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PRODUCTION OF FUEL-GRADE ETHANOL BY EXTRACTIVE DISTILLATION EMPLOYING THE SALT EFFECT

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ABSTRACT

Salt-effect distillation is a novel variation on extractive distillation in which the agent added to the column to effect the separation is a salt rather than a liquid. The technique, which can offer major energy savings over conventional processing, is attracting renewed interest at present, primarily in the dehydration of ethanol for gasoline-alcohol fuel blends. Details of the technique, its chemistry, its advantages and problems, and some of its applications are discussed. More detailed reviews are cited.

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1. INTRODUCTION

1.1 Extractive Distillation

Extractive distillation is a technique employed to separate systems which are either impossible (due to the existence of an azeotrope) or uneconomical (due to excessively low relative volatility) to separate by normal fractional distillation. An added liquid, termed a "third component" or "separating agent", is fed to the column, where its molecules tend to form association complexes in the liquid phase with the molecules of the two feed components, thus lowering both of their volatilities. The separating agent is chosen such that its molecules will associate preferentially with the molecules of the less volatile feed component over those of the other. As a result, the volatility of the less volatile component will be lowered by an amount greater than that of the more volatile component, thus raising the relative volatility of the system and thereby reducing the number of ideal stages required to achieve the separation. If an azeotrope exists, its composition will be altered. If a sufficient amount of sufficiently selective separating agent is added, the azeotrope may be shifted to the extent that one of the feed components can be collected in the distillate in relatively pure composition, or even shifted to the extent of being eliminated completely. Separating agents which are found to be selective toward the less volatile of the two feed components usually tend to be of quite low volatility themselves, and as a result concentrate primarily in the liquid phase inside the column. A subsequent distillation operation is required to recover the separating agent from the bottom product stream for recycle, while the lesser amount of separating agent appearing in the vapor phase is stripped from the overhead by an additional section of

column, referred to as a "knockback section", placed above the point on the column where the separating agent is fed. Extractive distillation is costly compared with normal fractional distillation, a consequence of both increased capital costs and increased energy costs arising largely from the requirement for recovery and recycle of the separating agent, which normally must be used at very high concentration to achieve its desired effect.

1.2 Extractive Distillation by Salt Effect

In certain systems where solubility considerations permit, it is possible to use a salt dissolved into the liquid phase as the separating agent in place of the normal liquid additive. The attraction of the salt-effect distillation technique lies in its potential for greatly reduced energy requirements compared with conventional extractive and azeotropic distillation processes.

The salt effect in vapor-liquid equilibrium, which briefly stated, is the ability of a nonvolatile electrolyte dissolved in a mixed solvent to alter the composition of the equilibrium vapor without itself being present in the vapor, has been known to chemical investigators since the Middle Ages[1]. Yet industrial applications based on the effect, including extractive distillation employing a salt as the separating agent, have been relatively neglected. In the United States for instance, the only current, large-scale application other than those pending in gasohol applications has been in nitric acid production[2]. On the other hand, the journals of the former Soviet Union countries demonstrate the existence of a strong and continuing interest in the effect itself and in its industrial applications, where the technique is referred to as "salt rectification". Much interest is also evident in the literature from Japan and China.

Figure 1 demonstrates a typical flowsheet for salt-effect distillation. The salt, which must be soluble to some extent in both feed components, is fed at the top of the column by dissolving it at a steady rate into the boiling reflux just prior to entering the column. The salt, being nonvolatile, flows entirely downward in the column, residing solely in the liquid phase. Therefore, no

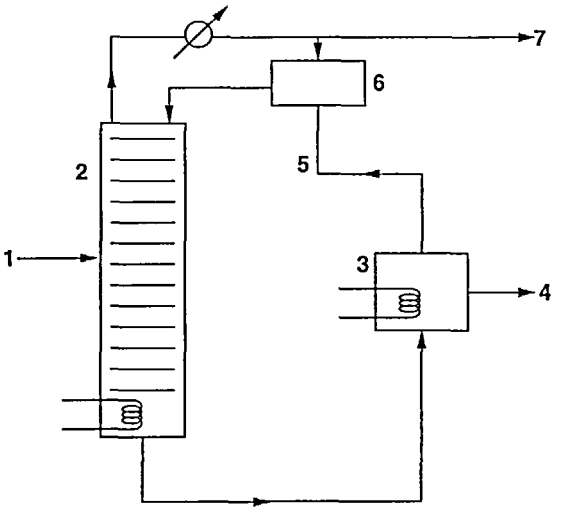

FIGURE 1
Basic flowsheet for salt-effect distillation. Feed stream (1), extractive distillation column (2), salt recovery operation (3), bottoms product (4), salt recycle (5), dissolving chamber (6), overhead product (7).

knockback section is required above the separating agent feedpoint to strip agent from the overhead product. Recovery of the salt from the bottoms product for recycle is by either full or partial drying, rather than by the subsequent distillation operation required with liquid separating agents.

