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Research Note

Effect of anaerobic co-digestion of grape marc and winery wastewater on energy production

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Abstract

Vineyard waste and wastewater generated from wineries present significant risks to the environment if untreated. In this study, we carried out anaerobic co-digestion of grape marc and winery wastewater for energy production in the form of methane and material recovery. The results showed that milling the grape marc prior to being suspended in winery wastewater and inoculated with activated sludge in a 15 weeks batch digestion resulted in the production of 5.04 MJ/kg VS of energy compared with 0.97 MJ/kg VS from the non-milled treatment. Milling of grape marc also showed reduced CO₂ emission, 4.95 l/kg VS compared with 9.96 l/kg VS from non-milled samples. The results suggest that physical pre-treatment of grape marc results in bioenergy production which is comparable to many other organic waste streams such as cattle slurry, hen litter and food processing waste offering significant potential for utilisation by the winery industry.

Keywords: Anaerobic digestion; Biogas production; Grape marc; Methane production; Winery wastewater. **Abbreviations:** VS_Volatile solids; DW_Dry weight; LOI_Loss on Ignition.

Introduction

Grape marc, the residue of skins and pips remaining after fermentation and wastewater are the primary wastes generated by the wine industry. These wastes represent a risk to the environment when released without appropriate treatment. For example, grape marc initiates a number of environmental hazards such as surface and ground water pollution, foul odours, fly and pest infestation (Australian Bureau of Statistics 2010-2011; Ying et al., 2012).

On the other hand winery wastewaters contain high concentrations of nutrients such as different classes of organic compounds, nitrates and phosphates (Bustamante et al., 2005a; Mosse et al., 2010). Pollution of surface water with this nutrient laden wastewater can lead to eutrophication with adverse impacts on biotic components. As a result, the discharge, irrigation or reuse of winery wastewaters cannot be undertaken without prior treatment (Melamane et al., 2007).

Recent stringencies in environmental standards, advances in modern chemistry and biotechnology, along with the industrial interest in waste minimization have resulted in a growing interest in exploitation of wine industry residues. Several methodologies have been studied in an effort to effectively manage the wine waste. These include composting, pyrolysis, ozonation and wet oxidation. While all these treatments aim to achieve a significant reduction in the level of organic matter, solids and inorganic load, costs associated with the construction, maintenance and operation of the infrastructure required are often seen as prohibitive, especially for small wineries (Bilgili et al., 2009; Bertran et al., 2004). The search for sustainable treatment systems which are capable of minimizing energy consumption has encouraged the use of anaerobic biological systems. Methane generated from anaerobic digestion has proven to be equivalent to heat and ethanol in terms of efficiency, cost and environmental impact while converting waste streams to energy (Parawira et al., 2008). The residues of the digester can also be used in agriculture as a secondary fertilizer due to the increased availability of nitrogen and the advantageous short-term fertilization effect (Sieling et al., 2013). Anaerobic treatment also minimizes the survival of pathogens which is important for using the digested residue as fertilizer.

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Several laboratory and pilot-scale studies of anaerobic digestion have demonstrated that this process is particularly well adapted to effluents with a high load, such as those from distilleries, which have additional favourable wine characteristics such as high organic content, relatively high temperature and good biodegradability (Mata-Alvarez et al., 2000). The characterization of the biogas potential of these wastes is a necessary step before they can be used in anaerobic digestion. One such test for assessing the biodegradability of waste materials is the Biochemical Methane Potential (BMP) test (Bilgili et al., 2009). The BMP tests provide an inexpensive and repeatable method to make relative comparisons of the anaerobic digestibility and potential biogas production between various substrates (Lesteur et al., 2010). The aim of this study was to evaluate the bioenergy potential of winery waste stream through the anaerobic digestion of grape marc and winery wastewater generated during wine production and through the application

of the standardized BMP test, for the first time compare the bioenergy potential of grape marc with those obtained from other organic wastes stream. The result of this study will provide essential information regarding the bioenergy potential of this important waste stream. In addition, the production of energy from winery waste will also reduce the amount of waste and prevent environmental contamination.

