



# Dynamic optimisation of the aroma production in brewing fermentation

Ioan-Cristian Trelea, Mariana Titica, Georges Corrieu

## ► To cite this version:

Ioan-Cristian Trelea, Mariana Titica, Georges Corrieu. Dynamic optimisation of the aroma production in brewing fermentation. *Journal of Process Control*, 2004, 14 (1), pp.1-16. 10.1016/S0959-1524(03)00007-6 . hal-01537106

**HAL Id: hal-01537106**

**<https://agroparistech.hal.science/hal-01537106>**

Submitted on 16 Jun 2017

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

# Dynamic optimisation of the aroma production in brewing fermentation

Ioan Cristian TRELEA<sup>1</sup>✉, Mariana TITICA<sup>2</sup>, Georges CORRIEU<sup>1</sup>

<sup>1</sup>UMR Génie et Microbiologie des Procédés Alimentaires, 78850 Thiverval-Grignon, France

E-mail: trelea@grignon.inra.fr

<sup>2</sup>CESAME, UCL Bâtiment Euler, Avenue Georges Lemaître 4-6, B-1348, Louvain la Neuve, Belgium

## Abstract

Several key compounds for the final beer flavour (higher alcohols, esters, vicinal diketones) are produced during the alcoholic fermentation phase. The paper demonstrates the possibility of obtaining various desired final aroma profiles and reducing the total process time using dynamic optimisation of three control variables: temperature, top pressure and initial yeast concentration in the fermentation tank. The optimisation is based on a sequential quadratic programming algorithm, on a dynamic model of the alcoholic fermentation and on an aroma production model. The robustness of the optimal control profile with respect to model uncertainty is discussed.

## Keywords

Beer, aroma compounds, constrained non-linear optimal control, model uncertainty

## Introduction

The alcoholic fermentation is an important stage in the beer production process. During this phase, fermentable sugars present in the brewing wort are transformed to ethanol and several aroma compounds important for the final beer flavour are produced. The contribution of the alcoholic fermentation phase to the final beer flavour depends on the wort composition, on the yeast strain and on the operating conditions. Industrial operating conditions for most existing beer brands were determined empirically and are confidential. The aim of this work is to demonstrate the possibility of optimising the fermentation temperature profile, the top pressure profile and the initial yeast concentration based on organoleptic and economic criteria. The organoleptic criterion takes into account target concentrations of the following compounds believed to be important for the final beer flavour [1]: two higher alcohols (isoamyl alcohol and phenyl ethanol), three esters (ethyl acetate, ethyl hexanoate and isoamyl acetate) and one vicinal diketone (diacetyl). The economic criterion is based on process time minimisation.

Previous work on the optimisation of the brewing process was concerned with the computation of the temperature profile alone. Gee and Ramirez [2] used Pontryagin's minimum principle for the temperature profile optimisation which insured a minimum fermentation time and a maximum ethanol production. Not surprisingly, they found that the fermentation temperature take its maximum possible value at any time. This purely economic optimum could be anticipated from the existing knowledge of the alcoholic fermentation process but is not used in practice because of its undesired effect on the aroma composition. Andres-Toro *et al.* [3] used an optimisation technique based on genetic algorithms and introduced the idea of aroma targets (for ethyl acetate and diacetyl) in addition to fermentation time minimisation. They obtained a non-trivial temperature profile where high temperatures were still favoured, however.

The present contribution is an extension and a generalisation of the results of Titica *et al.* [4]. Dynamic optimisation is used to compute flexible control profiles instead of two-stage controls considered previously. Limitations of the process model that made it unsuitable for simulation in time-varying operating conditions were removed. The possibility of reducing the fermentation time substantially without altering the final aroma profile was demonstrated for a beer previously obtained in constant operating conditions. The feasibility (using a single yeast strain and wort composition) of four commercial beer aroma profiles is discussed and the limitations of the aroma control using the three considered operating conditions alone are pointed out. Finally, the robustness of the final aroma concentrations and of the optimal operating conditions with respect to unavoidable modelling errors is analysed.

## Materials and methods

### *Experimental*

The alcoholic fermentation and the aroma production models were built and validated on data coming from nine laboratory scale experiments. Experiments were carried out in a 15 L, 0.5 m high, stainless steel bioreactor (LSL Biolafitte, France) filled with 12 L of wort, under gentle agitation (100 rpm). Preliminary experiments showed that mechanical agitation was needed to compensate the absence of the natural agitation that occurs in large scale brewing (10 m high tanks or higher) due to CO<sub>2</sub> release. The lager wort and the industrial yeast strain, *Saccharomyces cerevisiae* var. *uvarum*, were provided by the Institut Français de Brasserie et Malterie (IFBM, France). The conditions of the experimental runs R1 to R4 and R6 to R9 were selected according to a 2<sup>3</sup> experimental design. The three factors were the fermentation temperature (10 and 16°C), the top pressure (50 and 800 mbar) and the initial yeast concentration (5 and 20 million cells/mL). The run R5 was performed in intermediate operating conditions (13°C, 450 mbar and 10 million cells/mL).

The concentrations of ethanol, isoamyl alcohol, phenyl ethanol, ethyl acetate, ethyl hexanoate and isoamyl acetate were determined by gas chromatography [5] coupled with mass spectrometry in the case of diacetyl [6]. The evolved carbon dioxide was recorded with a gas meter (Schlumberger, France), with a resolution better than 0.5% of the total amount of gas produced in each experiment. The measurements describing the alcoholic fermentation (ethanol, wort density, CO<sub>2</sub> production, refractive index and fermentable sugar concentration) were reconciled using well-established stoichiometric relationships [7]. The yeast cell concentration was determined with a particle counter (Coulter Z1, Coultronics, France). Three counts were performed at 3 and 3.5 µm and the logarithmic average of the six counts was taken.

### *Alcoholic fermentation model*

The optimisation of the operating conditions with respect to the specified criterion is an iterative process. In each iteration candidate solutions are tested using a process model. The process model used in this work consisted in an alcoholic fermentation model coupled with an aroma production model.

The alcoholic fermentation model is a modified version of a previously reported one [8]. The main modification concerns the description of the CO<sub>2</sub> transfer between the wort and the headspace. In [8] it was assumed that CO<sub>2</sub> was dissolved in the wort until saturation and released afterwards. This assumption was reasonable for constant operating conditions, but seems questionable in the dynamic optimisation context when variable temperature and

top pressure profiles are usually obtained. The stated assumption was replaced by a rigorous mass balance of the CO<sub>2</sub> in the wort and in the headspace and a mass balance of the air in the headspace. Solubility of the air (N<sub>2</sub> and O<sub>2</sub>) in the wort was found negligible as far as the mass balance was concerned.

The alcoholic fermentation can be described equivalently by the fermentable sugar consumption, ethanol production, wort density decrease or CO<sub>2</sub> production [7]. The CO<sub>2</sub> was selected because it is the most convenient variable to be measured on-line [9][10]. The model was constructed by analogy with classical microbial growth kinetics with substrate limitation and product inhibition:

$$\frac{dC_p(t)}{dt} = \nu(\theta(t), C_d(t)) \cdot \frac{S(t)}{K_s + S(t)} \cdot \frac{1}{1 + (E(t)/K_E)^2} \cdot (C_p(t) + K_X X_0) \quad (1)$$

$$C_p(0) = 0 \quad (2)$$

$$E(t) = Y_{E/C} C_p(t) \quad (3)$$

$$S(t) = S(0) - Y_{S/C} C_p(t) \quad (4)$$

The rate of the alcoholic fermentation was described by the rate of CO<sub>2</sub> production  $dC_p/dt$ . Simultaneously, ethanol ( $E$ ) is produced and fermentable sugars ( $S$ ) are consumed, with constant yields ( $Y_{E/C}$  and  $Y_{S/C}$  respectively). The initial fermentation rate, when  $C_p = 0$ , is proportional to the initial yeast concentration  $X_0$ .

The “specific” fermentation rate  $\nu$  was expressed as [11]:

$$\nu(\theta, C_d) = K_\nu \exp(K_{\nu\theta}(\theta - \theta_{typ}) - K_{\nu C}(C_d - C_{d,typ})) \quad (5)$$

For small temperature variations ( $\pm 3K$ ) compared to the typical absolute fermentation temperature (286K), the given relationship is a close approximation of the Arrhenius law. A similar dependence was assumed for the dissolved CO<sub>2</sub> ( $C_d$ ). The “typical” temperature and dissolved CO<sub>2</sub> values are those of the central point of the experimental design (run R5) and correspond to usual values for lager beer making. For modelling purposes, it was assumed that the produced CO<sub>2</sub> ( $C_p$ ) is transferred into the solution, because of the large contact area between the yeast cells and the wort. The mass transfer between the solution ( $C_d$ ) and the headspace ( $C_h$ ), depends on the partial CO<sub>2</sub> pressure in the headspace ( $p_c$ ) and on the tank geometry and agitation through a kinetic constant ( $\tau$ ):

$$\frac{dC_d(t)}{dt} = \frac{dC_p(t)}{dt} - \frac{1}{\tau} [C_d(t) - C_{sat}(\theta(t), p_c(t))] \quad (6)$$

$$C_d(0) = 0 \quad (7)$$

$C_{sat}$  is the equilibrium CO<sub>2</sub> concentration in the wort at a given temperature ( $\theta$ ) and at a given partial headspace pressure ( $p_c$ ). It was determined from tables provided by Institut Français de Brasserie et Malterie and approximated by the following empirical formula:

$$C_{sat}(\theta, p_c) = K_C p_c \frac{\theta + \theta_{abs}}{\theta_{abs}} e^{-K_{C\theta}\theta} \quad (8)$$

