Bioprocess Engineering 18 (1998) 289–291 © Springer-Verlag 1998

Evaluation of ethanol evaporation losses in acetic acid fermentations

L.E. Romero, D. Cantero

Abstract Three different operation systems have been employed at laboratory, pilot plant and industrial scales. Developed experimentation has demonstrated that quantified ethanol losses minimize significantly when operating at low temperatures with low aerations and when mainly working with the closed system.

1 Introduction

Evaporation of volatile compounds during acetic fermentation processes is one of the main causes of reduced yields on the industrial scale. Batch operation of large capacity plants (10,000–200,000 l) is common in this type of process. In this situation, given the need for extensive aeration of the medium, a large amount of air saturated with volatile components leaves the fermenter. Generally speaking, losses of ethanol due to evaporation in industrial fermentation can result in overall reductions of 10% to 30% compared to the stoichiometric yield, depending on the working temperature [1].

Given the increasing importance of this process in the fermentation industry, an in-depth study of operation systems minimizing evaporation losses is needed.

2 Materials and methods

2.1 Microorganism

Every experiment used a submerged culture of the microorganism strain classified taxonomically as *Acetobacter aceti* ATCC 15973, one of the most widely used in the industrial production of vinegar.

2.2 Fermentation medium

The substrate used in all the experiments was a complex natural medium made up of a young wine from the Jerez production area, with the following constituents: ethanol: 70–90 g/l; total acidity: 15-20 g of tartaric acid/l; sugars: 1–2 g/l; higher alcohols; 0.5–1.0 g/l; volatile esters: 1–5 mg/l; pH: 2.9–3.1; sulphur dioxide: 60–70 mg/l. This medium was sterilized for 20 minutes at 120 °C, and the pH later fixed at 4 with KOH 1M, to ensure the most suitable conditions for microorganism growth.

2.3 Assay methods

The acetic fermentation experiments were monitored by taking samples at regular time intervals, and performing the following determinations: concentration of ethanol, by gas chromatography [2] and concentration of acetic acid, by potentiometric measurement [3]; pH, concentration of dissolved oxygen and temperature of the fermentation medium was continuously recorded by the appropriate equipment.

2.4 Inoculation

In all the experiments, the fermenter was inoculated by adding, a volume equivalent to 10% of the total capacity of previously prepared inoculum, to the initial sterile medium. This inoculum was made up of a medium with similar characteristics to that of the fermentation, showing a high rate of growth of *Acetobacter aceti*; preparation was made by parallel acetic fermentation in incubation chambers. Before each inoculation, the stability of the operating variables and the correct operation of the control devices were checked.

2.5 Experimental design

Schematic diagrams of the three sets of equipment used in the experimentation are shown in Figs. 1, 2 and 3. The simplest of the three is the open system (Fig. 1); this one consists of an automatic, thermostatically controlled fermenter equipped with both mechanical agitation and aeration completely open to the atmosphere; automatic control is assumed by the PID computer system.

The semiclosed fermentation system (Fig. 2) is similar to the previous one but adapted by adding two columns of 5 cm ID and 100 cm height, each with 1 cm Raschig rings and by filling them up to a height of 70 cm. The gas outflow from the fermenter is passed through the first column (the absorption column) which contains water; this water is then recirculated through the second column (the desorption column), through which the clean air prior to being introduced into the fermenter flows. Water is

Received: 29 April 1997

L.E. Romero, D. Cantero
Biological and Enzymatic Reactors Research Group,
Department of Chemical Engineering,
Food Technology and Environmental Technologies,
Faculty of Sciences, University of Cadiz,
11510. Puerto Real Cadiz, Spain
e-mail: luisenrique.romero@uca.es

Correspondence to: L.E. Romero

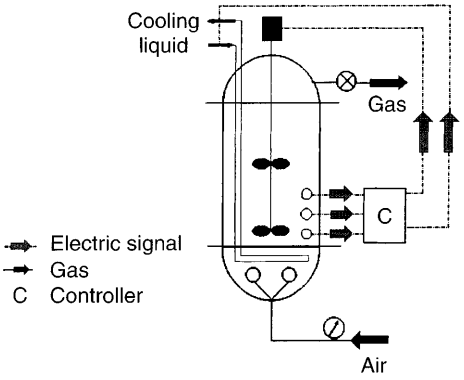

Fig. 1. Open system schematic diagram

recirculated by means of a peristaltic pump. At the same time, an electronic gauge controls the level of water in the columns. The third type of fermentation equipment used (Fig. 3) is the closed system, consisting of a fermenter which operates in a closed gas circuit, thus, preventing the leakage of volatile compounds into the gas outflow. Discrete quantities of oxygen are injected into the recirculation gas flowing into the fermenter, to compensate for the consumption due to the biomass. The system is fitted with a dissolved oxygen electrode and a controller for the number of injections needed to maintain dissolved oxygen at the required level, this one operating within a tolerance of ±10% of the set level.

