



Contents lists available at ScienceDirect

Waste Management

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

Review

Citrus essential oils and their influence on the anaerobic digestion process: An overview

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ARTICLE INFO

Article history:

Received 12 January 2014

Accepted 26 June 2014

Available online xxxxx

Keywords:

Anaerobic digestion

Limonene

Citrus waste

Orange waste

Citrus essential oils

ABSTRACT

Citrus waste accounts for more than half of the whole fruit when processed for juice extraction. Among valorisation possibilities, anaerobic digestion for methane generation appears to be the most technically feasible and environmentally friendly alternative. However, citrus essential oils can inhibit this biological process. In this paper, the characteristics of citrus essential oils, as well as the mechanisms of their antimicrobial effects and potential adaptation mechanisms are reviewed. Previous studies of anaerobic digestion of citrus waste under different conditions are presented; however, some controversy exists regarding the limiting dosage of limonene for a stable process (24–192 mg of citrus essential oil per liter of digester and day). Successful strategies to avoid process inhibition by citrus essential oils are based either on recovery or removal of the limonene, by extraction or fungal pre-treatment respectively.

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Table 1
Chemical composition of citrus waste.

Parameter (units)	Citrus pulp De Blas et al. (2010)	Dried citrus pulp Bampidis and Robinson (2006)	Citrus pulp Calsamiglia et al. (2004)	Citrus pulp silage Bampidis and Robinson (2006)	Orange waste Mahmood et al. (1998)	Orange peel Kammoun Bejar et al. (2012)	Orange peel Morton (1987)
Water content (%)	10.8	11.7	82.5	79.0	79.02	74.8	72.5
Ashes (% d.m.)	7.1	5.87	6.25	5.5	3.78	3.313	2.9 · 10 ⁻³
Protein (% d.m.)	6.4	7.37	8.29	7.3	6.53	8.015	5.45
Fat (% d.m.)	1.6	3.43	3.32	9.7	–	0.955	0.73
Fibre (% d.m.)	13.3	–	14.1	–	10.59	42.129	–
Starch (% d.m.)	0.5	–	2.90	–	<1.00	–	–
Sugar (% d.m.)	22.8	–	20.3	–	15.00	46.649	–
pH	–	–	3.93	–	4.30	–	–
Ca (% d.m.)	1.50	1.49	0.93	2.04	–	1.201	0.58
P (% d.m.)	0.12	0.35	0.15	0.15	–	–	0.07
Na (% d.m.)	0.08	0.08	0.08	0.09	–	0.312	0.01
Cl (% d.m.)	0.05	0.08	0.05	–	–	–	–
Mg (% d.m.)	0.14	0.15	0.14	0.16	–	0.156	–
K (% d.m.)	0.85	2.51	0.68	0.62	–	0.222	0.77
S (% d.m.)	0.11	0.07	0.13	0.02	–	–	–
Cu (mg/kg d.m.)	6.0	6.7	–	6	–	11.28	–
Fe (mg/kg d.m.)	220	230	–	160	–	15.85	8
Mn (mg/kg d.m.)	12	7.7	–	7	–	–	–
Zn (mg/kg d.m.)	9	14	–	16	–	18.67	–
Co (mg/kg d.m.)	–	0.16	–	0.16	–	–	–

“–”: no data available at the referred paper.

1. Introduction

1.1. Citrus waste

Citrus waste mainly consists of: (a) waste generated by the juice manufacturing industry, consisting of peel and pressed pulp; (b) fruit discarded for commercial reasons (damaged fruit, as example); and (c) fruit discarded due to regulations that limit production. This material is not allowed to enter the food chain and therefore is considered to be waste.

The amount of waste generated depends on the harvest, since it is a fraction of the total amount of fruit produced. Citrus processing for juice extraction produces around 500 tonnes of waste per 1000 tonnes of fruit processed (Lane, 1983a; Lohrasbi et al., 2010). The percentage of fruit discarded due to commercial or regulatory issues is more difficult to calculate, but it ranges from 2% to 10% depending on the type of citrus considered and environmental aspects, such as weather conditions. The most recent data correspond to the 2010/2011 season, when the total citrus production in the region of Valencia (eastern Spain) was 3.5 million of tons. Of this, around 0.4 million of tonnes (11% of the total production) was reported as losses. Another 0.4 million of tonnes was used in the industry, which could be expected to produce around 0.2 million of tonnes of waste (CAPA, 2011).

1.2. Citrus waste management and valorisation

Citrus waste typically has a low pH (3–4), high water content (around 80–90%) and high organic matter content (around 95% of total solids). These characteristics mean that citrus waste should not be disposed of in landfills according to European regulations (Council Directive 2008/98/EC of 10th November 2008 on waste). Traditionally, citrus waste from the juice manufacturing industry has been used as livestock feed, thanks to its nutritive value, which is similar to that of barley grain or sugar beet pulp. This is due to its high carbohydrate content, the significant proportion of cell wall components and its low degree of lignification (see Table 1). However, the juice manufacturing companies are currently facing waste management difficulties due to the market saturation.

Non-hazardous waste management schemes do not usually accept citrus waste for composting, due to its low pH, the presence of essential oils that inhibit the composting process and the fast biodegradation of this waste, which can cause anaerobiosis problems in compost piles.

Thermal treatment alternatives (incineration, gasification or pyrolysis) cannot be applied to citrus waste due to its high water content. Although they would be technically feasible, they would not be efficient from an energy or an economic point of view, since a previous dehydration step would be necessary.

The manufacture of bioethanol from citrus waste has recently been evaluated as a valorisation alternative. A bioethanol yield of 50–60 L tonne⁻¹ of waste (Boluda-Aguilar and López-Gómez, 2013) was obtained, equivalent to 294–352 kW h tonne⁻¹ of waste. However, the investment necessary to set up a bioethanol plant is great: around 600 € tonne⁻¹ of waste treated per year (Sánchez-Segado et al., 2012), compared with the 75–200 € tonne⁻¹ year⁻¹ necessary for a biogas plant (Cavinato et al., 2010; Karellas et al., 2010; Sorda et al., 2013). From the perspective of energy, this valorisation option is not as efficient as methane (biogas) production through the anaerobic digestion of citrus waste. The biochemical methane potential (BMP) of citrus peel is between 0.46 and 0.64 m³_{CH₄} kg_{VS}⁻¹ (Gunaseelan, 2004; Kaparaju and Rintala, 2006; Koppa and Pullammanappallil, 2013). That is equivalent to 78–110 m³_{CH₄} tonne⁻¹ of waste with 18% total solids (TS) and 95% volatile solids (VS, dry matter basis) and to 737–1040 kW h tonne⁻¹ of waste: 1.5–2 times higher than the values obtained with bioethanol. In addition, by using co-digestion strategies, other by-products could be co-treated with the orange waste, thereby contributing to integral waste management within the producing area.

The methane production in the mesophilic semi-continuous anaerobic digestion of citrus waste ranges between 0.21 and 0.29 m³_{CH₄} kg_{VS}⁻¹ (Lane, 1984; Srilatha et al., 1995). Higher values of 0.3–0.6 m³_{CH₄} kg_{VS}⁻¹ have been reported at thermophilic conditions (Kaparaju and Rintala, 2006; Martín et al., 2010).

For all these reasons, anaerobic digestion is a sound alternative for valorisation of citrus waste. However, its citrus essential oil (CEO) content inhibits the bioprocess. The objective of the present work is to review the effect of CEO on the anaerobic digestion and to analyse the pre-treatments that make it feasible.

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