

In Situ Two-Stage Gas Stripping for the Recovery of Butanol from Acetone-Butanol-Ethanol (ABE) Fermentation Broths

Rebeca Díez-Antolínez¹, María Hijosa-Valsero¹,
Ana I. Paniagua-García¹, Xiomar Gómez²

¹Centre of Biofuels and Bioproducts, Instituto Tecnológico Agrario de Castilla y León (ITACyL), Villarejo de Órbigo, Spain

²Natural Resources Institute (IRENA), Universidad de León, León, Spain



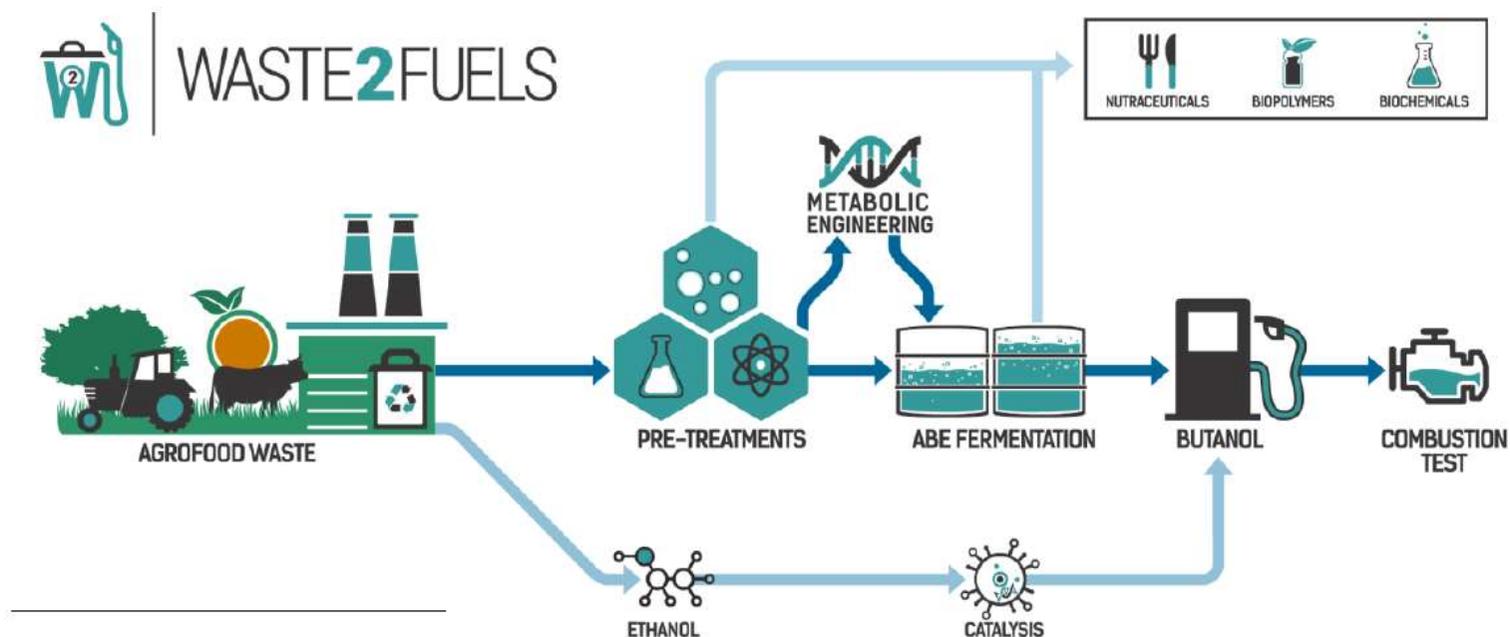
INTRODUCTION



Waste2Fuels project

Sustainable production of next generation biofuels from waste streams

- Twenty participants from Spain, Italy, Austria, Ireland, United Kingdom, Greece, Germany, France and Israel.
- From 1 January 2016 to 31 December 2018.