The evolution of the CO<sub>2</sub> concentration in the headspace ( $C_h$ ) is given by :

$$\frac{dC_h(t)}{dt} = \frac{\gamma}{\tau} [C_d(t) - C_{sat}(\theta(t), p_c(t))] - \frac{\gamma C_h(t)}{C_h(t) + C_a(t)} \Phi_g(t) \quad (9)$$

$$C_h(0) = 0 \quad (10)$$

where  $\gamma$  is the ratio of the wort volume to the headspace volume,  $C_a$  is the air concentration and  $\Phi_g$  the total gas outflow rate ( $\text{CO}_2 + \text{air}$ ). The  $\text{CO}_2$  outflow rate is proportional to the  $\text{CO}_2$  mass fraction in the headspace. The air concentration in the headspace diminishes with a rate proportional to the air mass fraction:

$$\frac{dC_a(t)}{dt} = -\frac{\gamma C_a(t)}{C_h(t) + C_a(t)} \Phi_g(t) \quad (11)$$

$$C_a(0) = \frac{p_{atm} M_a}{R(\theta(0) + \theta_{abs})} \quad (12)$$

The partial  $\text{CO}_2$  ( $p_c$ ) and air ( $p_a$ ) pressures were calculated using the perfect gas law. The technologically important variable is the total pressure ( $p$ ):

$$p_c = \frac{C_h}{M_c} R(\theta + \theta_{abs}) \quad (13)$$

$$p_a = \frac{C_a}{M_a} R(\theta + \theta_{abs}) \quad (14)$$

$$p = p_c + p_a \quad (15)$$

where  $R$  is the perfect gas constant,  $M_c$  is the  $\text{CO}_2$  molar mass and  $M_a$  is the equivalent molar mass of the air.

The parameter values of the alcoholic fermentation model are reported in Table 1. The differences with previously reported values [8][12] are due to the way in which the dissolved carbon dioxide was estimated.

## Aroma production model

A mathematical model for some aroma compounds produced during the alcoholic fermentation, and considered important for the final beer flavour, was reported previously [11][12]. The same model was used for optimal control, with the following modifications: (i) In the new version, the operating conditions affect the yields versus  $\text{CO}_2$  in a multiplicative, rather than additive way. (ii) The two-phase yield for the ester production was replaced by a single-phase, monotonically increasing yield. (iii) The model parameters were identified based on the whole pool of experiments, rather than separately for each experiment. This allowed a statistical analysis of the reliability of the parameter estimates. (iv) Parameters not significantly different from zero were removed from the model, i.e. set to exactly zero.

**Higher alcohols.** Two higher alcohols were considered in this work, based on their organoleptic thresholds in beer [1]: the isoamyl alcohol (*IAL*) and the phenyl ethanol (*PHL*). Their production rate was related to the alcoholic fermentation rate, with yields ( $Y_{i/C}$ ) depending on the operating conditions: temperature ( $\theta$ ), dissolved  $\text{CO}_2$  ( $C_d$ ) and initial yeast concentration in the wort ( $X_0$ ):

$$\frac{dA_i(t)}{dt} = Y_{i/C}(\theta(t), C_d(t), X_0) \frac{dC_p(t)}{dt} \quad i \in \{IAL, PHL\} \quad (16)$$

$$A_i(0) = 0 \quad (17)$$

$$Y_{i/C}(\theta, C_d, X_0) = W_{i,1} \exp[W_{i,2}(\theta - \theta_{typ}) + W_{i,3}(C_d - C_{d,typ}) + W_{i,4}(X_0 - X_{0,typ}) + W_{i,5}(\theta - \theta_{typ})(C_d - C_{d,typ}) + W_{i,6}(\theta - \theta_{typ})(X_0 - X_{0,typ}) + W_{i,7}(C_d - C_{d,typ})(X_0 - X_{0,typ}) + W_{i,8}(\theta - \theta_{typ})(C_d - C_{d,typ})(X_0 - X_{0,typ})] \quad (18)$$

**Esters.** The three selected esters were the ethyl acetate (*ETA*), the ethyl hexanoate (*ETX*) and the isoamyl acetate (*IAA*). Their production rate was related to the alcoholic fermentation rate, as in the case of higher alcohols, but the yield increased with fermentation progress:

$$\frac{dA_i(t)}{dt} = Y_{i/C}(\theta(t), C_d(t), X_0) C_p(t) \frac{dC_p(t)}{dt} \quad i \in \{ETA, ETX, IAA\} \quad (19)$$

$$A_i(0) = 0 \quad (20)$$

The effect of the operating conditions on the yields ( $Y_{i/C}(\theta, p, X_0)$ ) had the same mathematical expression as in the case of the higher alcohols (Equation 18).

**Vicinal diketones.** The two important diketones in beer are the diacetyl and the pentanedione. In the considered experiments, the pentanedione concentration was always lower than the organoleptic threshold [12] and hence only the diacetyl model was considered for the optimal control. A detailed analysis of this model was given previously [12]. The diacetyl was simultaneously produced and reduced during the alcoholic fermentation:

$$\frac{dA_{DIA}(t)}{dt} = Y_{DIA/C}(t) \frac{dC_p(t)}{dt} - W_{DIA,1} \exp[W_{DIA,2}(\theta - \theta_{typ})] \quad (21)$$

$$A_{DIA}(0) = 0 \quad (22)$$

The production yield ( $Y_{DIA/C}$ ) asymptotically decreased to zero. An empirical relationship describing this behaviour was established:

$$\frac{dY_{DIA/C}(t)}{dt} = -W_{DIA,3} \frac{dC_p(t)}{dt} Y_{DIA/C}(t) \quad (23)$$

$$Y_{DIA/C}(0) = W_{DIA,4} \quad (24)$$

**Statistical significance of the model coefficients.** The significance test was based on the so-called Mallows statistic, which establishes a trade-off between the fit to experimental data and the model complexity, i.e. number of adjustable parameters [14]. This criterion says that a parameter should be retained in the model only if it reduces the residual variance sufficiently compared to the estimated measurement variance. For each aroma compound ( $i$ ), the Mallows statistic ( $I_i$ ) was minimised over the various combinations of  $n_i$  nonzero parameters:

$$I_i = \frac{1}{\sigma_i^2} \sum_k (A_i(t_k) - a_i(t_k))^2 + 2n_i \quad i \in \{ETA, ETX, IAA, IAL, PHL, DIA\} \quad (25)$$

Here  $a_i(t_k)$  are the measured concentrations and  $A_i(t_k)$  are the concentrations predicted by the model with  $n_i$  nonzero parameters. The measurement variance  $\sigma_i^2$  was estimated as the residual variance of the complete model, with all nonzero parameters.

The numeric values of the coefficients appearing in the aroma production model, together with their confidence limits, are reported in the Table 2.

## Optimal control problem

The optimal control of the fermentation process consisted in the selection of the operating conditions that minimised an overall criterion  $Q$ , which reflected the desired product quality and plant operation mode. Additional requirements were introduced via a set of constraints that admissible solutions must satisfy. The operating conditions considered in this work are the wort temperature  $\theta$ , controlled by the cooling rate  $\Phi_\theta$ , the

top pressure  $p$ , controlled by the gas outflow rate  $\Phi_g$ , the initial yeast concentration in the wort  $X_0$ , and the total fermentation time  $t_f$ . The optimal control problem is stated as:

$$\{t_f, \Phi_\theta(t)_{t \in [0, t_f]}, \Phi_g(t)_{t \in [0, t_f]}, X_0\} = \arg \min Q \quad (26)$$

The “arg min” symbol means the “the values which minimise”. The optimisation criterion and the constraints are detailed below.

## Optimisation criterion

The considered problem is a multiobjective optimisation: approaching the five aroma targets as close as possible, reducing the final diacetyl concentration and the total processing time, as well as smoothing out the temperature and the top pressure profiles. The overall optimisation criterion is a weighted sum of the nine partial criteria. The weights specify the desired trade-offs between possibly conflicting objectives. In order to simplify the selection of the weights, all partial criteria were scaled by physically meaningful quantities. Thus, the partial criteria and the associated weights are dimensionless quantities of order of unity.

**Approaching the aroma targets.** The final concentrations of the five aroma compounds, believed to be important for the final beer flavour, have to be as close as possible to their respective targets:

$$Q_i = W_i \frac{(A_i(t_f) - A_{i, \text{trg}})^2}{A_{i, \text{tol}}^2}, \quad i \in \{ETA, ETX, IAA, IAL, PHL\} \quad (27)$$

Here  $Q_i$  is the partial criterion associated to the aroma compound  $i$ ,  $W_i$  is the associated weight,  $A_i(t_f)$  is the aroma concentration at the end of the fermentation,  $A_{i, \text{trg}}$  is the target concentration, and  $A_{i, \text{tol}}$  is the accepted tolerance. Since there is no point in trying to approach the target closer than either the model accuracy or the perceived difference, the tolerance was selected as:

$$A_{i, \text{tol}}^2 = \sigma_i^2 + A_{i, \text{org}}^2 \quad (28)$$

where  $\sigma_i$  is the model prediction accuracy (Table 2) and  $A_{i, \text{org}}$  is the organoleptically significant difference in the concentration of the compound  $i$  taken as 10% of the organoleptic threshold of the considered compound in beer [1]. An alternative way of expressing the tolerance would be using the largest of the two values  $\sigma_i$  and  $A_{i, \text{org}}$ . The tolerance provides a natural scaling of the associated optimisation criterion.

