreatment favored the kinetics of the process compared to whole biomass digestion, promoting lower carbon

intensity and yielding a potential economic gain [42]. In another work, the authors [79] investigated the performance of SE using coffee husk biomass. In this study, lower temperatures and longer times were selected as operational conditions. Indeed, the optimized parameters were 120 °C for 60 min, which corresponded to an SF of 2.37. These conditions provided the highest methane yield of 144.96 NmL/g of volatile solids but led to a lower removal percentage of cellulose, hemicellulose and lignin [79] when compared against other results [42]. The lower results obtained in [79] are in agreement with previously published reports that suggest the negative impact of longer process at lower temperatures on the sugar conversion when SE is applied [79]. In a different study, methane and biogas production yields were evaluated applying SE to reeds (*Phragmites australis*). This vegetal and aquatic species is so highly productive that it can even be considered as invasive. Reeds were treated with SE at SF ranging between 2.47–4.83. The results revealed that SE at 200 °C for 15 min had an excellent impact on the degradation and biodegradability of reed biomass and increased the specific methane yield by 89% in comparison to the non-treated sample. This improved experimental yield implies a final production of 677 L of biogas per kg of volatile solids [81]. In another work, wheat straw was assessed as a potential carbon source for its further use in biotechnological processes such as biofuel production, among others. The proposed strategy to optimize the SE pretreatment parameters included the evaluation of the results obtained in the range of 5 to 20 min and 180–200 °C. Additionally, the raw material was impregnated with water directly in the reaction chamber of the SE device. Under the severe experimental conditions of 200 °C for 10 min the highest total saccharide yield (~50 g/L) was achieved. Nevertheless, the best yield for glucose, 41.2 g/L, required the increase of this optimal SE time for another 5 min, while the maximal yield for xylose, 18.9 g/L, was reached at a lower temperature, 190 °C, but still for 10 min. These conditions also increased the production of the inhibitors, especially acetic and formic acids, although at concentrations 10 times lower than the saccharides, <3 g/L. Therefore, SE was considered a great approach to treat wheat straw to recover monosaccharides [68]. The authors conducted a pilot-scale investigation of SE batch reactor pretreatment of olive prunings to maximize the total glucose yield [80]. The optimization was performed using the application of mathematical and statistical analyses based on response surface methodology (RSM). The highest glucose yield (86%) and thereby the highest total sugars yield was achieved at the severity of 4.41 (210 °C, 15 min and 20 bar). Under these experimental parameters 19.76 g of glucose/100 g of dry raw material were obtained, and 28.25 g of total sugars/100 g of dry raw material were obtained. These outcomes indicate that 1 kg of raw material would allow the production of 144.1 g of ethanol [80]. Comparison of results among works developed with SCB and other raw materials is complex due to the variability of units used to express data. However, we found that, regarding the recovery of glucose, the application of SE to olive pruning biomass (19.76 g of glucose/100 g of dry raw material) and to SCB (16.1 g of glucose/100 g of dry raw material) provided quite similar outcomes [37]. Another work used the same data analysis technique, RSM, to optimize the production of fermentable sugars extracted from hornbeam wood (*Ostrya carpinifolia*). An overall yield of 67.8% of sugar content was reached under an SF of 3.97. This would correspond to a theoretical ethanol yield of 251 L/ton of dry hornbeam feedstock [82]. SE pretreatment was also applied to assess the degradation of sorghum polysaccharides, which is meant to favor the process by reducing the cellulose, hemicellulose, and lignin content, thereby favoring the utilization of enzymes or microorganisms in fermentation. SE enhanced the ethanol fermentation rate, and a maximum ethanol yield of 20.5 g/100 g was reached under the SE pressure of 1.5 MPa, which was 2.41 times greater than the control. Some of the SCB works that have expressed their efficiency in the same units as the sorghum's study indicated that the maximum concentration of ethanol produced was 0.42 and 0.511 g ethanol/g glucose [36,47]. Hence, outcomes seem to point out that SCB has much higher potential to produce ethanol.

In this way, and considering the data available, SE seems to have potential application as pretreatment of lignocellulosic biomass other than SCB for bioethanol production. Even though the scarce comparison of data seems to point to SCB as a more efficient raw material, the above discussed results demonstrate the versatility of this thermo-mechanical pretreatment for a wide variety of raw materials in which positive results regarding both fermentable sugars and cellulose, hemicellulose and lignin content reduction were observed.

5. Complexity of Applying Steam Explosion to Biorefinery Processes

One of the most recurring complexities is that, as the SE process progresses, there is a simultaneous release of soluble inhibitory by-products, such as furfural and 5-hydroxymethylfurfural (HMF) or lignin-derived by-products such as vanillin, syringaldehyde and other phenolic compounds [85].