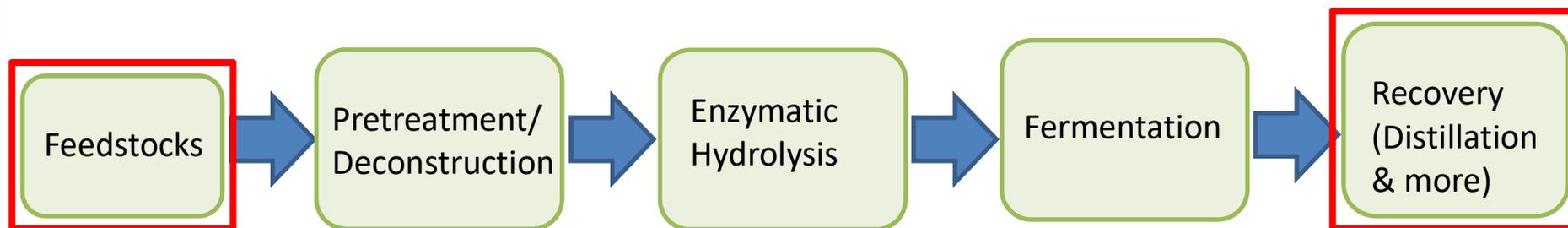


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Overview of ABE fermentation process



| Pros of Butanol as Biofuel | Challenges of ABE fermentation |
|----------------------------|--|
| High energy content | High cost of traditional feedstock |
| Low water absorption | Clostridia strains don't ferment naturally cellulosic substrates |
| Low vapour pressure | High cost of pretreatment/deconstruction processes |
| Less corrosive | Substrate and product inhibition processes |
| Low volatility | Low productivity |
| Good blending ability | High cost of solvent recovery processes |

Alternative Feedstock: AgroFood Wastes (AFWs)



Solvent Recovery step



Typical distillation ABE recovery unit in batch processes

Alternative In situ Product Recovery (ISPR) techniques in research

Gas Stripping

Vacuum Fermentation

Pervaporation

Liquid-Liquid Extraction

Perstraction (Membrane Extraction)

Adsorption



OBJECTIVES



Objectives:

- To improve the ecoefficiency of butanol recovery using two-stage gas stripping.
- To optimise one-step stripping working conditions (feed temperature, gas flow and refrigeration temperature) for a maximal simultaneous butanol selectivity (α_B) and butanol recovery efficiency (η_B).
- To assess the effect of in-situ gas stripping in ABE fermentation bacteria (*Clostridium beijerinckii* CECT 508).