**Minimising the diacetyl concentration.** Diacetyl is an undesired compound in the finished beer. No target was defined for this compound because the lowest possible value is desired in practice (ideally zero). Minimisation of the diacetyl concentration at the end of the main fermentation is an economically important goal, since it may result in the reduction of the cost of the subsequent processing (dedicated “diacetyl rest” and/or lagering phases). Mathematically, this goal was expressed as:

$$Q_{DIA} = W_{DIA} \frac{A_{DIA}(t_f)}{A_{DIA, \text{typ}}} \quad (29)$$

where  $A_{DIA}(t_f)$  is the diacetyl concentration at the end of the alcoholic fermentation and  $A_{DIA, \text{typ}}$  is a typical value of the final diacetyl concentration, used as a scaling constant.

**Minimising the fermentation time.** The reduction of the processing time is desirable from an economic point of view. No target value was given for the fermentation time. The optimisation algorithm was free to select the shortest possible fermentation time compatible with the other requirements:

$$Q_t = W_t \frac{t_f}{t_{typ}} \quad (30)$$

where  $t_f$  is the final fermentation time and  $t_{typ}$  is a typical value for the final time, used for scaling.

**Smoothing out the temperature and top pressure profiles.** Technologically, a smooth plant operation is usually desired. Smooth temperature profile requires a smaller peak cooling power. A smooth top pressure profile avoids discontinuities in the CO<sub>2</sub> outflow, facilitating its recovery. Numerically, penalising zigzagging profiles favours robust convergence to a global optimum. The smoothing objective was expressed as a penalty of the curvature (second time derivative) of the temperature and top pressure profiles. For numerical computations, the profiles were sampled at equally spaced time points. The scaling factors were chosen to make the curvature criteria invariant with respect to the total fermentation time and to the range of the variables:

$$Q_\theta = W_\theta \frac{1}{n_t - 2} \sum_{k=2}^{n_t-1} \left( \frac{\theta_{k+1} - 2\theta_k + \theta_{k-1}}{\theta_{\max} - \theta_{\min}} \right)^2 \quad (31)$$

$$Q_p = W_p \frac{1}{n_t - 2} \sum_{k=2}^{n_t-1} \left( \frac{p_{k+1} - 2p_k + p_{k-1}}{p_{\max} - p_{atm}} \right)^2 \quad (32)$$

Here  $n_t$  is the total number of sampling points,  $\theta_k$  and  $p_k$  are wort temperature and total headspace pressure at the sampling point number  $k$ .

**Overall optimisation criterion.** The overall optimisation criterion is the sum of the nine partial criteria. It reflects the best possible trade-off between generally conflicting optimisation goals:

$$Q = \sum_{i \in \{ETA, ETX, IAA, IAL, PHL, DIA, t, \theta, p\}} Q_i \quad (33)$$

## Technological constraints

The operating conditions (temperature, top pressure and initial yeast concentration) were limited to the model validity range, which is the range of the operating conditions of the experimental design that provided data for model identification and validation. Additionally, the initial top pressure must equal the atmospheric pressure  $p_{atm}$ , since it can be increased only by the CO<sub>2</sub> produced by fermentation:

$$\theta(t) \in [\theta_{\min}, \theta_{\max}], \quad t \in [0, t_f] \quad (34)$$

$$p(t) \in [p_{atm}, p_{\max}], \quad t \in [0, t_f] \quad (35)$$

$$p(0) = p_{atm} \quad (36)$$

$$X_0 \in [X_{0\min}, X_{0\max}] \quad (37)$$

On most fermentation tanks, the temperature is controlled using a cooling jacket. The associated control variable, namely the cooling rate  $\Phi_\theta$ , can only be positive since no heating device usually exists. Its amplitude is limited by the maximum available cooling power or the maximum possible heat transfer rate:

$$\Phi_\theta(t) \in [0, \Phi_{\theta\max}], \quad t \in [0, t_f] \quad (38)$$

The top pressure is generally controlled by a valve. The corresponding control variable is the outflow gas rate  $\Phi_g$ , which is also positive and limited by the valve diameter:

$$\Phi_g(t) \in [0, \Phi_{g\max}], \quad t \in [0, t_f] \quad (39)$$



The alcoholic fermentation is considered finished when the residual fermentable sugar concentration reaches a specified value, corresponding to the so-called wort “attenuation” limit:

$$S(t_f) = S_{res} \quad (40)$$

This constraint defines the fermentation end time  $t_f$ . The residual concentration  $S_{res}$  is generally known for a given wort and yeast strain.

Numerical values of the optimal control problem parameters are reported in Table 3. The target aroma concentrations considered in this work are given in Table 4. The aroma profiles of the five sorts of beer listed in Table 4 are discussed in the “Results and discussion” section below.

### **Dynamic optimisation algorithm**

The considered optimal control problem is a dynamic one, since the unknown control variables are functions of time. The optimisation criterion contains linear and quadratic terms in the state variables. The constraints are linear in the state and control variables, except the top pressure constraint, which involves products of state variables ( $C_d(t)\theta(t)$  and  $C_a(t)\theta(t)$ ). However, the main nonlinearity comes from the dynamic model of the fermentation process.

Taking into account these characteristics of the control problem, a variant of the sequential quadratic programming (SQP) algorithm was used for solving it [15], coupled with a collocation (time discretisation) technique [16] and a safeguarded line search based on a “confidence region” method [17]. The main steps of the algorithm were:

**Step 0. Initialisation.** A non-optimal but admissible (with respect to the constraints) solution was determined before entering the main algorithm. The initial fermentable sugar concentration is determined from the desired ethanol concentration in the finished beer ( $E_{trg}$ ):

$$S(0) = S_{res} + \frac{Y_{S/C}}{Y_{E/C}} E_{trg} \quad (41)$$

Random but admissible set-point profiles were generated for temperature and top pressure. A random admissible value was generated for the initial yeast concentration. The model equations were solved using simple proportional controllers to follow the set-points:

$$\Phi_\theta(t) = K_{p\theta}(\theta(t) - \theta_{set}(t)) \quad (42)$$

$$\Phi_g(t) = K_{pg}(p(t) - p_{set}(t)) \quad (43)$$

The fermentation end time ( $t_f$ ) was determined as the moment when the fermentable sugar concentration fell below the specified limit ( $S_{res}$ ). The resulting control variables (cooling rate and gas outflow rate) and the resulting fermentation end time were used as starting points for the optimisation.

**Step 1. Discretisation of the dynamic problem.** Let  $x$  be the vector of the  $n_x = 12$  state variables and  $u$  the vector of the  $n_u = 2$  control variables:

$$x = [C_p \quad A_{ETA} \quad A_{ETX} \quad A_{IAA} \quad A_{LAI} \quad A_{PHL} \quad A_{DIA} \quad Y_{DIA/C} \quad \theta \quad C_d \quad C_h \quad C_a]^T \quad (44)$$

$$u = [\Phi_\theta \quad \Phi_g]^T \quad (45)$$

The state and control variables were sampled at a finite number ( $n_t = 49$ ) of time points [16]. This reduced an infinite-dimensional problem to a finite-dimensional one, which could be handled by a numerical computer. The optimisation criterion, the constraints and the dynamic model equations were discretised on the same time grid.

**Step 2. Construction of a local optimisation sub-problem.** The SQP method requires local approximation of the non-linear optimisation problem by a quadratic one with linear constraints, using limited Taylor series expansion. In the standard SQP method the quadratic approximation is made to the Lagrangean function. Its Hessian matrix includes curvature information on both the objective function and the constraints [15]. In this application the Hessian matrix of the objective function alone was used. The objective function being truly quadratic, its second order Taylor series expansion was exact. The discretised dynamic model equations were treated as additional constraints [15]. The limited Taylor series expansion being valid only locally, a limited search region around the current solution was established, using a set of “box” (min-max) constraints [17].

**Step 3. Solution of the local optimisation sub-problem.** Commercial software, based on an “active constraint set” method was used to solve the quadratic optimisation problems with linear constraints [18]. This software did not exploit the special structure of the collocation equations and was unable to solve a problem of moderate size ( $2 + n_t(n_u + n_x) = 688$ ). The number of variables was reduced to  $2 + n_t n_u + n_x = 112$  by solving the linearized collocation equations explicitly. The reduction step involved a structurally well conditioned and sparse matrix, making the computation both fast and accurate.

**Step 4. Convergence test.** The non-linear state equations were solved using the determined control variables. The value of the optimisation criterion was computed and the satisfaction of the constraints was checked. If the control variables, the state variables and the optimisation criterion were modified by less than a pre-specified amount, and if all constraints were satisfied, then calculations were halted. Else a new iteration was made, starting with step 1.

The algorithm was always run several (~10) times with various random initialisation in step 0. Robust convergence to the same optimum was observed. Occasionally, local optima in form of zigzagging control profiles were encountered. The control smoothing terms (Equations 31 and 32) were found very useful in avoiding these local optima. A detailed mathematical description of the algorithm is available from the authors on request.

## Results and discussion

### ***Alcoholic fermentation model validation***

The main measured and simulated variables in the alcoholic fermentation model are presented in Figure 1 for the run R5, not used for the identification of the model coefficients. The carbon dioxide evolution rate is predicted reasonably well. The evolution rate is zero for the first 24 hours: during this time period the produced  $\text{CO}_2$  is partly dissolved in the wort and partly accumulated in the headspace increasing the top pressure. The top pressure increases until the set-point is reached and then remains constant being controlled by the outflow valve. The partial air pressure remains constant until the outflow valve is opened and then decreases, the air being evacuated from the tank along with the  $\text{CO}_2$ . The partial  $\text{CO}_2$  pressure equals the total one when the air was evacuated completely. The ethanol production, proportional to the cumulated  $\text{CO}_2$  production, is predicted almost perfectly in this run. It appears from the model simulation that the dissolved  $\text{CO}_2$  exceeds its saturation

limit by about 50% during the most active fermentation period (between 50 and 100 hours). Dissolved CO<sub>2</sub> could not be measured directly in the considered experiments but this value is consistent with data found in the literature [19] and strongly depends on the tank geometry through the parameter  $\tau$  in Equations 6 and 9. Taking into account the CO<sub>2</sub> super-saturation is the main evolution of the model considered in this paper compared to previous work [8]. It has a moderate impact on the aroma concentrations predicted by the model, particularly in time-varying operating conditions when the top pressure is decreased quickly.

