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APPLICATION OF A GAS RECIRCULATION SYSTEM TO INDUSTRIAL ACETIC FERMENTATION PROCESSES

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ABSTRACT

This paper describes a gas recirculation system for the exhaust gases from the aerobic fermenters normally used in acetic fermentation processes. With the application of this procedure, it is possible to operate in a closed system, so preventing the large losses of fermentation yield due to evaporation which occur in open systems. In addition, this system reduces losses of volatile organoleptic compounds (ethanol, acetic acid and ethyl acetate, among others) so enabling the product to be incorporated into processes for the manufacture of high quality vinegars.

INTRODUCTION

Acetic fermentation can be defined as a thermodynamically enhanced biological oxidation process in which ethanol is partly oxidised by acetic acid bacteria to give acetic acid and water (Suárez-Lepe, 1990).

In general, aerobic fermentation processes use of fermenters with stirring devices to disperse O_2 in the medium. O_2 may enter in pure form or as a gas mixture and suitable devices are used to ensure an efficient transfer of gas to the liquid. Part of the O_2 is consumed by the microorganisms, the remainder is evacuated, with other gases, from the fermenter. The removal of these gases causes a significant loss of volatile components which are withdrawn from the system in the flow of gases.

In the particular case of the acetic fermentation, this loss of volatile components affects both the substrate, ethanol, and the final products, acetic acid, ethyl acetate, etc.. As a result, a loss is seen in fermentation yield and in the quality of the products obtained.

The present project optimizes and implements a special fermentation system of gas recirculation, described already in the literature for other processes (Ferrer and Clotet, 1980), but applying it here to the acetification process. In this system, some elements of standard aerobic fermenters are altered, doing away with the release of gases, thereby largely avoiding loss of volatile compounds.

MATERIAL AND METHODS

The following is a description of the methods of analysis and the equipment used for the experiment.

Experimental equipment. The fermentation system used is shown in diagrammatic form in Figure 1. Its characteristics are as follows:

- A cylindrical fermentation vessel, with a hemispherical base and crown. The working volume of the equipment used was 5 litres (laboratory scale) and 10,000 litres (semi-industrial scale).
- A stirrer drive with an automatic speed regulator.
- A temperature sensor.
- A glass electrode for continuous pH measurement.
- A polarographic dissolved oxygen sensor.
- A foam level sensor.
- An air diffusion system using microporous stainless steel diffusers.
- A liquid and gas sampling port.
- Four baffles throughout the length of the fermenter.
- Filters for the input and output of air to the fermenter, fitted with a non-return system.

Because the biomass present consumes oxygen from the flow of gas, it is necessary to inject small amounts of oxygen at a frequency depending on the microbial growth phase (Ferrer and Clotet, 1980). The replacement of oxygen in the medium is governed in this system by a polarographic dissolved oxygen sensor submerged in the medium. The dissolved oxygen concentration is thus regulated with a proportional-integral-differential (PID) controller so that, when the concentration drops below the set point, the oxygen injection device is automatically activated and when such a concentration is exceeded, the oxygen input is stopped, maintaining recirculation at all times.

The control element consisted of a multiple-input controller for the connection of the sensors and an interface to enable data recording by a computer. Likewise, the

interface had outputs enabling it to control pH, dissolved oxygen, temperature and the incorporation of anti-foam substances. Oxygen was injected through an ON/OFF type solenoid valve connected to an oxygen supply, and the gases were recirculated with a vacuum/pressure pump.

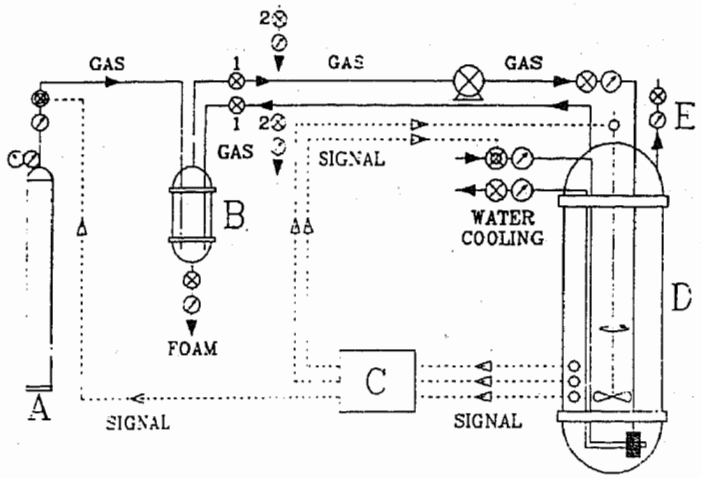

Figure 2. Scheme of the experimental equipment used (laboratory and semi-industrial scales). A: oxygen supply unit. B: gas mixer and foam trap. C: automatic control equipment. D: discontinuous aerated fermenter. E: pressure safety valve. Open gas system: valves 1 closed and 2 open. Gas recirculation system: valves 1 open and 2 closed.

Optimal operating conditions. All the experiments were operated with aeration rates of 0.5 vvm and a stirrer speed of 400 rpm; these conditions ensured that oxygen transfer coefficients from the gaseous to the liquid phase were sufficient, as seen in Table 1, to sustain the necessary addition of oxygen to the fermentation medium. These transfer coefficients were determined by the static absorption-desorption method (Atkinson and Mavituna, 1987).