Results and Discussion

Biogas and methane production

Cumulative biogas production was calculated by summing the observed daily gas production values within the experimental period (15 weeks) (Fig.1). Milling the grape marc increased the total biogas production by 1.5- fold (Fig. 2). Particle size greatly influences the rate of biodegradation (Izumi et al., 2010; Taherzadeh and Karimi 2008) by increasing the susceptibility to enzymatic hydrolysis, possibly by weakening the lignocellulosic bonds in grape seeds (Raheman and Mondal 2012; De la Rubia et al., 2011).

Statistical analysis showed that the treatment containing milled grape marc had a significant difference ($P \le 0.05$) in the weekly biogas production compared to that in treatment containing non-milled grape marc. This can be attributed to the increase in surface area brought about by milling.

Similar to biogas production, reactors containing milled grape marc resulted in a greater methane production, with an average methane concentration of 41% per week as compared to the average concentration of 29% in the treatment containing non-milled grape marc. This relates to an energy potential of 31.0 MJ/kg VS and 6.7MJ/kg VS from the digesters containing milled and non-milled grape marc respectively. There was a 5-fold increase in the total energy yield in digesters with milled grape marc as compared to that in the non-milled treatment (Fig 3). The results obtained in this study indicate that particle size reduction increased substrate utilization for methane fermentation. Literature shows that reduction of substrate particle size increases the susceptibility to enzymatic hydrolysis which in turn increases biodegradability (Mais et al., 2002; Muller et al., 2007). It has also been shown that ground grape marc gives better results in terms of methane production (Bertran et al., 2004).

Non-milled grape marc generated less methane but produced double the amount of CO_2 in comparison to the treatment containing milled grape marc (Fig 3). This shows that apart from the increased recovery of green energy in form of methane, milling of grape marc will substantially reduce the carbon and environmental footprints by reducing the amount of CO_2 generated during the anaerobic digestion of grape marc and winery wastewater.

Biogas production potential

A variety of waste sources generate huge quantities of solid waste containing a sizeable proportion of biodegradableorganic matter (Kothari et al., 2010). Since the key factors affecting gas yields are organic matter content and percentage of dry matter content, different feedstock produce different amounts of biogas. A comparison of biogas production potential of some feedstock (animal slurry from cattle, pigs and chickens and residues from food processing industries, reported in literature) with biogas production potential of codigested grape marc and winery wastewater is presented in Fig 4. Of these, hen litter shows the highest amount of biogas production (30 l/ kgDW). In comparison, potential biogas production efficiency of anaerobic digester containing milled grape marc and winery wastewater is 19 l/kg DW, which is significantly higher than those from most other potential feedstock (Fig 4).

Digestate utilization

An optimum carbon-to-nitrogen ratio is required during microbial degradation as microorganisms require C for growth and N for protein synthesis. A C/N ratio of approximately 30 is considered optimal for the metabolism of microorganisms (Ferrer et al., 2001; Moldes et al., 2007). The C/N ratio in all the digesters at the start of the experiment was approximately 30.

Bustamante et al. (Bustamante et al., 2005b) have confirmed the potential of biosynthetically originated waste products for use in agriculture when they are subjected to some chemical and/or biological treatment. Ferrer et al. (2001) also successfully used biodegraded grape marc as an organic fertilizer to increase the corn dry matter. Treatments containing milled grape marc show a higher decrease in the dry matter content of the digestate as compared to that in the non-milled treatments, enhancing their potential to be used as a fertilizer. A study carried out by Amon et al. (2006) has shown that digestate with a reduced dry matter content, makes the slurry spread more evenly which can thus infiltrate more rapidly into the soil. Brunetti et al. (2012) showed the beneficial effects of anaerobically digested grape marc on the quality of soil organic matter, demonstrating that it promotes the humification process of soil organic matter, increasing its stability and agronomical efficiency. Anaerobic digestion is also able to inactivate weed seeds, bacteria, viruses, fungi and parasites in the feedstock which is of great importance when the digestate is used as fertilizer.