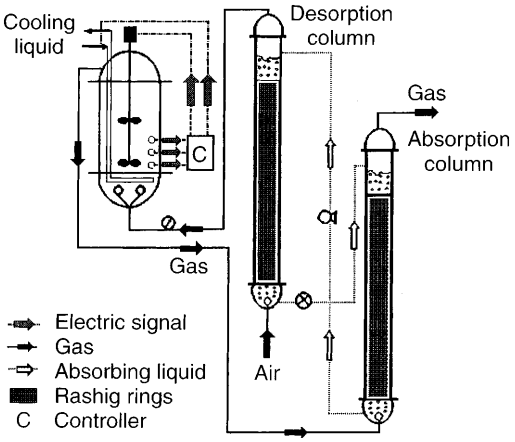

Fig. 2. Semiclosed system schematic diagram

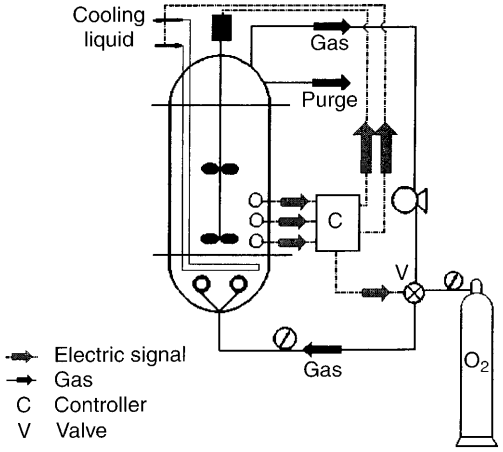

Fig. 3. Closed system schematic diagram

3
Experimental results and discussion

Results obtained from global materials balances may be discussed by studying ethanol evaporation losses, fermentative yields and yield losses evaluation; as well as the acetification rates for the equipment employed. Detail of the equipment are given in Table 1.

3.1
Ethanol evaporation losses

Results obtained from global material balances may be discussed by studying ethanol consumption, acetic acid production, real ethanol losses and theoretical ethanol losses (Fig. 4).

As the equipment used proves, the overall ethanol consumption is greater than produced acetic acid. The remaining ethanol constitutes evaporation losses. Figure 4 shows both the real ethanol evaporation losses deduced from the material balance and the theoretical evaporation losses calculated by means of a modified UNIFAC method developed within our Biological and Enzymatic Reactors Research Group.

In the open systems it may be checked that real evaporation losses at three studied scales are very similar to the theoretical UNIFAC predictions [4]. When either semiclosed or closed systems are employed, real data and UNIFAC data have been found to disagree. This is due to the fact that only temperature and molecular data are

Table 1. Summary of the experiment conducted. The concentration ranges of ethanol and acetic acid indicated provide the values at the beginning and end of the experiment

Scale	System	Temp. (°C)	Air (vvm)	Ethanol (E_0–E_f) (g/l)	Acetic acid (A_0–A_f) (g/l)
Laboratory (5 l)	Open (L1)	26 °C	0.2	80–25	0.4–33
	Semiclosed (L2)	26 °C	0.2	80–25	0.4–33
	Open (L3)	26 °C	0.05	80–0	1.5–58
	Semiclosed (L4)	26 °C	0.05	80–0	1.5–58
Pilot plant (1,000 l)	Open (Pp1)	30 °C	0.2	48–15	40–74
	Closed (Pp2)	28 °C	0.2	47–22	40–70
Industrial (10,000 l)	Open (I1)	20 °C	0.0032	50–20	40–80
	Open (I2)	25 °C	0.0032	50–20	40–80

