



## Research article

# Evaluation of the treatability of a winery distillery (vinasse) wastewater by UASB, anoxic-aerobic UF-MBR and chemical precipitation/adsorption



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## ABSTRACT

A multi-stage pilot-scale treatment cycle consisting of an Upflow Anaerobic Sludge Blanket reactor (UASB) followed by an anoxic-aerobic Ultra Filtration Membrane Bio Reactor (UF-MBR) and a post treatment based on chemical precipitation with lime or adsorption on Granular Activated Carbons (GAC), was applied in order to evaluate the treatment feasibility of a real winery distillery wastewater at laboratory and bench scale. The wastewater was classified as high strength with acidic pH (3.8), and concentrations of 44,600, 254, 604 and 660 mg/l for COD<sub>tot</sub>, total nitrogen, total phosphorous and phenols, respectively. The UASB reactor was operated at Organic Loading Rates (OLR) in the range 3.0–11.5 kgCOD<sub>tot</sub>/m<sup>3</sup>/d achieving treatment efficiency up to 97%, with an observed methane production of 340 L of CH<sub>4</sub>/kgCOD. The MBR system was operated with an organic load in the range 0.070–0.185 kgCOD/kgVSS/d, achieving a removal up to 48%, 67% and 65% of the influent COD, total nitrogen and phenols, respectively. The combination of UASB and UF-MBR treatment units was not effective in phosphate and colour removal assigning to further chemical precipitation and adsorption processes, respectively, their complete removal in order to comply with legal standards for wastewater discharge. Subsequently, the optimization of the investigated treatment chain was assessed by applying a chemical precipitation step upstream and downstream the UASB reactor, and a related treatment unit cost assessment is presented in view of a further technological scale-up.

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## 1. Introduction

Processes for wine production generate organic and inorganic pollution mostly associated with solid wastes and liquid effluents. The liquid effluents usually referred as “winery wastewater” are mainly originated in washing operations during grape harvesting, pressing and first fermentation phases of wine processing (Rodríguez-Chueca et al., 2017; Ioannou et al., 2015; Lucas et al., 2010; Mulidzi, 2010; Mosteo et al., 2007), as well as a result of the distillation process applied to wine processing residues (e.g. vinasses). As a consequence, volumes and pollution loads greatly

vary over the year demanding that the treatment system must be versatile to face both the loading regimen and stream fluctuation (Bolzonella et al., 2010).

Winery wastewater contains large amounts of biodegradable organics in addition to relatively small concentrations of recalcitrant compounds such as polyphenols, organic acids and sugars and relatively low presence of solids and nutrients (Serrano et al., 2011; Braz et al., 2010). According to Andreottola et al. (2005) and Beck et al. (2005), the readily biodegradable COD represents the most relevant fraction of total COD with values ranging between 71.4% and 85%, respectively. The high percentage of this fraction is due to the prevalent presence of ethanol and, to a smaller extent, sugars and organic acids (Andreottola et al., 2009). The concentration of slowly biodegradable COD varies from 2.9% to 9.4% of total COD while the un-biodegradable soluble fraction resulted quite different

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in Andreottola et al. (2005) and Beck et al. (2005) probably due to the different approaches used for COD fractionation.

Nowadays, several winery wastewater treatment technologies are available and they involve, in most cases, the use of biological processes (Ioannou et al., 2015; Chai et al., 2014; Mulidzi, 2007; Thanikal et al., 2007; Brucculeri et al., 2005; Petruccioli et al., 2002) (see Table 1S, Supplementary material). Such processes ensure a significant removal of the organic content although the presence of recalcitrant compounds frequently makes the complete winery wastewater treatment impossible (Ioannou et al., 2015).

A common, simple and relatively low-cost solution may be represented by the co-treatment of municipal and winery wastewater in conventional activated sludge processes (Andreottola et al., 2009; Fernández et al., 2007; Pathe et al., 2002). Possible problems such as bulking phenomena or decrease of sludge settleability suggested the proposition of dedicated plant including aerobic/anoxic processes using suspended biomass (activated sludge, membrane bioreactors, MBR, sequencing batch reactors, SBR), aerobic biofilm systems (conventional rotating biological contactors, RBC, innovative fixed bed biofilm reactors, FBBR, or moving bed biofilm reactors, MBBR, SBBR), anaerobic processes using suspended biomass (conventional anaerobic digesters or anaerobic sequencing batch reactors, ASBR), anaerobic biofilm systems using granules (Upflow Anaerobic Sludge Blanket, UASB), hybrid systems (Up flow Sludge Blanket Filter, USBF) (Moletta, 2005; Andreottola et al., 1998) and constructed wetlands (Andreottola et al., 2009).

The only anaerobic processes, many of which summarized in Table 1S (see Supplementary material), are not able to ensure compliance with final discharge standards set by the law. Consequently, they are generally used as pre-treatments thus requiring additional treatments downstream.

