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Rethinking ethanol separation in continuous spirit production from a process intensification perspective

Anton A Kiss*

Abstract

Continuous neutral-alcohol and food-grade ethanol production is an energy-intensive process in which distillation and rectification alone account for 50–85% of total plant energy use. While significant advances in separation technology have emerged over the past two decades, the spirits industry continues to rely predominantly on multi-effect distillation configurations developed decades ago. This perspective paper evaluates intensified separation technologies for large-scale continuous production of food-grade ethanol. Special emphasis is on practical applicability within the constraints of spirit manufacturing, namely food-grade compatibility, congener management and retrofit feasibility. The ethanol–water azeotrope at 95.63 wt% (96.47 vol%) at atmospheric pressure remains the key thermodynamic challenge, though only for the high-strength distillate/neutral-alcohol stage as most finished spirits (bottled at ca 40 vol%) never approach it. Breaking or avoiding it without using (food-grade-incompatible) mass separating agents requires an actual rethinking of process design. Technologies are assessed against a technology readiness level (TRL) framework. Cyclic distillation and heat pump-assisted distillation are identified as near-term, deployment-ready solutions offering 30–70% energy savings at TRL 7–9. HIDiC, suitable for new plant designs, can reach up to 77% savings. Hybrid distillation–membrane separation is the most promising medium-term pathway combining high purity and low energy demand. Pass-through distillation emerges as a disruptive option enabling concurrent fermentation and alcohol recovery. A decision framework linking feed concentration, target purity, plant scale and capital constraints is proposed to guide technology selection for both new plants and retrofit applications. The findings highlight that the spirits sector is well positioned to reduce its carbon footprint and operating costs by adopting proven process intensification technologies that have demonstrated industrial viability in other sectors.

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Keywords: spirit production; food-grade alcohol; energy efficiency; cyclic distillation; heat pump-assisted distillation; hybrid distillation–pervaporation; congener management; decarbonisation

INTRODUCTION

The spirits industry is a manufacturing sector operating at large scale with substantial energy demands that translate into operating costs and environmental impact. At the heart of every distilled spirit (e.g. whisky, vodka, rum, gin, brandy and liqueurs) there is a simple operation, namely the separation of ethanol from water. Yet despite its apparent simplicity, this separation is thermodynamically constrained, energy-intensive and subjected to quality and safety requirements of food-grade production. This restricts the choice of separation method.

Ethanol separation serves different objectives depending on the product class targeted, and this shapes which separation technologies are appropriate. We distinguish four main classes: (1) *traditional batch-distilled spirits* (e.g. pot-still whisky, cognac, artisanal fruit brandies), where congener retention (not removal) is essential to product character and where batch distillation remains the dominant technology; (2) *continuous neutral alcohol* (e.g. vodka, gin base, industrial rum base), produced in continuous multicolumn rectification trains targeting extensive congener

removal; (3) *non-anhydrous food-grade ethanol*, produced similarly to the previous class but to a specified hydrous purity (typically ≥ 96 vol%) without requiring complete dehydration; and (4) *anhydrous fuel-grade ethanol* (> 99 wt%), included here only as a comparative reference point, since much of the published separation literature addresses this class. This perspective paper concerns classes (2) and (3), namely continuous, large-scale neutral and food-grade alcohol production, and explicitly excludes class (1), traditional batch spirit production, from the technology assessment that follows. Class (4) is referenced only where fuel-ethanol literature provides relevant comparative data.

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Within this scope, production of alcohol from biobased feedstocks involves several stages: pretreatment of raw materials, fermentation of sugars to yield a dilute ethanol broth and separation of the alcohol–water mixture. The last stage in spirit production is the most energy-demanding step in the entire process. As illustrated in Fig. 1, distillation and purification account for roughly 70% of thermal energy use in a typical ethanol plant.^{1,2} Multiple studies report that distillation alone uses 50–85% of total plant energy, which is about 5.6 MJ L⁻¹ out of a total 11.1–12.5 MJ L⁻¹ for current designs.^{2,3} Recent plant-wide analyses of nine operational distilleries report total specific energy use in the range 1.73–4.05 kWh L⁻¹ alcohol (ca 6.2–14.6 MJ L⁻¹), which is consistent with the earlier estimates and highlights the variability and improvement potential across sites.⁴ For an industry producing billions of litres annually, this is a large energy burden and an opportunity for improvement.

Distillation is among the oldest and most widely used separation technologies in the chemical industry, valued for its simplicity, robustness and effectiveness.⁵ However, its thermodynamic efficiency is notoriously low. For the ethanol–water system, an additional complication arises in the form of a minimum-boiling azeotrope at 95.63 wt% (96.47 vol%) ethanol, which sets an upper limit on the achievable product purity by classic distillation.⁶ The vapor–liquid equilibrium (VLE) of this system, described reliably by the NRTL thermodynamic model,^{7,8} reveals that separation becomes more difficult at compositions approaching the azeotrope (Fig. 2). In that range, it requires more stages, higher reflux ratios and thus greater energy input.

Spirits production differs from other ethanol applications (e.g. fuel-grade bioethanol) as the separation task is not simply about water removal. During the distillation run, ethanol and water act as carriers for a large number of volatile aroma compounds, collectively known as congeners. These include higher alcohols (fusel oils), esters, aldehydes and organic acids, whose distribution across the heads, heart and tails fractions (typical in a batch distillation) determines the flavour profile and quality of

the final product.⁹ Yet, as shown later, most ethanol separation studies report performance only for binary feeds, leaving congener behaviour largely undocumented. Methanol is a toxic compound produced from the demethoxylation of pectin during fermentation, and must be carefully managed and removed.⁹ Ethanol forms azeotropes with many compounds whose boiling points are close to its own, for example benzene, butyraldehyde, ethyl acetate and thiophene. These cannot be quantitatively removed by simple distillation and pose both health and sensory concerns.¹⁰ Critically, the azeotropic entrainers and extraction solvents used in fuel-grade bioethanol dehydration (e.g. benzene, cyclohexane or glycols) are strictly incompatible with food-grade standards and thus excluded from spirits manufacturing.

Despite decades of research into alternative separation technologies, the spirits industry has been slow to adopt new approaches. Most production facilities operate multi-effect distillation trains (e.g. pressure-cascaded beer and rectification columns with vapor-side reboilers and inter-column heat recovery) based on designs established in the mid-20th century, which achieve steam consumptions of 3.0–4.2 kg of steam per litre of 96° Gay-Lussac (%vol at 20°) ethanol.^{6,11} The reasons for this conservatism are well understood. Capital investment is substantial, product quality is crucial, regulatory frameworks are strict and the industry's customer base is highly sensitive to any change in organoleptic properties. Yet, none of these factors is a rational technical justification for the status quo. Technologies required to reduce separation energy by 30–77% are commercially available, food-grade compatible and operating today in other industrial sectors. The barrier is not technical risk, but institutional inertia and the absence of sector-specific technoeconomic guidance. The combination of rising energy costs, tightening carbon emission targets and a rapidly maturing process intensification (PI) toolkit creates a time-sensitive case for rethinking the separation design at the core of spirits manufacturing.

To make this opportunity concrete, we adopt here a reference production scale of 300 000 hL yr⁻¹ (30 × 10⁶ L yr⁻¹ or ca 24 ktpy),

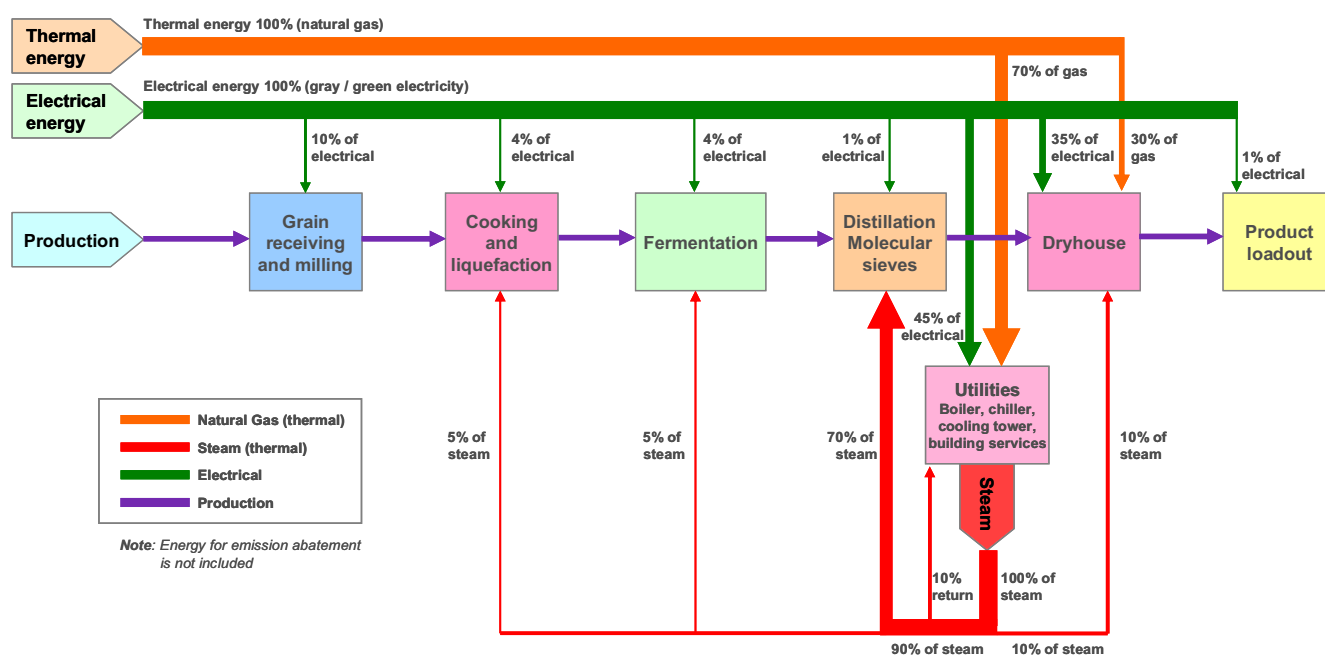


Figure 1. Energy flows through a modern ethanol plant (adapted from Meredith¹).

distilling a 10 vol% feed to 96.2 vol% product, representative of a large-scale continuous neutral spirit distillery. At this scale, distillation and purification use about 247.8 TJ of thermal energy annually ($11.8 \text{ MJ L}^{-1} \times 70\% \times 30 \times 10^6 \text{ L yr}^{-1}$), equivalent to a CO_2 footprint of roughly $13\,800 \text{ t yr}^{-1}$ when generated from natural gas steam, or about 545 g CO_2 per litre of spirits produced. This assumes a CO_2 emission factor of $\text{ca } 56 \text{ kg CO}_2 \text{ GJ}^{-1}$ for natural gas combustion at $\text{ca } 85\%$ efficiency, leading to $66 \text{ kg CO}_2 \text{ GJ}^{-1}$ of steam delivered. A 30% energy reduction through cyclic distillation alone would eliminate $\text{ca } 4150 \text{ t CO}_2 \text{ yr}^{-1}$, and deploying heat pump-assisted distillation would save $\text{ca } 8300 \text{ t CO}_2 \text{ yr}^{-1}$ from a single facility. For an industry producing billions of litres annually, the sector potential is of a different order of magnitude.

This perspective paper critically evaluates the landscape of available and emerging separation technologies for food-grade ethanol production, combining several criteria specific to spirits manufacturing: food-grade compatibility, congener management and retrofit feasibility for existing plant infrastructure. Technologies are assessed using a technology readiness level (TRL) framework, and a practical decision guide linking technology choice to plant-specific conditions is proposed. We also establish the thermodynamic, regulatory, compositional and economic constraints that any candidate technology must satisfy. The paper draws on open literature alongside industrial case studies to identify the most promising pathways towards a more energy-efficient, sustainable and economically competitive spirits industry. Note that this perspective is focused on continuous distillation, as used in large-scale spirit production. Ultimately, we posit that the spirits sector is in fact well positioned for rapid decarbonisation of its separation operations. Recent decarbonisation studies focused on the spirits sector emphasise primarily fuel switching and anaerobic digestion of by-products,⁴ while the present work complements that perspective by targeting the distillation core itself through process intensification.

