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Hydrogen production and TOC reduction from gasification of lactose by supercritical water

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ABSTRACT

New data were obtained for production of H₂ and reduction of total organic carbon (TOC) by gasification of lactose in supercritical water with(out) the presence of catalysts (NaOH, KOH and Na₂CO₃) in a continuous Inconel 625 flow reactor. The gasification reactions were carried out at 550–700 °C, 22.5 MPa, lactose concentrations in the range of 0.5–2.5 wt%, feed flow rates of 5 and 7 g min⁻¹ and a total reaction time of 30 min. The optimum condition for the production of H₂ yielded approximately 4.5 mol H₂/mol lactose with a TOC reduction of ~95%. In addition, supercritical gasification of whey was carried out using the same operating conditions as the lactose system. A reduction of 92% on the TOC and 0.45 molar fraction of H₂ production was achieved, similar to the gasification of lactose at a concentration of 0.5 wt%.

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Introduction

The well established dairy industry works as a single marketplace in the overall economy. However, effluents from industrialization of milk derived products are environmental and economic challenges due to significant organic matter content. Furthermore, various sanitization related compounds, such as acids, phosphates, alkaline (NaOH, KOH, and

others) and oxidizing agents are found in these effluents. For instance, bovine whey, a by-product of cheese processing, contains water (93–94% w/v), lactose (4.5–5.0% w/v), soluble proteins (0.7–0.9% w/v), and minerals (0.6–1.0% w/v) [1]. Therefore, cheese whey effluent has approximately 90% of water and can be harnessed energetically through the generation of hydrogen by supercritical gasification [2,3]. Water has unique properties as a reaction medium in its supercritical state above its critical temperature and pressure ($T_c \geq 374$ °C,

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$P_c \geq 22.1$ MPa) [4]. Supercritical water gasification (SCWG) of biomass and industrial wastewater is an efficient and attractive method to produce hydrogen due to its energy efficiency with low generation of pollutants [5,6]. In supercritical gasification, a biomass with high water content can be used for natural conversion of hydrogen [7]. The water in the gasification process is not only a solvent but also a reagent that allows hydrogen atoms to form H_2 [4,8]. Furthermore, the addition of alkalis (NaOH, KOH, $Ca(OH)_2$), carbonates (Na_2CO_3 , K_2CO_3) and bicarbonate ($NaHCO_3$) in SCWG promotes hydrogen production at low temperatures modifying near-critical water conditions [9–12].

There are few studies in the literature on gasification in supercritical water of effluents of the dairy industry [5,6,12]. Sögüt et al. [5,6] conducted a study focused on the partial oxidation of whey using SCWG in continuous flow tubular reactor with hydrogen peroxide as oxidant at 400–650 °C, residence times of 6–21 s, total organic carbon (TOC) of 56 g L^{-1} , and 25.0 MPa with TOC removal of approximately 99%. Muangrat et al. [12] studied gasification using a closed batch autoclave reactor of whey in subcritical water at 300–390 °C, 9.5–24.5 MPa, 5 g min^{-1} , reaction time up to 120 min, whey concentration of 5 wt%, and with addition of NaOH and H_2O_2 , reaching 40% of theoretical hydrogen concentration.

In the present work, supercritical water gasification (SCWG) of lactose was performed at conditions that maximized the production of hydrogen gas (H_2). Alkali catalysts (NaOH, KOH) and carbonate (Na_2CO_3) were used to improve the production of H_2 via the water-gas shift reaction [10]. TOC analysis was carried out to quantify total carbon in the inlet and outlet streams of the reactor in all experimental runs allowing the evaluation of the gasification reaction efficiency. Finally, whey gasification at the same lactose operating conditions was conducted with the purpose of testing the proposed methodology in a complex system and providing some information to support future scale-up of gasification process.

Experimental

Materials

Lactose monohydrate (>98.0 wt%) was purchased from Anidrol (Diadema-SP, Brazil). KOH, NaOH, Na_2CO_3 , were obtained from Synth (Diadema-SP, Brazil). All used chemicals and solvents were of analytical grade. Cheese whey was provided by Latco Ltda (Cruzeiro do Oeste-PR, Brazil).