These by-products generated during the SE process can be basically divided into three main categories: weak acids, furanic derivatives, and phenolic compounds [56]. Weak acids, such as acetic acid, vanillic acid, benzoic acid or syringic acid, are likely to be found in the pretreated hydrolysates [86]. In fact, the release of acetic acid from hemicellulose is the cause of the low pH that is often found in the slurry of the SE product [55,56]. In this sense, the previously mentioned SF is key for the regulation of by-products formation. A negative correlation between SF and acid pH has been demonstrated. In fact, an increased SF can reduce the concentration of acetic acid since several conditions prompt the hydrolysis of acetyl groups of hemicelluloses to a greater extent. The release of the acetyl groups allows their removal from the biomass before the hydrolysis stage starts and so the increment of the pH [20]. Reaching optimal pH conditions is critical to optimize the SE pretreatment since an acidified raw material exposed to high temperatures during SE might promote the degradation of monosaccharides and generate furanic compounds, such as HMF [56]. HMF is a degradation by-product of the hexose (glucose or fructose) sugars contained in the hemicellulosic fraction of the biomass. HMF may subsequently be transformed into 5-arylaminomethyl-2-furanmethanol, 5-hydroxymethylfuroic acid, furfuryl alcohol, levulinate ester and formic and levulinic acids [86,87].

Finally, the phenols which are naturally present in vegetal biomass can be released during the degradation of lignin or from the breakdown of carbohydrate monomers [56]. The concentration and variety of phenolic compounds produced vary depending on the substrate loading and the type of biomass. The most abundant representatives are hydroxycinnamic acid derivatives (p-coumaric acid, cinnamic acid, ferulic acid, chlorogenic acid, caffeic acid and rosmarinic acid), gallic acid and tannins [85,86]. Yields of degradation by-products vary widely from study to study according to the conditions and the feedstock involved [67].

The formation of inhibitory by-products during the SE pretreatment is a disadvantage that is necessary to minimize [46]. In some cases, a catalyst such as H₂SO₄ is added to further increase the yield of the hydrolysis stage [88]. In this way, the application of SE has led to degradation of hemicellulosic sugars as well as the incomplete disintegration of the structure linking lignin and carbohydrates. Moreover, the formation of different inhibitor compounds during the SE pretreatment negatively affects the efficiency of the process [89], so the optimization of different parameters including the SF is a key step to overcome these shortcomings. Nonetheless, certain other shortcomings associated with the method have also been suggested, such as the limited depolymerization of cellulose [71]. The most repeated advantages and disadvantages in the use of this pretreatment method are compiled in Table 4.

Table 4. Advantages and disadvantages of steam explosion (SE) pretreatment.

| Advantages | Ref. |
|---|---------|
| Alternative method for the reutilization of agro-industrial by-products to create value-added products | [57] |
| Solubilization of hemicellulose into monomers and oligomers enhances microbial enzymatic hydrolysis | [81] |
| Strong ability to compromise carbohydrate recovery and cellulose hydrolysis performances | [43] |
| Further processing of phenolic monomers presents in lignin | [90] |
| Elevated solid loadings are operable by large-scale autocatalyzed steam pretreatment | [76] |
| Absence of organic solvents and corrosive chemicals | [70] |
| Water saving technique just 1.5 kg of water is needed to treat 1 kg of biomass | [67] |
| No or little downsizing is needed | [91] |
| Low-energy treatment: <70% energy requirements to reach same particle size than mechanical processes | [57,92] |
| Physical pretreatments generate no toxic by-products; they are sulfur-free processes | [3,90] |
| Relatively economic because of no external catalyst requirement | [20,87] |
| Broad applicability: high short-term efficacy, industrial scale-up and affordable technique | [57] |
| Numerous scientific reports strongly support outcomes and continuously disclose new application fields | [65] |
| SE has seen as the technology most closely approaching commercialization | [93] |
| Disadvantages | |
| Challenging control of strength and consistency of treatment that may degrade other effective sugars components and occurrence of Maillard reaction; therefore, the treatment cannot be uninterrupted | [57] |
| Hemicellulose fraction may be partially degraded due to severe pretreatment conditions | [88] |
| Potential capacity of destroying cellulose connection | [57] |
| Over-degradation of hemicellulose and cellulose may create inhibitory by-products limiting the effectiveness | [85] |
| Poor pulp yields necessitate further research into the application of pretreatment technique | [90] |
| Some studies have found no correlation between crystallinity and sugar conversion after SE pretreatment | [84] |
| Commercial application still under development and has not been proved yet | [90] |
| The process can lead to re-condensation of lignin | [57] |