MAIN CHALLENGES

Thermodynamic constraints of ethanol–water separation

The ethanol–water binary system is one of the most studied separation problems in the chemical process industry. As shown in

Fig. 2, the VLE of this system reveals a pronounced pinch region at high ethanol concentrations, where the equilibrium curve approaches the 45° diagonal and the driving force for separation becomes very small. This behaviour culminates in a minimum-boiling homogeneous azeotrope at 95.63 wt% (96.47 vol% or 89.4 mol%) ethanol at atmospheric pressure, boiling at 78.2°C .⁶ At this point the composition of liquid and vapor phases is identical, and no further enrichment is achievable by classic distillation regardless of the number of stages employed. Note that the azeotrope is only a practical constraint for production targeting $\geq 95 \text{ vol\%}$ ethanol, as the majority of finished spirits (typically bottled at $\text{ca } 40 \text{ vol\%}$) do not require azeotrope-breaking technology at all. This constraint applies to the intermediate high-strength distillate/neutral-alcohol production step, not the final product.

Figure 3 provides a framework for understanding the implications of this by dividing the separation task into several operational zones: a stripping zone at low ethanol concentrations

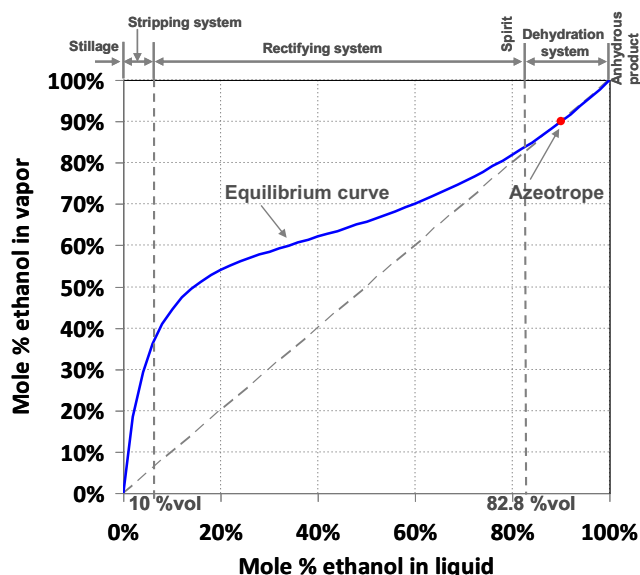


Figure 3. Structuring the distillation system strategy for ethanol–water separation.

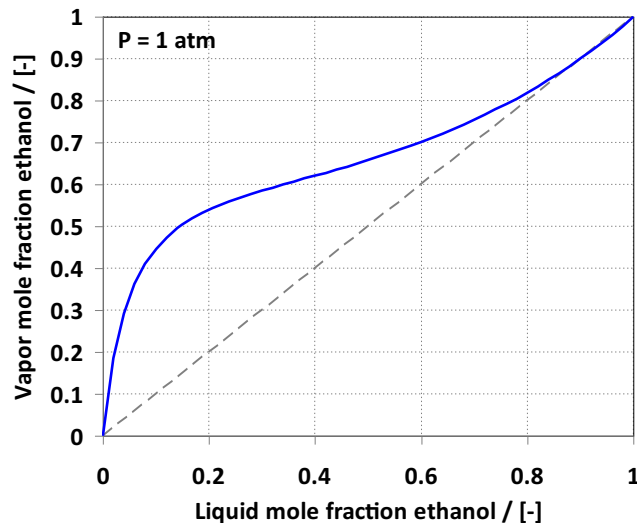
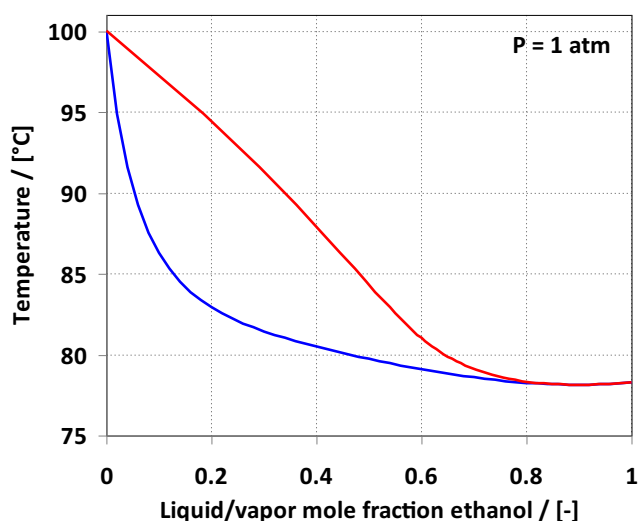


Figure 2. T – xy and y – x diagrams (molar fractions) for ethanol–water mixture (NRTL thermodynamic model).

(dilute broth, in the range of 5–15 wt%), a rectifying zone approaching the azeotrope (up to around the usually minimum acceptable hydrous ethanol mass fraction of 92.5 wt% or *ca* 82.8 mol%) and a dehydration zone for producing anhydrous ethanol beyond the azeotropic composition of *ca* 95.63 wt% or 89.4 mol%.⁶ This zonal perspective is essential for technology selection, as different separation technologies are optimally suited to different concentration ranges, and the energy penalty increases sharply in the rectifying zone as the azeotrope is approached. Crucially, for spirits production, the legal minimum product specification of 96 vol% (95.3 wt%) sits just below the azeotropic composition, meaning the dehydration zone is not required. This is a major simplification as compared to fuel-grade bioethanol production, where anhydrous ethanol (>99 wt%) is the target.

Key physicochemical properties governing separation behaviour are summarised in Table 1.¹² The difference in boiling points between ethanol (78.23 °C) and water (100 °C) is the primary driving force exploited by distillation. But the relatively small gap (combined with strong hydrogen-bonding interactions and non-ideal liquid phase) makes this one of the more energy-intensive binary separations encountered in the food and beverage industry. The vapor pressure of ethanol (5.95 kPa at 20 °C) is nearly twice that of water (3.17 kPa at 25 °C), reflecting a relative volatility that (while favourable at low concentrations) diminishes rapidly as the azeotrope is approached.

The practical implication is frequently overstated in the academic literature. Although the ethanol–water system is demanding in energy terms, it is routinely handled at scale in today's plants, so the challenge is not feasibility but efficiency.

Food-grade constraints

A critical distinction between spirits production and the far more extensively studied field of fuel-grade bioethanol separation is the absolute requirement for food-grade compatibility of all process materials and separation agents (as summarised in Table 2). In bioethanol production, breaking the ethanol–water azeotrope to reach anhydrous purity is routinely accomplished by azeotropic distillation using light entrainers (e.g. benzene or cyclohexane), or by extractive distillation with heavy solvents (such as ethylene glycol), both of which are proven at industrial scale.¹¹ These approaches are entirely prohibited in spirits manufacturing, where the product is destined for human consumption and where any trace of toxic solvent contaminants is unacceptable under food safety regulations. This constraint has key practical consequences.

Liquid–liquid extraction requires solvents (e.g. octanoic acid, octanol or iso-octanol) that are difficult to remove completely and whose food-grade status has limited approval and uncertain acceptability for large-scale potable usage.^{14,15} Also azeotropic distillation with light entrainers forming heterogeneous azeotropes is excluded, as the entrainers themselves pose health risks or sensory contamination risks.¹⁰ Pressure-swing adsorption (PSA) using zeolite 3A molecular sieves is a notable exception, as it is capable of producing anhydrous ethanol and fully food-grade compatible, as the zeolite does not contaminate the product chemically.¹⁶ However, the spirits industry does not require anhydrous purity, which reduces the attractiveness of PSA as a main separation technology.

Congener management

Congener management objectives differ between traditional batch spirits (controlled retention, outside the scope of this perspective) and continuous neutral-alcohol/food-grade production

(extensive removal, the focus here). Beyond the binary ethanol–water separation challenge, spirits production requires the simultaneous management of a complex mixture of volatile coproducts. During fermentation, a spectrum of congeners is generated alongside ethanol: fusel oils (propyl, butyl and amyl alcohols and their isomers), esters (mainly ethyl acetate), aldehydes (acetaldehyde), organic acids and methanol.^{9,11} These compounds partition between liquid and vapor phases, form binary and ternary azeotropes with ethanol and water and must be selectively concentrated and removed at specific points in the column train to achieve both purity and the desired flavour profile.¹⁰

From a product-quality standpoint, some congeners (esters and higher alcohols) are desirable in controlled amounts as they contribute to the characteristic aroma of each spirit category. However, others (e.g. high methanol, excessive fusel oils, sulfur compounds) are strictly constrained by regulation and off-flavour limits (Table 1). Dissolved CO₂ from fermentation is usually flashed off in upstream steps and does not materially affect the thermodynamic design of the distillation train, so it is not a major focus in this analysis. The challenge here is not to eliminate congeners, but to shape their profiles within relatively narrow, product-specific specification windows, despite variability between feedstocks and fermentation conditions.

Methanol is not a fermentation by-product in the strict sense but is released during mash processing via enzymatic demethoxylation of pectin, and its concentration in fruit spirits can be significant.⁹ Its boiling point (64.7 °C) is sufficiently different from that of ethanol to allow removal by conventional rectification, but this requires a dedicated demethylation column operating at high reflux ratios (adding to the overall energy usage). The practical implication is significant as any alternative separation technology considered for spirits production must prove that it preserves or enhances the ability to manage congener profiles. This is a constraint that has received insufficient attention in the literature on ethanol separation, which largely focuses on the binary ethanol–water system.^{3,10}

We argue that the systematic neglect of congener management in the PI literature is a critical gap between academic research and industrial applicability in this field. No alternative separation technology should be proposed for spirits production without explicit experimental proof of its behaviour towards the full congener spectrum. Yet, the overwhelming majority of published work on ethanol separation considers only the binary ethanol–water system. This is a barrier to industrial adoption that the research community should address as a first priority.

Note that rigorous models (e.g. customised NRTL parameters including key higher alcohols, esters and acids) have been shown to reproduce industrial spirits distillation behaviour with good accuracy.⁸ Such models indicate that accounting for congeners typically increases the calculated separation energy by *ca* 7% compared with idealised binary ethanol–water models.¹⁷ This reinforces the need to validate PI options under realistic multi-component conditions rather than relying on binary simulations alone.

