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Research paper

Anaerobic digestion of nine varieties of grape pomace: Correlation between biochemical composition and methane production

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ABSTRACT

In the present study, nine different varieties of deseeded grape pomace were analyzed for the biochemical composition of the raw material (total solids, volatile matter, total chemical oxygen demand, cellulose, hemicellulose, lignin, total soluble compounds and polyphenols). The biological methane potential of the substrates was assessed in batch anaerobic digesters. The results showed that the cumulative methane yields varied widely and ranged from 102 to 300 $dm^3\ kg^{-1}$ of total solids, calculated at standard gas conditions of 273 K and 101.5 kPa. The anaerobic biodegradability of the total chemical oxygen demand varied between about 30% to a maximum of 69%. The data obtained were exploited by principal component analysis. The study revealed that lignin and cellulose, explaining almost 63% of the total variance in the data, are the main factors limiting the degradability of the selected grape pomace. The effects of the other parameters, which represent nearly 32% of the total variance, were fully reviewed. In particular, no significant correlation was found between the polyphenol content and the methane yield.

1. Introduction

Nowadays, a large fraction of the world's total energy demands is supported by non-renewable fossil resources such as coal, oil, and natural gas [1]. These resources are not only limited in supply but also have adverse effects on the environment due to the emission of greenhouse gases into the atmosphere. Bioenergy, especially biogas produced through the anaerobic digestion (AD), is considered to be one of the highly promising alternatives to fossil-derived energy due to several inherent and significant merits [2].

The development and the use of vegetal biomasses as energy source are currently encouraged worldwide. According to an estimation reported by the international organization of vine and wine, almost 2800 hm^3 of wine were produced worldwide in 2015. The European Union participated in almost 60% of the total production with Italy, France and Spain as three main European producing countries [3]. On another note, Lebanon is one of the oldest sites of wine production in the world [4]. The number of Lebanese wineries has increased from 5 in 1998 to over 30 today. Winemaking process generates large amounts of solid by-products, in particular Grape Pomace (GP) which consists of about 75% of pulp and 25% of seeds. GP are produced during a short period of harvesting, which boosts the source of biomass in the production area. During storage on harvesting sites, the buildup of phenolic compounds is responsible for decreasing the pH of the pomace as well as raising its resistance to biological degradation [5]. An important environmental problem resides in the leachates of tannins and other compounds, which may lead to oxygen depletion in the soil and ground waters that can affect topical fauna and flora [6]. In the last decades, the interest in the recovery and exploitation of GP has become a global issue. Its conversion into methane is a promising possibility given that the current methods of valorization (cathecin polymers or anthocyan colorants extraction, oil, ethanol conversion, …) have limited markets and can absorb only a limited portion of the residue generated.

The recovery of these by-products, using the anaerobic digestion, was evaluated by The French Institute of Vine and Wine (IFV, Le Grau du Roi, France). The results showed that the methane potential of GP depends on factors like grape colors (red and white) and storing process [7]. Literature review showed that the grape chemical composition is influenced by the environmental factors and the grape varieties. In a recent study, the chemical composition of red and white GP originating from different French areas of production was assessed. Large variability was observed in relationship with the color and the origin [8].

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Research was conducted to investigate the influence of grape variety on the lignocellulosic fractions of grape stalks. Results showed that the grape variety significantly influences the composition of the raw material [9]. However, the influence of biochemical parameters on the AD of different GP varieties has never been investigated for their valorization as an alternative energy source. It is worth noting that the biochemical fractions are chemically linked. Their interactions in the lignocellulosic matrix are crucial to the recalcitrance of the biomass and have a direct influence on methane production [10]. Due to that, using linear correlations to describe the interaction between methane yield and biochemical components cannot provide a highly significant relations in accordance with the AD theory [11]. A recent research indicated that methane production cannot be investigated by a single parameter, a combination of many variables might be necessary to allow a better comprehension of the interactions [12].

To define the proceeding of this research, batch trials were carried out in a previous study to assess the methane potential of full GP, pulps and seeds separately (variety Cabernet Franc 2013, France). Results showed that pulps have the highest methane production, followed by full GP and seeds [13]. Regarding the low methane potential of seeds, the aim of this research is the characterization and the valorization of different varieties of deseeded GP, as agro-industrial wastes, to generate green energy. The main objectives were to define the main chemical components of nine varieties of deseeded GP originating from different French and Lebanese wine-growing areas (Total Solids (TS), Volatile Solids (VS), total Chemical Oxygen Demand (COD), cellulose, hemicellulose, lignin, total soluble compounds and polyphenols) and to evaluate the anaerobic biodegradability and the maximal methane production of the samples in batch mode. The possible correlations between the parameters were investigated using statistical methods.

