

# A Review on Various Methods for Distillation of Isopropyl Alcohol (IPA) and Water

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**ABSTRACT:** Distillation is the process used for separation of two or more component based on their boiling point. IPA is used in various industries as a raw material. IPA and water forms azeotrope with a composition of about 67.28 mol% IPA and 32.72 mol% water at 1 atm and 80.18°C. This article presents methodologies used for the distillation of IPA and Water. The main work of this paper is to study various distillation methods used for the separation of IPA and Water: viz. Extractive distillation using Dimethyl Sulfoxide as entrainer, Azeotropic distillation using cyclohexane as solvent, fixed adsorptive distillation, Hybrid method vapour permeation/distillation using zeolite membrane. This paper shall help the policy maker to find the various methods for the distillation process of IPA and Water.

**KEYWORDS:** Distillation, IPA, Water, Azeotrope, process.

## I. INTRODUCTION

Distillation columns are one of the most widely used processes in the chemical and petroleum industries for the separation of chemical components into more or less pure product streams. This separation of the components is done based on differences in volatilities i.e. tendencies to vaporize among various chemical components. Distillation is one of the most energy intensive processes in the chemical and petrochemical industries [1]. In a distillation column, the component with low boiling point is removed from the top of the column and the components with high boiling point are removed from the lower part of the column. Since distillation has a broad application area, the separation technology depends on raw materials, products and separating operation of a variety of requirements [7]. Various distillation processes are used for the separation of IPA and water. Azeotropic and extractive distillation are an important unit operation in today's processes [5]. Extractive and Azeotropic distillation use a third component to break the azeotrope from a two-component system. The third component is used for azeotropic distillation, then it is known as solvent and if used for extractive distillation then it is known as entrainer. Entrainer and solvent play a very vital role in the distillation process. It is used to make a boiling point difference in the components whereas the selection criteria depends on the behaviour of the system [24].

Fixed adsorptive distillation method can be used to overcome the problems faced by azeotropic distillation. Even though if we use an infinite number of stages or even if we increase the height of packing the azeotrope form cannot be broken due to the existence of a pinch point. Fixed adsorptive distillation technique can be used to break or to pass the azeotropic point [27]. In past decades, another process which has gained much interest in the chemical industry is pervaporation (PV) process [13]. The technology offered by membrane separation has many advantages over mass transfer processes which is already existing [21]. We can also use pervaporation for dehydrating chemicals which is heat sensitive [17]. Vapour permeation and Pervaporisation are closely related only the difference is feed is in vapour phase in vapour permeation and liquid in pervaporisation. Compared to pervaporisation, Vapour permeation has the advantage that there is no phase change occurs while going from the feed to the permeate side. IPA has various industrial uses. IPA can be manufactured by two processes: indirect and direct hydration of propylene [8]. For industrial use the IPA must have a water content less than 0.5% [25].

## II. METHODS FOR DISTILLATION OF IPA AND WATER

### 1. FIXED ADSORPTIVE DISTILLATION

Fixed adsorptive distillation is a process in which both distillation and adsorption work simultaneously. Fixed adsorptive distillation uses packing which consists of two columns of adsorptive bed and two Raschig rings, which again

consist of inter-bed of adsorbent which is filled with silica gel. When the condensate from condenser get out from the top it get split into two stream in which one passes through the silica gel adsorption bed and one is bypass. In the first column the distillate produce has a composition slightly below azeotropic composition. Silica gel which shows surface polarity is present in the first column adsorb only selective component i.e. water, so that only pure IPA passes through the column. In the second adsorption bed the IPA from the first column is distils to more pure form.

This method can overcome the azeotropic point. It is found that if the feed composition and the ratio of flow is high, then the purity of second column product will be high. It is different from other methods of azeotropic distillation where the third component is directly added into the solution to form a mixed slurry like mixture. Whereas in Adsorptive distillation the additional component is placed separately. So that there is no need to separate the addition component and the solution. The adsorbent present in the bed is fixed and as the time passes it get saturated. Due to which adsorption by the adsorbent become difficult and the process could not take place further. By minimizing the flow of distillate through the adsorption bed or by increasing the bypass we can avoid undergoing saturation in short time. If the flow is high better mixing will be done above azeotropic point, but the saturation speed will increase. If the flow is low then saturation speed will decrease and the mixture passing into the second column will not be above azeotropic point. Two beds are used simultaneously in which if one is saturated the other one start working, and the first one is regenerated. However, basically fixed adsorptive distillation can also be operated continuously as many applications in industries [27].