Retrofit requirements

The main industrial challenge is economic and operational rather than thermodynamic. The majority of spirits distilleries worldwide operate existing capital-intensive infrastructure, with distillation column trains representing fixed investments with operational lifetimes of 30–50 years.¹ The practical question for most operators is not *what is the theoretically optimal separation process?*

Table 1. Key physicochemical properties and regulatory status of ethanol, water and main congeners in fermentation-derived spirits feeds

Compound	Class	Boiling point (°C) ^a	Vapour pressure at 20 °C (kPa) ^a	Rel. volatility <i>versus</i> ethanol ^a	Azeotrope with ethanol ^b	EU legal limit (mg L ⁻¹ AA) ^c	Typical conc. range (mg L ⁻¹ AA) ^d	Main removal mechanism in classic column train ^d
Water	Solvent	100.0	2.34	<1 (→0 near azeo.)	Min.-boiling azeo. at 95.63 wt% EtOH, 78.2 °C	n/a	n/a	Stripping column/bottom product
Ethanol	Product	78.2	5.95	1.00 (ref.)	–	Target product	n/a	–
Methanol	Head impurity	64.7	12.8	~2.2–3.0	No binary azeo. with EtOH. Ternary azeo. reported with EtOH–water	10 mg L ⁻¹ AA (vodka, neutral spirit). Up to 1000 mg L ⁻¹ AA (fruit spirits, e.g. plum)	4000–13 500	Dedicated demethylation column. High reflux ratio. Light-ends side draw
Acetaldehyde	Head aldehyde	20.2	98.7	>> 1 (~20–40)	Binary azeo. with EtOH at ~1.1 wt% AcAlD, 77.8 °C	≤1000 mg L ⁻¹ AA (neutral spirit)	~10–450	Heads removal. Partial condenser. Light-ends draw at top of rectification column
Ethyl acetate	Head ester	77.1	9.7	~1.5–2.0	Binary azeo. with EtOH at ~30.8 wt% EtAc, 71.8 °C. Ternary azeo. with EtOH–water	≤13 000 mg L ⁻¹ AA (fruit spirits). ≤1300 mg L ⁻¹ AA (neutral spirit)	~100–6600	Heads fraction removal. Side draw near top of rectification column
Diethyl acetal	Head acetal	103.6	2.1	~0.8–1.0	Binary azeo. with EtOH at ~47 wt%, 78.0 °C	No specific EU limit (monitored under aldehydes)	~0.46–3.3	Co-removed with heads fraction. Sensitive to column temperature profile
1-Propanol (<i>n</i> -propanol)	Light fusel	97.2	1.9	~0.7–0.9	Binary azeo. with water at 71.7 wt%, 87.7 °C. Ternary azeo. with EtOH–water	Combined fusel oils ≤2000 mg L ⁻¹ AA	~3200–4800 (jointly under higher alcohols)	Side draw in rectification zone (intermediate volatility). Fusel oil column
Isobutanol (2-methyl-1-propanol)	Fusel oil	107.9	0.95	~0.4–0.6	Binary azeo. with water at 66.8 wt%, 89.0 °C	Combined fusel oils ≤2000 mg L ⁻¹ AA	~3200–4800 (jointly under higher alcohols)	Side draw in lower rectifying zone. Fusel oil decanter/wash column
Isoamyl alcohol (3-methyl-1-butanol)	Fusel oil	131.1	0.28	~0.15–0.25	Binary azeo. with water at 49.2 wt%, 95.1 °C. Ternary azeo. with EtOH–water	Combined fusel oils ≤2000 mg L ⁻¹ AA	~3200–4800 (jointly under higher alcohols)	Main fusel oil side draw. Intermediate rectifying tray. Fusel oil wash column. Liquid–liquid phase split
Amyl alcohol (1-pentanol)	Heavy fusel	138.0	0.18	~0.10–0.18	Binary azeo. with water at 45.2 wt%, 95.8 °C	Combined fusel oils ≤2000 mg/L AA	~3200–4800 (jointly under higher alcohols)	Heavy fusel side draw near base of rectifying section. Fusel decanter

Table 1. Continued

Compound	Class	Boiling point (°C) ^a	Vapour pressure at 20 °C (kPa) ^a	Rel. volatility versus ethanol ^a	Azeotrope with ethanol ^b	EU legal limit (mg L ⁻¹ AA) ^c	Typical conc. range (mg L ⁻¹ AA) ^d	Main removal mechanism in classic column train ^d
Acetic acid	Organic acid	117.9	1.57	~0.25–0.40	Binary azeo. with water (not with EtOH). Heterogeneous azeo. at 97.4 wt% AcOH	No specific EU limit	~400–1400 (reported as total acids)	Bottom/tails fraction. Remains mostly in liq. phase at low concentrations
Furfural	Aldehyde	161.7	0.24	~0.08–0.12	Binary azeo. with water at 35.0 wt%, 98.3 °C	No specific EU limit (limit as aldehydes in some national regulations: up to 40–50)	~1.4–18	Tails fraction removal. Base of column or bottom product. Some removal <i>via</i> heads due to ternary behaviour at low conc.
Ethyl hexanoate/ethyl caprate	Heavy esters	167–245	<0.1	<0.05	No significant azeo. with EtOH	No specific EU limit	Trace tails-fraction	Remain in liquid phase (tails). Removed with fusel oil or bottom stream
Benzaldehyde	Aromatic aldehyde	178.1	0.13	~0.05–0.08	No strong azeo. with EtOH	No specific EU limit. Sensory threshold ~0.35 mg L ⁻¹ in spirit	~0.2–24 mg	Tails fraction. Difficult removal by distillation alone. Relevant mainly for fruit distillates
Hydrogen sulfide/DMS compounds	Sulfur compounds	–60 to 37	Very high	>> 1	Volatile. No relevant azeo.	No EU limit. Organoleptic threshold: 0.002–0.05 mg L ⁻¹	Not routinely quantified. Managed by organoleptic threshold, not conc. limit	Heads removal. Stripping column. Copper contact in still

Relative volatility (α) is given relative to ethanol at *ca* 78°C and atmospheric pressure. α values are approximate and concentration-dependent. Values are given for the rectifying zone (50–90 wt% ethanol). EU legal limits are from Regulation (EU) 2019/787 on spirit drinks (expressed as mg per litre of pure alcohol, mg L⁻¹ AA = anhydrous alcohol) unless otherwise noted. Category-specific limits apply. The values shown are for the most restrictive category (neutral spirit/vodka) unless fruit spirit limits are noted. 'Combined fusel oils' refers to the sum of 1-propanol, 2-methyl-1-propanol, 2-methyl-1-butanol and 3-methyl-1-butanol. All removal mechanisms and legal limits shown apply to continuous neutral-alcohol/food-grade production. Traditional batch-distilled spirits, where congener retention (not removal) is the objective, are outside the scope of this table and this perspective.

^a Source of boiling points, vapor pressure, relative volatility: Kadlec *et al.*¹²

^b Azeotrope composition/temperature data: Madson⁶ and Onuki *et al.*¹⁰

^c EU legal limits (mg L⁻¹ AA): Regulation (EU) 2019/787.

^d Congener class definition, typical concentration range, and removal mechanism: Spaho,⁹ Puentes *et al.*,⁸ Raičević *et al.*,¹³ Decloux and Coustel.¹¹

Table 2. Comparison of continuous neutral-alcohol/food-grade ethanol production and fuel-grade bioethanol production

Dimension	Continuous neutral-alcohol/food-grade production	Fuel-grade bioethanol production
Target purity	Non-anhydrous, typically ≥ 96 vol% (95.3 wt%). Legal minimum sits just below the azeotrope (95.63 wt%)	Anhydrous, typically > 99 wt%. Requires breaking the azeotrope
Permitted separation agents	Food-grade compatible only. No toxic entrainers or solvents permitted	Light entrainers (benzene, cyclohexane) or heavy solvents (ethylene glycol) permitted for azeotropic/extractive distillation
Food-safety requirements	Strict food-safety regulation. Zero tolerance for toxic residues in product. Regulated under spirits-drink frameworks (e.g. EU Regulation 2019/787)	No food-safety constraint. Determined by fuel-quality specifications (e.g. ASTM D4806)
Congener/methanol management	Extensive congener removal required (fusel oils, esters, aldehydes). Methanol strictly limited (e.g. ≤ 10 mg L ⁻¹ AA for vodka/neutral spirit) via dedicated demethylation columns	Congener removal not a primary design objective. Methanol content unregulated for fuel use
Sensory-quality requirements	Central to product acceptance. Flavour/aroma profile must meet category-specific organoleptic standards	Not applicable. No sensory specification
Retrofit considerations	Existing multicolumn trains (30–50 year asset lives) constrain technology choice. Retrofit compatibility a key selection criterion	Retrofit less constrained by food-contact/sensory requirements. Broader range of entrainer-based technologies available for upgrade
This comparison excludes traditional batch-distilled spirits, where congener retention (not removal) is the objective, and it is intended to clarify why separation technologies and reference data developed for fuel-grade bioethanol dehydration cannot be directly transposed to the continuous neutral-alcohol/food-grade case addressed in this perspective.		

But rather *what can be implemented within or alongside existing equipment at acceptable cost and without disrupting product quality?* This need for technologies that can be integrated into existing plants is a key evaluation criterion that differentiates this perspective from broader reviews of ethanol separation technology. Technologies that require new column designs or highly integrated configurations face higher barriers to adoption than those that can be implemented as add-ons or internals replacements to existing column shells.^{5,18,19}

In our view the retrofit imperative is actually an opportunity. As discussed in the next section, cyclic distillation and heat pump-assisted distillation (HPAD) retrofits require no new column shells, no changes to process chemistry and no regulatory requalification of the product. The capital and operational barriers are lower than those faced by the industry when adopting stainless steel vessels, automated control systems or clean-in-place decades ago (all adopted at scale). In economic terms, installing cyclic trays or/and a heat-pump loop is typically an internals-plus-auxiliaries project, requiring only a fraction of the investment associated with fabricating, installing and integrating an entirely new column train. Literature case studies consistently report that the additional CAPEX of such retrofits is recovered within a few years via 20–40% reductions in energy-related operating costs.

TECHNOLOGY ASSESSMENT

The separation technologies available for food-grade ethanol production cover a wide range of maturity levels, operational principles and applicability to the spirits context. This section organises the technology landscape into several tiers based on TRL. This was chosen as the main criterion as it determines the deployment timeline and commercial risk profile, which is most relevant to an industrial producer. Each technology is evaluated against the key criteria established earlier: food-grade compatibility, congener management capability and retrofit feasibility. A qualitative comparison of all technologies is provided in Table 3.

Reference-process definition and baseline conventions

Quantitative energy-saving claims in this paper are reported relative to two explicit baselines, since spirits producers operate a mix of both. The primary baseline is single-effect atmospheric distillation without heat integration, consuming 3.0–4.2 kg of steam per litre of 96° GL ethanol.^{6,11} This is the baseline against which all percentage savings for cyclic distillation, HPAD (MVR/TVR/VC/CRHP), HIDIc, HD-PV and PTD are expressed unless explicitly stated otherwise. The secondary, intermediate baseline is multi-effect distillation (2–3 effects), which is already implemented in many industrial plants today and reduces thermal energy demand by roughly 40–60% relative to the single-effect baseline through inter-column heat recovery and vapor-side reboilers. Because a plant already operating multi-effect distillation has a smaller further-savings potential than one operating a single-effect column, the savings of other technologies are reported against the single-effect baseline, with savings relative to the multi-effect baseline annotated in parentheses where the underlying case-study data allow this second comparison to be made.

Deployment-ready technologies

These are technologies with demonstrated industrial viability, for which the primary barrier to adoption is commercial awareness and willingness to invest rather than technical risk. The commercial maturity of cyclic distillation and HPAD is further corroborated by about 20 patents filed over the past two decades covering tray designs, compressor configurations and integrated column internals, providing an independent confirmation of the TRL 7–9 classification beyond the peer-reviewed literature.

