



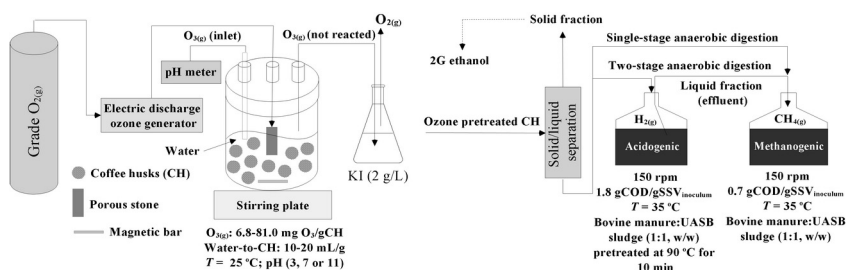
Production of biogas (methane and hydrogen) from anaerobic digestion of hemicellulosic hydrolysate generated in the oxidative pretreatment of coffee husks



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GRAPHICAL ABSTRACT



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ABSTRACT

Ozone pretreatment of coffee husks (CH) was evaluated to generate hydrolysates for biogas production and to preserve cellulose of the solid phase for 2G ethanol production. Pretreatment variables included liquid-to-solid ratio (*LSR*), *pH* and specific applied ozone load (*SAOL*). Considering single-stage anaerobic digestion (AD), the highest methane production (36 NmL CH₄/g CH) was achieved with the hydrolysate generated in the experiment using *LSR* 10 mL/g, *pH* 11 and *SAOL* 18.5 mg O₃/g CH, leading to 0.064 kJ/g CH energy recovery. Due to the presence of toxic compounds in the hydrolysate, the addition of powdered activated carbon (4 g/L) to the reactor enhanced biogas production, leading to 86 NmL CH₄/g CH yield and 0.58 kJ/g CH energy recovery. When two-stage AD was applied, methane production resulted in 49 NmL CH₄/g CH, with additional 19 NmL H₂/g CH production, resulting in a net 0.26 kJ/g CH energy recovery.

1. Introduction

For developing countries such as Brazil, lignocellulosic biomass is a promising alternative source because it is renewable and abundant (Couto et al., 2004). According to the Brazilian National Supply Company (CONAB), Brazil is the world's largest exporter of coffee and, consequently, generates a considerable amount of waste (coffee husks).

It is estimated that Brazilian coffee production in 2017 generated about 2.7 million tons of coffee husks as waste (CONAB, 2017) that represent a natural, cheap and abundant source of lignocellulosic biomass that can be used for sustainable production of bioenergy, biofuels and bio-products of high added values. Therefore, from an environmental point of view, the production of second-generation (2G) bioethanol and biogas from coffee husks would be an interesting and strategic

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alternative. Some studies for the use of coffee husks have been made, such as direct use as a fuel in farms, animal feed, solid state fermentation and biodiesel production; however, considering the high amount of waste generated, there is still a need to seek better alternatives and more profitable uses.

Lignocellulosic biomass presents a high resistance to chemical and biological degradation, which is mainly explained by the presence of a complex interaction between the major compounds such as lignin, hemicelluloses and cellulose in its structure. As a result of the structural complexity presented by this type of biomass, its use as a raw material for the production of bioenergy such as biogas and bioethanol requires a pretreatment step. The main objective of this step is to disaggregate or disrupt interactions within the lignocellulosic complex (reduce particle size, increase surface area and pore volume, reduce lignin content and decrease cellulose crystallinity), facilitating access and enzymatic and microbiological digestibility (Mussatto & Dragone, 2016).

Ozonation has been used as a pretreatment technique for different types of biomass to improve their biodegradability, for example, wheat straw, oats, barley, rice, sugarcane bagasse, grasses and sawdust from different tree species. Moreover, ozonation has been used as a treatment technique in cellulose and paper industry in the bleaching stage aiming at removing lignin residues and fragments from the fiber surface (Hung & Sumathi, 2004; Travaini et al., 2014). However, no studies were found that evaluated the use of oxidative pretreatment for adding value to coffee husks through the production of renewable fuels, which shows the relevance of the present study.

In the oxidation of lignocellulosic biomass major compounds with ozone, the most susceptible material to degradation is lignin, followed by hemicelluloses, with little preference for cellulose (Taherzadeh & Karimi, 2008). Thus, ozone becomes effective, breaking the association between several components of lignocellulosic biomass and producing a substrate with better reactivity for enzymatic hydrolysis (Kumar et al., 2009; Sun & Cheng, 2002; Taherzadeh & Karimi, 2008). The mechanisms of organic matter oxidation by the application of ozone can be classified in direct (molecular reaction) or indirect (radical reaction). The direct mechanism called ozonolysis (Criegee mechanism) occurs when ozone molecule (O_3) promotes an electrophilic addition to a double bond (π) between carbons, forming ozonides that decompose into carbonyl compounds and hydrogen peroxide in the presence of water. Due to its electrophilic nature, ozone also reacts with structures with high electron density such as aromatic compounds, promoting addition to the double carbon-carbon bonds and causing ring opening and formation of unsaturated byproducts with carbonyl and/or carboxylic functional groups (Mussatto & Dragone, 2016). The indirect mechanism prevails in alkaline media or in the presence of some agents (hydrogen peroxide, UV radiation) that lead to the formation of radicals such as hydroxyl ($\cdot OH$), superoxide ($O_2^{\cdot -}$) and hydroperoxide (HO_2^{\cdot}) (Gottschalk et al., 2010). These radicals are very reactive and not selective, and oxidation is considered an advanced oxidative process (AOP) that has aroused considerable interest in several applications (Mussatto & Dragone, 2016; Nascimento et al., 1998).