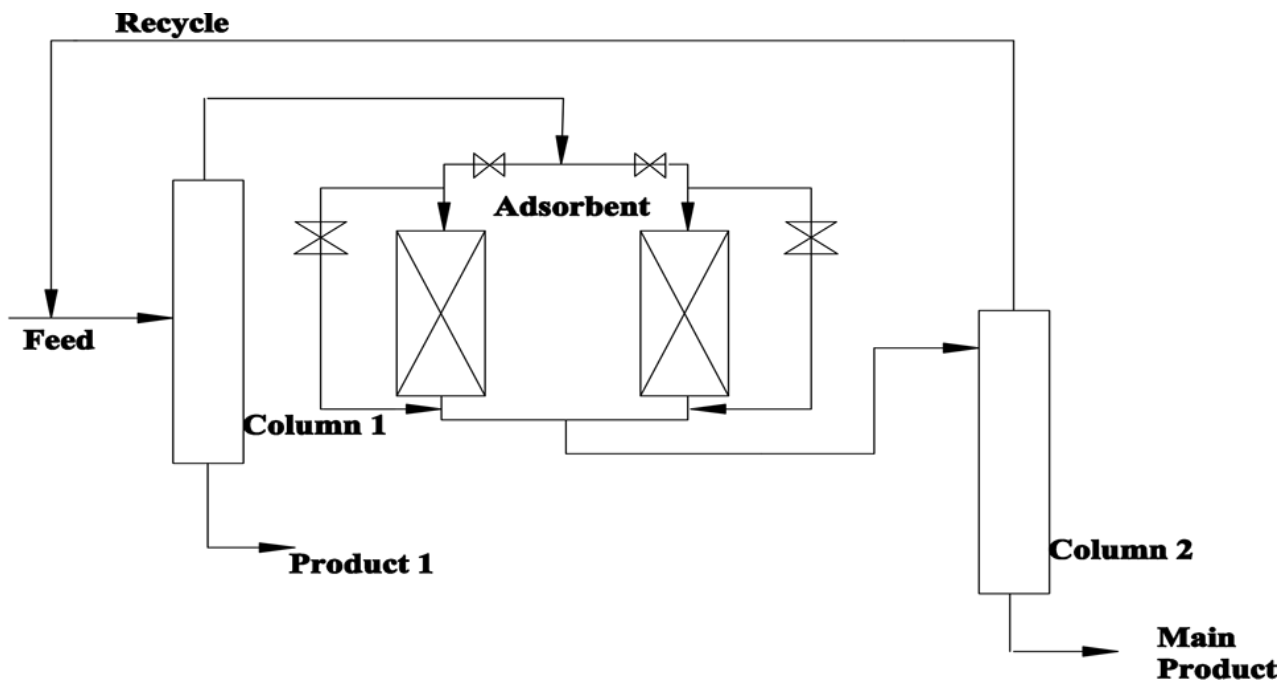


Figure 1: Fixed adsorptive Distillation

## 2. EXTRACTIVE DISTILLATION

Before designing the distillation column entrainer study must be done. In extractive distillation process, the choice of entrainer is one of the vital factors for separating effectively [31]. The entrainer must satisfy the condition and should easily overcome the azeotropic point for a feasible separation to be obtained [26]. The use of third component makes extractive distillation differ from batch distillation. The amount of entrainer to be feed to distillation column should be studied properly for economical purpose and optimization for distillation column. While selecting entrainer and column design detailed calculation can be time consuming and the data require for calculation is more. As a substitute distillation residue curves should be used [15].

IPA and water forms azeotrope and to break this azeotrope entrainer should be used. In the distillation of IPA and water, DMSO can be used as an entrainer. DMSO has been used for fiber-spinning solvent [3]. In the first distillation column IPA-Water mixture along with DMSO is feed into the rectifying section above the entrainer feed. DMSO alters the relative volatility of IPA-Water mixture due to which IPA moves towards the top and Water remain downward in

the distillation column [2]. In the rectifying section IPA separates from DMSO and the pure IPA is distilled as product. In the stripping section Water and DMSO is present which is feed to the second column which is known as entrainer recovery column. In this column simple separation take place where pure IPA is taken out from the top and the DMSO is recycle from the bottom to the first distillation column. Some of the entrainer could be loss during process so a makeup stream of entrainer is provided. The product obtain from this method consist of 99.99 mole % of IPA and 99.9% of water [22].