Cyclic distillation

Cyclic distillation is readily available to spirits producers operating conventional tray columns.²⁰ Unlike classical distillation (where liquid and vapor flow at the same time through the column), cyclic distillation operates through two alternating phases: a vapor flow period in which vapor rises through the column while

Table 3. Qualitative comparison of separation processes for food-grade ethanol production in spirits manufacturing versus single-effect distillation baseline

Technology	Key advantages	Main drawbacks	Energy use (versus classic)	TRL	Food-grade compatible	Ref.
Conventional distillation/rectification	Simple, robust, well-understood. No additives required. Full congeners management via side draws	Low thermodynamic efficiency (5–18%). High steam consumption (3.0–4.2 kg L ⁻¹). Large equipment footprint	Baseline	9	Yes	6, 11
Azeotropic distillation	Can break azeotrope. Proven at industrial scale	Requires light entrainers (benzene, cyclohexane). Toxic and incompatible with food-grade standards. Azeotrope limit applies	Similar or higher	9	No	11
Extractive distillation	Effective azeotrope breaking. Proven at scale	Heavy solvents (ethylene glycol, etc.) not food-grade. Solvent recovery column required	Similar	9	No (most solvents)	11, 15
Pressure swing/vacuum distillation	No solvents. Azeotrope disappears below ~11.5 kPa. Proven technology	Large equipment. High reflux ratio. Deep vacuum costly. Large steam consumption	Similar or higher	9	Yes	16
Cyclic distillation	~30% energy and CapEx savings. Retrofit compatible. No additives. Full congener management kept	Requires specific tray internals and control system. Limited supplier base (single provider). Much of the supporting pilot- and industrial-scale literature is co-authored by that provider (MaletaCD)	–30%	7–9 (industrial-scale data)	Yes	20, 18, 19, 21
MVR heat pump	40–70% energy savings. Retrofit compatible. Proven at scale	Mechanical compressor required. Higher CapEx. Less effective for large temperature lifts	–40 to –70% (0 to –40% versus multi-effect baseline)	9	Yes	22, 23
TVR heat pump	Lower CapEx than MVR. Simple steam ejector. Retrofit compatible	Lower energy savings than MVR. Steam ejector requires high-pressure steam	–10 to –30%	9	Yes	22
HIDiC	Up to 77% energy savings. Proven at industrial scale (SuperHIDiC)	Cannot retrofit existing columns. High CapEx. Specialised design required	–50 to –77% (–15 to –55% versus multi-effect baseline)	9 (SuperHIDiC, by Toyo Eng.)	Yes	24, 25
Pervaporation	No azeotrope limitation. Low energy for dehydration. No solvents	Cannot produce high purity alone. Membrane fouling by congeners. Limited lifetime	–30 to –50% (dehydration only)	6–9 (component suppliers)	Yes	26, 27
Hybrid distillation–membrane separation	<50% of azeotropic distillation energy. No mass separating agents. Food-grade compatible	Membrane behaviour with congeners undercharacterized. Moderate CapEx. TRL variable by configuration	–50 to –60% versus AD/ED	6–9 (component-level)	Yes	28, 29, 30
Pressure-swing adsorption (PSA)	Anhydrous purity achievable (<50 ppm H ₂ O). Food-grade (zeolite 3A). Proven at scale	Only needed for anhydrous ethanol (not required for spirits). Two-column cycling complexity	–20 to –40% (dehydration)	9	Yes	16
Liquid–liquid extraction (LLX)	Effective for dilute feeds. Proven technology	Two-step process. Cannot reach high purity alone. Food-grade solvent options very limited	Similar	6–9	Limited	15
Hybrid distillation–LLX			–30 to –35%	6–7	Limited	14, 15

Table 3. Continued

Technology	Key advantages	Main drawbacks	Energy use (versus classic)	TRL	Food-grade compatible	Ref.
Pass-through distillation (PTD)	Significant savings for feeds > 10 mol% ethanol. Reduced total investment (~34%) and OpEx (~32%) Up to 50% energy savings. Low-temperature operation. Concurrent fermentation possible. Food-grade brine Equipment size reduction by 4–7 times. Fast start-up. Suitable for modular/offshore units	Solvent food-grade compatibility uncertain. Two-step process. Additional distillation for solvent recovery TRL 6 – pilot scale only. Brine management required. Limited industrial track record	–40 to –50%	6 (DryStill pilot)	Yes	31, 32
HiGee distillation	Equipment size reduction by 4–7 times. Fast start-up. Suitable for modular/offshore units	No energy savings. Additional electricity for rotor. Mechanical complexity. Reliability concerns	+5 to +10%	6–9 (≤5 for ethanol–water)	Yes	33
Membrane distillation (MD)	No solvents. Low-grade heat usable. Applicable to dilute streams	Not suitable as standalone for high-purity ethanol. Membrane wetting risk. Low flux	Similar or higher	4–7	Yes	27
Microwave-assisted/ MMD	Enhanced flux and separation factor versus conventional MD. Reduced energy versus conventional MD	TRL 2–4. No industrial validation. No quantified energy savings at process scale	Speculative	2–4	Unknown	34, 35
MW-induced spray distillation	Pilot-scale validated. Relative volatility of ethanol more than doubled versus classic	No process-scale energy/cost data. Pilot-scale only, single reported study	Speculative (no process-scale data)	3–4	Unknown	36
Microbubble distillation stripping	Separation factor ~ 12.3 at 10 vol %. Low liquid temperature. Suitable for thermally sensitive broths	TRL 3–5. Continuous operation not demonstrated. Energy economics unquantified	Speculative	3–5	Likely	37, 38

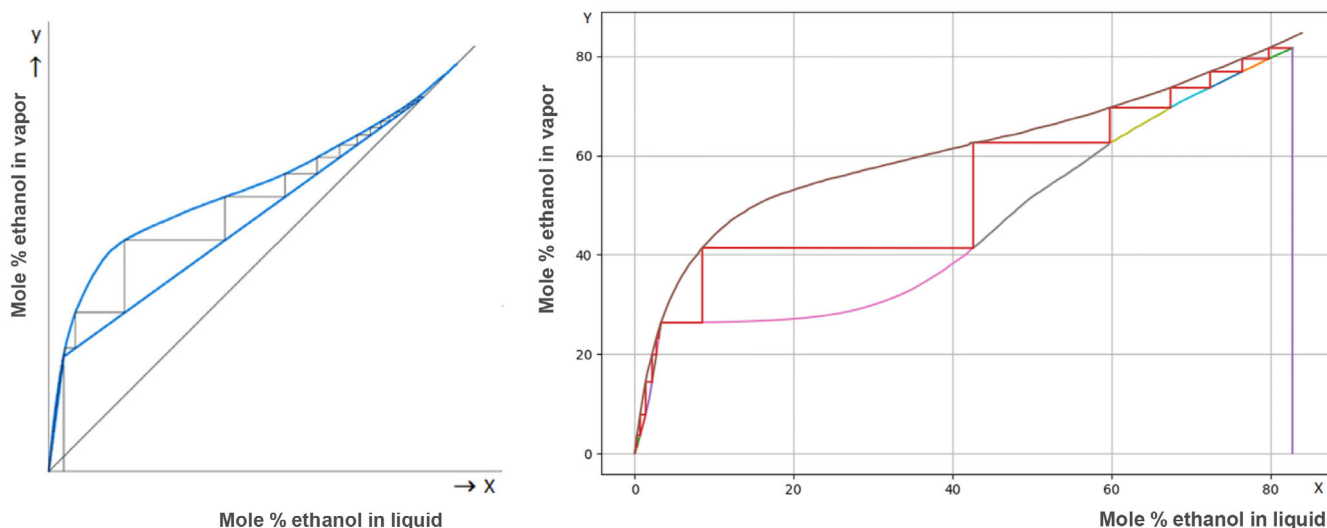


Figure 4. Y-x diagrams for ethanol-water separation for classic distillation (left) and cyclic distillation (right).¹⁹

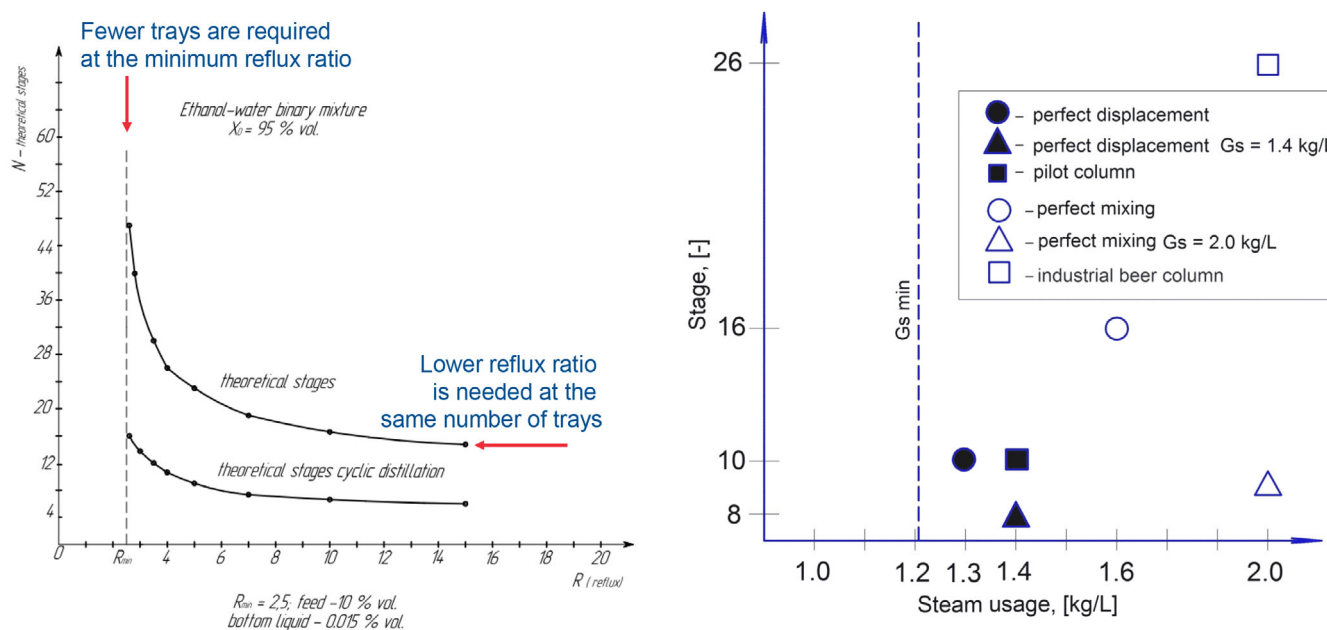


Figure 5. Ethanol-water separation efficiency in a cyclic distillation column (perfect displacement mode) versus classic distillation (perfect mixing mode).¹⁸

liquid remains stationary on each tray, followed by a liquid flow period in which all liquid drops simultaneously to the tray below while vapor flow is interrupted.^{18,19} This separate phase movement restructures the mass transfer mechanism on each tray, eliminating the back-mixing losses inherent in conventional operation and delivering a steeper operating line in the VLE diagram (Fig. 4).¹⁹ The practical consequences are substantial.

For a given separation specification, cyclic distillation requires fewer theoretical stages than classic operation, typically 1.5–2 times fewer trays, with apparent Murphree tray efficiencies over 200% as cyclic operation fundamentally changes the contacting pattern compared to the crossflow assumption underlying the classical definition.²⁰ It also uses *ca* 30% less steam, translating to equivalent reductions in both capital expenditure (CapEx)

and operating expenditure (OpEx). Figure 5¹⁸ demonstrates this advantage, showing the improved separation efficiency and reduced steam consumption relative to a classical column operating on the same ethanol–water system. Critically, the technology is fully food-grade compatible. No solvents or mass separating agents are involved, and the separation mechanism is purely thermodynamic. Congener management is preserved, as the tray-by-tray liquid movement allows side draws at specific tray positions in the same manner as conventional columns.