Temperature was kept constant at 25 ± 1°C; in all cases, the initial concentrations of ethanol and acetic acid were 70 g/litre and 10 g/litre respectively.

Table 1. Experimental values of $k_L a$ (h^{-1}) at laboratory scale (Gómez et al. 1993)

Agitation (rpm)	Aeration (vvm)		
	0,05	0,2	0,5
100	--	14	67
400	16	36	118
700	39	61	111

Raw material. The fermentation substrate in all the experiments was a complex natural medium comprising a young wine from the wine-making region of Jerez (Spain). This medium was sterilised at 120°C for 20 min and then the pH was ajusted to 4.0 with 1M KOH. All experiments used a submerged culture of one of the main vinegar-isolated strains, classified as Acetobacter aceti ATCC 15973.

Analytical methods. During the experiments, in order to monitor the different variables, samples were taken from the fermenters at regular time intervals. The concentrations of ethanol, acetic acid and ethyl acetate were analyzed in each case by gas chromatography (Sanz, 1987). Total acidity was also calculated in each sample, by potentiometric evaluation up to pH of 7, using 0.3M NaOH as titrant (Amerine and Ough, 1980).

EXPERIMENTAL RESULTS

For the conditions described above, experiments were carried out in open systems at both laboratory and pilot plant levels, in order to make the appropriate comparisons. Fermentation yields (the quotient of the net acetic acid obtained divided by the ethanol consumed) from the different fermentation systems are shown on Table 2.

Table 2. Comparison of the fermentation yields (%) between open and recirculation systems.

	FERMENTATION YIELDS	
LEVEL	OPEN SYSTEM	RECIRCULATION SYSTEM
Laboratory	70	100
Laboratory	60	100
Pilot Plant	70	99

On the other hand, ethyl acetate is a compound which forms in appreciable quantities in the acetic fermentation. Because of its high volatility and low solubility in water, it can be used as a recovery index for yield losses in the system. In this way the detection of high ethyl acetate concentrations in the system would make clear the efficacy of the air re-utilization system.

Figure 2 shows the general tendencies of the ethyl acetate concentration in an experiment, at laboratory scale, carried out in two fermenters in parallel, one with an air recirculation system and the other open (without recirculation).

As can be seen, the general tendency is an increase as fermentation proceeds, to a maximum, followed by a drop. This pattern is due basically to the displacement of the

dynamic esterification balance, which is confirmed by the calculations of ethyl acetate concentration in balance with the concentrations of ethanol and acetic acid present at any time.

The following are the thermodynamic data for this reaction: $\Delta G^{\circ}_{25^{\circ}C}$ = 2,65 kcal/mol and K_{eq} = 0,0114 (Hill, 1977). However, with these data and the ethanol and acetic acid concentrations, slightly higher concentrations are to be expected of ethyl acetate in solution. The reason why the real concentration detected is lower may be due fundamentally to the gaseous phase maintained in recirculation with the equipment. On the other hand, the maxima of both curves, while displaced in time, appear in both fermenters when they are at the same pH and ethanol and acetic acid concentrations. This confirms the dependence of the ester concentration on the concentrations of alcohol and the acid present in solution. In general, the maximum ethyl acetate concentration thus appears when the product of the ethanol and acid concentrations is a maximum, as a result of the esterification balance referred to.

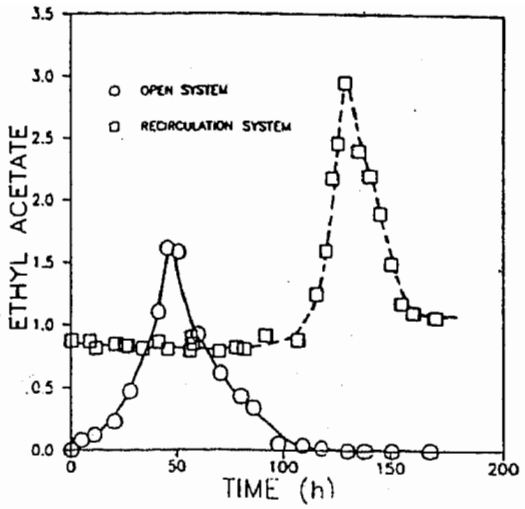

Figure 2. Evolution of ethyl acetate concentrations in systems with and without gases recirculation.

Finally, the maximum values attained in this case were: 2.5 g/litre for the system with recirculation, and 1.6 g/litre for the open system, from which it is deduced that the efficiency of recovery of volatile substances from the system with air recirculation is high in comparison with open systems.

The high initial levels of ethyl acetate in the system with air re-utilization are due in large part to the operational dynamics itself: in other words, the semi-continuous charge/discharge process at the end of each fermentation favoures the accumulation of that compound in the system.

CONCLUSIONS

From the results obtained, it is seen that the application of the gas recirculation system to acetic fermentation processes in a submerged culture is satisfactory. On the one hand, the system examined minimizes the losses of volatile compounds (ethanol and ethyl acetate) during the process and, on the other hand, fermentation yields of close to

100% are obtained. These results suggest the use of closed systems in preference to open systems where evaporation losses represent between 30% and 40% of the final yield.

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