

## BIOGAS PRODUCTION OF WINEMAKING WASTE IN ANAEROBIC FERMENTATION PROCESS

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**Abstract.** Biogas production from winemaking waste – yeast biomass and wine residue containing substrate (wine lees) was investigated. Laboratory scale 700 ml batch anaerobic digesters were used. The highest biogas yield reached in this study from local wine lees was  $855.5 \text{ l}\cdot\text{kg}_{\text{VS}}^{-1}$ ;  $774.5 \text{ l}\cdot\text{kg}_{\text{TS}}^{-1}$  or  $204.5 \text{ l}\cdot\text{kg}_{\text{COD}}^{-1}$ . Conclusions – (i) local wine lees are suitable substrate for biogas production in anaerobic fermentation process; (ii) local wine lees are easily degradable substrate with low buffering capacity.

**Key words:** anaerobic fermentation, wine, yeast, biogas.

### Introduction

The European Union (EU) is the leading producer of wine. Producing some 175 million hl every year, it accounts for 45 % of wine-growing areas, 65 % of production, 57 % of global consumption and 70 % of exports in global terms [1]. Wine growing and making is also traditionally an important sector of agriculture in Mediterranean region. Wine production represents around 10 % of the value of agricultural production in France, Italy, Austria, Portugal, Luxembourg and Slovenia, and a little less in Spain [2].

The process from grape harvesting to bottled wine is complex and can include multiple processing and fermentation stages. Wineries produce a number of biological wastes [3]. The residues generated in wine-making include grape stalk, grape pomace and wine lees [4]. Grape pomace and wine lees were used in spirit distillery till a stricter regulation (EC Regulation 1493/1999) was introduced in the EU. These residues have also been used for animal feed as well as direct field fertilization. Winery residues are not a significant environmental problem, although they currently remain an unused organic material [5].

Wineries demand a great quantity of energy during the three months that winemaking time lasts, which involves a high installed power capacity. This energy is used mainly for refrigerating the must in fermentation, which reaches approximately to 35.39 kcal per liter of must [6]. Winery can cover up to 45 % of its energy requirements for the winemaking time utilizing the grape pomace that it generates itself [6]. Although winemaking waste has sufficient biogas potential its usage for energy production is not widespread.

### Materials and methods

#### Construction of bioreactor

Batch glass bioreactors with the total capacity 0,7 liter were used in the current study. The schematic diagram of the system is presented in Fig. 1.

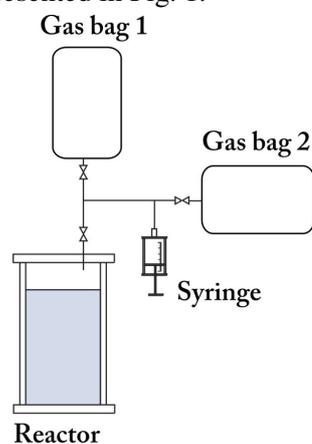


Fig. 1. Schematic diagram of batch anaerobic bioreactor used in the study

Reactors were incubated in the thermostat at temperature  $37 \pm 0.5$  °C. Two plastic gas-tight bags with the capacity of one liter were used: No 1 for gas initial collection and No 2 for collection after measurement of the volume. The volume of the produced gas was measured with a plastic syringe with precision  $\pm 2$  ml.

### **Description of winemaking waste**

Winemaking waste - lees were obtained from a homemade wine maker in Madona county. Wine lees are deposits of dead yeast and other solid particles that precipitate to the bottom of a vat of wine after fermentation and ageing. Wine lees were collected in 1,5 l plastic containers and stored in refrigerator at 4 °C for further use. Particular wine lees were mainly formed of mixture of yeast biomass, volatile organic acids, ethanol and water. Some characteristics of wine lees used in this study are summarized in Table 1.

Table 1

**Characteristics of wine lees used in this study**

Parameter	Value	Unit
Total solids	41.4 $\pm$ 0.1	g·kg <sup>-1</sup>
Volatile solids	37.3 $\pm$ 0.2	g·kg <sup>-1</sup>
COD	156 000 $\pm$ 11 000	g·kg <sup>-1</sup>
pH	3.2 $\pm$ 0.1	-

### **Seed material**

The seed material was obtained from 50 liter anaerobic continuously stirred tank reactor (CSTR) from own laboratory with plant biomass mixture as substrate. The reactor was operated at 50 days HRT, 37 °C with stable pH  $7.5 \pm 0.2$  without pH control. The content of methane in biogas was  $55 \pm 1$  %. Fresh inoculum from 50-liter reactor was used for startup procedure.

