Bioresource Technology 217 (2016) 137–140

Contents lists available at ScienceDirect

Bioresource Technology

journal homepage: www.elsevier.com/locate/biortech

Short Communication

Recovery of carboxylic acids produced during dark fermentation of food waste by adsorption on Amberlite IRA-67 and activated carbon

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HIGHLIGHTS

- Two adsorbents are evaluated for the recovery of carboxylic acids from dark fermentation broth.
- Dark fermentation was performed on batch, without pH control, without addition of external inoculum.
- Lactic, acetic and butyric acids were recovered from fermentation broth.

ARTICLE INFO

Article history:
Received 30 December 2015
Received in revised form 7 February 2016
Accepted 9 February 2016
Available online 11 February 2016

Keywords:
Adsorption
Dark fermentation
Food waste
Platform chemicals
Carboxylic acids

ABSTRACT

Amberlite IRA-67 and activated carbon were tested as promising candidates for carboxylic acid recovery by adsorption. Dark fermentation was performed without pH control and without addition of external inoculum at 37 °C in batch mode. Lactic, acetic and butyric acids, were obtained, after 7 days of fermentation. The maximum acid removal, 74%, from the Amberlite IRA-67 and 63% from activated carbon was obtained from clarified fermentation broth using 200 $g_{adsorbent}/L_{broth}$ at pH 3.3. The pH has significant effect and pH below the carboxylic acids pKa showed to be beneficial for both the adsorbents. The un-controlled pH fermentation creates acidic environment, aiding in adsorption by eliminating use of chemicals for efficient removal. This study proposes simple and easy valorization of waste to valuable chemicals.

1. Introduction

The statistics from the World Bank (Hoornweg and Bhada-Tata, 2012) show that the world annual solid waste generation was approximately 1.3 billion tonnes in 2012. Landfilling of food waste causes environmental degradation, producing greenhouse gases plus limited landfilled space is also an issue associated with it. The efficient resource recovery from the food waste is required in order to minimize the environmental issues associated, and furthermore it will create value from waste. The use of bioprocesses arises as an alternative to this aim. Dark fermentation, a bioprocess, can convert the organic waste into valuable chemicals as carboxylic acids, e.g. lactic, propionic, butyric, acetic, and valeric acid (Bastidas-Oyanedel et al., 2015; Agler et al., 2011). Dark fermentation does not require sterile operating conditions, moreover is a mixed culture fermentation capable to use carbohydrate, protein and lipid portions of the waste (Jankowska et al., 2015).

One of the main barriers in this process is the recovery of the carboxylic acids from the fermentation broth. Thus, integration of recovery processes with dark fermentation is a crucial step. The adsorption to recover carboxylic acids is a simple technique with easy integration with fermenter (Bayazit et al., 2011). Adsorption can be employed in two ways, either direct addition of adsorbent in the fermenter (Gao et al., 2011) or in a separated step (Garrett et al., 2015). The latter is found to be more feasible because the direct addition of the adsorbent in the fermenter results in the adsorption of substrate which reduces the adsorption capacity of the adsorbent (Zhou et al., 2013).

The main objective of this work is to study the effect of pH and adsorbent dose on the carboxylic acids recovery in batch operation from dark fermentation broth of food waste. It was used two types of adsorbents, Amberlite IRA-67 and activated carbon, in a separate step from fermentation. The reason of selection of these two adsorbents is their low market value and affinity towards organic acids. However, there have been limited studies of their adsorption behavior for the fermentation products.

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http://dx.doi.org/10.1016/j.biortech.2016.02.035

2. Methods

2.1. Food waste

Synthetic food waste was prepared instead of real food waste in order to reproduce experimental results. The synthetic food waste contained cooked rice, as carbohydrate source, and commercial dog food pellets, as proteins and lipids source. Cooked rice and dog food pellets were mixed in 50% total solids (TS) each. The calculated dry base composition of the synthetic waste food has 68.5% of carbohydrates, 12% of proteins, 5% of fats. The resulting synthetic food waste has similar composition of kitchen waste used by Zhang et al. (2008). Previous to the fermentations, tap water is added to set the TS of the synthetic food waste to 100 g_{TS}/L.

2.2. Dark fermentation

Batch fermentations, in triplicate, were carried in glass bottles, with a working volume of 2 L. After adding the synthetic food waste, the bottles where sparged with nitrogen gas to obtain anaerobic conditions. Bottles were capped with rubber stoppers and brewing airlocks, which allows gases to escape while preventing air from entering the bottles. The fermentation initial pH was 6.8. The bottles were kept at 37 °C, without stirring, and without pH control for 7 days. No external inoculum and nutrients were added (Favaro et al., 2013; Wang et al., 2014).