Regarding lignocellulosic material, the SCB pretreatment at lab-scale is generally performed in batch reactors, while industry pretreatment is performed in continuous reactors for a more competitive process. However, there are some limitations when SE pretreatments are run in a continuous manner, including the maintenance of the steam pressure inside the reactor during the process without producing leakage, the continuous feeding of the reactor, and the evaporation of sugars that affects the mass balance [94]. For example, one study evaluated the hydrothermal humification and decomposition effects of SE using broccoli as vegetable waste. The results showed that SE affected the physicochemical properties of broccoli residues, such as lignin re-condensation and degradation of the amorphous cellulose region, which were accompanied by porous structure destruction and browning phenomena. However, it was concluded that SE promotes the degradation of easily biodegradable feedstocks and the subsequent reactions of polycondensation, aromatization and nitrogen fixation [95]. When scaling-up bioethanol production, it is crucial to select the optimal conditions to obtain the maximum yields of fermentable sugars. In this way, the intensity of the combined severity factor (CSF) range affects the monomer and oligomer production. In this sense, the authors of [94] designed a pilot scale study for the SCB pretreatment with SE autocatalyzed and catalyzed with sulfuric acid in a 65 L steam gun reactor. The authors applied CSF ranging from 0.37 to 1.12. The results of this pilot scale design showed that lower CSFs leads to lower xylose recovery and higher oligomer recovery. This direct correlation between CSFs and xylo-oligosaccharide production makes possible the identification of the critical value of the CSFs, where oligosaccharides start to decrease instead of increase. This is a key factor for the industrial scale design since both the biomass slurry and the cellulolytic enzyme cocktails must be tailored depending on the oligosaccharide concentration to achieve the maximum efficiency of the process. This pilot scale experiment also showed the correlation between a high severity pretreatment and the d-gluconolactone production as well as its by-products that may negatively affect the enzyme performance in the enzymatic hydrolysis step [94].

Another similar study assessed xylose production after pretreatment of lignocellulosic material at pilot scale. An optimized SE pretreatment of 12 bar(g), 3 min, 1.2% phosphoric acid and 500 g substrate yielded up to 94% initial xylose release in hydrolysates with low levels of fermentation inhibitors. In addition, subsequent fermentation assessment of the hydrolysates demonstrated that the hydrolysates are non-toxic. This study supports the adaptability of the SE pretreatment to experimental conditions. In fact, different biomasses allowed the development of a cost-effective experimental design proposal. This setup reduced the formation of undesirable by-products as well as the operation time and the number of experiments required [66]. The industrial scale-up of SE is based on the specific features of the process such as the lower dielectric constant of water, the short residence time and the chemical-free extraction, therefore making it a convenient method to isolate high-value compounds such as sugars from biomass. These types of applications merit being further pursued as an environmentally friendly replacement for conventional solvent extraction methods. However, it is important to be aware that when the biomass is extremely variable, the need to optimize the treatment used for its fractionation is increased [58].

6. Conclusions

A new era of biofuel production has been promoted in recent years due to the current necessity to reduce carbon emissions generated by conventional fuels. In this way, alternative pathways have been developed, among them bioethanol production using lignocellulosic biomass is one of the most convenient considered approaches. However, a pretreatment step of the biomass is needed to achieve efficient bioethanol production. Thus, SE has been widely studied as a potential thermomechanical technique to be incorporated along the biofuel production process. SE is a pretreatment method that uses high temperatures and pressure conditions to achieve sugar releasement in a fast and cost-effective manner. SE is also characterized because of its several advantages, such as wide applicability, high efficiency and friendly environmental features despite its conventionality. Although different matrices can be used for bioethanol production, SCB has awakened special interest since sugar commercialization generates considerable amounts of biomass considered by-products, the elimination of which is currently an issue. Several studies have shown positive results obtained in terms of both sugar conversion and the content reduction in cellulose, hemicellulose and lignin when SE was applied in SCB. Thus, bioethanol production using this methodology seems to be a suitable new pathway to be considered. Moreover, the scaling-up feasibility of this process has also been considered from a techno-economic point of view, with positive results such as high yield of glucose and hemicellulose in a two-step process, lignin transformation and hemicellulose solubilization and profitability. Meanwhile, the principal complexities faced by industries applying this pretreatment method are the partial degradation of hemicellulose, the generation of toxic compounds and the necessity of acid catalysts for the efficiency of the process with high-lignin content material. However, these challenges are being addressed through the combination of severity factors, optimization of the process and the design of predictive statistical models. To give further approaches to SE pretreatment, other matrices were also considered, showing equivalent results to those obtained with SCB, which reaffirms the potential application of this methodology. Therefore, considering the current data available, bioethanol production using SE as a pretreatment of SCB may be a potential production process for 2G biofuels.

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