Several variations on the process are possible. For example, an azeotrope-containing system could be separated by first taking it almost to the azeotrope by normal fractional distillation without salt, then across the former azeotrope point by a single-stage or few-stage extractive distillation with salt present, and then to final purity by further distillation without salt.

2. ADVANTAGES AND DISADVANTAGES OF A SALT

Liquid and salt separating agents work in more-or-less similar manners: the most common mechanism is that their solution

species (molecules or ions) exert forces of attraction on the molecules of the feed components in the liquid phase, resulting in the formation of association complexes, or clusters. If the attraction is selective to one feed component over the other, relative volatility will be altered (raised, if the attraction is selective to the less-volatile component; lowered, if selective to the more). A principal advantage of a salt over a liquid, however, is that salt ions are typically capable of causing much larger alterations in the volatility relationship between feed components than even the most polar of liquid additives. As a result, much less separating agent may be required, with resulting economies in separating agent inventory, in equipment sizing, in the agent recovery and recycle capacity required, and particularly in the overall energy requirements for the process.

In our own laboratory for example, we have been able to produce 99+ mol % ethanol from even dilute aqueous ethanol feedstocks in a 12-tray, bubble-cap column using only 6 mol % potassium acetate in the liquid phase[3]. The azeotrope was completely eliminated even at this very low salt concentration. To place this concentration in context, it must be noted that most conventional azeotropic and extractive distillation processes for producing anhydrous ethanol require very much larger concentrations of separating agent, ranging anywhere from a 1:1 up to a 5:1 mole ratio of liquid separating agent to ethanol-water feed even with concentrated feedstocks. For example, Black and Ditsler[4] reported separating agent-to-feed ratios ranging from 2.5:1 to 4:1, using ethylene glycol as the extractive distillation separating agent. This range is of the order of 50 times as high as that reported above for the salt potassium acetate to achieve a similar effect. Dobroserdov[5,6] observed that the addition of calcium chloride at the low concentration of 1 mol/L in the liquid phase of the ethanol-water system reduced the number of ideal stages required in order to concentrate 14.3 mol % ethanol feed solution to 86.0 mol %, from 18.5 to only 3.5 ideal stages, a more than fivefold reduction. Recently, Barba, Brandani, and Di Giacomo[7] demonstrated the

production of 97.5 mol % ethanol from a 22 mol % ethanol-water feedstream, in a 10-stage packed column using calcium chloride dissolved into the reflux at a concentration of 2.6 mol %. Other examples are given in the industrial applications described below.

A second major advantage of a salt separating agent over a liquid lies in the complete nonvolatility of the former. As a result, all of the salt separating agent will reside in the liquid phase within the column, and will leave the column entirely in the bottom product stream. Since no portion of the salt will distribute itself into the vapor phase, and assuming that normal precautions against entrainment are taken, the overhead product will be completely free of separating agent without either a knockback section or any additional purification required. In our previously-mentioned extractive distillation work in the ethanol-water system with potassium acetate[3], for example, a potassium ion test sensitive to one part in 16,000 was unable to detect any salt presence in the overhead product.

A significant disadvantage of a salt compared with a liquid separating agent is that the number of systems for which an effective agent can be found are much more limited by solubility considerations in the former case, particularly noting that the salt must have significant solubility in the reflux stream (that is, in the more volatile as well as the less volatile feed component). Another is the general disadvantage associated with solids handling compared with liquids handling.

3. SALT EFFECT IN VAPOR-LIQUID EQUILIBRIUM

When nonvolatile electrolytes such as salts are dissolved into a mixed solvent consisting of two (or more) liquid components, one or both of which may be polar, many of the solution properties of the mixed solvent are affected. These may include activity coefficients, solubilities, liquid-phase structure, equilibrium vapor composition, and others. At the heart of the changes in macroscopic properties lie basic and complex solution phenomena, including a variety of ion-solvent interactions involving one or

both ion species and one or both solvent species, solvent-solvent interactions, self-interactions within individual components present, and the various effects that such composition-dependent phenomena can have on liquid structure and related physicochemical and thermodynamic properties. For example, in a relatively small number of systems, reduction in the mutual solubilities of the two feed components caused by the presence of the salt is sufficiently large to cause formation of a second liquid phase.