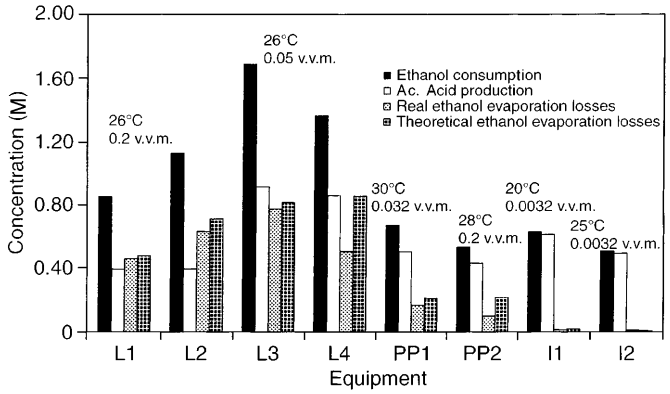

Fig. 4. Data of ethanol consumption, acetic acid production, real ethanol losses and theoretical ethanol losses for the equipment used

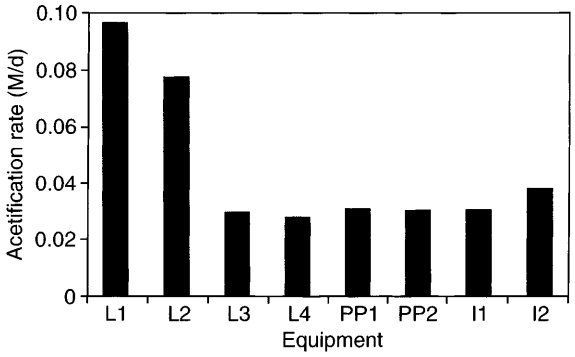

Fig. 6. Data of acetification rate for the equipment used

reflected in the UNIFAC method, but not the characteristics of the physical system such as volatile recuperation equipment.

If a comparison among open, semiclosed and closed systems is established, it is possible to conclude that the closed system [5] is more effective than the others even at unfavourable conditions (pilot plant, high aeration: 0.2 v.v.m. and high temperature: 28 °C). In addition, semiclosed systems appear to be useful at laboratory scale, at low temperature: 26 °C and at low aeration rate: 0.05 v.v.m.

3.2
Fermentative yields and yield losses

Fermentative yield and yield losses can be evaluated if both the reaction stoichiometry (1:1) and the portion of ethanol transformed into acetic acid are taken into consideration and, likewise with it, if acetic acid evaporation is understood as negligible to material balance. Moreover, the sum of these factors must be 100%.

Figure 5 puts forward these parameters, being an increment in fermentative yields when operation scale is

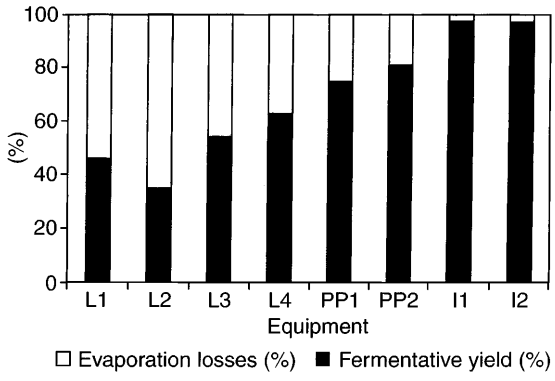

Fig. 5. Data of evaporation losses and fermentative yields for the equipment used

increased the typical trend observed. This is mainly due to the low aeration rates employed at large operation scales. It must be emphasized that for the case of pilot plant semiclosed systems, such operation at high aeration rates: 0.2 v.v.m. and high temperatures: 28 °C makes a fermentative yield of 81% possible.

3.3
Acetification rate

Acetification rate constitutes an essential factor in the production of vinegars in industry and, in accordance with this, this paper evaluates acetification rates considering the quantities of acetic acid produced per day (M/d).

Results gathered in Fig. 6 show that, except for the case of 0.2 v.v.m. laboratory experiments, all experiments developed at three operation scales present similar acetification rate values (between 0.029 and 0.038 M/d).

4
Conclusions

Results obtained clearly show that, in order to predict the different physical systems accurately, the UNIFAC method calls for modifications (Gómez, 1994). The closed system at unfavourable operation conditions is the most effective one concerning the avoidance of significative ethanol losses. Semiclosed systems appear to be useful at laboratory scale, low temperature: 26 °C and low aeration rate: 0.05 v.v.m.

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