2. Materials and methods

2.1. Materials

The GP varieties (*Vitis vinifera*) were collected from different wineries located in France (Fr) and Lebanon (Lb) as indicated in Table 1. Samples about 5 kg of each substrate were collected and transported to the laboratory. On arrival, the seeds and stalks fragments were removed manually from the set of GP. The raw material was stored at −20 °C until utilization. An active inoculum was collected from the digester of the waste-water treatment plant of Saint-Brieuc city in France, operating under mesophilic conditions. The particulate matter (> 1 mm) was removed from the inoculum by passing through sieve in order to ensure better homogeneity and to improve the reproducibility of the tests. The latter was subjected to a starvation period of one week before starting of the experiments.

2.2. Analyses

Total Solids (TS) content were determined by dry weight in oven at 105 °C until constant weight. Afterwards, Volatile Solids (VS) were determined in a muffle furnace after 4 h at 550 °C and then cooled in a desiccator and weighed. Total Chemical Oxygen Demand (COD) was measured using Merck COD Spectroquant® test, range 500–10,000 g m^{-3}, and by a spectrophotometer NOVA 60 (Merck, Germany). The matter was ground using a blender (Waring blender 8011EG, Waring Commercial, USA). It is considered that all organic matter is oxidized with the hot sulfuric solution of potassium dichromate and with silver sulfate as the catalyst after 2 h at 150 °C.

2.3. Van Soest fractionation

The characterization of structural polysaccharides and lignin was carried out by the method of Van Soest [14–16] which can extract the total soluble compounds with a neutral detergent solution in excess, acting on the sample for 1 h at 100 °C. This detergent solution is composed of Na_2HPO_4, sodium tetraborate, α-amylase, sodium EDTA, sodium lauryl sulfate and sodium sulfite. These products were provided by Merck (Darmstadt, Germany). The extracted fraction is separated from the insoluble neutral detergent fibers by filtration. Following the action of the neutral detergent, hemicellulose is extracted by an excess of acid detergent solution (cetyltrimethyl ammonium bromide 20 kg m^{-3}, 98 kg m^{-3} of H_2SO_4) acting for 1 h at 100 °C. The extracted

Table 1
Grape pomace varieties, origins and collection.

Samples	Grape variety	Collect time	Age (months)	Geo-coordinates	Altitude (m)	Origin and collection	Region and country
S1 S2	Cabernet Sauvignon (C.S Fr) Merlot	October 2014	11	45° 19′ 15.913″ N 0° 57′ 19.053″ W	6	- Civil society for agricultural holdings (SCEA Birot et Fils, Gaillan en Médoc) - GP collected directly after the pressing of the grapes	Bordeaux, France
S3 S4	Chardonnay Pinot noir	October 2014	11	49° 2′ 15.023″ N 3° 57′ 5.672″ E	78	- Civil society for manufacturing and commercialization of winemaking products (Sofralab, Magenta) - GP collected directly after the pressing of the grapes	Champagne, France
S5 S6 S7	Marselan Syrah Cabernet Sauvignon (C.S Lb)	September 2014	10	33° 49′ 35.245″ N 35° 53′ 33.405″ E	909	- Wine company (Château KSARA) - GP collected directly after the pressing of the grapes	Bekaa, Lebanon
S8	Cabernet Franc (C.F)	October 2014	11	47° 12′ 25.949″ N 0° 25′ 51.392″ W	59	- Wine company (Domaine Des Acacias) - GP collected directly after the pressing of the grapes	Tigné-Layon, France
S9	Ensiled GP	May 2014	18	47° 16′ 1.927″ N 0° 30′ 12.769″ W	28	- Agricultural and industrial distilleries: UAPL (Union Agricole Pays De Loire) - After the pressing of the grapes, GP was distilled, ensiled for seven months and then collected. The ensiling operation includes the lactic acid fermentation, the loading into a silo, the compaction and sealing to exclude air and finally the storing.	Thouarcé, France