Membrane technologies, combined with conventional activated sludge-based processes, have been extensively studied over the years. They represent an interesting solution especially for small companies as highlighted in Bolzonella et al. (2010).

Other technological solutions are based on the combination of different processes, even in a single reactor, as already experienced in Andreottola et al. (2005), Farina et al. (2004), Petropoulos et al. (2016) and Molina et al. (2007), Akunna and Clark (2000), Basset et al. (2016) and Andreottola et al. (2002) with reference to FBBR, ASBR, hybrid USBF, GRABR, AnMBR and SBBR, respectively. However, organic matter removal is not the only task for winery wastewater treatment, since nitrogen, phosphorous compounds, and colour also need to be tackled following a multiple objectives approach (Ioannou et al., 2015).

In this regard, literature shows several examples such as Amaral-Silva et al. (2016) who integrated ferric coagulation, Fenton reaction and activated sludge for phosphorus removal. De Gisi et al. (2016) highlighted the use of granular activated carbons (GAC) as well as alternatively low-cost sorbents for colour removal from different wastewater. Regarding membrane technologies, Bolzonella et al. (2010) adopted a MBR system based on microfiltration (MF) for the biological activated sludge phase, while real cases based on the use of ultrafiltration MBR (UF-MBR), are rather limited. Sheldon and Erdogan (2016) have recently applied an UF-MBR for soft drink production wastewater, with different characteristics respect to the winery ones.

In this context, with the intent of strengthening the current knowledge, the article deals with the verification of the treatability of a real winery distillery (vinasse) wastewater through the application of a treatment cycle consisting in UASB, anoxic-aerobic UF-MBR, and post treatment steps (chemical precipitation with lime or alternatively GAC adsorption), in order to comply with the discharge standards set by the Italian and European regulations. More specifically, the following sub-goals have been investigated:

(i) Identification of the optimal operational set-up and related criticalities of the UASB reactor as well as verification of the process efficiency with reference to high organic loading rates (OLR) in the range 6.2–11.5 kg COD/m<sup>3</sup>/d; (ii) Identification of the optimal operational set-up and related criticalities of the anoxic-aerobic UF-MBR system, assessing the individual contributions referable to the biological process and the membrane separation process; (iii) Identification of the main process parameters of chemical precipitation with lime and GAC adsorption, intended as post-treatment solutions; (iv) Optimization of the treatment cycle by using chemical precipitation with lime upstream and downstream the UASB treatment unit.

## 2. Materials and methods

### 2.1. Experimental plan

The experimental trials were carried out on a real winery wastewater. A total amount of about 2 m<sup>3</sup> was collected from a distillery located in South Italy (Sicily) and, after a preliminary characterization, the inlet wastewater was properly stored into a stirred and refrigerated (2–4 °C) tank, also used as feeding unit.

The pilot scale tests involved three treatment steps arranged in series, as following reported (see Fig. 1a): (i) anaerobic treatment; (ii) anoxic-aerobic treatment; (iii) chemical and physical post-treatment. Regarding the first treatment step, an UASB reactor was used with the intent of maximizing the biogas production to be used for energy purposes. As second treatment step, an anoxic-aerobic MBR system equipped with an ultrafiltration membrane was realized and fed with the clarified fraction of the UASB effluent (digestate), with the main scope to provide a residual COD removal as well as a reduction of N and P content. The third step consisted in two separate treatment options: chemical precipitation based on the use of lime, and GAC adsorption. The main scope of such treatments was the residual COD, phosphorous and colour removal in order to comply with the effluent standards for wastewater discharge set by the European (Directive 91/271/EEC) and Italian Law (Legislative Decree No. 152/2006).

Each treatment step was operated separately, the effluent of each treatment unit being stored and refrigerated in order to be fed to the downstream one. At first, the UASB process was operated for 4 months, and the effluent was stored in a refrigerated tank during last 3 operation weeks before being fed to the MBR section; the MBR was operated for 4 months and the effluent was collected and stored during last 2 months of operation and then used for further chemical treatments. The whole experimental tests were carried out for 10 months and structured according to the time schedule as shown in Fig. 1b.

### 2.2. Inlet wastewater characterization

The real wastewater consists of vinasse resulting from the grape distillation process. Wastewater was firstly characterized by means of chemical-physical analyses, as reported in Table 1, showing an acidic pH and a high organic matter concentration, with an almost all soluble COD including a recalcitrant fraction which is, at least in part (e.g. tannic content), responsible of the dark colour as discussed hereinafter.

### 2.3. Reactor set up

The bench scale UASB system consists in a cylindrical reactor made of Plexiglas with an approximate volume of 24 L (H = 75 cm, D = 22 cm); a series of manual ball valves in stainless steel have been inserted on its surface in order to allow (i) the removal of the

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