From early investigations on, an empirical relationship between the effect of the salt on equilibrium vapor composition and its solubilities in the two components of the mixed solvent has been observed in many systems. The resulting model is based on two principal observations: first, that the equilibrium vapor composition will be enhanced in the component in which the salt is less soluble; and second, that the magnitude of the enhancement depends both on the amount of salt present in solution (which is limited by its solubility in the liquid phase), and on the magnitude of the difference between its solubilities in the two feed components.

The vapor-liquid equilibrium (VLE) diagrams that follow reflect this commonly-observed pattern. In each case the system consists of ethanol-water containing a dissolved salt, the system is boiling, and pressure is constant at atmospheric. Equilibrium vapor composition (y) is plotted against liquid composition (x), with both expressed as mole fraction ethanol. Liquid composition is presented on a salt-free basis in order to allow direct comparison of the VLE curve for the salt-containing system with that for ethanol-water alone, the latter being shown as a dashed line for reference purposes. In the first four figures to follow, the salt is present at saturation. In the fifth, it is present at a constant concentration below that of saturation in either feed component.

Figure 2 demonstrates the effect of ammonium chloride[8]. This salt is quite soluble in boiling water (21.0 mol %) but almost insoluble in boiling ethanol (0.005 mol %). It is seen to cause a significant enhancement in ethanol concentration, the component in

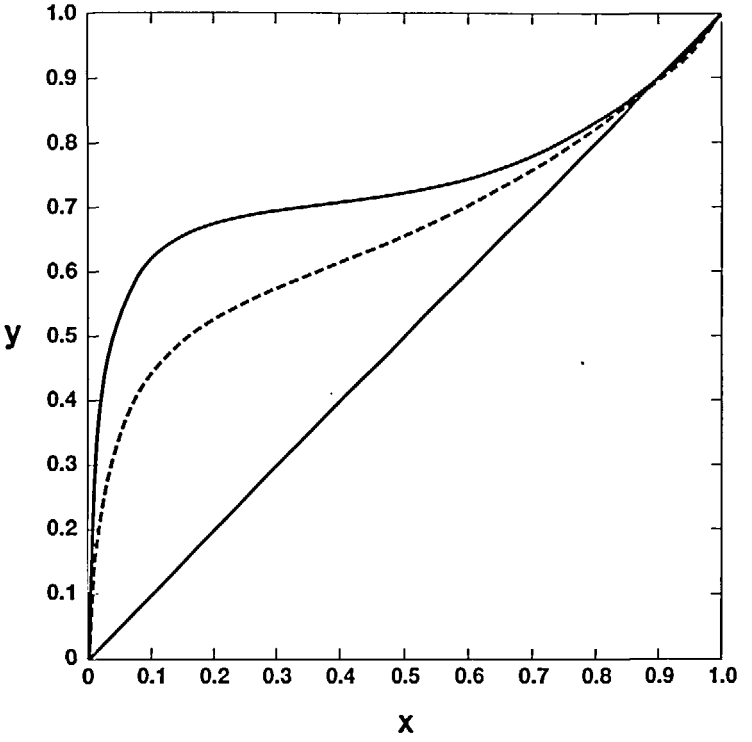

FIGURE 2
Ethanol-water VLE with ammonium chloride at saturation.

which it is less soluble, in the equilibrium vapor, but the effect is observed mainly in the water-rich region where a substantial amount of salt is present in solution. The effect almost disappears in the azeotrope region where salt solubility is very low.

Figure 3 presents the effect of barium nitrate[8] a salt of relatively low solubility in boiling water (2.4 mol %) and essentially no solubility in boiling ethanol. Not surprisingly, the effect on VLE is small, and only noticeable in the water-rich region. Nevertheless, what little effect visible is an ethanol enhancement in the equilibrium vapor.