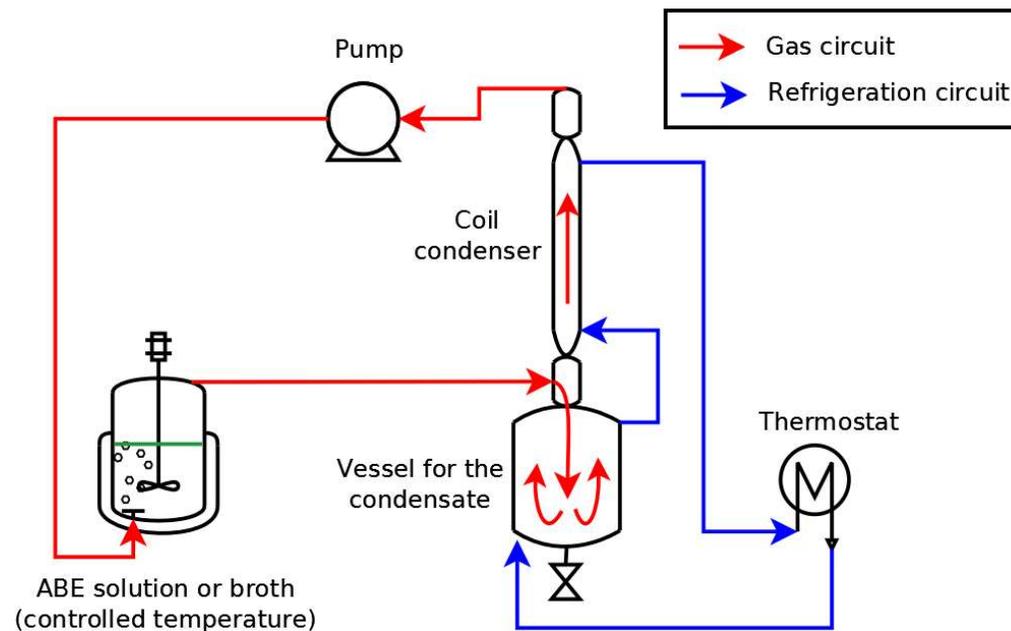


EXPERIMENTAL SECTION

Gas stripping optimization



- Feed solution: a synthetic aqueous solution A:B:E (3:6:1) (5 g/L acetone, 10 g/L butanol and 1.67 g/L ethanol).
- Gas stripping setup scheme:



- Response Surface Methodology (RSM) experimental design.
- Experimental validation of RSM equations at various operation times (4-18h).



Gas stripping optimization

- **Response Surface Methodology (RSM) experimental design**
 - 20 experiments and included 8 cube points, 6 central points and 6 axial points ($\alpha = 1.68179$).
 - 3 working conditions: feed temperature (T_{feed}), gas flow (G) and refrigeration temperature ($T_{\text{ref.}}$).
 - Optimum combination to maximize the response values (butanol selectivity (α_B) and butanol recovery efficiency (η_B)):

$$\alpha_i = \frac{y_i / (1 - y_i)}{x_i / (1 - x_i)}$$

$$\eta_i = \frac{m_i C}{m_i F} \times 100$$

- **Two-stage gas stripping**
 - The aqueous phase (8 % (w/w) butanol) subjected to a 2nd gas stripping.

Fed-batch fermentation system coupled with gas stripping



Fermentation broth:

Substrate: Cheese whey ($L_0 = 40$ g/L) with nutrient supplementation.

Volume: 500 mL.

Microorganism: *Clostridium beijerinckii* CECT 508T.

Bacterial density: $7 \cdot 10^8$ cell/mL.

Stripping conditions:

Initial ABE composition: 3.02 g/L acetone; 12.04 g/L butanol; 0.23 g/L ethanol; 0.99 g/L acetate; 1.15 g/L butyrate.

Working conditions:

- Time: 4h.
- T_{feed} , G , T_{ref} : optimum RSM results.

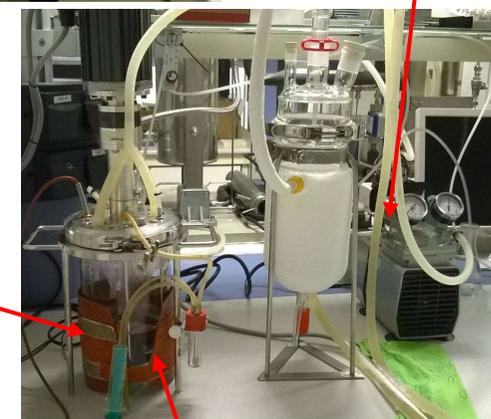
Antifoaming was added.

Experimental gas stripping setup



Thermostat

Fermenter,
ABE broth





RESULTS

Gas stripping optimisation



- Estimated RSM optimal working conditions were experimentally validated at various gas stripping operation times (4-18 h) with an synthetic aqueous medium and a fermentation broth to improve global energy efficiency.
- Alternative feed temperatures ($T_{\text{feed}} = 35^{\circ}\text{C}$) more suitable for bacteria were tested.
- A two-stage gas stripping was proposed to highly concentrate the condensate in butanol.
- The effect of gas stripping conditions on bacterial development was assessed.