### ***Aroma production model validation***

Measured and simulated concentrations of the aroma compound considered in this work are reported in Figure 2 for the experimental run R5, not used for model derivation. The concentrations of five out of the six compounds are predicted reasonably well taking into account the scatter of the experimental data. The concentration of isoamyl acetate is significantly overestimated, however. This was already the case with previous models [11]. The measured final isoamyl acetate concentration in run R5 is one of the lowest among all runs, while the operating conditions have intermediate values. This is probably due to anomalous measurements for this particular experiment: in later runs, carried out in conditions similar to run R5, final isoamyl acetate concentrations close to 1.5 mg/L were obtained. The diacetyl is simultaneously produced and reduced during the fermentation run. The production rate decreases gradually and approaches zero at 50 hours. The diacetyl concentration reaches a maximum when the reduction rate equals the production rate and declines exponentially when the production rate becomes negligible.

### ***Producing an existing beer at a lower cost***

This section illustrates the reduction of the production cost for an existing sort of beer without altering its aroma profile. By “aroma profile” is meant here the set of final concentrations of the two higher alcohols and three esters considered in this work. The target aroma concentrations were those measured at the end of the run R5, that is at the central point of the experimental design. The simulated experiment corresponding to run R5 is shown in Figure 3(A). With constant temperature (13°C), constant top pressure (450 mbar above the atmospheric pressure) and a typical initial yeast concentration (10 million cells/mL) the target aroma profile is reproduced well, illustrating the adequacy of the model. The predicted fermentation time is 121 hours, in agreement with the experimental value (Figures 1 and 2).

The result of applying the optimisation algorithm to the same target aroma profile is illustrated in Figure 3(B). The final predicted aroma profile matches the target equally well but the fermentation time is reduced to 81 hours, that is by 33%. This is achieved by increasing the average fermentation temperature and the initial yeast concentration. The aromatic equilibrium of the final beer is maintained, however, using variable operating conditions (slightly decreasing temperature and uniformly increasing top pressure) by taking advantage of the time-varying fermentation rate and of the time-varying ester yields. The final diacetyl concentration was the same as in run R5 (0.5 mg/L): the highest diacetyl degradation rate due to higher temperature was compensated by a shorter fermentation time.

### ***Producing new beer flavours with the same yeast strain and wort***

The measured aroma concentrations in four existing beer brands (coded B1 to B4, Table 4) were given as targets to the optimal control algorithm. The results for the beers B2 and B3 are shown in Figure 4. Beer B2 has a relatively high concentration of higher alcohols and a low ester concentration, while the opposite is true for beer B3. Figure 4 shows that both aroma profiles can be achieved using the same wort and yeast strain but different operating conditions: Beer B2 needs a high initial yeast concentration, a high fermentation temperature (except for the final phase) and a bell-shaped top pressure profile, while the aroma profile of beer B3 requires roughly the opposite: low initial yeast concentration, V-shaped temperature profile and a low top pressure for most of the fermentation time. Note that beer B2 is produced much faster than beer B3 (91 hours instead of 152) mainly because of the higher average temperature and of the higher initial yeast concentration. This illustrates the fact that the selected weights in the optimisation criterion (Table 3) favour the reproduction of the desired target aroma profile compared to time minimisation, which is only a secondary objective: for beer B3, the fermentation time was increased as much as needed to obtain the desired final aroma composition.

It is expected, however, that not *all* aroma profiles can be reproduced with the given yeast strain and wort, even if the operating conditions are selected optimally in their admissible ranges. Two examples are given in Figure 5 for the aroma targets of beers B1 and B4 (Table 4). The difficulty of achieving the aroma profile B1 comes from the fact a low concentration of ethyl hexanoate can not be obtained simultaneously with a high concentration of isoamyl acetate. The sensitivity of these compounds to the operating conditions (coefficients  $W_{i,2}$  to  $W_{i,8}$ , Table 2) are very similar, meaning that their concentrations can not be manipulated independently. The best possible solution found by the optimisation algorithm (Figure 5(A)) is a compromise where the final concentration of isoamyl acetate is lower than required and that of ethyl hexanoate is higher. The concentrations of ethyl acetate and isoamyl alcohol are reproduced correctly, however, and that of phenyl ethanol is in the tolerance domain. The situation is worse for the aroma profile B4 (Figure 5(B)) because the target concentration of ethyl hexanoate is even lower and the concentration of isoamyl acetate even higher. The only compound for which the target is achieved in this case is the isoamyl alcohol.

### ***Final aroma profile robustness with respect to model uncertainty***

Due to the finite amount of experimental data available for model identification and to unavoidable measurement error the beer fermentation model parameters (Tables 2 and 3) can only be determined with limited accuracy. The uncertainty on the model parameters was expressed as a joint probability distribution and determined numerically (based on the so-called local Fisher information matrix) during the model fitting process [20]. The performance robustness was tested for the B2 beer by applying the pre-computed optimal operating conditions (temperature profile, top pressure profile and initial yeast concentration) to 20 models with parameters drawn at random from their respective probability distributions. The 95% confidence domain of the final aroma concentrations is shown in Figure 6(A). The confidence domain was determined as the range between the 2.5 and the 97.5 percentile of the calculated values. It appears from Figure 6 that the final aroma concentrations stay within their admissible tolerance limits despite model uncertainty. This is consistent with the selection of the tolerance limits that are always larger than the model accuracy (Equation 28).

## **Optimal control strategy robustness with respect to model uncertainty**

The sensitivity of the optimal control profile to the model uncertainty was investigated for the B3 beer by performing the optimal control calculation for 20 models with parameters drawn at random from their respective probability distributions. The 95% confidence domains of the final aroma concentrations and of the associated operating conditions (temperature, top pressure and initial yeast concentration) are shown in Figure 6(B). The confidence domain was determined as the range between the 2.5 and the 97.5 percentile of the calculated values. It appears that similar optimal control policies are obtained consistently despite model parameter variations. A V-shaped temperature profile, a bell-shaped top pressure profile and a low initial yeast concentration seem to be truly characteristic to the considered combination of yeast strain, wort composition and target aroma profile. The exact values of the operating conditions are not critical, however. For example, in a practical implementation,  $\pm 0.5^{\circ}\text{C}$  variations in the wort temperature,  $\pm 200$  mbar variations in the top pressure or  $\pm 0.5$  million cells/mL variations in the initial yeast concentration would not affect the final beer flavour significantly.

## **Conclusion**

A dynamic model of the beer fermentation process, including an alcoholic fermentation model and an aroma production model, was designed and validated. It is based on nine fermentation runs with operating conditions (temperature, top pressure and initial yeast concentration) selected according to a  $2^3$  experimental design. The model was used for the dynamic optimal control of the beer fermentation process. A primary goal was to approach pre-specified final aroma targets (two higher alcohols and three esters simultaneously) as close as possible and a secondary objective was to reduce the fermentation time and the final concentration of an undesired aroma compound (diacetyl).

An optimal time-varying control policy allowed the reduction of the fermentation time of an existing sort of beer, previously produced in constant operating conditions, by 33% while preserving the final aroma concentrations of the considered compounds. The optimal control strategy also allowed the reproduction of aroma profiles of two existing commercial beers (with different higher alcohols / esters ratios) by means of the operating conditions alone, i.e. using the same yeast strain and wort. Two other existing aroma profiles could not be reproduced satisfactorily (with the same yeast strain and wort) because some aroma compounds had similar sensitivities to the considered operating conditions and could not be manipulated independently.

The sensitivity of the final aroma concentrations and of the optimal control policies with respect to modelling errors was explored numerically by generating random sets of model coefficients from their joint probability distributions estimated during the model identification phase. Achievable aroma profiles stayed within the tolerance limits and consistent optimal control profiles were obtained despite model uncertainty.

## **References**

- [1] S. Engan, Organoleptic threshold values of some alcohols and esters in beer, *Journal of the Institute of Brewing* 78 (1972) 33-36.
- [2] A. Gee, W.F. Ramirez, Optimal temperature control for batch beer fermentation, *Biotechnology and Bioengineering* 31 (1988) 224-234.