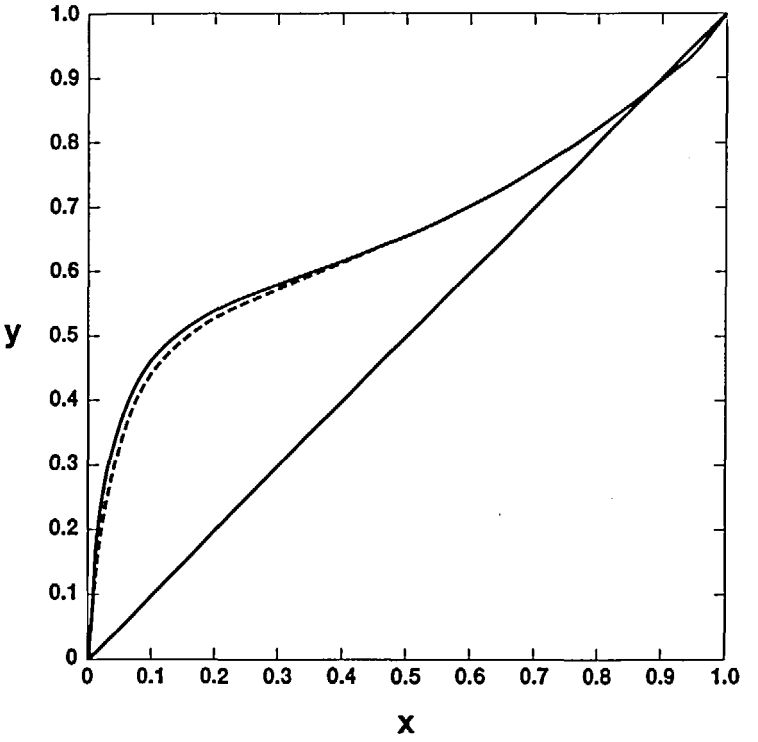

FIGURE 3
Ethanol-water VLE with barium nitrate at saturation.

The dramatic effect of potassium acetate[9] on VLE in the ethanol-water system is shown in Figure 4. Potassium acetate has the largest effect on VLE of any salt yet reported on the ethanol-water system. Its solubility in boiling water is extremely high (49.4 mol %, that is, almost mole for mole), while its concentration in boiling ethanol, while considerably less, is still substantial (10.6 mol %). Relative volatility is increased by four to five times in the water-rich region, while the effect lessens somewhat but is still very significant in the ethanol-rich region. The azeotrope has not only been shifted in the ethanol-rich direction

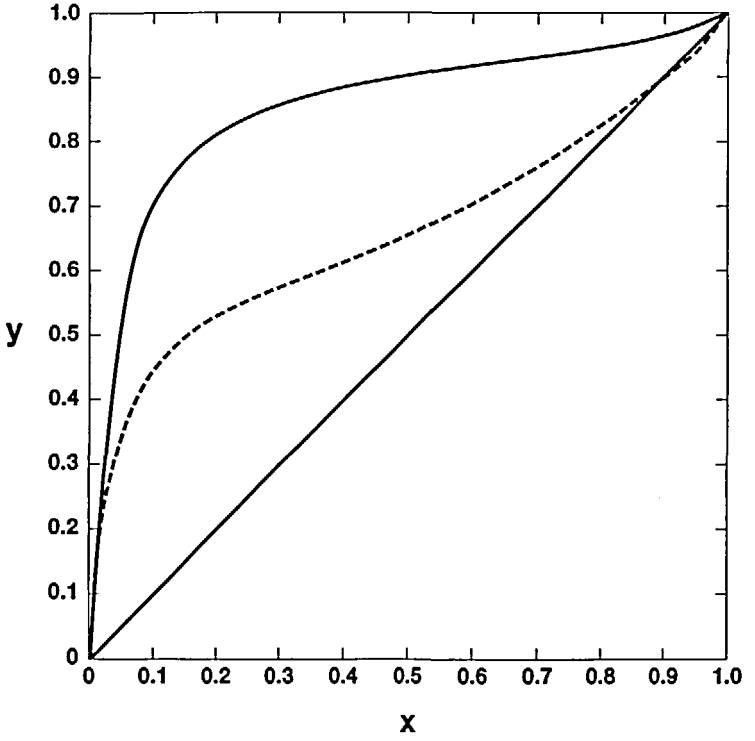

FIGURE 4
Ethanol-water VLE with potassium acetate at saturation.

but has been moved all the way to the 100 mol % ethanol point, and thereby totally eliminated.