Gas stripping optimisation



Response Surface Methodology (RSM) experimental design

The number of variables to be optimised was reduced to three in order to simplify the process.

| RSM variables to be optimized | |
|-------------------------------|-----------|
| T feed (°C) | 25 to 50 |
| Gas flow rate (L/min) | 2.6 to 10 |
| T refrigeration (°C) | -20 to -5 |

| Fixed parameters | |
|-----------------------|-----------|
| Time (hours) | 18 |
| ABE composition (g/L) | 5:10:1.67 |
| Volume (mL) | 500 |

According to the RSM mathematical estimations:

Optimal working conditions:

$T_{\text{Feed}}: 60\text{ }^{\circ}\text{C}$

Gas Flow rate: 1.34 L/min

$T_{\text{ref}}: 5\text{ }^{\circ}\text{C}$

Simultaneous response:

Separation factor, $\alpha_B = 6.9$

Efficiency, $\eta_B = 82.9\%$

Gas Stripping optimisation



Response Surface Methodology (RSM) experimental design

Validation of the RSM model

The optimal estimated values for T_{feed} (60 °C), G (1.34 L/min) and T_{refr} (5 °C) were experimentally validated at different stripping times.

| Stripping time (h) | Selectivity, α | | | Recovery efficiency, η (%) | | | Concentration in the distillate (g/L) | | |
|--------------------|-----------------------|-------|------|---------------------------------|-------|-------|---------------------------------------|-------|-------|
| | A | B | E | A | B | E | A | B | E |
| 18 | 1.74 | 3.61 | 3.64 | 41.67 | 84.57 | 86.95 | 9.41 | 34.71 | 6.34 |
| 10 | 3.66 | 5.52 | 5.32 | 59.16 | 86.76 | 86.65 | 19.23 | 51.32 | 9.46 |
| 4 | 6.28 | 10.36 | 7.74 | 51.30 | 79.79 | 64.24 | 32.55 | 92.14 | 13.69 |

Concentrations above water solubility value. Two phases appear.

Shorter stripping times increase selectivity (α) without drastically reducing recovery efficiency (η).

Modification of RSM gas stripping optimum conditions

Optimal working conditions:

T_{Feed} : 35 °C

Gas Flow rate: 1.34 L/min

$T_{\text{ref.}}$: 5 °C

Simultaneous response:

Separation factor, $\alpha_B = 4,2-5,7$

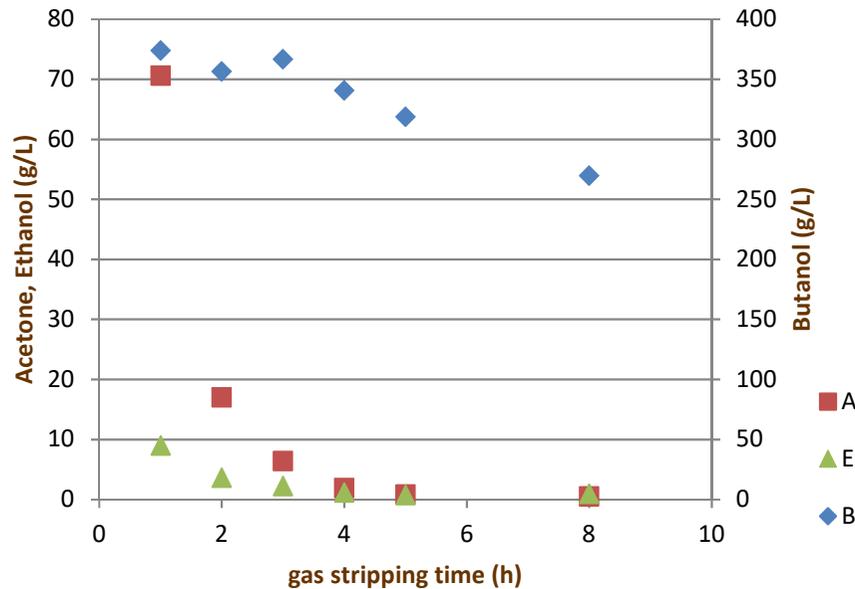
Efficiency, $\eta_B = 15,3-35,9 \%$

Two-stage gas stripping

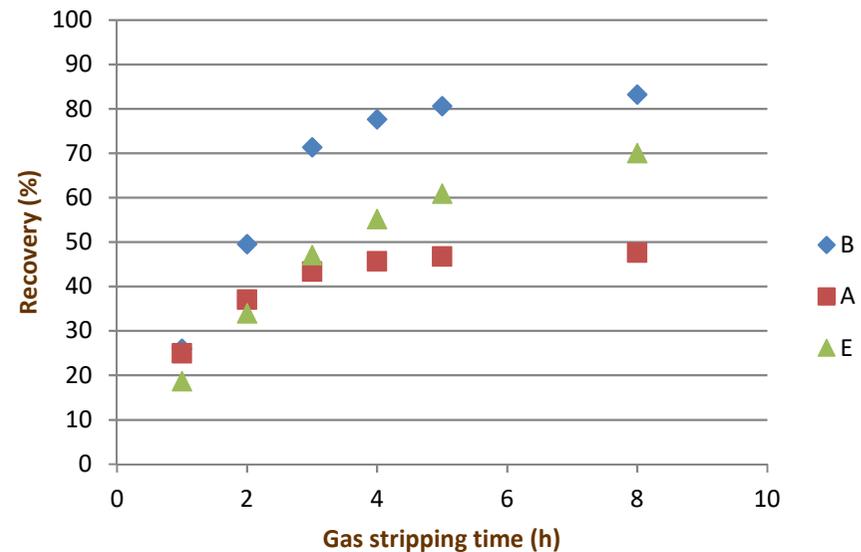


The condensate collected from the 1st gas stripping was subjected to a 2nd gas stripping.

Dynamic evolution of parameters during 2nd gas stripping:



a) Dynamic evolution of ABE solvent concentration in the condensate during second-stage gas stripping



b) Dynamic evolution of ABE solvents recovery during second-stage gas stripping.

Fed-batch fermentation system coupled with gas stripping

Fermentation broth

- Gas stripping conditions ($T_{\text{feed}} = 60\text{ }^{\circ}\text{C}$, Gas flow = 1.34 L/min, $T_{\text{refr}} = 5\text{ }^{\circ}\text{C}$) were tested to check butanol recovery and cell viability.
- Stripping time: 4 h.