- [3] B. Andres-Toro, J.M. Giron-Sierra, J.A. Lopez-Orozco, C. Fernandez-Conde, Evolutionary optimisation of an industrial batch fermentation process, ECC'97 European Control Conference, Brussels, Belgium 3 (1997) WE-EG5 615.
- [4] M. Titica, I.C. Trelea, A. Cheruy, optimisation of fermentation operating conditions with a view to control beer aroma profile, CAB-8: 8<sup>th</sup> International Conference on Computer Applications in Biotechnology, June 24-27 (2001) Quebec, Canada
- [5] L.E. Stenroos, K.J. Siebert, M.C. Meilgaard, Gas chromatography determination of beer volatiles by carbon disulphide extraction: improved technology, data handling and interpretation, Journal of the American Society of Brewing Chemists 32 (1976) 4-13.
- [6] S. Landaud, P. Lieben, D. Picque, Quantitative analysis of diacetyl, pentanedione and their precursors during beer fermentation by an accurate GC/MS method. Journal of the Institute of Brewing 104 (1998) 93-99.
- [7] I.C. Trelea, L. Latrille, S. Landaud, G. Corrieu, Reliable estimation of the key variables and of their rates of change in alcoholic fermentation, Bioprocess and biosystems engineering 24 (2001) 227-237.
- [8] I.C. Trelea, M. Titica, S. Landaud, E. Latrille, G. Corrieu, A. Cheruy, Predictive modelling of brewing fermentation: from knowledge-based to black-box models. Mathematics and Computers in Simulation 56 (2001) 405-424.
- [9] S.I. Daoud, B.A. Searle, On-line monitoring of brewing fermentation by measurement of CO<sub>2</sub> evolution rate, Journal of the Institute of Brewing 96 (1990) 297-302.
- [10] G. Corrieu, I.C. Trelea, B. Perret, On-line estimation and prediction of density and ethanol evolution in the brewery, MBAA Technical Quarterly 37(2) (2000) 173-181.
- [11] M. Titica, S. Landaud, I.C. Trelea, E. Latrille, G. Corrieu, A. Cheruy, Modelling of higher alcohol and ester production kinetics based on CO<sub>2</sub> emission, with a view to beer flavour control by temperature and top pressure. Journal of the American Society of Brewing Chemists, 54(4) (2000) 167-174.
- [12] I.C. Trelea, S. Landaud, L. Latrille, G. Corrieu, Prediction of confidence limits for the diacetyl concentration during beer fermentation, Journal of the American Society of Brewing Chemists 60 (2002) in press.
- [13] M.C. Meilgaard, D.S. Reid, K.A. Wyborski, Reference standards for the beer flavour terminology system. Journal of the American Society of Brewing Chemists, 36 (1982) 119-128.
- [14] C. L. Mallows, Some comments on  $C_p$ , Technometrics, 15 (1973) 661-675.
- [15] J.T. Betts, Practical methods for optimal control using nonlinear programming, Society for Industrial and Applied Mathematics, Philadelphia (2001) 190 p.
- [16] W.H. Press, S.A. Teukolsky, W.T. Vetterling, B.P. Flannery, Numerical recipes in C, Chapter 17: Two point boundary value problems, Cambridge University Press, 1997, pp. 762-772.
- [17] P.E. Gill, W. Murray, M.H. Wright, Practical optimisation, Academic Press, London 1981.
- [18] T. Coleman, M.A. Branch, A. Grace, Optimisation toolbox for Matlab: User's guide, The MathWorks Inc., 1999, pp. 2.23-2.32.
- [19] I. Daoud, R. Dyson, J. Irvine, R.C. Cuthbertson, Practical experience of on-line monitoring of evolved CO<sub>2</sub> from production fermenters, Proceedings of the 22<sup>nd</sup> European Brewing Convention, Zurich, Germany (1989) 323-330.

458 [20] K. Bury, Statistical distributions in engineering, Cambridge University Press (1999) pp. 27-48.

## Nomenclature

Symbol	Units	Significance
$\theta$	°C	Wort temperature
$\theta_{abs}$	K	Absolute temperature conversion constant
$\theta_{max}$	°C	Maximum allowed fermentation temperature
$\theta_{min}$	°C	Minimum allowed fermentation temperature
$\theta_{set}$	°C	Set-point value for the fermentation temperature
$\theta_{typ}$	°C	Typical fermentation temperature
$\nu$	h <sup>-1</sup>	“Specific” CO <sub>2</sub> production rate
$\sigma_i$	mg L <sup>-1</sup>	Residual standard deviation of the model for the aroma compound $i$
$\tau$	h	Rate constant for CO <sub>2</sub> transfer between wort and headspace
$\gamma$	L L <sup>-1</sup>	Wort volume to headspace volume ratio
$\Phi_\theta$	°C h <sup>-1</sup>	Wort cooling rate. For a specified amount of wort, it corresponds to a cooling power
$\Phi_{\theta max}$	°C h <sup>-1</sup>	Maximum achievable wort cooling rate.
$\Phi_g$	g L <sup>-1</sup> h <sup>-1</sup>	Gas outflow rate from the tank (CO <sub>2</sub> + air), per litre of wort
$\Phi_{g max}$	g L <sup>-1</sup> h <sup>-1</sup>	Maximum achievable gas outflow rate from the tank, per litre of wort
$A_{DIA}$	mg L <sup>-1</sup>	Diacetyl concentration
$A_{DIA,typ}$	mg L <sup>-1</sup>	Typical diacetyl concentration at the end of the alcoholic fermentation
$A_i$	mg L <sup>-1</sup>	Concentration of the aroma compound $i$
$A_{i,org}$	mg L <sup>-1</sup>	Organoleptically significant difference in the concentration of the aroma compound $i$
$A_{i,tol}$	mg L <sup>-1</sup>	Tolerance in achieving the concentration of the aroma compound $i$
$A_{i,trg}$	mg L <sup>-1</sup>	Target concentration for the aroma compound $i$
$C_a$	g L <sup>-1</sup>	Amount of air per litre of headspace
$C_d$	g L <sup>-1</sup>	Dissolved CO <sub>2</sub> concentration
$C_{d,typ}$	g L <sup>-1</sup>	Typical dissolved CO <sub>2</sub> concentration
$C_h$	g L <sup>-1</sup>	Amount of gaseous CO <sub>2</sub> per litre of headspace
$C_p$	g L <sup>-1</sup>	Produced CO <sub>2</sub> per litre of wort
$C_{sat}$	g L <sup>-1</sup>	Dissolved CO <sub>2</sub> concentration at equilibrium
$DIA$		Diacetyl
$E$	g L <sup>-1</sup>	Ethanol concentration in the wort
$E_{trg}$	g L <sup>-1</sup>	Target ethanol concentration at the end of the alcoholic fermentation
$ETA$		Ethyl acetate
$ETX$		Ethyl hexanoate
$I_i$	none	Akaike’s information criterion for the aroma compound $i$
$i$	none	Index of an aroma compound. One of { $ETA$ , $ETX$ , $IAA$ , $IAL$ , $PHL$ }
$IAA$		Isoamyl acetate
$IAL$		Isoamyl alcohol
$k$	none	Sampling time index



$K_v$	$\text{h}^{-1}$	“Specific” $\text{CO}_2$ production rate constant
$K_{v\theta}$	$^{\circ}\text{C}^{-1}$	Sensitivity of the $\text{CO}_2$ production rate with respect to temperature
$K_{vC}$	$\text{g}^{-1} \text{L}$	Sensitivity of the $\text{CO}_2$ production rate with respect to dissolved $\text{CO}_2$ concentration
$K_C$	$\text{g L}^{-1}$	Dissolved $\text{CO}_2$ constant
$K_{C\theta}$	$^{\circ}\text{C}^{-1}$	Sensitivity of the dissolved $\text{CO}_2$ with respect to temperature
$K_E$	$\text{g L}^{-1}$	Ethanol inhibition constant
$K_{p\theta}$	$\text{h}^{-1}$	Wort temperature controller parameter
$K_{pp}$	$\text{g L}^{-1} \text{h}^{-1}$ $\text{mbar}^{-1}$	Top pressure controller parameter
$K_S$	$\text{g L}^{-1}$	Substrate limitation constant
$K_X$	$\text{g L}^{-1} (10^6 \text{ cells mL}^{-1})^{-1}$	Initial $\text{CO}_2$ production rate constant
$M_a$	$\text{g mol}^{-1}$	Equivalent air molar mass
$M_c$	$\text{g mol}^{-1}$	$\text{CO}_2$ molar mass
$n_i$	none	Number of nonzero model parameters
$n_t$	none	Number of time sampling points
$n_u$	none	Number of control variables
$n_x$	none	Number of state variables
$p$	mbar	Total (air + $\text{CO}_2$ ) top pressure in the fermentation tank
$p_a$	mbar	Partial air pressure in the headspace
$p_{atm}$	mbar	Atmospheric pressure
$p_c$	mbar	Partial $\text{CO}_2$ pressure in the headspace
$p_{max}$	mbar	Maximum allowed top pressure in the fermentation tank
$p_{set}$	mbar	Set-point value for the top pressure in the fermentation tank
$PHL$		Phenyl ethanol
$Q$	none	Global optimisation criterion
$Q_{\theta}$	none	Partial optimisation criterion for the smoothing of the wort temperature profile
$Q_{DLA}$	none	Partial optimisation criterion for final diacetyl concentration minimisation
$Q_i$	none	Partial optimisation criterion associated to aroma compound $i$
$Q_p$	none	Partial optimisation criterion for the smoothing of the top pressure profile
$Q_t$	none	Partial optimisation criterion for the total fermentation time minimisation
$R$	$\text{mbar L mol}^{-1}$ $\text{K}^{-1}$	Perfect gas constant
$S$	$\text{g L}^{-1}$	Fermentable sugar concentration in the wort
$S_{res}$	$\text{g L}^{-1}$	Desired residual (final) fermentable sugar concentration in the wort
$t$	h	Fermentation time since yeast inoculation
$t_f$	h	Total alcoholic fermentation time
$t_{fmax}$	h	Maximum allowed total alcoholic fermentation time
$t_{typ}$	h	Typical duration of the alcoholic fermentation
$u$		Vector of the control variables