In all of the three examples provided above, the salt is more soluble in water than in ethanol, as is typical with most common salts. Mercuric chloride, however, is one of a few salts which are more soluble in boiling ethanol (5.8 mol %) than in boiling water (3.7 mol %), and its effect[10] is shown in Figure 5. Here, as the solubility data would predict, equilibrium vapor composition is enhanced in water rather than in ethanol, with the azeotrope being shifted in the water-rich rather than in the

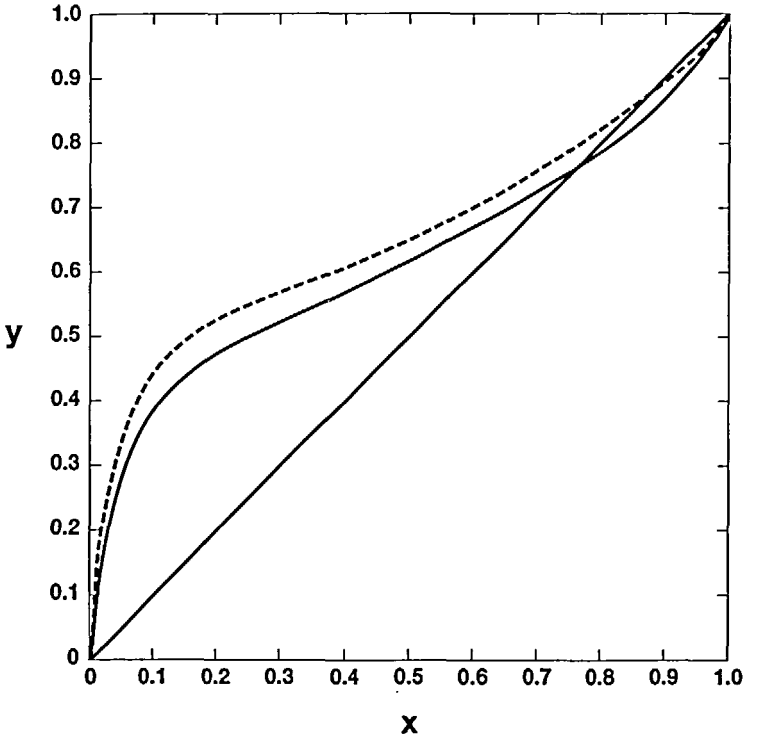

FIGURE 5
Ethanol-water VLE with mercuric chloride at saturation.

ethanol-rich direction. The effect is observed to be exerted throughout most of the mixed-solvent composition range because the salt, while more soluble in ethanol than in water, is substantially soluble in both components.

Most investigators have measured salt effect on VLE in systems by maintaining salt concentration at saturation throughout the mixed-solvent composition range, presumably in order to record the largest effect on equilibrium vapor composition that the particular salt can have at each liquid composition throughout the particular system. This condition, however, would not be represen-

tative of that existing in an extractive distillation column employing a salt separating agent. Assuming the salt were fed at or near saturation in the boiling reflux stream entering the column at the top, and assuming that "constant molal overflow" applied in the column, then molal salt concentration in the liquid phase would remain essentially constant from tray to tray as the column is descended. Therefore, the VLE curve to be used in the design of such a column should have salt concentration at a constant rather than at saturated concentration. It is possible to calculate salt effect at fixed concentrations from VLE given for salt at saturation, provided that a reasonably reliable salt-effect correlation equation[11] for the system in question is available.

Figure 6, as an example of VLE at constant rather than saturated concentration, is for ethanol-water containing calcium chloride, measured by Nishi[12]. Calcium chloride, like potassium acetate, while more soluble in boiling water (21.1 mol %) than in boiling ethanol (9.1 mol %), is significantly soluble in both. As the solubility data would predict, calcium chloride shifts the azeotrope in the ethanol-rich direction, and at the concentration employed in this case, all the way to total elimination. Nishi's salt concentration, 16.7 wt %, unfortunately was held constant on a weight basis, rather than on a mole basis as the concept of constant molal overflow would require. (The constant 16.7 wt %, converted to a mole basis, ranges from 3.2 mol % in pure water to 7.7 mol % in pure ethanol).

Models of salt effect on VLE based on solubilities alone can only be empirical at best, because they ignore other obviously-relevant parameters as, for example, ion charges, ion radii, and the degree of dissociation of the salt in the mixed solvent. Several systems have been identified[13] in which the observed salt effect is actually opposite to that which solubility-only models predict, suggesting that structure-breaking as well as structure-making mechanisms may be at work in these systems, with the predominating effect in some even changing from one mechanism to the other as mixed-solvent composition is varied.