Apparatus and experimental procedure

The experimental apparatus scheme used for hydrogen production is shown in Fig. 1. The system consists of a continuous flow reactor built in Inconel 625, a preheater, a condenser, and tubings of 316 stainless steel.

The experimental procedure consisted of lactose aqueous solution preparation, where lactose quantities were weighed with a high precision scale (Ohaus Analytical Standard, USA, uncertainty of 0.0001 g). The lactose-water mixture contained in a glass reservoir (1) was suctioned by a high pressure pump,

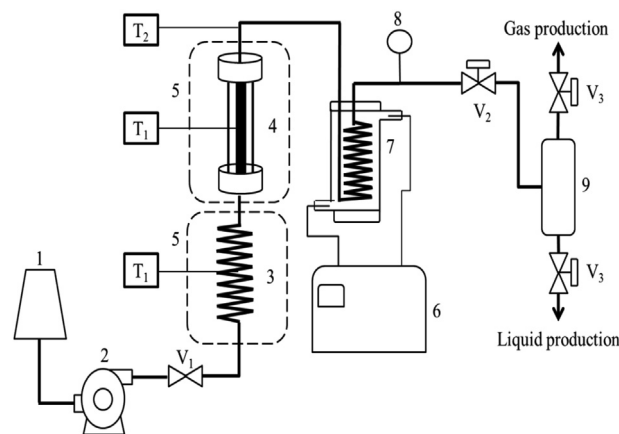


Fig. 1 – Scheme of the SCWG apparatus. (1) Reservoir for the feed. (2) High pressure pump. (3) Preheater. (4) Reactor. (5) Oven. (6) Thermostatic bath. (7) Condenser. (8) Manometer. (9) Phase separator. (T₁) Temperature controller of electrical resistance. (T₂) Universal temperature indicator. (V₁) One-way valve. (V₂) Back-pressure regulator valve. (V₃) Needle valve.

HPLC type (2). The mixture was transferred through a stainless steel tube 316 ($L = 1.5$ m, I.D. = 3.175 mm) to the valve V_1 (one-way valve) and then distributed to the preheater (3), ($L = 6$ m, I.D. 3.175 mm). The properly heated reaction mixture (50 °C) entered the reactor (4) ($L = 0.2$ m, I.D. = 13 mm). Both, the heating of the reactor and the preheating were carried out through two bipartite ovens (5), each equipped with two resistors in infrared ceramic (Corel, São Paulo-SP, Brazil) with power of 1000 W. A universal PID control (Novus, Porto Alegre-RS Brazil) connected to a thermocouple type J (accuracy ± 1.0 °C) (T_1) in contact with the preheater and reactor was used to activate the resistors. After passing through the reactor, the mixture flowed through a stainless steel tube 316 ($L = 2.5$ m, I.D. = 3.175 mm) for heat exchange, with temperature monitoring performed by a temperature indicator (Novus, Porto Alegre-RS Brazil) connected to a thermocouple type T (accuracy ± 1.0 °C) (T_2). Then, the mixture entered the condenser (7) containing a coil of stainless steel 316 ($L = 2.5$ m, I.D. = 3.175 mm) for cooling at 10 °C. After, the mixture reached the back-pressure valve (V_2) for pressure adjustment observed on the gauge (8). In the separator (9), gas and liquid were separated, where the liquid phase comprised water, unreacted lactose and other products, and the vapor phase was composed of gaseous reaction products such as H_2 , CO_2 , CH_4 , CO , C_2H_2 and C_2H_6 . Liquid effluent and solid residue obtained from each experiment were not analyzed in terms of their content. The TOC was measured in the liquid effluent.

Analysis

The composition of the gas produced during the lactose degradation was determined by gas chromatography (CG Agilent 7890A, USA) using a column of 30 m \times 0.535 mm \times 20 μm (HP-PLOT Molesieve/5A columns, USA), helium as a carrier gas, and two detectors in series, one of them being a thermal conductivity detector (TCD) and the

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