The pretreatment processes of lignocellulosic biomass can generate a solid fraction, rich in cellulose and a liquid fraction, which consists of hemicellulosic hydrolysate, rich in oligomers and C_5 sugars, such as xylose and arabinose. Biogas production by anaerobic digestion of liquid fraction is a technological option to be considered in order to maximize energy recovery from lignocellulosic biomass (Baeta et al., 2016a,b; Barakat et al., 2012; Travaini et al., 2016) and the generated solid fraction can be used for the production of 2G ethanol after passing through a stage of enzymatic hydrolysis followed by alcoholic fermentation.

Thus, the aim of this study was to investigate and optimize the ozonation process of coffee husks as a pretreatment technique for the production of hydrolysates (liquid fraction) that would be used in the production of biogas (CH_4 and/or H_2) via anaerobic digestion.

2. Material and methods

2.1. Chemicals

Cyclohexane, ethanol (99.5%), hydrochloric acid (36–37 wt% in water), sodium hydroxide and sodium thiosulphate were purchased from Synth (Brazil). Sulfuric acids (95–98% and 99.999%) were purchased from Synth and Sigma-Aldrich (Brazil). Chromatography grade standards cellobiose, D-glucose, D-xylose, L-arabinose, acetic acid, formic acid, propionic acid, isobutyric acid, butyric acid, valeric acid, isovaleric acid, 5-hydroxymethyl-2-furfuraldehyde (HMF) and 2-furfuraldehyde (FF) were purchased from Sigma-Aldrich (Brazil). Oxygen (purity 99.99%) used during ozonolysis was purchased from White Martins/Praxair (Brazil). The powdered activated carbon (PAC) was purchased from Synth (Brazil).

2.2. Coffee husks

Coffee husks (CH) were collected at Jangada farm, Criminoso, Lavras, Minas Gerais, Brazil. They were previously dried (under sunlight) to remove moisture until a less than 10% moisture content was achieved. After drying, CH were ground (42–60 mesh, 0.355–0.250 mm) in a knife mill (Marconi, model MA048) and stored at room temperature prior to use.

2.3. Characterization of biomass and fractions obtained in oxidative pretreatment

The quantification of the main components of CH and of the solid and liquid fractions generated after pretreatment were performed according to the standard methodologies. Ash content (inorganic) was determined according to the standard methodology “Ash in wood, pulp, paper and paperboard” (TAPPI T211 om-02). In this method, three samples of CH (1.000 g, on dry-weight basis) were weighed into porcelain crucibles, transferred to a muffle furnace, and heated to 25 °C from 525 °C at a heating rate of 1.25 °C/min and kept at 525 °C for 2 h. Quantitative determination of extractives in CH was performed according to TAPPI T204 cm-07. In this method, three samples of CH (10.000 g, on dry-weight basis) were weighed into paper cartridges and continually extracted with a mixture of cyclohexane and ethanol (1:1, v/v) for 24 h using a Soxhlet apparatus. The insoluble lignin content in the solid fractions was determined according to the standard methodology “Determination of acid-insoluble lignin in biomass” (NREL LAP-004). In this method, three samples of CH (0.700 g, on dry-weight basis) were weighed into glass cylindrical tubes and 10.7 mL of 72% (w/w) H_2SO_4 solution was added to the tubes. The mixture was magnetically stirred at 25 °C for 2 h. Then, 400 mL of distilled water is added to each tube. The tubes were placed into an autoclave for 1 h at 121 °C. The liquid and solid fractions obtained after hydrolysis were separated by vacuum filtration on Büchner glass crucibles. The solid fraction (insoluble lignin) was dried in an oven at 80 °C for 4 h while the liquid fraction (soluble carbohydrates and carbohydrate degradation products) were analyzed by high performance liquid chromatography (HPLC). Ash content in insoluble lignin was determined as previously described. Soluble lignin was analyzed on a UV-Vis spectrophotometer (NREL LAP-004).

The concentration of sugars (cellobiose, glucose, xylose and arabinose), organic acids (acetic and formic) and sugar degradation products (FF and HMF) were determined by HPLC in a Shimadzu HPLC system equipped with Aminex HPX 87H column (300 × 7.8 mm Bio-Rad) maintained at 55 °C (Shimadzu column oven, model CTO-30A) using refractive index detector (Shimadzu, model RID-6A) for sugars and a UV-Vis detector (Shimadzu, model SPD-10AV) set at wavelengths of 210 nm and 274 nm for organic acids and sugar degradation products (FF and HMF), respectively. The mobile phase was composed of 5 mmol/L sulfuric acid at a flow rate of 0.6 mL/min.

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