



Combining reverse osmosis and ion-exchange allows beet distillery condensates to be recycled as fermentable dilution water



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HIGHLIGHTS

- Recycling of beet distillery condensates as fermentation water is investigated.
- The removal of target inhibitory compounds is examined.
- Combined physical purification processes are compared to single ones.
- Multistage fermentation is implemented to assess fermentability.
- Combining RO with IE allows 87.5% of perfectly fermentable water to be recovered.

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ABSTRACT

Combinations of physical purification processes, i.e. anion-exchange, adsorption, and reverse osmosis were evaluated and compared with single ones for their ability to remove target inhibitory compounds from distillery condensates with the purpose of reusing condensates as fermentation water. Performances of the treatments were evaluated through analyses of residual target compounds and batch and continuous fermentation experiments. Reverse osmosis on BW30 membrane (Dow Chemical) at 25 bar transmembrane pressure and volume reduction ratio (VRR) = 8 followed by anion-exchange (weak Amberlite FPA 51 resin, Dow) was the most efficient process to decrease all inhibitory target compounds (formic, acetic, propanoic, butanoic acids and 2-phenethyl alcohol) present in a distillery condensate below their detection or quantification limit. Water recovery was 87.5%. Such treated condensate proved convenient for reuse as fermentation water. Fermentation tests run in a multistage device exhibited yeast viability and ethanol production performances (concentration and global productivity) equivalent to the blank for a final ethanol concentration of 80 g L⁻¹ close to practical value encountered in distilleries.

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1. Introduction

Whatever the raw feedstock is (starch- or sugar-based), ethanol production consumes large quantities of water and gives rise to stillage, a highly polluting wastewater. Reducing water consumption through stillage recycling is a growing matter of concern. However, direct recycling can only be very partial given the risks of adverse effects on alcoholic fermentation [1–3].

Biological treatment through anaerobic and aerobic steps as currently practised in India or Asia especially does not allow stillage to comply with discharge standards and a fortiori to be considered for recycling [4];

although further purification by nanofiltration was claimed to produce potentially reusable water, this was not assessed experimentally [5]. Recently, a two-stage anaerobic treatment (UASB) coupling thermophilic and mesophilic sludges was proposed on cassava vinasses [6–8] and the treated water could be recycled at 80% at least without disturbing fermentation over several cycles. However, biological treatments are expensive, energy consuming and lack flexibility [6]. Several works have focused on the interest of membrane technology to recover reusable water directly from stillage: ultrafiltration was studied with contradictory results on recycling [9,10]. Coupling nanofiltration and reverse osmosis was proved to give water clean enough to be rejected but recycling as dilution water was not considered [11].

Some countries have generalised a different strategy for stillage management based on thermal concentration. In France, beet stillage is commonly concentrated for the production of fertilisers or cattle

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feed, allowing water to be recovered as condensates. However, in spite of significant efforts on the design of the concentration units, the quality of condensates remains not sufficient for a safe recycling and they are currently land-disposed after purification in ponds, corresponding to €3000/day expense for a distillery producing 500 m³ alcohol day⁻¹ [12]. The need for better water management imposes more intensive and cheaper purification treatments. Surprisingly, although condensates should be easier to purify and valorize than raw stillage, there is, to our knowledge, very few work published on it, at the exception of ours.

In our research team, condensate purification through physical processes i.e. ion-exchange, adsorption and reverse osmosis, has been studied for several years. Condensates contain significant quantities of potentially inhibitory volatile compounds [13]. Acetic acid is the major one (1–2 g L⁻¹) but other aliphatic acids, alcohols, aromatic compounds and furan derivatives are present with assessed inhibitory effect even at low concentrations. Nine molecules: C1 to C6 acids (formic, acetic, propanoic, butanoic, pentanoic and hexanoic), 2,3-butanediol, furfural and 2-phenethyl alcohol were selected as “targets”. Actually, none of the aforementioned processes proved able to remove all the target compounds but based on their different selectivity principles, combinations could perhaps achieve complete removal. Besides, some questions are still pending: is total purification necessary? Does the absence of target molecules warrant that condensates are innocuous? Which molecules are especially toxic? Up to what threshold should they be removed? Indeed, toxicity levels for target molecules in single solutions were determined [14]. But these levels can hardly be used as requirements for condensate quality because of synergetic effects between inhibitors and the influence of the purity of the fermentation medium. Besides, ethanol has also well-known toxic effects at a high concentration. Fermentation experiments in conditions representative of industrial application are therefore necessary.

The objective of this article is to investigate combinations of different physical detoxification processes and to compare them with single ones in order to propose a new and reliable process for condensate purification and re-use as dilution water. Purification efficiency will be evaluated and fermentability will be assessed through batch and continuous fermentation experiments. Comparing the different treatments should help to relate fermentability with the concentration of inhibitors and to define a limit below which no toxicity effect on fermentation is observed.