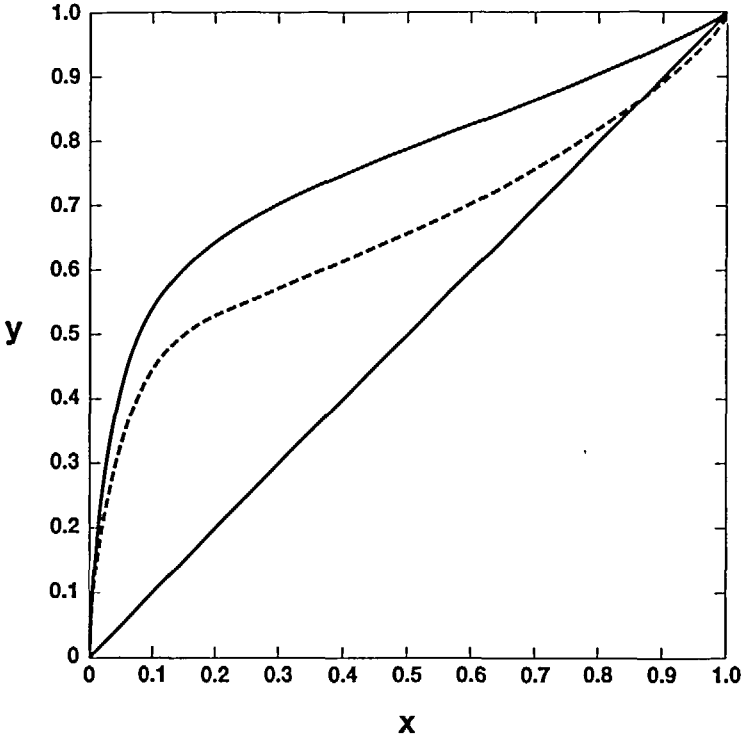

FIGURE 6
Ethanol-water VLE with calcium chloride at constant concentration of 16.7 wt %.

Attempts at theoretical treatment are further hindered by the serious complication that the degree of dissociation of a salt may change within a given system as mixed-solvent composition is varied, potentially affecting all of the system interactions involved. For example, a particular salt may dissociate fully in boiling water, but only partially in boiling ethanol. The effects on VLE caused by each type of salt particle (its molecules and its two varieties of ion species) are probably all different, and the relative proportions in which they exist in solution will change when the degree of dissociation changes. Because of such complexi-

ties, attempts at correlation and prediction of salt effect on vapor-liquid equilibrium have achieved only modest success so far[11].

4. EXAMPLES OF INDUSTRIAL APPLICATIONS

The relative lack of exploitation of salt-effect distillation by industry can be attributed, at least in part, to the extremely complex liquid-phase phenomena occurring in such systems, and to the resulting difficulties in modelling them in order to develop design equations.

Concerns have also been expressed that salt precipitation from solution could occur within the extractive distillation column, plugging the trays. However, with a salt more soluble in the less rather than in the more volatile feed component as is the normal case, precipitation is most unlikely to occur. The explanation is that the salt, while at essentially constant molal concentration in the liquid phase descending the column ("constant molal overflow"), becomes a progressively lower percentage of saturation in the liquid phase, and therefore progressively more soluble in the liquid phase, as the column is descended. No problems with salt precipitation are known to have been reported in the literature.

Some of the few major applications to date are described below.

4.1 HIAG Process

It may not be generally recalled that most automotive gasoline sold in Europe from the late 1920s to the Second World War was gasohol, containing approximately 10% ethanol produced from indigenously-grown sugar beets to serve as an extender for gasoline in order to reduce its high costs. A process known as HIAG (Holz Industrie Actien Geselleschaft), licensed by DEGUSSA and based on patents registered by Adolph Gorhan during the late 1920s and early 1930s, employed a salt-effect extractive distillation technique to concentrate ethanol to absolute (99.8%+) for use in this application[14]. About 100 HIAG plants were built, first in Europe and later worldwide, by DEGUSSA between 1930 and 1951, with production capacities ranging up to 150,000 L/day. The salt consisted of a