### **Experiment methodology**

Three reactors with different initial concentrations of wine lees – 10 %, 15 % and 20 % were set up. One reactor with seed material alone was used as control. In each reactor 500 g of seed material were used. In reactors with the test substrate 55, 88.2 or 125 g of wine lees were added correspondingly. The prepared mixtures were carefully mixed and headspace volume flushed with CO<sub>2</sub> to ensure anaerobic conditions. The volume of the produced gas was measured two times per day. The composition of gas was determined periodically.

### **Analytical methods**

The gas composition was determined by a gas analyzer (Gasboard-3200L, Wuhan Cubic Optoelectronics). Total solids (TS) were determined by drying the sample in the laboratory oven (60/300 LFN, Snol) at 105 °C till constant mass reached. The sample weight was determined by the laboratory balance (GF-3000, A&D). Volatile solids (VS) were determined by keeping the dried samples in a laboratory furnace (8.2/1100, Snol) at constant temperature of  $550 \pm 5$  °C during the period of 0.5 hours.

### **Results and discussion**

Gas production started soon after the experiment set-up. Cumulative biogas production is presented in Figure 1. Maximal yield was reached on the third day and did not significantly change during the rest period. This observation indicates that organic matter of wine lees is easy degradable. The methane content of all reactors was 55 – 60 %. The largest amount of biogas per unit of volatile solids added was in the reactor with 10 % of wine lees. The lowest biogas yield in the reactor with 20 % concentration can be explained by low pH of wine lees that negatively influence the stability of the process.

Biogas yields are presented in Table 2. During the study it was determined that the highest biogas production from wine lees per unit of added solids during 6 days was from 10 % concentration –  $855.5 \text{ l} \cdot \text{kg}_{\text{VS}}^{-1}$  ( $774.5 \text{ l} \cdot \text{kg}_{\text{TS}}^{-1}$ ;  $204.5 \text{ l} \cdot \text{kg}_{\text{COD}}^{-1}$ ). The biogas yield results in this study are in compliance

with the reports of other scientists. Bayerische Landesanstalt für Landwirtschaft reports biomethane yield  $661.6 \text{ l} \cdot \text{kg}_{\text{TS}}^{-1}$  from yeast of brewery waste [7]. Fountoulakis et. al. [5] studying winery waste from Mediterranean region reported  $147 \text{ l}_{\text{CH}_4} \cdot \text{kg}_{\text{COD}}^{-1}$ .