A complete industrial flowsheet for food-grade ethanol production using cyclic distillation is shown in Fig. 6,^{19,21} including a multicolumn train (C1–C7) with dedicated columns for stripping, rectification, extractive dilution, fusel oil removal and methanol separation. This flowsheet demonstrates that the technology

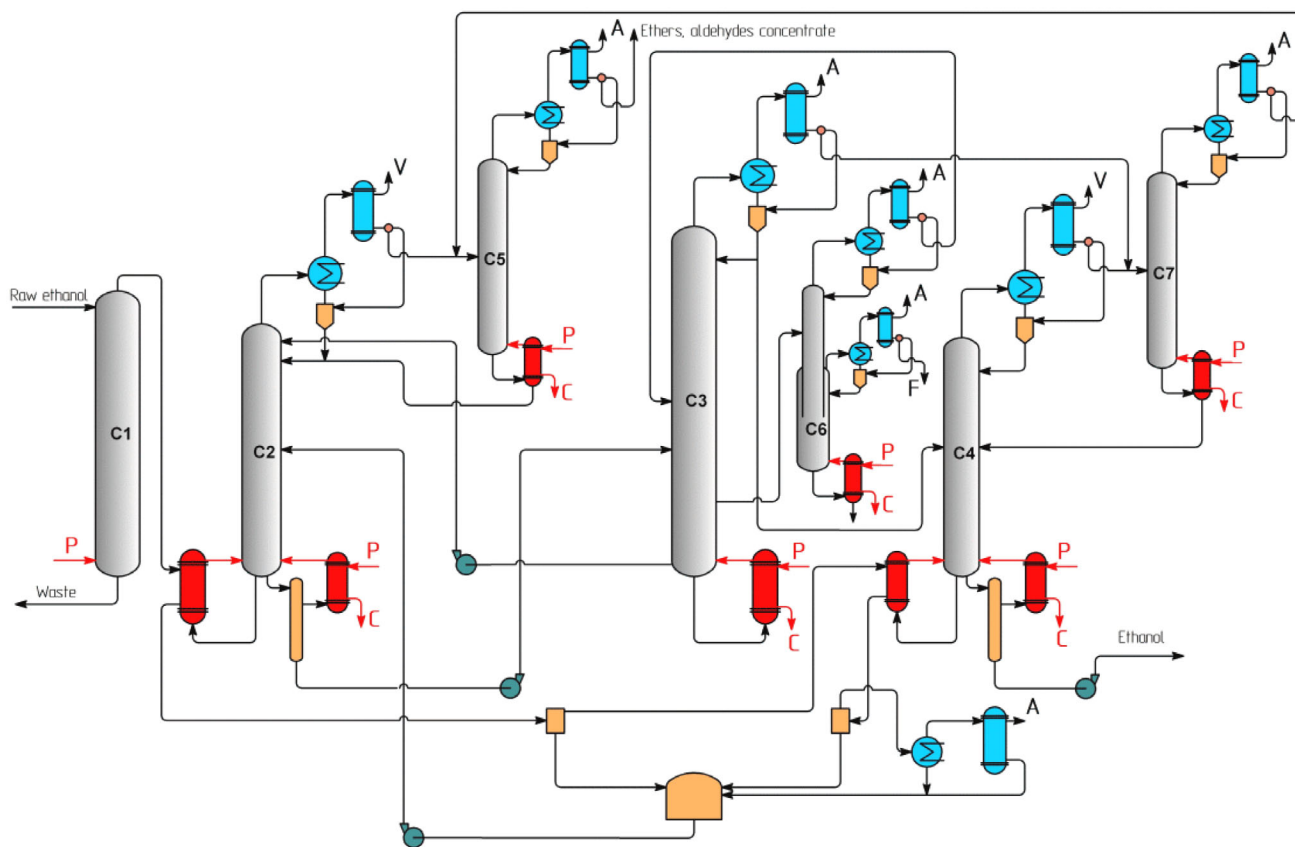


Figure 6. Flowsheet of an ethanol food-grade production plant: C1 – beer column, C2 – hydro-selection column, C3 – rectification column, C4 – column for end-cleaning, C5 – column for concentrating impurities, C6 – fusel column, C7 – methanol column, P – steam, C – condensate, A – atmospheric, V – vacuum, F – fusel alcohol.^{19,21}

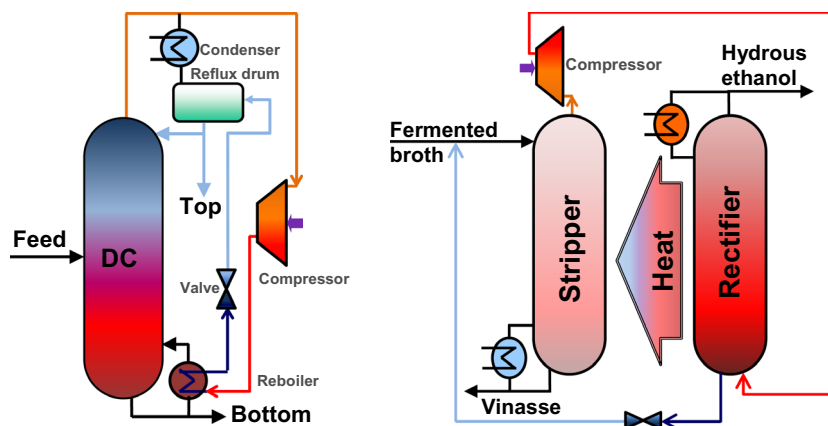


Figure 7. Schematic representation of MVR (left) and HIDiC (right) configuration for hydrous ethanol production (adapted from Kiss *et al.*,²² Kiss and Olujic²⁴).

integrates naturally into the conventional multicolumn architecture of spirits plants. Most significantly for existing producers, cyclic distillation can be implemented as a retrofit (e.g. columns of 5–42 trays, diameters 0.4–1.7 m) by replacing conventional tray internals with purpose-designed cyclic operation trays and adding the cyclic control system, without replacing the column shell.^{18,19} Industrial-scale operating data from a continuous food-grade alcohol plant confirmed energy reductions of *ca*

30% alongside a 3–4% increase in alcohol yield (due to a reduction from the 3–6% losses down to *ca* 1–1.5% typical of a well-optimised column) due to higher separation purity, with impurity removal at higher concentrations reducing waste output, in line with theoretical predictions.¹⁹ This confirms that cyclic operation can enhance congener removal and ethanol yield simultaneously.

Cyclic distillation is a compelling near-term opportunity for spirits separation, due to the combination of proven industrial

scale, 30% energy savings, full food-grade compatibility, retrofit applicability and documented experience in the food-grade alcohol sector. However, it should be noted that industrial supply is currently concentrated in a single commercial provider (associated with much of the cited pilot/industrial literature), so broader independent validation and additional suppliers would strengthen the evidence base.

Heat pump-assisted distillation

Conventional distillation is thermodynamically inefficient because it consumes high-quality steam in the reboiler and rejects the heat uselessly in the condenser. HPAD addresses this waste by upgrading the low-temperature condenser heat and returning it to the reboiler, thereby significantly reducing external steam demand.²² For the ethanol–water system, the small temperature difference between the reboiler and condenser (ca 22 °C, Q/W ratio > 16) means that the coefficient of performance (COP) of the heat pump is favourable, making HPAD very attractive for this application.

Several configurations are applicable to spirits production. Mechanical vapor recompression (MVR) takes the overhead vapor directly, compresses it and uses it as the reboiler heat source (Fig. 7, left). This is the simplest and most energy-efficient configuration, delivering energy savings of 40–70% relative to conventional operation and proven at industrial scale (TRL 9).²³ Thermal vapor recompression (TVR) uses a steam ejector rather than a mechanical compressor, offering lower capital cost and simpler maintenance at the expense of somewhat lower energy efficiency, with savings of 10–30%.²² Vapor compression (VC) using a closed refrigerant cycle offers greater flexibility when the overhead vapor is unsuitable for direct recompression, at the cost of a somewhat lower coefficient of performance than MVR.²³ Compression–resorption heat pump (CRHP) replaces the single-component working fluid with a binary mixture (typically NH_3 – H_2O) that allows wet compression and a gliding profile that better matches the column's temperature gradient. This reduces the compression work required for a given heat duty and extends the economically viable temperature lift range beyond that of standard MVR, making it attractive for column configurations with a larger reboiler–condenser temperature difference.²³

Heat pump-assisted technologies are fully food-grade compatible and retrofit-friendly. An existing column can be upgraded by adding the compressor loop and some heat exchangers without modifying the column itself. Pinch analysis is recommended prior to implementation to identify the optimal integration point and quantify achievable savings for a specific column configuration.²² Heat pump-assisted retrofits should be considered standard for any new column investment in spirits production of the order of several million litres per year (typically $>5 \times 10^6 \text{ L yr}^{-1}$). The energy savings of ca 40–70% are achieved with mature, compressor technology that operates at similar scales (e.g. in refrigeration, industrial gases and large-scale food processing). From a cost and decarbonisation perspective, HPAD shifts part of the energy demand from boiler fuel to electricity, so the net economic and CO_2 benefit depends on the local electricity–fuel price ratio and grid carbon intensity.

Internally heat-integrated distillation column (HIDiC)

HIDiC takes the heat pump concept further by integrating the heat exchange internally between the rectifying and stripping sections of the same column, which are operated at different pressures. The rectifying section operates at higher pressure and thus

higher temperature than the stripping section, enabling direct heat transfer from rectification to stripping section.²⁴ As illustrated in Fig. 7(right), this configuration eliminates or drastically reduces the external reboiler duty. For ethanol–water separation, energy savings of up to 77% have been demonstrated relative to conventional distillation.^{24,25} This shows that a higher column and compressor investment is typically recovered within 3–6 years due to the high energy savings.

HIDiC is proven at industrial scale (TRL 9), with several commercial implementations by Toyo Engineering in Japan under the SuperHIDiC brand, though these installations are in other separation duties (e.g. high-purity alcohols, hydrocarbons) and no SuperHIDiC unit has yet been reported for ethanol–water spirits separation specifically. It is fully food-grade compatible and delivers the highest energy savings of any thermally based intensification route. However, HIDiC requires purpose-designed column equipment and cannot be implemented as a retrofit to existing column shells. Thus it is mainly a technology for new plant construction or major capital renewal cycles. Literature case studies indicate that HIDiC typically requires a modest increase in column and compressor capital cost but reduces reboiler duty by roughly 40–70% as compared to a conventional distillation column, so that the operating savings dominate lifecycle economics.²⁴

Given that SuperHIDiC's only reported industrial application is outside ethanol production, and given ongoing debate in the literature regarding HIDiC's advantages relative to external heat-pump integration,^{24,39} HIDiC should be evaluated case-by-case against HPAD options at the design stage, rather than assumed as a default. An industry that constructs a new conventional distillation column train today, without evaluating either HIDiC or HPAD, risks locking in decades of higher-than-necessary energy use and associated carbon cost.

Pressure swing adsorption

For completeness, PSA with zeolite 3A is mentioned as the only adsorption-based technology that is both food-grade compatible and commercially mature (TRL 9). Its operating principle based on selective water adsorption on molecular sieving, requires no solvents, leaves no chemical residue in the product and is proven at large industrial scale. Its limitation in the spirits context is the scope, as PSA addresses only the dehydration zone (>95.6 wt% ethanol), a zone that most spirits producers do not need to enter. For a producer requiring anhydrous purity, PSA is the right technology choice. But for the majority of spirits operations, it is irrelevant to the primary energy-reduction objective.

Near-term technologies

These technologies are validated at pilot or demonstration scale (TRL 5–7) and are approaching industrial readiness, but require further development or commercial scale-up before they can be considered standard industrial practice in spirits production.

Hybrid distillation–membrane separation (pervaporation and vapor permeation)

Hybrid distillation–pervaporation (HD-PV) combines classic rectification with membrane-based pervaporation in a tightly integrated flowsheet. As shown in Fig. 8, the distillate stream from the rectification column (near the azeotropic composition) is fed to a hydrophilic pervaporation membrane module, which selectively permeates water while retaining ethanol in the retentate.²⁶ The retentate, enriched beyond the azeotrope, is recycled to the column, while the water-rich permeate is processed separately.

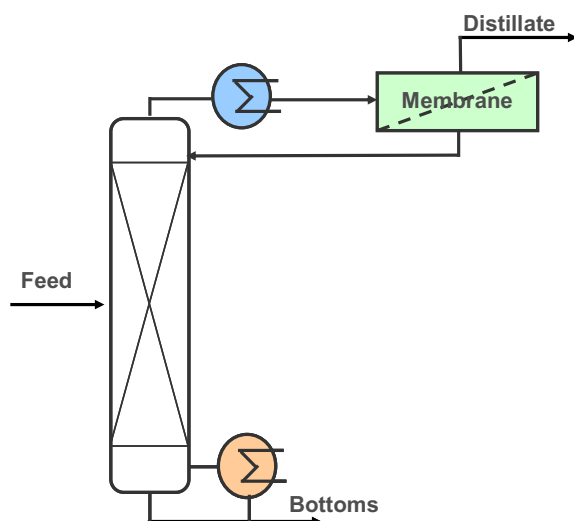


Figure 8. Hybrid distillation–pervaporation process (adapted from Tgar-guifa and Abderafi²⁶).