$W_{\theta}$	none	Weight associated to the smoothing of the wort temperature profile
$W_{DLA}$	none	Weight associated to the minimisation of the final diacetyl concentration
$W_{DLA,j}$	none	Diacetyl production and reduction model coefficients
$W_i$	none	Weight associated to the partial optimisation criterion $Q_i$
$W_{i,j}$	none	Aroma production model coefficient $j$ for compound $i$
$W_p$	none	Weight associated to the smoothing of the top pressure profile
$W_t$	none	Weight associated to the minimisation of the total fermentation time
$x$		Vector of the state variables
$X_0$	$10^6$ cells mL <sup>-1</sup>	Initial yeast concentration in the wort
$X_{0\ min}$	$10^6$ cells mL <sup>-1</sup>	Minimum allowed initial yeast concentration in the wort
$X_{0\ max}$	$10^6$ cells mL <sup>-1</sup>	Maximum allowed initial yeast concentration in the wort
$Y_{DLA/C}$	mg g <sup>-1</sup>	Diacetyl versus CO <sub>2</sub> production yield
$Y_{E/C}$	g g <sup>-1</sup>	Ethanol versus CO <sub>2</sub> production yield
$Y_{i/C}$	mg g <sup>-1</sup>	Yield of aroma compound $i$ versus CO <sub>2</sub>
$Y_{S/C}$	g g <sup>-1</sup>	Fermentable sugar versus CO <sub>2</sub> yield

**Table 1.** Numerical values of the parameters in the alcoholic fermentation model

Para meter	Units	Value (95% confidence limits)	Source
$\theta_{abs}$	K	273.16	Unit conversion: degrees Celsius to Kelvin
$\theta_{typ}$	°C	13	Central point of the experimental design
$\tau$	h	3	Separate experiment
$\gamma$	L L <sup>-1</sup>	4	Experimental protocol
$C_{d,typ}$	g L <sup>-1</sup>	2.76	Central point of the experimental design, Equation (8)
$K_v$	h <sup>-1</sup>	0.0446 (0.0413 ... 0.0481)	Maximum likelihood, runs R1-R4 and R6-R9
$K_{v\theta}$	°C <sup>-1</sup>	0.132 (0.127 ... 0.137)	Maximum likelihood, runs R1-R4 and R6-R9
$K_{vC}$	g <sup>-1</sup> L	0.074 (0.034 ... 0.113)	Maximum likelihood, runs R1-R4 and R6-R9
$K_C$	g L <sup>-1</sup>	0.0283	Tables supplied by professional brewer association
$K_{C\theta}$	°C <sup>-1</sup>	-0.0335	Tables supplied by professional brewer association
$K_E$	g L <sup>-1</sup>	25.5 (22.2 ... 29.2)	Maximum likelihood, runs R1-R4 and R6-R9
$K_S$	g L <sup>-1</sup>	3	Reference [11]
$K_X$	g L <sup>-1</sup> (10 <sup>6</sup> cells mL <sup>-1</sup> ) <sup>-1</sup>	0.145 (0.122 ... 0.172)	Maximum likelihood, runs R1-R4 and R6-R9
$M_a$	g mol <sup>-1</sup>	28.8	20% O <sub>2</sub> + 80% N <sub>2</sub> by volume
$M_c$	g mol <sup>-1</sup>	44	Chemical formula of CO <sub>2</sub>
$p_{atm}$	mbar	1013	760 mm Hg
$R$	mbar L mol <sup>-1</sup> K <sup>-1</sup>	83.1	Perfect gas constant
$Y_{E/C}$	g g <sup>-1</sup>	1.028 (1.013 ... 1.043)	Reference [7]
$Y_{S/C}$	g g <sup>-1</sup>	1.884 (1.834 ... 1.934)	Reference [7]

**Table 2.** Numerical values of the parameters in the aroma production model

(In parenthesis: 95% confidence limits)

Source: maximum likelihood estimation based on data from runs R1-R4 and R6-R9

Parameter	Aroma compound ( <i>i</i> )					
	ETA	ETX	IAA	IAL	PHL	DIA
$\sigma_i$	2.73	0.0201	0.224	8.08	4.78	0.106 (0.092 ... 0.125)
$W_{i,1}$	0.0426 (0.0386 ... 0.0470)	0.00027 (0.00023...0.00031)	0.0040 (0.0037 ... 0.0044)	1.91 (1.80 ... 2.04)	0.83 (0.75 ... 0.91)	0.0102 (0.0083 ... 0.0124)
$W_{i,2}$	0.148 (0.123 ... 0.172)	0.162 (0.126 ... 0.198)	0.143 (0.119 ... 0.167)	0	0.054 (0.026 ... 0.082)	0.176 (0.129 ... 0.222)
$W_{i,3}$	-0.838 (-1.001 ... -0.675)	-0.651 (-0.797 ... -0.506)	-0.754 (-0.876 ... -0.632)	-0.080 (-0.147 ... -0.012)	-0.11 (-0.22 ... -0.003)	0.203 (0.162 ... 0.255)
$W_{i,4}$	0	-0.045 (-0.066 ... -0.025)	-0.034 (-0.047 ... -0.021)	0.015 (0.008 ... 0.022)	0.011 (0.001 ... 0.021)	0.175 (0.110 ... 0.390)
$W_{i,5}$	0	0	0	0.052 (0.033 ... 0.070)	0.036 (0.001 ... 0.071)	NA
$W_{i,6}$	0.0041 (0.0015 ... 0.0066)	0.011 (0.005 ... 0.017)	0.0046 (-0.0007 ... 0.0099)	0.0040 (0.0018 ... 0.0062)	0.0052 (0.0020 ... 0.0084)	NA
$W_{i,7}$	0	0	0	-0.023 (-0.032 ... -0.013)	-0.018 (-0.032 ... -0.005)	NA
$W_{i,8}$	0	0	0.0046 (-0.0015 ... 0.0106)	0	0	NA

NA = not applicable

**Table 3.** Numerical values of the optimal control problem parameters.

Parameter	Units	Value	Parameter	Units	Value	Parameter	Units	Value
$\theta_{max}$	°C	16	$K_{p\theta}$	$\text{h}^{-1}$	5	$W_{ETX}$	none	1
$\theta_{min}$	°C	10	$K_{pp}$	$\text{g L}^{-1} \text{h}^{-1} \text{mbar}^{-1}$	$5 \times 10^{-4}$	$W_{IAA}$	none	1
$\Phi_{\theta max}$	°C $\text{h}^{-1}$	0.5	$n_t$	none	49	$W_{IAL}$	none	1
$\Phi_{g max}$	$\text{g L}^{-1} \text{h}^{-1}$	2	$p_{max}$	mbar	1813	$W_{PHL}$	none	1
$A_{DIA,typ}$	$\text{mg L}^{-1}$	0.1	$S_{res}$	$\text{g L}^{-1}$	1	$W_p$	none	$2 \times 10^{-4}$
$A_{ETA,org}$	$\text{mg L}^{-1}$	2.5	$t_{f max}$	h	200	$W_t$	none	0.1
$A_{ETX,org}$	$\text{mg L}^{-1}$	0.02	$t_{typ}$	h	100	$X_{0 min}$	$10^6$ cells $\text{mL}^{-1}$	5
$A_{IAA,org}$	$\text{mg L}^{-1}$	0.20	$W_{\theta}$	none	$2 \times 10^{-4}$	$X_{0 max}$	$10^6$ cells $\text{mL}^{-1}$	20
$A_{IAL,org}$	$\text{mg L}^{-1}$	5	$W_{DIA}$	none	0.1			
$A_{PHL,org}$	$\text{mg L}^{-1}$	4	$W_{ETA}$	none	1			

**Table 4.** Target ethanol and aroma compound concentrations

Beer sort	$E_{trg}$ g L <sup>-1</sup>	$A_{ETA,trg}$ mg L <sup>-1</sup>	$A_{ETX,trg}$ mg L <sup>-1</sup>	$A_{IAA,trg}$ mg L <sup>-1</sup>	$A_{IAL,trg}$ mg L <sup>-1</sup>	$A_{PHL,trg}$ mg L <sup>-1</sup>
R5 <sup>a</sup>	40	15	0.115	1.0	77	34
B1 <sup>b</sup>	38	20	0.135	3.1	67	37
B2 <sup>b</sup>	48	18	0.086	1.6	96	47
B3 <sup>b</sup>	48	28	0.225	3.8	70	30
B4 <sup>b</sup>	40	28	0.125	4.1	62	22

<sup>a</sup> Final ethanol and aroma concentrations measured in run R5

<sup>b</sup> Ethanol and aroma concentrations measured in commercial beers

## Figure legends

Figure 1. Alcoholic fermentation model validation. Data from run R5, not used for model identification. Measured values (o) and simulated values (—).

Figure 2. Aroma production model validation. Data from run R5, not used for model identification. Measured values (o) and simulated values (—).

Figure 3. Fermentation time reduction of an existing beer without changing the final aroma profile. **(A)** Constant operating conditions corresponding to run R5. **(B)** Time-varying operating condition determined by the dynamic optimisation algorithm. Top: aroma concentrations at the end of the alcoholic fermentation. Bottom: operating conditions for the alcoholic fermentation process. Target values (o), admissible range ( $\cdot \cdot \cdot$ ) at  $\pm 2A_{i,tol}$  and predicted values (—).

Figure 4. Different final aroma profiles obtained with the same yeast strain and wort. **(A)** Beer B2. **(B)** Beer B3. Top: aroma concentrations at the end of the alcoholic fermentation. Bottom: operating conditions for the alcoholic fermentation process. Target values (o), admissible range ( $\cdot \cdot \cdot$ ) at  $\pm 2 A_{i,tol}$  and predicted values (—).

Figure 5. Aroma profiles impossible to achieve with the considered yeast strain and wort. **(A)** Beer B1. **(B)** Beer B4. Top: aroma concentrations at the end of the alcoholic fermentation. Bottom: operating conditions for the alcoholic fermentation process. Target values (o), admissible range ( $\cdot \cdot \cdot$ ) at  $\pm 2 A_{i,tol}$  and predicted values (—).