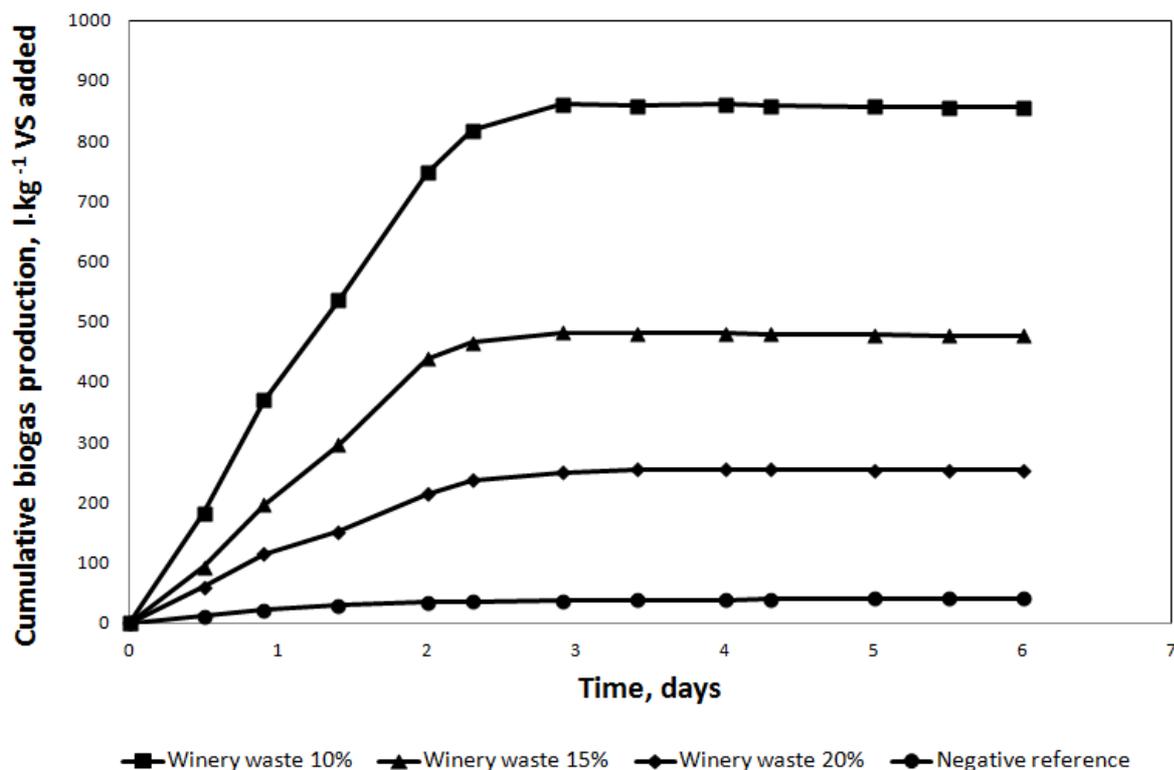


Fig. 2. Cumulative biogas production during experiment

Table 2

Biogas yields from winemaking waste with different initial concentration

Concentration	Yield			Start pH	Final pH
10 %	$855.5 \text{ l} \cdot \text{kg}_{\text{VS}}^{-1}$	$774.5 \text{ l} \cdot \text{kg}_{\text{TS}}^{-1}$	$204.5 \text{ l} \cdot \text{kg}_{\text{COD}}^{-1}$	7.15	7.18
15 %	$477.2 \text{ l} \cdot \text{kg}_{\text{VS}}^{-1}$	$432.0 \text{ l} \cdot \text{kg}_{\text{TS}}^{-1}$	$114.1 \text{ l} \cdot \text{kg}_{\text{COD}}^{-1}$	7.02	6.56
20 %	$253.7 \text{ l} \cdot \text{kg}_{\text{VS}}^{-1}$	$229.7 \text{ l} \cdot \text{kg}_{\text{TS}}^{-1}$	$60.7 \text{ l} \cdot \text{kg}_{\text{COD}}^{-1}$	6.92	5.28

For concentrations 15 % and 20 % final pH was lower than optimal for methane production. It can be concluded that in concentrations above  $\geq 15$  % wine lees are rapidly fermented in organic acids resulting in pH lowering and subsequent methane production decrease.

Reduction of TS and VS are also important indicators to illustrate degradability of substrate. Concentrations of solids in all reactors containing the test substrate as well as reference are summarized in Table 3. In all tested concentrations of wine lees TS and VS reduction was greater than one for the reference. This observation indicates that wine lees do not inhibit anaerobic fermentation process and are suitable for biogas production.

Table 3

Solid concentration in reactors before and after fermentation

Concentration	Total solids			Volatile solids		
	Before, $\text{g} \cdot \text{kg}^{-1}$	After, $\text{g} \cdot \text{kg}^{-1}$	Reduction, %	Before, $\text{g} \cdot \text{kg}^{-1}$	After, $\text{g} \cdot \text{kg}^{-1}$	Reduction, %
10 %	$14.9 \pm 0.9$	$9.7 \pm 0.3$	35.3	$11.5 \pm 0.5$	$5.8 \pm 0.2$	49.5
15 %	$16.4 \pm 0.8$	$9.8 \pm 0.5$	40.2	$12.9 \pm 0.5$	$5.7 \pm 0.3$	55.8
20 %	$17.9 \pm 0.8$	$9.6 \pm 0.1$	46.1	$14.4 \pm 0.5$	$5.5 \pm 0.2$	61.8
Reference	$12.0 \pm 1.0$	$8.8 \pm 0.3$	27.0	$8.6 \pm 0.5$	$5.9 \pm 0.1$	31.5

\*Standard deviations are calculated from 3 independent tests

Taking into account that values in Table 3 are a sum of the seed material and winemaking waste solids, recalculation was carried out to determine solid changes for winemaking waste alone. For this purpose the values in Table 3 were subtracted by the corresponding solid value from the reference reactor. The results from this operation are presented in Table 4.

Table 4

#### Calculated values of winemaking waste solids before and after fermentation

Concentration	Total solids			Volatile solids		
	Before, g·kg <sup>-1</sup>	After, g·kg <sup>-1</sup>	Reduction, %	Before, g·kg <sup>-1</sup>	After, g·kg <sup>-1</sup>	Reduction, %
10 %	2.9	0.9	69.5	2.8	-0.1	103.9
15 %	4.4	1.0	76.2	4.3	-0.2	104.2
20 %	5.9	0.9	85.4	5.7	-0.4	107.2

Although TS reduction for 15 % and 20 % concentrations is greater than for 10 %, the total biogas yield is lower due to low final pH. Negative values of VS after fermentation and reduction over 100 % indicate that fermentation of wine lees can improve degradation of other substrates (in this time solids from seed material).

These are the first results on biogas production possibilities from winemaking waste from Madona county. Further research is needed to determine the optimal concentration in different co-fermentation mixtures, preferably with substrates with high buffering capacity.

#### Conclusions

1. Local wine lees are suitable substrates for biogas production in the anaerobic fermentation process.
2. Local wine lees are easily degradable substrates with low buffering capacity.

#### Acknowledgements

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