Materials and Methods

Sample preparation

Grape marc (10 kg, from the production of red wine) and winery wastewater (10 L, from wastewater holding tank prior to discharge or treatment) were obtained from a vineyard in Adelaide, Australia. The obtained grape marc was homogenized to a paste using a kitchen blender (particle size 1-2 mm). For trials involving the study of the effect of milling, homogenized wet grape marc was further crushed using a mortar and pestle (particle size 100-200µm). A mesophilic inoculum (seed sludge) was collected from a working mesophilic anaerobic digester at a wastewater treatment plant in Glenelg, South Australia. The inoculum was kept at 4°C prior to use. Prior to anaerobic digestion tests, the inoculum was incubated for 48 h at 35°C to allow stabilization and to deplete the residual biodegradable organic material.

Experimental set-up

The BMP100 test was carried out in accordance with the Guidance on monitoring MBT and other pre-treatment processes for the landfill allowances schemes. Laboratory scale anaerobic digesters were set up and kept at a constant temperature of 35°C. The digesters consisted of 7 Schott bottles (500 ml). Each digester was equipped with one port to transfer the biogas to a collection cylinder, fitted with gas opening valves. An air suction pump (115VAC, ENVCO, Australia) was used to fill the collection vessel with acidified (pH 4) water. The digesters were incubated in a water bath for a period of 15 weeks.



Fig 1. Cumulative Biogas production (ml/g VS) in various Anaerobic Digesters during BMP100 test. — - Milled grape marc and winery wastewater, - - Non-milled grape marc and winery wastewater.



Fig 2. Total Biogas production (l/ kg DW) in the Anaerobic Digesters during BMP100 test. [■] - Milled grape marc and winery wastewater, [■] - Non-milled grape marc and winery wastewater.



Fig 3. Total Methane (l/kg VS) and Carbon dioxide (l/kg VS) production in different treatments during BMP100 Test. Total Methane, IIII B - Total Carbon dioxide.



Fig 4. Comparison of Potential Biogas production efficiency from different feedstocks (Data obtained from Warburton, D (Warburton 1997) and compared to this study. Values expressed are in l/ kg DW.

Co-digestion of grape marc and winery wastewater was carried out to study the effect of milling the grape marc on the biogas production. The two treatments studies included: A) milled grape marc and winery wastewater, B) non-milled grape marc and winery wastewater. Mesophilic inoculum alone was used as a baseline negative control to determine and rule out the CH₄ production resulting from the inoculum. Both the treatments were tested in triplicate. In all test digesters, prepared wet grape marc samples (80 g) (equivalent to 22 g loss on ignition) (LOI) were mixed with wastewater (222 ml). This mixture was then de-oxygenated by sparging with N₂ gas for 5 min. Each digester had a pH of 7.5 which was regularly measured (Eutech PH510) and maintained (using Na₂CO₃). The temperature was measured using a portable thermometer and each digester was mixed daily to ensure contact between bacteria/enzymes and substrates.

Test monitoring

Analytical assays were run on the biogas produced and the test materials before and after the anaerobic digestion process. Grape marc was analysed for the properties related to biogas production before and after the anaerobic digestion process. The total carbon, total nitrogen and dry matter content were measured using standard methods (Mishra et al. 2001). All chemical analyses were carried out in triplicate and the average values considered for discussion. A gasometric method which uses a volume displacement device was used to quantify biogas. Biogas production was determined daily by measuring the headspace of each digester by an acidic water displacement volumetric method as previously described (US Department of State 2011). The final values obtained for total biogas production were calculated to standard temperature and pressure (STP) conditions for comparison with other studies. Biogas produced was analysed weekly for CH₄ and CO₂. A Photo Ionization Detector (PID, iBrid MX6) (Fantozzi and Buratti 2011) and Kitagawa precision tubes (Dai and Blanes-Vidal 2013) were used for measuring CH₄ and CO₂ respectively. T test was performed using Microsoft Excel to examine the

significance of the weekly differences of biogas and methane production across the two treatments.

Conclusion

In this study, the effect of anaerobic co-digestion of grape marc and winery wastewater on energy production was evaluated. The results showed that milling of wastes led to a significant increase in biogas yield and a significant decrease in dry matter content of digestate as well as CO_2 generation during the anaerobic digestion of milled grape marc compared to non-milled grape marc treatment. This study showed that anaerobic co-digestion of grape marc and winery wastewater is a promising technology for energy productions from wastes.

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