Figure 6. Sensitivity of the final aroma concentration and of the optimal control strategy to modelling errors. **(A)** The optimal control policy for beer B2 was applied without change to 20 possible model parameter sets. **(B)** The optimal control policy for beer B3 was recomputed for 20 possible model parameter sets. Top: aroma concentrations at the end of the alcoholic fermentation. Bottom: operating conditions for the alcoholic fermentation process. Target values (o), admissible range ( $\cdot \cdot \cdot$ ) at  $\pm 2 A_{i,tol}$  and 95% confidence intervals for the predicted values (■).

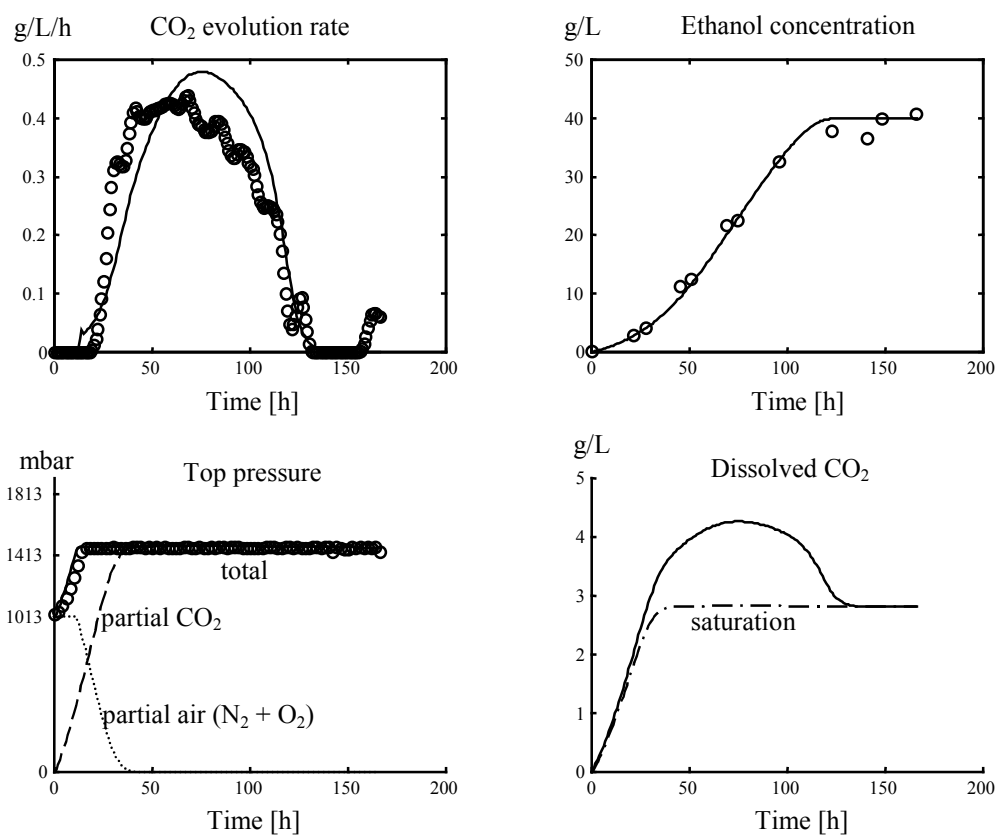


Figure 1



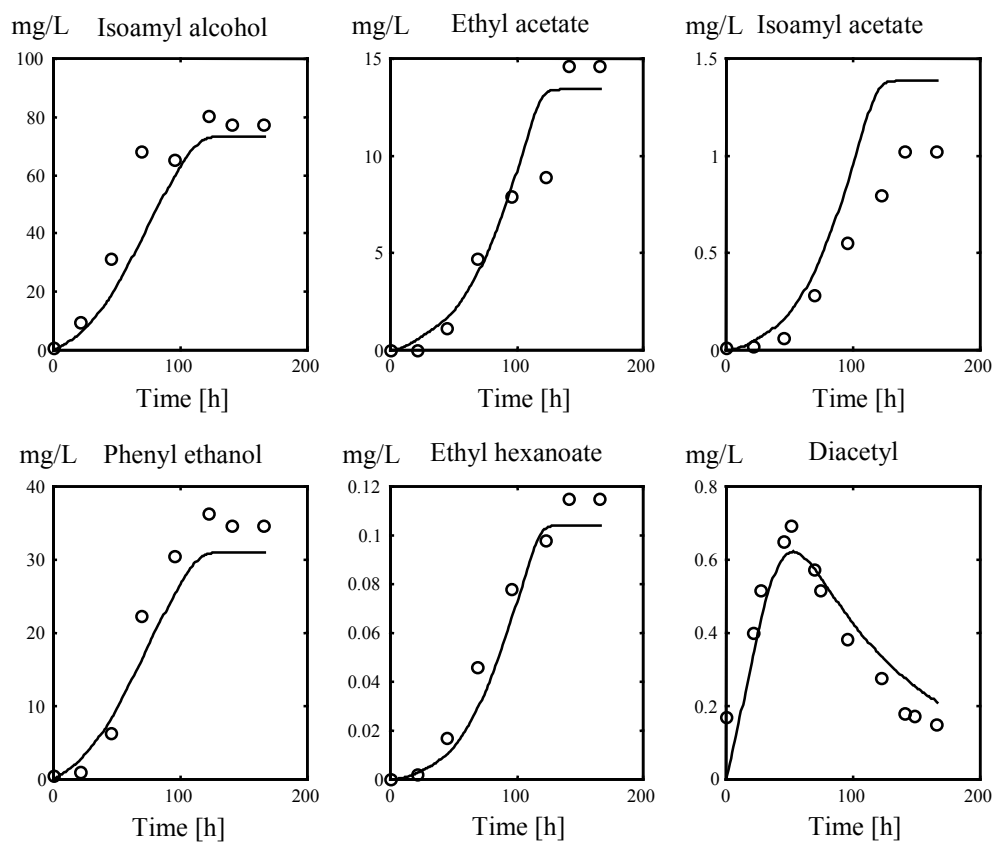


Figure 2

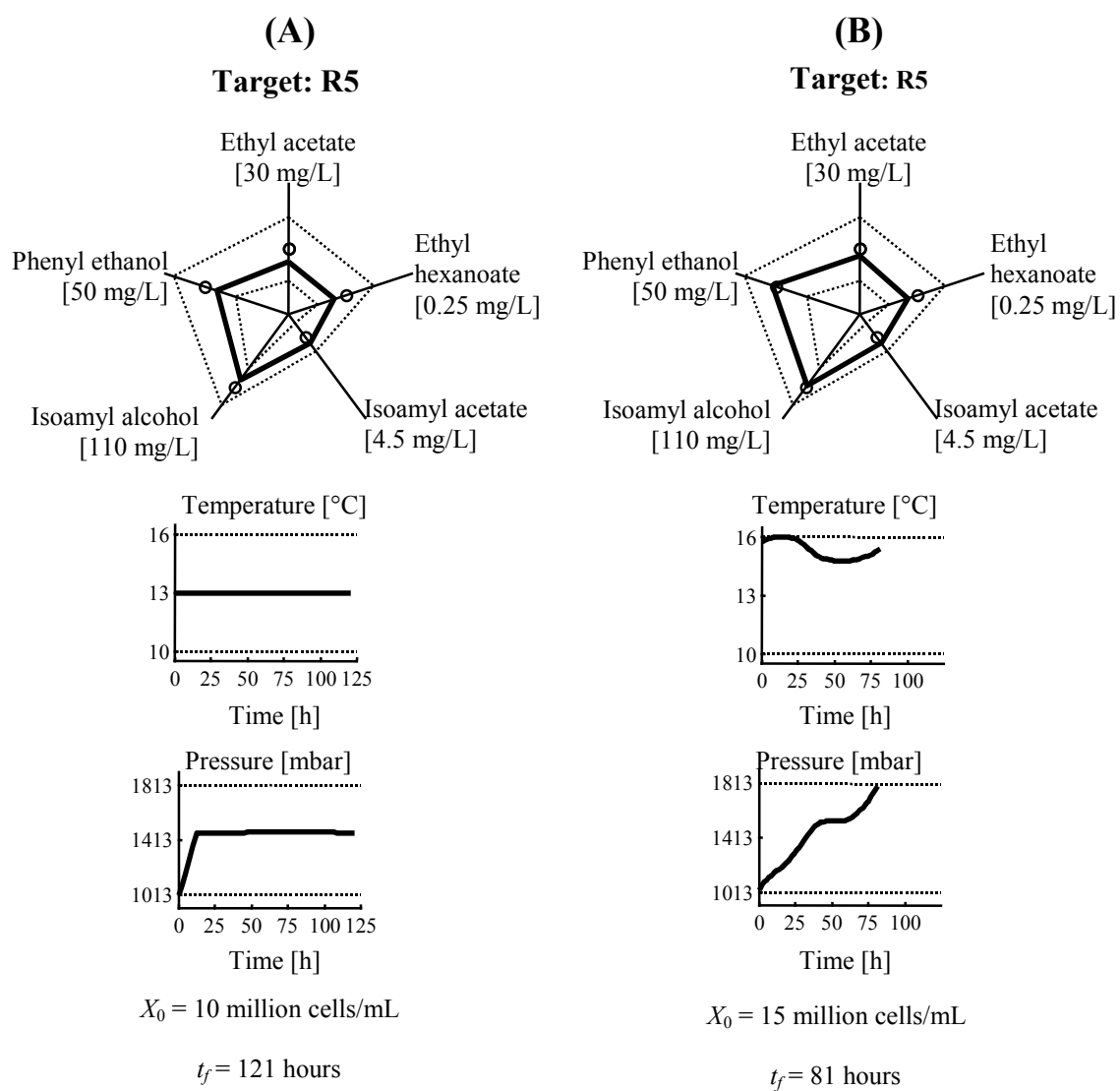


Figure 3.