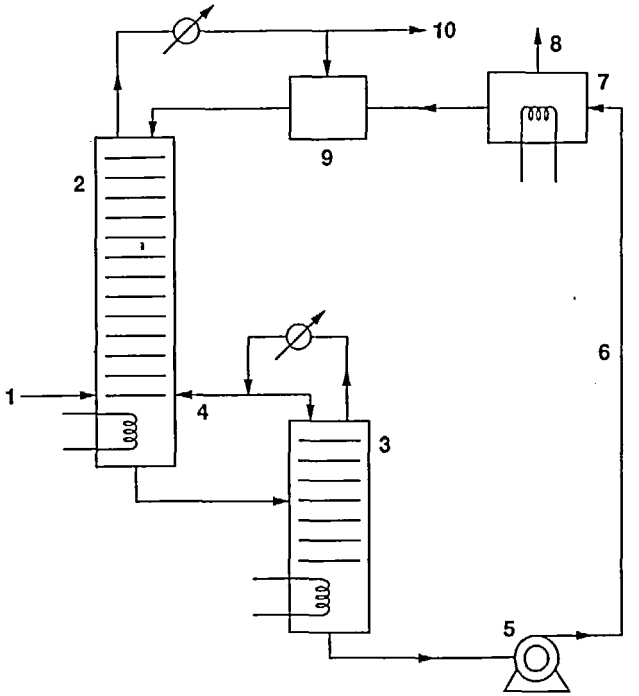

FIGURE 7
HIAG process for production of absolute alcohol. Ethanol-water feed stream (1), extractive distillation column (2), stripping column (3), residual ethanol return (4), recycle pump (5), salt-water recycle (6), dehydrator (7), water removal (8), dissolving chamber (9), ethanol product stream (10).

mixture of two acetates. The flowsheet for the HIAG process is shown in Figure 7.

The feedstream, consisting of near-azeotropic ethanol-water solution concentrated from fermentate by conventional distillation, is fed to the reboiler of the extractive distillation column. Anhydrous, fused salt from recycle is dissolved into the reflux stream entering the top of the column. "Absolute" (anhydrous) alcohol at 99.8 wt % purity, entirely free of salt, is produced at the overhead. The bottoms stream, consisting of salt, water, and ethanol traces is fed to a small stripping column to recover

residual ethanol which is then returned to the reboiler of the main column. The aqueous salt solution from the bottom of the stripping column is pumped to a dehydration unit, where the solution is heated to 300 C by superheated steam, bringing the salt to a molten (fused) state in order to achieve total dehydration. The molten, dehydrated salt then flows to a mixing chamber where it is dissolved into the hot reflux.

A major feature of the HIAG process is that the salt remains in fluid condition, either dissolved or molten, throughout the entire process, thereby avoiding the problems commonly associated with solids handling.

DEGUSSA kept both the salt formulation and its concentration in solution proprietary. However, the salt is known to have been a 70/30 wt % mixture of potassium and sodium acetates. Although potassium acetate[9] is a considerably more effective salt than sodium acetate[15] in eliminating the ethanol-water azeotrope, the two salts form a low-melting (300 C) eutectic at the 70/30 ratio, allowing this particular ratio to be brought to fused condition for dehydration at the lowest possible temperature. The purpose was to avoid any possibility of "charring" (thermal decomposition) of these organic salts to occur during dehydration.

And judging from the extractive distillation experiments by Cook and Furter with potassium acetate referred to earlier[3], it can be concluded that the salt concentration employed in the HIAG process was likely quite small, probably only a few mole percent.

Despite its energy efficiency (DEGUSSA claimed a HIAG steam consumption of only 65 kg per hectolitre of absolute alcohol produced), the HIAG process was replaced progressively by less energy-efficient azeotropic distillation processes using liquid separating agents such as benzene, with the last HIAG plant ceasing operation in 1965, in Brazil.

4.2 IHI Process

The Ishikawajima-Harima Heavy Industries (IHI) company in Japan developed a salt-effect extractive distillation process for

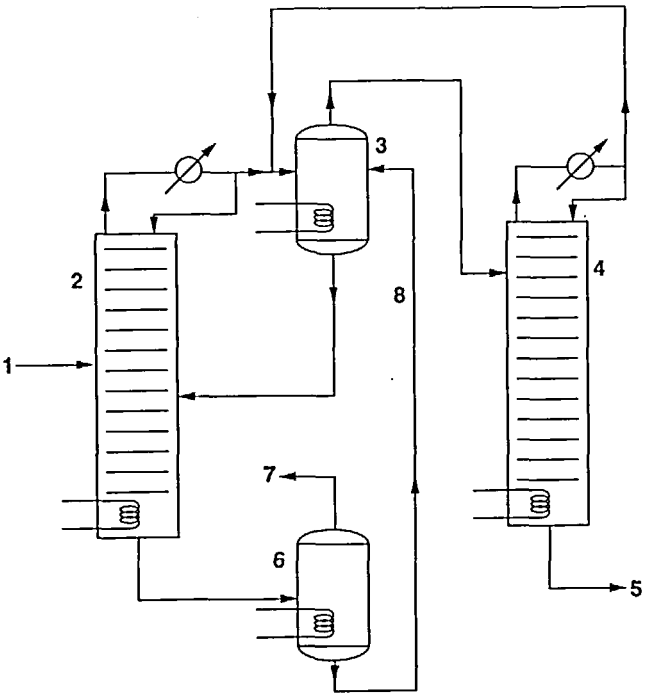

FIGURE 8
IHI process for isopropanol (IPA)-water separation. IPA water feed stream (1), first distillation column (2), evaporator (3), second distillation column (4), IPA product stream (5), concentrator (6), water-rich stream (7), salt recycle (8).

concentrating isopropyl alcohol (IPA) from aqueous solution, using calcium chloride as the separating agent in place of the conventional benzene[16]. Isopropanol-water exhibits an azeotrope at 69 mol % isopropanol under atmospheric pressure. The flowsheet for the IHI process is shown in Figure 8.