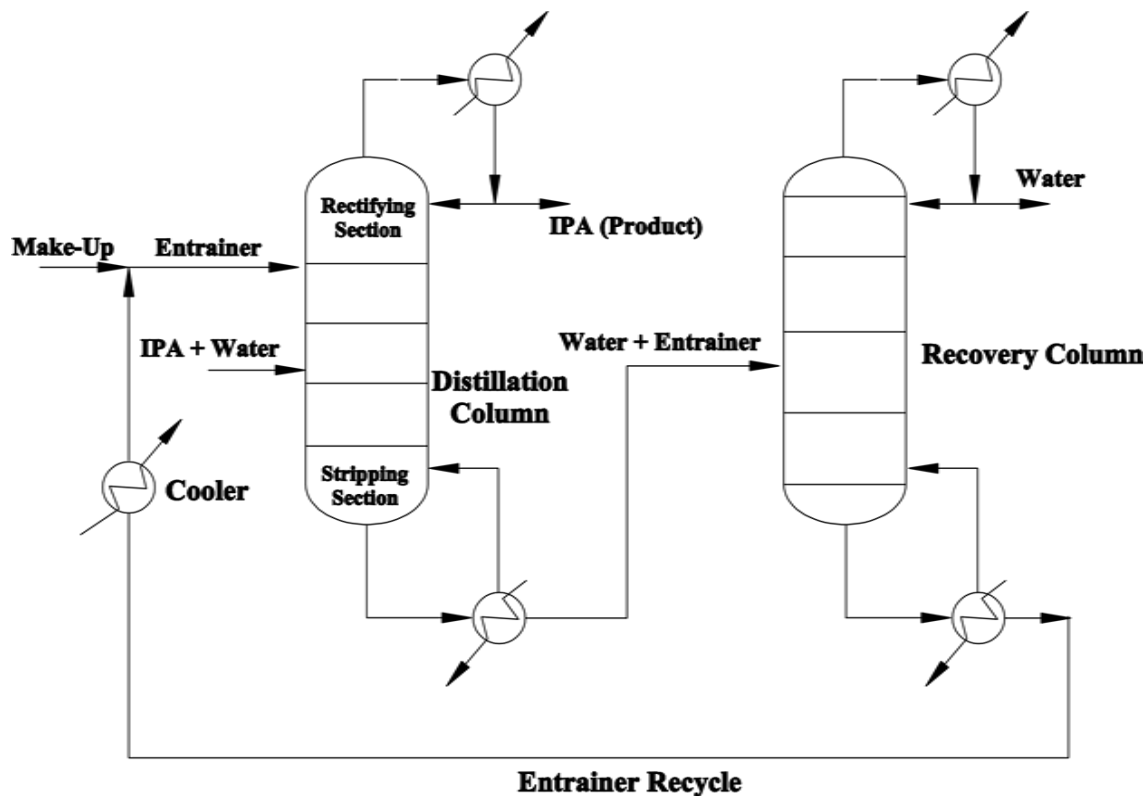


Figure 2: Extractive distillation

### 3. AZEOTROPIC DISTILLATION

Heterogeneous azeotropic distillation is one of the important process used in the chemical industry for the separation of azeotropes and close-boiling binary mixtures. Azeotrope is an ancient Greek word which means "to boil unchanged," where the liquid has a same composition as that of emitted vapour [28]. The third component added is known as solvent which break the azeotrope [9]. In the azeotropic distillation of IPA and water cyclohexane can be used as a solvent [20]. In the process high purity with maximum product recovery can be obtain [23]. In this process the third component added i.e. solvent forms an azeotrope with more volatile component to carry out the desired separation [4].

The feed with IPA-Water along with cyclohexane is added into the first column. The solution feed stage and the entrainer feed stage is calculated. Entrainer is added from above the feed stage. IPA is having more volatility called as a light key, while water has more relative volatility and called as heavy key [30]. Cyclohexane forms the azeotrope with IPA and distilled out from the top. Water is taken out from the bottom. Decanter is used in this process to separate two phase in the overhead product. The Aqueous distillate product rate helps to maintain the aqueous phase level. The level of organic phase in a decanter is maintained by an on-off control scheme using entrainer makeup flow rate [29]. The solvent from the decanter is recycled back to the distillation column while some part of IPA is used as a reflux and the remaining part is taken out as an overhead product [9].