2.3. Adsorption experiments

Two adsorbents were tested for the recovery of acids: (1) Amberlite® IRA-67 free base a weakly basic anion exchange resin with a particle size of 500–750 μm, and (2) granulated activated carbon Norit® type Darco® with a particle size of 12–20 mesh. Both adsorbents were obtained from Sigma–Aldrich and were used without any pre-treatment or purification.

The adsorption study consisted on 3 types of experiments, (1) single/multi acid adsorption, (2) pH effect, and (3) adsorbent dose effect. For all the 3 cases, the adsorption experiments were performed for 4 h at a constant temperature of 25 °C, in an orbital shaker using a stirring speed of 150 rpm. In previous experiments (data not shown) adsorption equilibrium was achieved before 4 h for all the cases.

2.3.1. Single and multi acid adsorption

Batch adsorption experiments were performed using: (1) pure acid solution (single acid), and (2) mixed carboxylic acids contained in the fermentation broth (multi acid). For the single acid experiment, individual carboxylic acid, i.e. lactic, acetic, or butyric acid, were used. For the multi acid experiment, clarified fermentation broth was used. The broth was centrifuged in 250 mL centrifuge bottles for 20 min at 3220 rcf. The centrifugation was done for the removal of suspended solid particles, avoiding the interaction of this debris with the adsorbents.

The single acid experiment was based on 3 solutions of pure acids, i.e. lactic acid, acetic acid, and butyric acid, while the multi acid experiment was based on clarified fermentation broth. All the 4 solutions were adjusted to pH 2. A volume of 50 mL of each solution was added to 5.0 g of each adsorbent.

The adsorption of different carboxylic acids, at equilibrium, is determined using Eq. (1):

$$Q = \frac{V * (C_0 - C_1)}{m} \tag{1}$$

where Q stands for adsorption capacity ($mg_{adsorbed\_acid}/g_{adsorbent}$), V is the solution volume (mL), C_0 and C_{eq} are the initial and equilibrium concentrations of carboxylic acids in solution (g/L), respectively, m is the mass of adsorbent (g).

2.3.2. pH effect on adsorption

The effect of pH was investigated for the multi component solution only, i.e. clarified fermentation broth (as in Section 2.3.1). The initial pH effect of the solution on the adsorption was performed adjusting the clarified fermentation broth pH using NaOH or HCl. Initial pH were 2.0, 3.3, 4.0 and 6.0. Note that pH 3.3 corresponds to the unadjusted pH clarified fermentation broth. After adjusting the initial pH. As in Section 2.3.3, 50 mL of each solution was added on 10 g of each adsorbent. The removal of carboxylic acids from the fermentation broth was determined using Eq. (2).

$$AR = \frac{C_0 - C_f}{C_0} \cdot 100 \tag{2}$$

where AR is the percentage of acid removal from the solution (%), C_0 and C_f are the initial and final concentrations of carboxylic acids in solution (g/L), respectively, and 100 is the coefficient for converting into percentage.

2.3.3. Adsorbent dose effect

In this experiment it was used the unadjusted pH clarified fermentation broth, pH 3.3. In this case 50 mL of solution was added to 1.0, 5.0 and 10.0 g of each adsorbent. The carboxylic acid removal from the broth was determined as in Eq. (2).

2.4. Carboxylic acids quantification

The concentrations of carboxylic acids were measured by 1260 HPLC (Agilent Technologies, USA) using Agilent Hi-Plex H column (65 °C), UV detector at 210 nm for organic acids, RID detector at 35 °C for ethanol. The mobile phase was 5 mM H_2SO_4 with a flow rate of 0.6 mL/min. External standards were used for calibration.

3. Results and discussion

3.1. Dark fermentation carboxylic acids composition

Lactic acid was the dominant dark fermentation product throughout the process with a maximum concentration of 11.6 g/L. It was followed by butyric acid with a concentration of 6.6 g/L and acetic acid with a concentration of 2.8 g/L. Valeric acid was also observed with a low concentration around 0.6 g/L. Ethanol and no other carboxylic acid was observed. The pH was initially 6.8 and dropped during the first 4 days of the fermentation to 3.3 where it remained throughout the rest of the fermentation (7 days). The production rate of lactic acid is highest because of acid tolerant ability of lactic acid bacteria, moreover acidic environment can decrease the hydrolysis rate of waste result in lower production of other acids (Veeken et al., 2000).

3.2. Comparison between single and multi-component adsorption

Table 1 shows the adsorption capacity (Q) and percentage of acid removal (AR), defined in Eqs. (1) and (2) respectively, of activated carbon and Amberlite IRA-67, in a single and multi-acid system. It is clear that the adsorption capacity is higher in single acid systems as compare with multi acid systems for both adsorbents, which indicates the competitive adsorption by other solutes present in the clarified fermentation broth. In the case of Amberlite IRA-67, the Q for lactic acid is not reduced but the percentage of acid removed decreases showing competitive adsorption by other components present in the system.