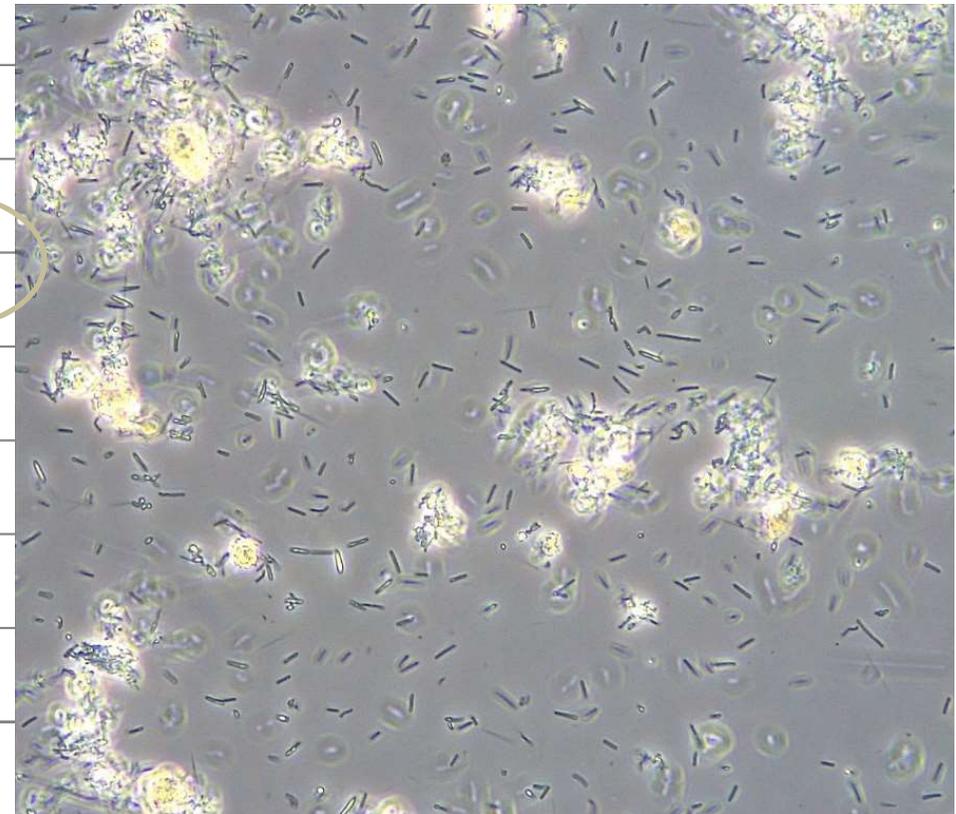
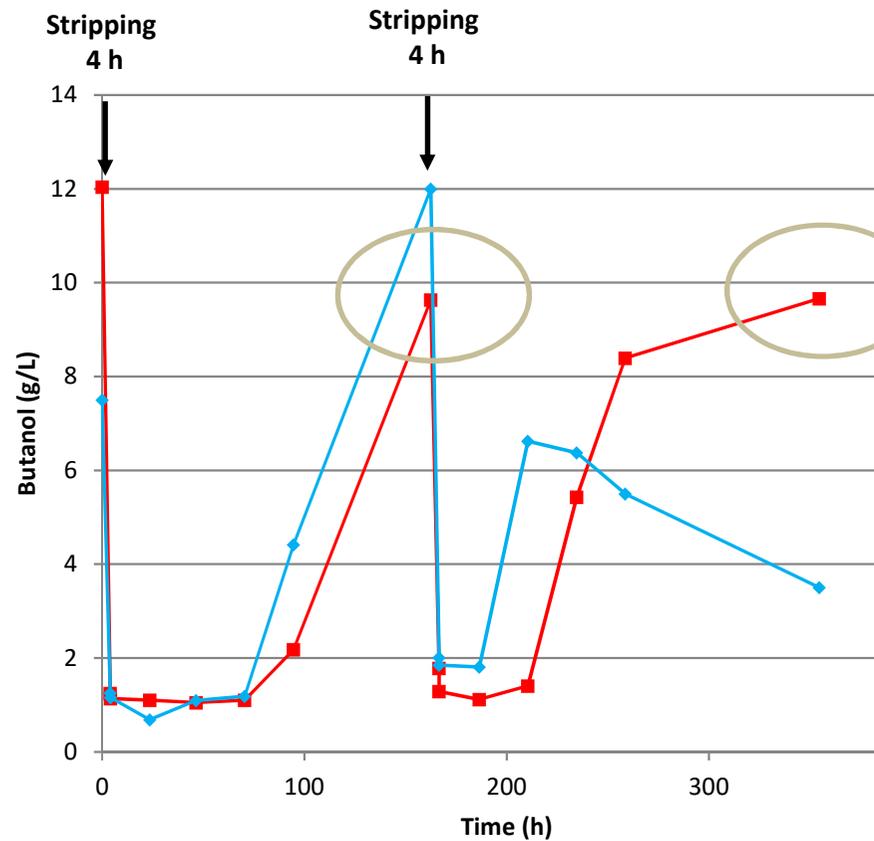
| | | 1 st Cycle | 2 nd Cycle |
|-----------------------------------|---|-----------------------|-----------------------|
| Selectivity, α | A | 4.65 | 4.79 |
| | B | 11.08 | 13.95 |
| | E | 9.32 | 17.11 |
| Recovery efficiency, η (%) | A | 27.60 | 25.41 |
| | B | 59.29 | 66.97 |
| | E | 55.83 | 91.99 |
| Concentration in condensate (g/L) | A | 13.89 | 22.16 |
| | B | 118.97 | 119.43 |
| | E | 2.14 | 4.94 |

Concentrations above water solubility value. Two phases appear.

Butylic phase (7% volume):
661.50 g/L B
Aqueous phase (93% volume):
77.08 g/L B

Fed-batch fermentation system coupled with gas stripping

Effect of gas stripping conditions on bacterial development





CONCLUSIONS

Conclusions



- Optimization of gas stripping parameters, including feed temperature, gas flow rate and refrigeration temperature are crucial for in situ butanol recovery.
- A two-stage gas stripping allows recovering highly concentrated butanol condensates, reducing energy consumption during the dewatering process.
- *Clostridium beijerinckii* CECT 508 strain resisted *in situ* gas stripping in fed-batch fermentation process but was negatively affected (too high feed temperature).



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