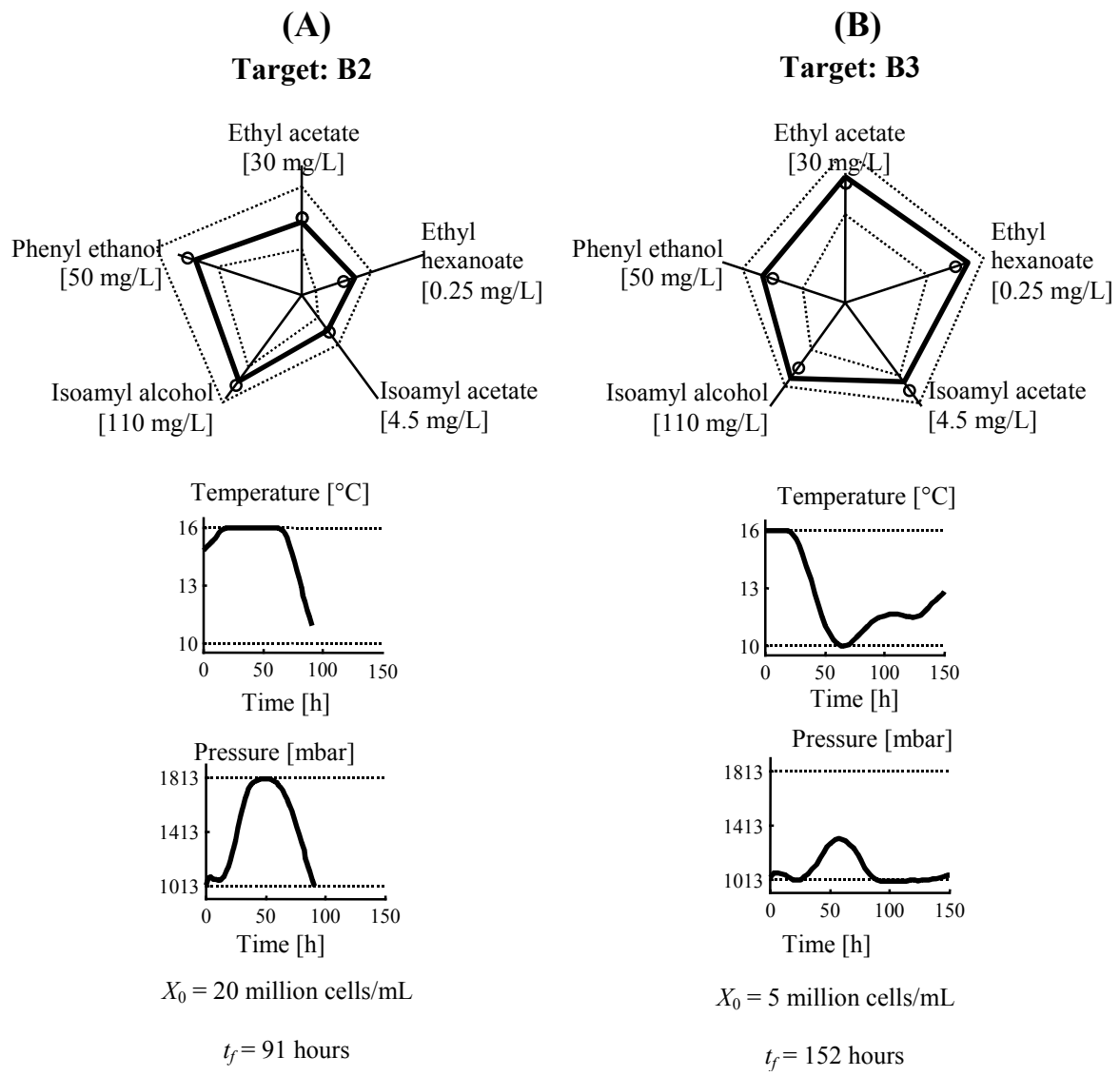


Figure 4.

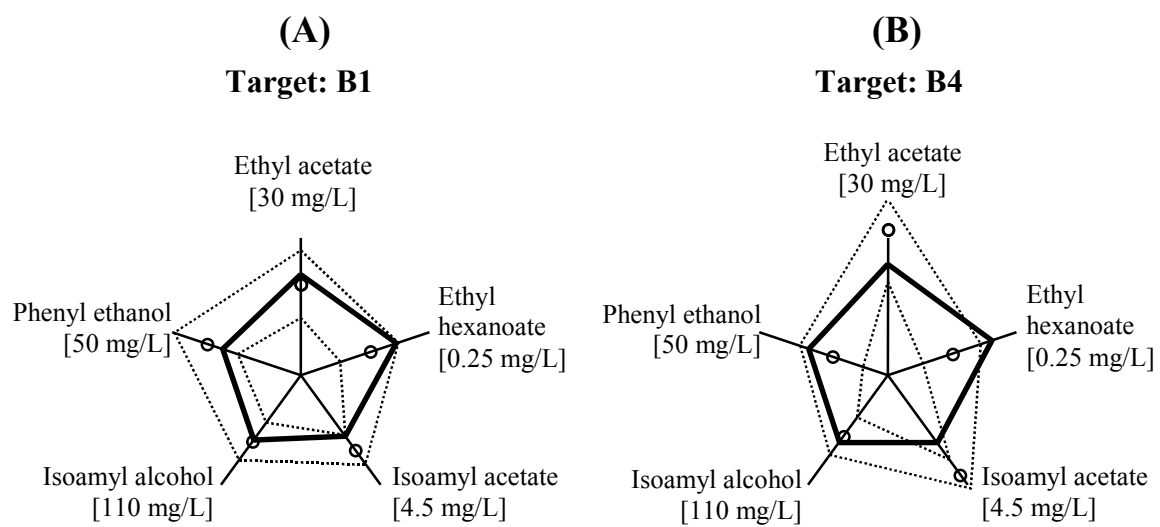


Figure 5.

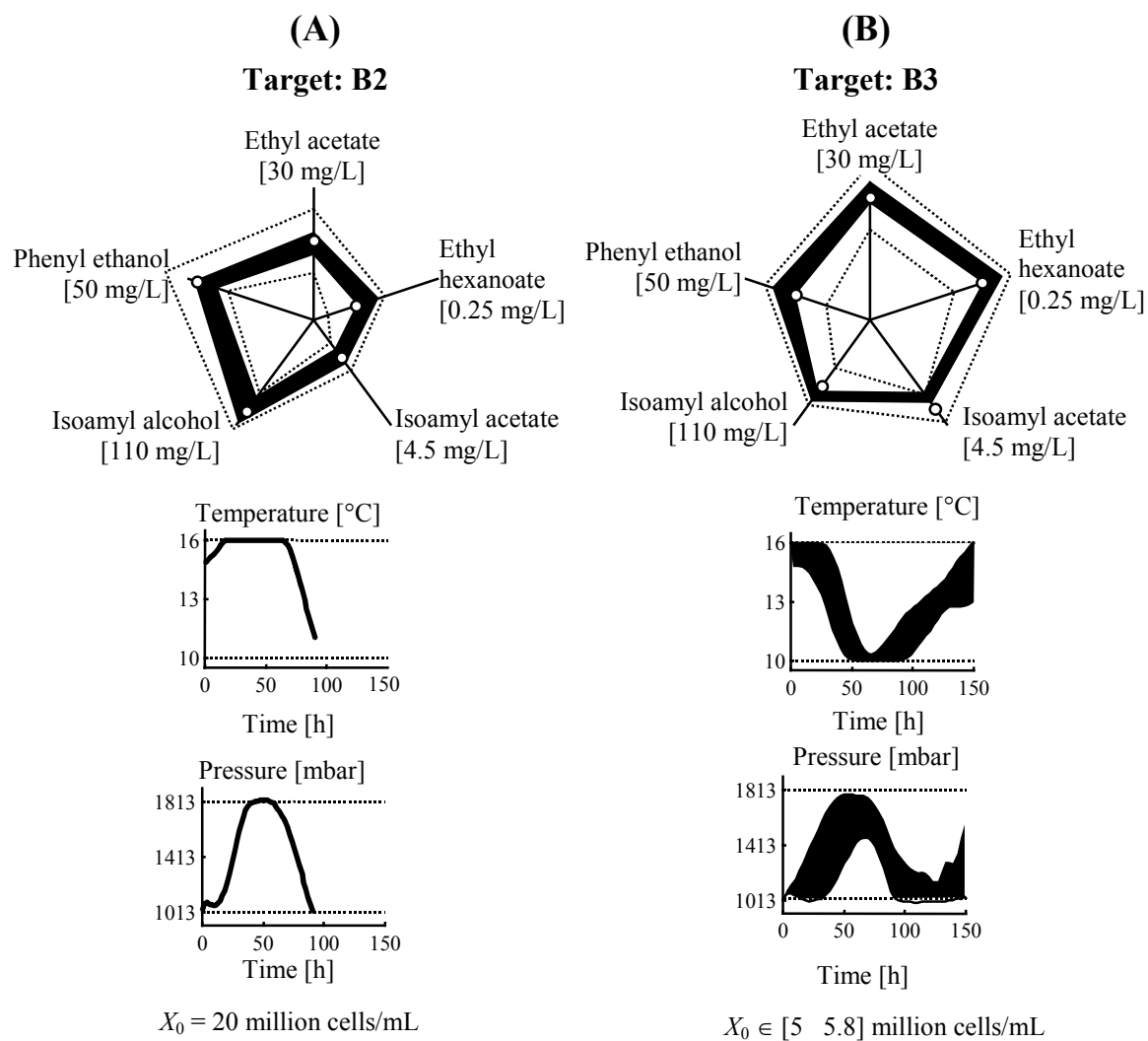


Figure 6.