This configuration effectively bypasses the azeotropic barrier without any mass separating agent, preserving full food-grade compatibility. An alternative to PV is reverse osmosis (RO). A feed pressure of up to 50 bar is not itself prohibitive, being comparable to that of seawater desalination (50–75 bar) with modest pressurisation energy. The limiting factor is instead the achievable ethanol concentration in the permeate/retentate at such pressures, which remains far below spirits-grade purity targets, making RO unsuitable as a standalone technology for this application.²⁷

Pervaporation and vapor permeation are frequently discussed together in the literature but differ in a key respect. Pervaporation processes a liquid feed directly across the membrane, whereas vapor permeation processes a vapor-phase feed, typically drawn from the rectification column's vapor stream at or near the azeotropic composition, avoiding the additional latent-heat penalty of condensing and re-evaporating the feed. This difference has key implications for both energy performance and technology maturity. Vapor permeation for bioethanol dehydration is commercially mature (TRL 9), with more than a dozen full-scale installations at US fuel-ethanol plants using Whitefox's ICE membrane system. Liquid-fed pervaporation validated specifically on congener-containing, spirits-grade feeds remains less mature (TRL 6), as the industrial vapor-permeation record to date is concentrated in fuel-ethanol dehydration (not in food-grade or spirits applications). The rest of this subsection distinguishes between the two where the underlying evidence differs.

In practice, HD-PV is most beneficial when the rectification column already produces a high-strength distillate. Hydrophilic PV membranes for ethanol dehydration have been evaluated on feeds of 82–96 wt% ethanol, with separation factors up to the 10^3 range, so they are well suited to polish ≥ 90 wt% feeds towards very high-purity product. The hybrid HD-PV configuration reduces the column's reflux ratio, lowering both reboiler duty and column diameter.²⁶ For spirits production, HD-PV is most relevant when product specifications approach or exceed the azeotropic composition, or when dehydration to high purity is required for spirit base preparation. Hybrid processes drawing on TRL 6–9 component technologies are available from established suppliers (e.g. Pervatech, DeltaMem, Sulzer, BORSIG Membrane Technology, GreenPebble Technologies), but fully

integrated HD-PV systems specifically validated for spirits-grade feeds remain at TRL 6.

Pilot-scale and technoeconomic studies on PV-assisted separations report energy savings of 30–50% compared to solvent-based azeotropic distillation.^{28,29} A recent study comparing multiple hybrid distillation–membrane configurations for bioethanol distilleries reported energy savings of up to about 70% relative to conventional distillation trains, with vapour permeation generally outperforming pervaporation.³⁰ Yet, relative to conventional rectification alone, the saving is lower and depends on the target purity: about 10–30% for purities near 90–95 wt%, rising towards 50–70% only when the hybrid configuration replaces extractive or azeotropic dehydration entirely. HD-PV becomes most energy-advantageous only when the product specification approaches or exceeds the azeotropic composition. The specific investment costs become competitive at medium-to-large scales when membranes achieve multiyear lifetimes.

The key technical challenge for food-grade applications is membrane durability and selectivity in the presence of congeners, as fusel oils, esters and aldehydes can foul or plasticise polymeric membranes. Fouling data of hydrophilic ethanol-dehydration membranes exposed to fermentation-derived spirits feeds are not yet available in the open literature. Yet, studies on hydrophobic membranes used in alcohol recovery from broths indicate that organic by-products can reduce pervaporation flux by 20–50%.³ This highlights the need to characterise the impact of congeners in spirits-grade feeds on membrane performance, under realistic fermentation-derived alcohol feeds.

Pass-through distillation

Pass-through distillation (PTD) is a sorption-assisted distillation process based on an open-loop absorption–desorption cycle that decouples the evaporation and condensation steps, allowing them to operate at different pressures and temperatures. As illustrated in Fig. 9,³¹ a liquid feed is evaporated at low pressure and low temperature, the vapor is absorbed into a concentrated hygroscopic brine solution (e.g. LiBr or LiCl) and the absorbed components are subsequently desorbed and condensed at higher pressure using external heat. The heat of absorption is recovered and reused for evaporation, enabling energy savings of up to 50% relative to conventional distillation when coupled with multi-effect distillation or mechanical vapor recompression.³¹

PTD is attractive for two reasons in the spirits context. First, the low operating temperatures make it suitable for thermally sensitive materials, preserving congener profiles that might be altered by high-temperature conventional distillation. Second, it enables concurrent alcohol recovery and fermentation, by the continuous removal of ethanol from the fermenter as it is produced. This circumvents product inhibition and can improve fermentation yield and efficiency.³² PTD has been validated at pilot scale by DryStill (Canada) and is currently at TRL 6, being a credible near-term option for new plant designs where concurrent fermentation–separation integration is desired.

Within the portfolio of options considered here, PTD is arguably the most disruptive concept as it shifts the relationship between fermentation and separation from sequential to concurrent. Deployment in spirits production will also require resolution of several practical issues specific to this context, including: food-contact approval of LiBr or LiCl brine under EU and FDA food safety frameworks, materials of construction for brine-side equipment (corrosion resistance), control of brine entrainment into the product vapor stream and minimum batch or fermenter size needed to make concurrent fermentation–separation integration

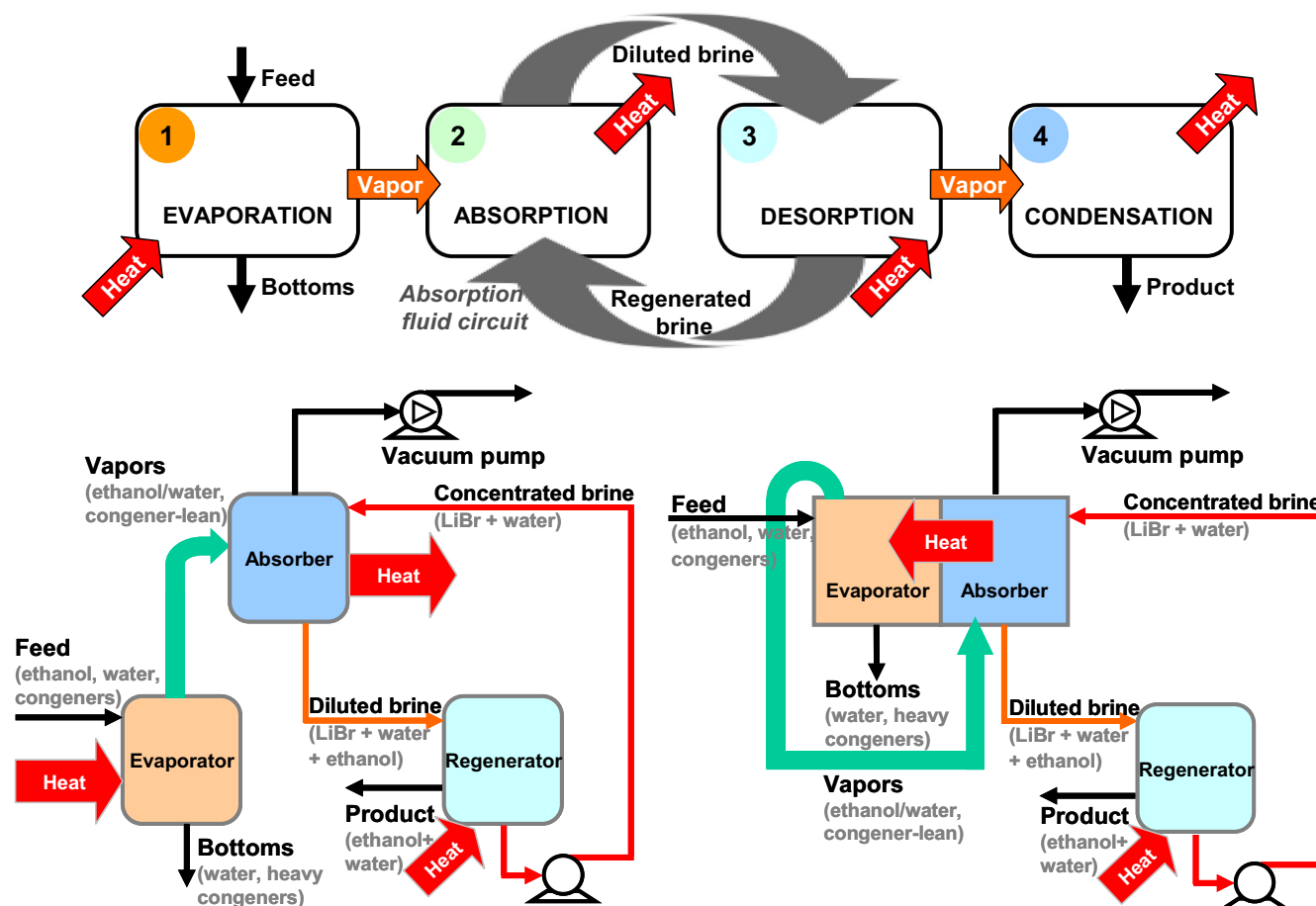


Figure 9. Working principle of PTD (top), and schematics of a PTD process (bottom): without and with thermally integrated evaporation and gas absorption.³¹

economically viable. As with extractive solvents, the brine's food-contact status must be established before industrial deployment. Unlike octanoic acid or glycols, LiBr/LiCl does not directly contact the vapor-phase product stream, but brine carryover/entrainment remains a food-safety risk requiring validation. These are engineering and regulatory challenges that should be resolved before the technology can be considered for food-grade deployment.

Emerging technologies

Several disruptive separation concepts (at TRL 2–5) have been reported in the literature that offer potentially major advantages but remain at early development stages, with insufficient experimental or simulation data to quantify energy savings or industrial feasibility reliably.

- HiGee distillation uses a rotating packed bed to replace gravitational force with centrifugal acceleration of $100\text{--}1000 \times g$, dramatically reducing equipment size by a factor of 4–7 and achieving HETP values as low as 2–8 cm compared to *ca* 30–60 cm for conventional packing.³³ For an ethanol–water system, experimental results with a rotating zigzag bed confirmed a height of a transfer unit of 2 cm. However, HiGee distillation offers no energy savings. On the contrary, additional electrical energy is required to drive the rotor. So far, the technology's mechanical complexity, rotating seals and reliability concerns have deterred major separation equipment suppliers

from commercialising it. It is best suited to niche applications where equipment footprint is the dominant constraint, such as offshore or modular mobile distillation units, and is at TRL 6–9 for specific applications.

- Microwave (MW)-assisted pervaporation and MW-induced membrane distillation exploit the different dielectric properties of ethanol and water under MW irradiation to enhance membrane flux and separation factors. Studies with carbon nanotube-immobilised membranes under MW heating reported ethanol fluxes of up to $11.3 \text{ L m}^{-2} \text{ h}^{-1}$ and separation factors of 13.7, which are increases of 46% and 102% respectively compared to conventional heating.³⁴ A more recent mechanistic study³⁵ on graphene oxide membranes reported substantial MW-induced increases in water and ethanol permeation flux, and distinguished dielectric heating effects on the bulk feed liquid from MW effects on molecules diffusing within the two-dimensional membrane channels themselves. This mechanistic separation is important for interpreting the carbon nanotube-based membrane results of Gupta *et al.*³⁴ and should guide future mechanistic studies of MW-assisted membrane separation for ethanol–water systems. While promising, these results are based on laboratory-scale experiments at low concentrations (5–15 vol% ethanol), and no quantified energy or cost comparisons at process scale are available. These technologies remain at TRL 2–4.
- MW-induced spray distillation addresses the low evaporation capacity and limited contact area that constrained earlier MW-induced distillation concepts, using a spray configuration

validated at pilot scale.³⁶ The relative volatility of ethanol in the azeotropic ethanol–water system was reported to more than double relative to conventional operation. While still at an early stage (TRL 3–4) and lacking process-scale energy/cost data, this pilot demonstration is a key step beyond laboratory-only MW-membrane concepts.

- Microbubble distillation stripping heats the gas phase rather than the liquid phase, generating a dense cloud of hot-air microbubbles that enhance ethanol removal from dilute fermentation broths while maintaining low liquid temperatures. This is beneficial for thermally sensitive feeds.³⁸ A separation factor of 12.3 at 10 vol% ethanol has been reported, and the technology shows promise for *in situ* bioethanol recovery from active fermenters. However, continuous operation has not yet been demonstrated, and the energy economics at industrial scale remain unquantified (TRL 3–5).