2. Physical detoxification processes and strategy for combined treatments

Anion-exchange resins have good capacities for organic acids and especially acetic acid. Ion exchange (IE) is a cost-effective and low energy consuming technology. Associated drawbacks (reactant consumption and waste production) can be significantly reduced by selecting a weak resin, optimising operating conditions and rationalising its integration into the global process. A previous study [15] showed that the weak anionic Amberlyst A21 (Dow) resin displays a maximal capacity close to 2 eq L⁻¹ for acetic acid in single solution; for a condensate containing 30 meq L⁻¹ of acetic acid and 3 meq L⁻¹ of butanoic acid (a major other acid), the stoichiometric volume is 37.8 BV (Bed Volume) and 56 BV, respectively. Amberlite FPA 51 (food-grade version of Amberlyst A21) achieves a total capacity of 1.08 eq L⁻¹ for acids in a condensate containing 15 meq L⁻¹ of acetic acid and lower concentrations of other target acids and shows a strong affinity for formic acid [16]; 37 BV of condensate (useful capacity 0.75 eq L⁻¹) may be processed before the breakthrough of acids detected by a rapid decrease of pH below 5 (the pH could therefore be a relevant indicator for process control).

However, neutral compounds such as 2-phenethyl alcohol or furfural are not or badly retained by IE making such treated condensate perhaps unsuitable for fermentation. Combining anion-exchange with

adsorption (Ads) should ensure total purification of condensates. Amongst different adsorbent polymers tested, food-grade Optipore SD2 (Dow) combined the best retention of target aromatic compounds (2-phenethyl alcohol and furfural) with a significant retention of acids [17]; stoichiometric volume was larger than 130 BV for 2-phenethyl alcohol and between 15 and 120 BV for the acids depending on the C-chain length and concentration. To limit the frequency of regeneration, adsorption should be implemented on acid-free condensate after a preliminary anion-exchange step.

The interest of reverse osmosis (RO) for removing inhibitory compounds in condensates was first assessed with “sea water” type membranes [18]. Looking for higher permeate fluxes and lower transmembrane pressure (TMP), “brackish water” type membranes were tried, especially CPA2 (or CPA3) and ESPA2 from Hydranautics and BW30 from Dow [19]. Two strategies (i) ESPA2/TMP = 10 bars and (ii) BW30/TMP = 25 bars were identified to meet an optimal permeate flux of 30 L h⁻¹ m⁻². Both membranes gave 100% rejection of the target neutral molecules of high molecular weight. Rejection of formic acid was 34% with BW30; rejection of acetic, propanoic and butanoic acids was 59, 80, 98% with ESPA2 and 85, 92, 100% with BW30, respectively, and it kept constant with increasing volume reduction ratio (VRR) up to 8. At equivalent permeate flux, BW30 displays better rejection than ESPA2 and should be preferred. Based on a 1500 ppm concentration in the feed and using a simple calculation algorithm presented in [20], an acetic acid mean permeate concentration of 371 and 452 ppm could be predicted for 75% (VRR = 4) and 87.5% (VRR = 8) water recovery, respectively. A further anion-exchange step would therefore be necessary to complete the removal of acids.

3. Material and methods

3.1. Condensate

Condensate was collected over a 14 working day period from Toury beet distillery (Cristal-Union, France). It was clear, pale yellow and slightly odorous.

3.2. Resins and membranes

Amberlite FPA51 (Dow Chemical) is a weak base anion-exchange resin based on a polystyrene matrix. According to the manufacturer, it contains at least 85% tertiary functional groups and the total capacity is ≥ 1.3 eq L⁻¹ under free base form. SD2 Optipore (Dow) is a polystyrene adsorbent with a specific area >800 m² g⁻¹. According to the manufacturer, it displays additional anion-exchange properties conferred by the presence of functional NR₂ groups. Ion-exchange capacity is >0.8 eq L⁻¹. Both chromatographic supports are food-grade. FPA51 was used under free base form after preliminary contacting with NaOH 1 N and thorough rinsing. SD2 was pre-treated by contact with ethanol and methanol, rinsed and contacted with NaOH as FPA51 to activate the ionic groups.

Table 1
RO membranes specifications.

Manufacturer	BW 30	ESPA2
	Dow chemical	Hydranautics
T _{max} (°C)	45 (35 at pH = 10)	45
P _{max} (bar)	41	41.4
pH range	2–11	3–11
NaCl rejection rate (%)	99.5*	99.6**

* At 15.5 bar and feed 2000 ppm NaCl.

** At 10.5 bar and feed 1500 ppm NaCl.

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