In the first distillation column, the more-volatile isopropanol is concentrated to just below the azeotrope composition. Its overhead product is then fed to an "evaporator" containing salt, in which the isopropanol concentration is carried across the former

azeotrope point by what essentially amounts to a single-stage extractive distillation conducted by salt effect. The overhead product from the evaporator, at 88 mol % isopropanol, with the isopropanol having now become the less volatile component, is fed to a second distillation column for concentration to 99%+ by distillation without salt. The salt-containing bottoms product from the evaporator is passed through the stripping section of the first distillation column to assist the separation, while the water-rich bottoms product from this column is fed to a second evaporator, termed a "concentrator", to remove most of the water and concentrate the salt for recycle to the first evaporator. The salt therefore recirculates within the process as a concentrated solution rather than in dry form. The overhead product from the second distillation column, consisting essentially of azeotropic isopropanol, is recycled as additional feed to the salt-containing stage. IHI has described a 20 tonne/day plant using this process, and claims a plant capital cost of only 56% and an energy requirement of only 45%, for a product of higher purity, compared with the conventional azeotropic distillation process using benzene as the separating agent. Higher tray efficiencies are also observed, and there is no formation of a second liquid phase as there is with benzene.

4.3 Nitric Acid Process

Hercules Inc. has been operating plants in the United States since 1957 for the production of nitric acid from dilute aqueous solution by extractive distillation, using the salt magnesium nitrate as the separating agent in place of the conventional sulfuric acid[2,17]. Hercules reports operating costs reduced by half primarily through energy savings, a 30 to 40% reduction in capital costs, a higher yield of product of higher quality, and less atmospheric pollution than for the conventional extractive distillation process using sulfuric acid. A similar process has been operated in Britain by Imperial Chemical Industries since 1960[18]. Like processes are believed to be in widespread use in Russia and other countries of the former Soviet Union.

5. THE AMERICAN GASOHOL PROGRAM

A continuing interest in the salt-effect extractive distillation technique for concentrating ethanol from azeotropic to absolute alcohol has existed since the early 1980s among alcohol producers and alcohol plant and equipment manufacturers in the United States, spurred by the promise of significant energy savings over conventional extractive or azeotropic distillation processes employing liquid separating agents. The current interest is gasohol, in which the ethanol used must be essentially anhydrous, but the future prospects for straight alcohol fuels are also very much in mind. A number of companies, located primarily in the mid-west, have been experimenting with the salt technique. One company located in Minnesota, for example, has been operating a 20,000 L/day plant successfully since 1981, using an acetate salt mixture as the separating agent. Several others are or have been in various stages of startup or operation. (The author has been involved in a consulting capacity with a number of these companies). The preferred salt tends to be either potassium acetate, or a mixture of acetates as in the HIAG process described earlier, but there are several other contenders including, most notably, calcium chloride. The preferred technique continues to be to concentrate ethanol close to the azeotrope by conventional distillation without salt, and then feed the solution to a salt-effect column for final concentration. The location of the alcohol-water feedpoint on the salt column has been reported to be a critical parameter. Various salt recovery and recycle schemes have been tested, including evaporation to dryness from the bottoms stream, recycle as a concentrated solution or a slurry by partial evaporation, and even recycle in fused (molten) condition following drying as in the HIAG process. Some early problems were reported with foaming in the column, but commercial antifoam agents were found to be effective.

6. SOURCES OF FURTHER INFORMATION

Major reviews of the literature on salt effect in vapor-liquid equilibrium and its applications in extractive distillation

are available[1,19], as is a review of correlation and prediction methods for the related phase equilibrium data[11]. A book containing VLE data for salt-containing systems has been published by Ciparis; the original version presenting data for 188 systems[20], and a more recent edition providing data for 287 systems[21]. A recent book by Ohe compiles data for 135 systems[22]. The technical aspects of extractive distillation processes employing salt effect have also been reviewed[6,23,24].

7. ACKNOWLEDGEMENTS

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