While all these technologies are scientifically interesting, they are unlikely to reach industrial deployment in spirits production within the next decade. This is in line with the technology-specific findings, namely that HiGee offers no net energy savings, MW-assisted membrane and microwave-induced spray distillation processes still lack process-scale energy data and microbubble stripping's energy economics are unquantified. Thus, these technologies are unlikely to deliver quantifiable energy benefits at industrial scale within the next decade, independent of their TRL. Instead of further academic study of these concepts in isolation, the research effort should be shifted towards the validation of higher TRL technologies under food-grade, congener-containing industrial conditions. The field has an excess of novel laboratory concepts and a deficit of industrial validation data, and this imbalance should be corrected as a matter of research priority.

DECISION FRAMEWORK FOR TECHNOLOGY SELECTION

The technology landscape includes solutions ranging from immediately deployable retrofits to speculative laboratory concepts. The breadth of options is a challenge and an opportunity for any spirits producer looking to reduce energy use and carbon footprint. Without a structured selection framework, there is a clear risk of either over-investing in immature technology or over-looking proven solutions. This section proposes a practical decision framework built around plant-specific variables that most strongly determine which technology or combination of technologies delivers the best outcome: (1) feed ethanol concentration, (2) target product purity, (3) plant scale and capital availability and (4) new plant design or a retrofit of existing infrastructure. Table 4 synthesises the decision framework into an explicit mapping between these variables and recommended PI technologies or combinations. For each region of this design space, the table indicates which technology provides the most favourable balance of energy savings, retrofitability and capital cost. Table 5 provides a summary of the quantitative performance data from industrial and pilot case studies behind this framework.

Feed concentration and target purity

Ethanol concentration of the fermentation broth entering the distillation train is the most basic process variable, as it determines the separation load and the number of theoretical stages required. Figure 3 provides the thermodynamic basis for this

dependency, showing that the stripping zone (5–15 wt% feed) and rectifying zone (up to ca 95.6 wt%) have distinct energy profiles and respond differently to PI interventions.⁶

For dilute feeds (5–10 vol% ethanol, typical of grain- or molasses-based fermentation), the stripping section dominates the energy use and the greatest gains are achievable by improving vapor–liquid contacting efficiency in this zone. Cyclic distillation is directly applicable here, delivering ca 30% steam savings with minimal process modification.¹⁸ HPAD is equally effective for this concentration range, with energy savings of 40–70% achievable when the temperature lift across the column is small, as is the case of the ethanol–water system.²²

For product purity approaching the azeotropic limit (>95 vol%), the rectifying zone becomes the energy bottleneck, and the reducing relative volatility in this region makes conventional intensification less effective. In this regime, hybrid distillation–membrane separation becomes most advantageous, as the membrane unit selectively removes water at compositions where distillation alone struggles.²⁶ It is important to note that most spirits producers targeting 96.2 vol% (95.3 wt%), just below the azeotrope, do not require azeotrope-breaking technology. For this majority, cyclic distillation and heat pump retrofits alone are sufficient and are the most cost-effective first step.

The widespread perception among spirits producers that azeotrope-breaking technology is required for high-quality rectification is untrue for the vast majority of production targets. This is especially the case for any plant targeting ≤96.2 vol% (95.3 wt%), which is the legal standard for hydrous ethanol and sits just below the azeotrope in the rectifying zone of the VLE diagram.⁶ As long as the required product purity remains at or below this level, conventional rectification (intensified with cyclic operation and/or heat pumps) is thermodynamically capable of meeting the specification without extractive or azeotropic entrainers. Yet, this misperception has led to unnecessary conservatism in technology selection and (in some cases) to the evaluation of extractive distillation (ED) and PSA options that are either food-grade incompatible or just redundant at this purity target. Correcting this issue is a practical contribution of this perspective, which directly reduces the perceived complexity of the technology selection problem for most spirits producers.

In practice, many spirit producers still operate close to the azeotropic purity and then dilute the product to bottling strength, which is thermodynamically suboptimal but historically driven by flavour management and regulatory practice. In the longer term, concepts such as PTD and membrane-assisted rectification could enable more flexible operating points (e.g. lower rectification purity combined with *in situ* or post-column adjustment) that reduce this processing waste without compromising product quality. However, such configurations have not yet been validated at industrial scale for spirits.

Plant scale and capital availability

The following scale thresholds are proposed as indicative benchmarks, derived from reported capital cost and payback period data in the literature.^{18,22,25} Plant-specific configurations, local energy prices and existing heat integration infrastructure will shift the economics in either direction, and site-specific techno-economic evaluation is recommended before any capital decision.

Plant scale significantly affects the economics of each technology option. At large industrial scale ($>10 \times 10^6$ L yr⁻¹), HPAD retrofits can reduce reboiler duty by roughly 40–60% and typically achieve payback times in the range of about 2–4 years,

Table 4. Comparison of PI technologies for food-grade ethanol separation in spirits production, in terms of retrofit compatibility, investment level, energy savings, payback period, minimum economic scale and congener management status

Technology	TRL	Retrofit to existing column? Retrofit compatibility score ^a	Investment level	Typical energy savings	Payback period (years)	Minimum economic scale	Congener management status
Cyclic distillation	7–9	Yes. Tray internals replacement, no new shell. RC-Score: 5	Low-medium (internals + control system)	~30% steam reduction. ~30% CapEx reduction for equivalent separation	2–4	>1 million L yr ⁻¹	Fully preserved. Tray-by-tray liquid flow maintains side draws for fusel oil, heads and methanol removal as in conventional columns
Mechanical vapour recompression (MVR) heat pump	9	Yes. Compressor loop + heat exchangers added externally. RC-Score: 5	Medium	40–60% reboiler duty reduction for ethanol-water (COP > 16. Temperature lift ~22°C)	2–4	>5 million L yr ⁻¹	Fully preserved – column internals and side draw configuration unchanged. No impact on congener separation
Thermal vapour recompression (TVR) heat pump	9	Yes. Steam ejector replaces/supplements reboiler steam. RC-Score: 5	Low-medium (ejector simpler than compressor)	10–30% steam reduction	2–4	>5 million L yr ⁻¹	Fully preserved. Column internals unchanged. No process chemistry modification
Vapour compression (VC) heat pump	8–9	Yes. Closed refrigerant cycle added externally. RC-Score: 4	Medium	30–50% reboiler duty reduction. Lower COP than MVR due to closed cycle	3–5	>5 million L yr ⁻¹	Fully preserved. Column internals unchanged. Refrigerant circuit fully isolated from product stream
Compression-resorption heat pump (CRHP)	7–8	Yes. Binary working fluid (NH ₃ –H ₂ O) loop added externally. RC-Score: 4	Medium-high (binary working fluid, more complex control)	40–65% reboiler duty reduction. Wider temperature lift range than MVR, suitable for larger ΔT columns	3–5	>10 million L yr ⁻¹	Fully preserved. Column internals and separation function unchanged. NH ₃ –H ₂ O working fluid fully isolated from product
HIDiC (internally heat-integrated distillation column)	9	No. Requires purpose-designed column. Not retrofittable to existing shells. RC-Score: 1	High (new column + compressor. ~20–30% higher column cost than conventional)	Up to 77% reboiler duty reduction for ethanol-water ²⁵ . Typically 40–70% in practice	3–6	>10 million L yr ⁻¹ (greenfield or major capital renewal)	Fully preserved. Thermally based intensification only. Column side draws for congener management can be designed into new column train
Hybrid distillation–membrane separation	6	Partial. Membrane module added downstream of existing rectification column. Existing column modified (reduced reflux). RC-Score: 3	Medium (membrane modules + skid. Established suppliers available)	30–50% versus AD/ED. Most beneficial when product purity approaches or exceeds azeotropic composition (>95 vol %)	4–8 (estimated, as spirits-specific data lacking)	>1 million L yr ⁻¹ (modular membrane skids available at smaller scale)	Data gap. Hydrophilic membranes (PVA, NaA zeolite, HybSi) validated for ethanol dehydration but not yet characterised for spirits-grade congener-containing feeds. Fusel oils and esters may foul or plasticise membranes. methanol behaviour in PV circuit unquantified

Table 4. Continued

Technology	TRL	Retrofit to existing column? Retrofit compatibility score ^a	Investment level	Typical energy savings	Payback period (years)	Minimum economic scale	Congener management status
Pass-through distillation (PTD)	6	Partial. New absorption-desorption circuit required. Can be integrated alongside existing fermentation and stripping equipment. RC-Score: 2	Medium (novel circuit, brine handling system required)	Up to 50% when coupled with multi-effect distillation or MVR	TBD. No industrial-scale economic data available	TBD. Minimum scale dependent on fermenter size and concurrent fermentation-separation integration target	Data gap. Congener behaviour in hygroscopic brine (LiBr/LiCl) uncharacterised. Brine selectivity between ethanol, water, methanol and fusel oils unknown. Food-contact approval of brine under EU/FDA frameworks not yet established. Industrial-scale demonstration with real spirits feeds required

Energy savings are relative to the single-effect distillation baseline (3.0–4.2 kg steam per litre of 96° GL ethanol), unless otherwise noted. For MVR, VC and CRHP, savings reflect reboiler duty reduction. Compressor electricity usage increases accordingly and must be factored into the site-specific cost-benefit calculation depending on local electricity-fuel price ratio. Payback periods are derived from literature case studies^{1,8,19,22,25} and are indicative for large continuous neutral spirit plants (>10 million L yr⁻¹). Actual payback depends on local energy prices, existing heat integration infrastructure and column configuration. Site-specific technoeconomic evaluation is recommended before any capital decision. Minimum economic scale thresholds are indicative benchmarks based on reported capital cost and payback data.

^a Retrofit compatibility score: 5 = drop-in internals/external-loop replacement requiring no new column shell or process requalification; 3 = partial retrofit requiring modification of existing column operation (e.g. reduced reflux) or an added external circuit; 1 = requires a purpose-built column, suitable only for new plant construction or major capital renewal. Scores reflect the qualitative retrofit descriptions and are intended as a practical screening aid for plant engineers, not a substitute for site-specific engineering assessment.

depending on local energy prices and heat-integration opportunities.²² HIDiC implementations, which can generally reduce energy use by up to 60–70% (*ca* 77% for ethanol–water) at the cost of a higher column and compressor investment, show somewhat longer but still attractive payback periods, typically of the order of 3–6 years for large continuous plants.²⁵

At medium scale ($(1–10) \times 10^6$ L yr^{−1}), cyclic distillation offers the best balance of capital cost and energy savings, as the retrofit investment (replacing tray internals and adding cyclic control) is modest relative to over 30% reduction in both CapEx and OpEx it delivers.^{18,19} PTD becomes attractive at this scale if concurrent fermentation–separation integration is desired, as it reduces the fermentation cycle time on top of distillation enhancement thus improving overall plant productivity.^{31,32}

At small and craft scale ($<1 \times 10^6$ L yr^{−1}), the capital threshold for compressor-based heat pumps and multicolumn cyclic systems may be prohibitive. Hybrid distillation–membrane separation at small scale is emerging as viable, with availability of modular membrane skids from established suppliers. The scale–technology mapping summarised above is integrated with the retrofit compatibility assessment in Table 4.

A key dimension absent from most technology comparison frameworks is the question of technology combinations. In practice, the optimal configuration for most large- and medium-scale continuous spirits plants is not a single technology but a package. For example, cyclic distillation combined with heat pump integration can address both tray-level mass transfer inefficiency and column-level thermodynamic inefficiency at the same time, delivering combined energy savings greater than either technology alone. For new plant designs at large scale, a combination of hybrid distillation–membrane separation (where high-purity dehydration is required) is the most energy-efficient configuration currently achievable.

Retrofit versus new plant design

The retrofit-versus-greenfield distinction is a decisive factor in technology selection, and it is most frequently overlooked in academic reviews of ethanol separation technology. As summarised in Table 4, the technologies recommended in this paper differ in their retrofit compatibility. Technologies are classified according to their suitability for implementation in existing plant infrastructure (retrofit) versus new plant design (greenfield), providing a practical decision guide. Energy savings are reported relative to a conventional multi-effect distillation baseline.

