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# Separation of isobutyl alcohol and isobutyl acetate by extractive distillation and pressure-swing distillation: Simulation and optimization

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**Abstract**

We have studied, simulated and evaluated economically two separation alternatives of a mixture made up of 52 mole% of isobutyl alcohol and 48 mole% of isobutyl acetate by means of a practical case of a plant to treat 12,000 Tm/year of the original mixture. The simulation has been carried out satisfactorily by means of a package of commercial software (Aspen HYSYS®) using the thermodynamic model UNIQUAC with binary parameters obtained experimentally by us.

The two processes evaluated (extractive distillation using *n*-butyl propionate as a solvent and pressure-swing distillation) have been optimized independently from each other and the best configurations have been evaluated economically. The simulation and economic evaluation of the two separation alternatives that we have considered allow us to conclude that, for a 12,000 Tm/year plant, the pressure-swing distillation is more attractive than the extractive distillation using *n*-butyl propionate as an entrainer.


*Keywords:* Extractive distillation; Pressure-swing distillation; Simulation; Isobutyl alcohol; Isobutyl acetate; *n*-Butyl propionate

## 1. Introduction

Isobutyl acetate (IBAc) is a solvent widely used in Chemical Industry. It is used alone or in solvent blends in applications including coatings, inks, adhesives, industrial cleaners and degreasers. The IBAc is produced by estherification of acetic acid with isobutyl alcohol (IBA). Final purification of acetate by traditional technologies is a relatively complex procedure due to the existence of a minimum boiling point azeotrope in the IBA + IBAc mixture at atmospheric pressure.

Azeotropes are non-ideal mixtures whose components are very difficult and, hence, expensive to separate. This can be overcome by several techniques including azeotropic and extractive distillation [1–3], reactive distillation [4,5], liquid–liquid extraction [6], adsorption [7], membrane pervaporation [8], salt addition [9] and pressure-swing distillation [10]. In this work, only extractive distillation (ED) and pressure-swing distillation (PSD) will be considered.

ED can be used to separate the components of an azeotropic mixture adding an agent (entrainer) that modifies the relative volatility of the mixture. Also, PSD can be used to recover pure components with a simple change in pressure, a fact which results in a change of the azeotropic composition, provided that it is pressure-sensitive.

Laboratory experiments in either extractive distillation or pressure-swing distillation are time-consuming and expensive because of the large number of parameters involved. It would be desirable to predict the experimental data with the help of available simulation programs. Computer simulations using commercial process simulators have been used with success as an aid for process development. They were used to set up the guidelines for further pilot experiments and moreover, to optimize the operating parameters governing the process at steady-state.

*Abbreviations:* BUP, butyl propionate; DMF, dimethylformamide; EC, extractive column; ED, extractive distillation; FCI, fixed capital investment (€$10^3$); HPC, high pressure column; IBA, isobutyl alcohol; IBAc, isobutyl acetate; LPC, low pressure column; PSD, pressure-swing distillation; RHD, reboiler heat duty (MJ/h); SRC, solvent recovery column; TAC, total annual costs (€$10^3$/year); VLE, vapour–liquid equilibrium

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**Nomenclature**

| | |
|---|---|
| $A$ | UNIQUAC binary interaction parameters (cal/mol) |
| $C$ | costs (€$10^3$/year) |
| $i_m$ | minimum acceptable rate of return |
| $i_r$ | fixed capital recovery rate (depreciation rate) |
| $K$ | vapour–liquid equilibrium constant |

*Greek letter*

| | |
|---|---|
| $\alpha$ | relative volatility |

*Subscripts*

| | |
|---|---|
| f | fixed |
| $i, j$ | primitive mixture components |
| S | solvent (entrainer) |
| v | variable |

*Superscript*

| | |
|---|---|
| $\infty$ | infinite dilution |

The synthesis and design of extractive distillation processes take place in two steps [11]. The first one involves the selection of one or more candidate solvents (which facilitate the separation by changing the relative volatilities in the mixture through physical or chemical interactions with the original components), and the choice of one or more column configurations. The second step, process design, involves the search for optimal process parameter values. The success of the second step depends on the solutions obtained for the first one because efficiency in extractive distillation is largely determined by the choice of a suitable entrainer.