For existing plants, the clear first-choice technologies are cyclic distillation and heat pump retrofits. They are implementable within existing column shells and piping configurations with limited plant downtime, no product chemistry changes and no regulatory requalification.^{5,18,19} For new plant construction or major capital renewal cycles, HIDiC should be evaluated as the primary design baseline due to its higher energy performance and proven industrial track record (as SuperHIDiC brand). Hybrid distillation–membrane separation should be included in the design scope where high-purity dehydration is required or where the medium-term upgrade pathway to TRL 7–9 is factored into the investment case.^{28,29}

Congener management

Congener management objectives differ between classic batch spirits (controlled retention) and continuous neutral-alcohol/food-grade production (extensive removal). A final selection criterion is the ability of the proposed technology to manage

congener profiles. This is unique to spirits production and absent from most technology comparison frameworks in the literature. Conventional multicolumn distillation trains handle congener separation through dedicated side draws, extractive dilution columns and demethylation columns.^{6,11} Any intensified technology must either replicate this functionality or be deployed in a configuration where classic columns handle congener management while the intensified technology addresses the energy-intensive ethanol–water bulk separation.

Methanol requires special attention as it is toxic and regulated separately. In classic trains, methanol is concentrated in a dedicated demethylation column operating at high reflux, typically handled downstream of the beer and rectification columns. Cyclic distillation and HPAD retrofits preserve this architecture. Cyclic distillation can be applied in demethylation columns to sharpen separation, while HPAD reduces steam demand without altering methanol split profiles. For HD-PV and PTD, methanol behaviour has not yet been quantified in detail. Until pilot data demonstrate that methanol is either retained in the ethanol product within legal limits or removed in a controlled side stream, these technologies should be applied only to sections of the flow-sheet that do not perform the primary methanol separation. For PTD specifically, an additional unknown is the selectivity of hygroscopic brine towards methanol relative to water and ethanol, as any preferential absorption of methanol into the brine-side recycle could either concentrate or dilute methanol in the product stream in unpredictable ways. This is an issue that must be addressed experimentally before the technology can be considered for any section handling methanol-containing feeds.

Cyclic distillation is fully compatible with side draw operations and preserves congener management capability within the column train.^{18,21} Heat pump retrofits (which modify the energy supply to existing columns without altering their internal separation function) are also suitable to congener management. Hybrid distillation–membrane separation, by contrast, requires careful attention to membrane behaviour in the presence of fusel oils and esters, whose interaction with hydrophilic membranes under industrial operating conditions is incompletely characterised.^{3,40}

The congener management criterion is the most underweighted factor in the current PI literature and most overweighed factor in industry conservatism. The academic community underweights it by ignoring congeners entirely, while practitioners overweight it by treating any perceived risk to congener profiles as an automatic veto on new technology. This knowledge gap (*i.e.* behaviour of food-grade-relevant congeners under intensified separation conditions) is the most urgent research priority for enabling the deployment of membrane-based hybrid technologies in the spirits sector.

Integrating the decision variables

The variables addressed in this section are interacting in the decision space. A small craft distillery retrofitting an existing column with a dilute grain feed arrives at a different optimal configuration (cyclic distillation or/and heat pump) than a large greenfield continuous plant with a concentrated molasses feed targeting maximum energy performance (HIDiC plus HD-PV). Table 4 synthesises the decision framework into an explicit mapping between these variables and recommended PI technologies or combinations. Figure 10 presents a simple technology selection, indicating which PI package is the most appropriate for each combination of feed concentration, target purity and retrofit versus greenfield context. This is intended as a practical high-level guide

Table 5. Performance of PI technologies in food-grade ethanol/spirits-context case studies

Technology	System	Scale/validation	Feed → product	Energy saving versus single-effect baseline	Residual CO ₂ intensity (kg CO ₂ L ⁻¹ AA) ^a	Comments	Ref.
Cyclic distillation	Food-grade EtOH	Industrial (0.4–1.7 m diameter columns). Full-scale operating data	~10 vol% → 96.2 vol%	~30% steam; +3–4% yield	0.382	Losses: 3–6% → ~1–1.5%	19
HPAD	Ethanol–water	Industrial-scale simulation	Various	40–60% reboiler duty	0.218–0.327 (MVR) 0.273–0.382 (VC) 0.191–0.327 (CRHP)	COP > 16 for EtOH–water	22
HIDIC	Ethanol–water (fuel)	Industrial-scale simulation	10 → >99 wt%	40–77% versus conventional	0.126–0.327	SuperHIDIC brand, Japan Fuel-grade study, not food-grade context	25
HD-PV	Dimethyl carbonate	Pilot-scale	Near-azeo. → >99%	10–30% (30–50% versus azeo. dist.)	0.382–0.491	Not food-grade directly	28
HD-PV	Bioethanol distillery	Simulation study (industrial-scale distillery)	Near-azeotropic distillate → high-purity ethanol	Up to ~70% versus classic distillation train (VP configuration outperforms PV)	0.382–0.491	Ethanol-specific. Simulation only, not yet validated on congener-containing spirits feed	30
PTD	Bioethanol	Pilot (DryStill)	Dilute → ~96%	Up to 40–50% (with MVR)	0.218–0.273	Bioethanol, not spirits feed	31

Baseline CO₂ intensity (0.545 kg CO₂ L⁻¹ AA) is calculated from the reference case (11.8 MJ L⁻¹ × 70% thermal share, at 66 kg CO₂ GJ⁻¹ delivered steam, derived from 56 kg CO₂ GJ⁻¹ natural-gas-combustion factor). This table uses delivered-steam basis for consistency. For MVR, VC and CRHP, compressor electricity demand is not included in this table's CO₂ figures (when using renewable electricity) – where compressor power is drawn from the grid, net CO₂ reduction will be lower depending on local grid carbon intensity.^a L AA = litre of absolute (100%) alcohol, conversion from litre of 96.2 vol% product uses a factor of 1.0395.

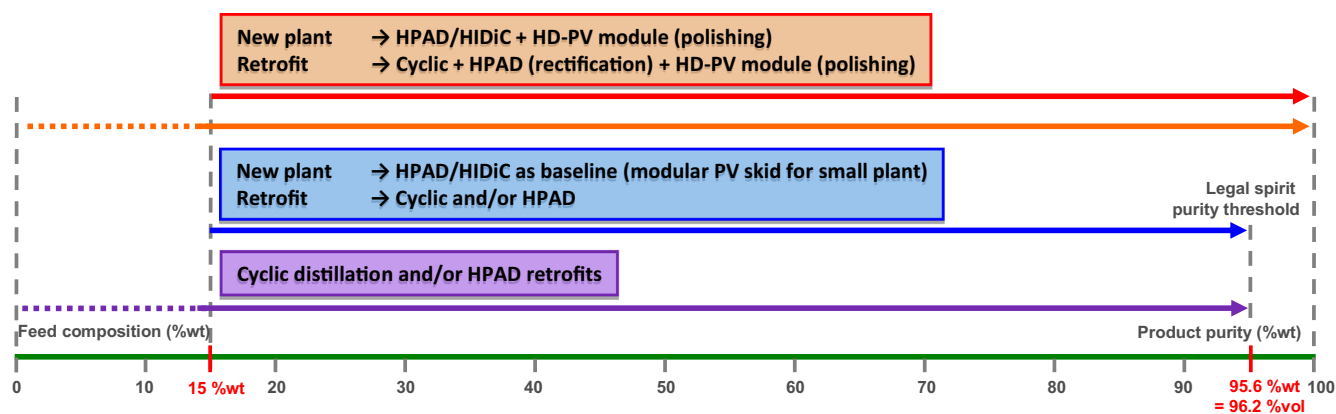


Figure 10. Selection of PI technologies for ethanol separation, based on the feed composition and target product purity.

for screening options rather than a detailed design algorithm, as site-specific technoeconomic analysis remains necessary for final decisions.

- **Feed ethanol concentration.** If below 15 wt%, the stripping section (not the rectifying zone) dominates the energy load. In this case, cyclic distillation or HPAD is directly applicable. Payback is typically just a few years at $>5 \times 10^6$ L yr⁻¹.
- **Target product purity.** If below 96.2 vol% then azeotrope-breaking technology is not required. Cyclic distillation and heat pumps are the recommended default package. No extractive distillation or PSA is needed. However, if purity >96.2 vol% is required then hybrid distillation–membrane separation enters the decision scope.
- **Plant scale and capital availability.** For large scale, HPAD or HIDiC (new plant) is fully cost-justified. Payback is <3 – 5 years for HPAD. For small/craft scale ($<1 \times 10^6$ L yr⁻¹) consider a modular PV skid.
- **Retrofit versus new plant design.** For retrofit, use cyclic distillation and/or HPAD. No new column shells, no regulatory requalification and limited plant downtime. For new greenfield plant consider HIDiC as main option.

CONCLUSIONS

The technology landscape for energy-efficient ethanol separation in spirit production is more mature than current industrial practice suggests. Cyclic distillation and HPAD are already commercially proven, food-grade compatible and capable of reducing distillation energy use by 30–77% relative to conventional configurations. They operate at industrial scale in adjacent sectors today, having established IP portfolios. Their performance in food-grade alcohol production has been reported at full industrial scale. Hybrid distillation–membrane separation is a medium-term upgrade path when approaching the azeotropic purity limit, with component-level TRL of 6–9 and integrated system validation underway. PTD (at TRL 6), offers the most disruptive option by enabling concurrent fermentation and alcohol recovery, with energy savings of up to 50% when coupled with multi-effect or MVR configurations. The technical case for change is clear, so the question is no longer whether these technologies work, but why they are not being deployed.

The immediate priority for any existing large-scale continuous distillery is a pinch analysis-guided evaluation of HPAD retrofit

feasibility across the column train. Payback periods of 2–4 years are typical at production scales above 10×10^6 L yr⁻¹, and the engineering risk is low. For medium-scale operations, cyclic distillation tray replacement delivers 30% steam savings and a 3–4% improvement in alcohol yield with no column shell modification and no product requalification. This is the lowest-barrier, highest-certainty intervention available. For new plant construction at any scale, (Super)HIDiC can be the baseline design target. Using conventional multi-effect distillation as the default design choice is a choice to lock in 30–50 years of avoidable energy cost and carbon liability, at a moment when both energy prices and carbon regulation are moving in one direction only. At a reference scale of 300 000 hL yr⁻¹, every year of delayed adoption of PI technologies represents about 8300 t of avoidable CO₂ emissions and an operating cost premium that keeps adding across the asset lifetime.

From a decarbonisation viewpoint, different PI routes align with different net-zero strategies. HPAD and HIDiC reduce boiler fuel demand by 40–70%, enabling partial electrification of distillation duty via compressor power, which can be supplied from low-carbon or renewable electricity. Cyclic distillation reduces thermal demand by *ca* 30% without changing the energy carrier, while PTD opens the possibility of low-temperature operation compatible with low-grade or renewable heat sources. The choice of technology for early adoption can be made in terms of energy efficiency and compatibility with future low-carbon utilities. For example, at a scale of 300 000 hL yr⁻¹ and assuming the compressor electricity is sourced from renewable generation, HPAD could reduce the distillation CO₂ footprint from *ca* 545 g CO₂ per litre to about 126–382 g CO₂ per litre, depending on the configuration. This is a reduction of about 30–75% in Scope 1 emissions and near-zero Scope 2 contribution from the compressor.

For the research community, the most important priority is the systematic experimental characterisation of intensified separation technologies under realistic food-grade, congener-containing fermentation-derived feeds. The majority of papers on ethanol separation are based on binary ethanol–water mixtures, which is a mismatch with the industrial reality. Most PI technologies have not been validated against the full congener spectrum present in spirits-grade feeds (e.g. fusel oils, esters, aldehydes, organic acids and methanol), at some realistic concentrations. Until this gap is closed, no equipment supplier can guarantee quality compliance, and no spirits producer can justify the investment. The field has an excess of new laboratory-scale separation concepts and a critical deficit of industrial-condition validation data.

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