In this work, based on the guidelines for the solvent screening, we have chosen three solvents: *N*,*N*-dimethylformamide (DMF), 1-hexanol and butyl propionate (BUP). DMF was recommended as a potential entrainer for alcohol–acetate azeotropic mixtures because of its high polarity [12] and 1-hexanol and BUP have been chosen because they are, respectively, in the same homologous series with one of the key-components [13]. Therefore, in order to be able to select the best solvent among them, we have carried out simulations with Aspen HYSYS® v3.2 of Aspen Technology Inc., using the binary interaction parameters correlated from experimental data obtained for all binaries involved [14–17]. According to the results obtained, the best solvent seems to be butyl propionate. Once the solvent has been selected, we have designed the separation sequence and optimized the operating parameters.

On the other hand, to investigate how the pressure-swing distillation works with the IBA + IBAc azeotropic system, we have done a simulation of the vapour–liquid equilibrium using DISTIL v5.0 of Hyprotech Ltd. at different pressures with the interaction parameters obtained from experimental VLE data [14]. Based on these results we have decided to carry out the design and optimization of the pressure-swing distillation process.

The aim of this work is to study the influence of the operation variable values and column configuration on the performance of the IBA + IBAc separation by extractive distillation with BUP as entrainer and by swing-pressure distillation, with the help of a commercial simulator (Aspen HYSYS® v3.2 of Aspen Technology Inc.). Finally, we have chosen the best alternative for the separation of the azeotropic mixture under study from the economic point of view.

## 2. Simulation

### 2.1. Problem definition

The two alternatives considered in this study (ED and PSD) were simulated with the same basic data. The feed is a mixture made up of 52 mole% of isobutyl alcohol and 48 mole% of isobutyl acetate, with a flow rate of 12,000 Tm/year; we took 8000 working hours per year, that is a mass flow of 1500 kg/h.

### 2.2. Property package

Computer simulation using commercial process simulators is a useful tool to predict qualitatively the influence of the operating variables on the column performance, provided that the interaction binary parameters for the studied mixture are available in their own data-bank. The accuracy of the simulated results is strongly dependent on the quality of the binary parameters for the liquid-phase activity coefficient models.

In this paper, the simulation was undertaken with HYSYS and DISTIL. UNIQUAC activity model was chosen because it was the most suitable, but unfortunately, no data exist in their own library to cover all possible interactions between the components studied in this work, so we have used the binary interaction parameters published by us in previous papers [14–17]. The parameters used are listed in Table 1.

### 2.3. Extractive distillation

#### 2.3.1. Solvent selection

Since the solvent is the core of extractive distillation, more attention should be paid on the selection of potential solvents. Of all possible entrainers that can be used for the separation of IBA and IBAc azeotrope mixture we have chosen three: *N*,*N*-dimethylformamide, 1-hexanol and butyl propionate. DMF was recommended as a potential entrainer for alcohol–acetate azeotropic mixtures because of its high polarity and 1-hexanol and BUP have been chosen because they are, respectively, in the same homologous series with one of the key-components.

Table 1
UNIQUAC binary interaction parameters

| Component $i$ | Component $j$ | $A_{ij}$ (cal/mol) | $A_{ji}$ (cal/mol) |
|---|---|---|---|
| IBA | IBAc | 116.17–0.53$T$[a] | 361.90–0.45$T$[a] |
| IBA | DMF | 56.603 | −155.920 |
| IBA | 1-Hexanol | 461.869 | −340.476 |
| IBA | BUP | 35.346 | 49.224 |
| IBAc | DMF | 551.320 | −281.210 |
| IBAc | 1-Hexanol | 316.172 | −227.902 |
| IBAc | BUP | 131.014 | −128.309 |

[a] $T$ is the temperature in Kelvin.