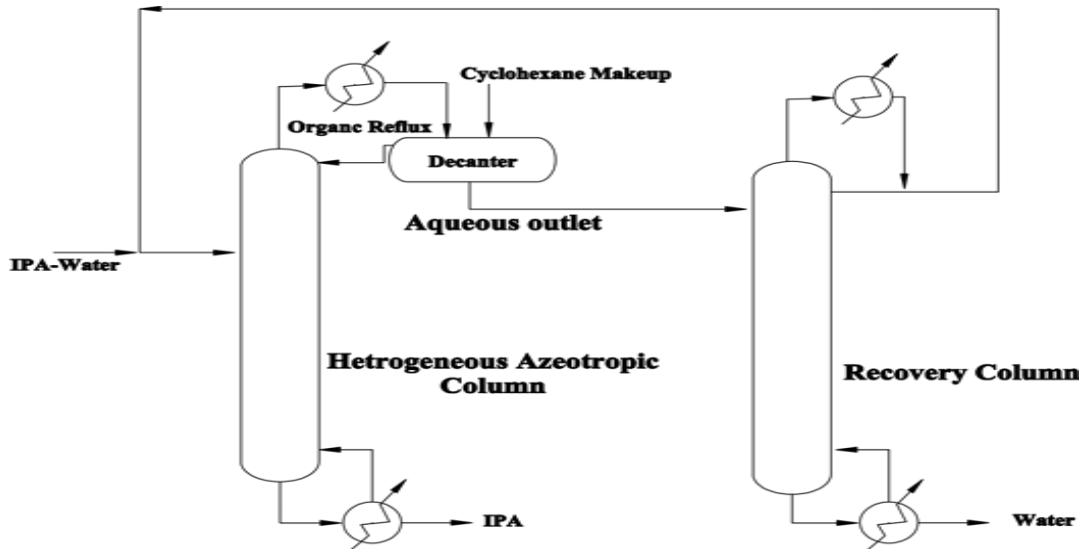


Figure 3: Azeotropic Distillation

#### 4. VAPOR PERMEATION USING ZEOLITE MEMBRANE

Vapour permeation by membrane separation has gain much important for the separation of azeotrope in chemical industry [10]. So the hybrid process can be used for overcoming the azeotropic point. It has another advantage over azeotropic and extractive distillation that it does not use the third component i.e. entrainer or solvent for separation[6]. Organic liquid can be dehydrate using vapour permeation and can be carried out by using polymeric membrane [16]. In this method the separation depends on the relative affinity of the components for the membrane and not on the relative volatility. Different types of membrane can be used for the vapour permeation process like: polymeric membranes, and inorganic membranes, ceramic membranes [12].

##### 4.1 Process of synthesis of zeolite membrane

The membrane synthesis shows high flux and high selectivity for permeation. The azeotrope form by IPA and water can be easily break by the membrane and the process is known as 'total recovery improvement for IPA [14].

Various process has been used for the synthesis of zeolite membrane by various scientist which is discuss below:

##### A. Synthesis process used by Masakazu Kondo, Mitsuru Komori[17]

A gel used for the synthesis of NaA zeolite was prepared according to the procedure reported by Masakazu Kondo, Mitsuru Komori. The gel consist of aluminium hydroxide, sodium silicate, deionized water and sodium hydroxide. As a substrate of zeolite membrane Tubular porous supports is used. The support has inner diameter of 9mm and outer diameter of 12mm with length equal to 800mm. Before beginning the synthesis process, pretreatment of substrate is done to increase the formation of zeolite. SiC paper is used to polish the substrate and seed crystal is used for coating. In the next step hydrothermal treatment is done in the gel at 100°C for 3.5 h. Then at reduce pressure the membrane is dried at 70°C and washed with distil water. Result were analysed using X-ray diffraction (XRD) with CuK radiation, scanning electron microscopy (SEM) for surface morphologies and electron probe microanalysis (EPMA) for compositions of the specimen.

##### B. Synthesis process used by Yoshio Morigami a, Masakazu Kondo[16]

Porous tubular support with outer diameter of 12mm and length of 80cm is used for the synthesis of zeolite membrane. Hydrothermal synthesis method is used for the preparation of membrane. The mixture of dissolving sodium hydroxide, aluminium hydroxide in distilled water is used for the preparation and known as aluminate solution. And by dissolving the sodium silicate in distilled water, silicate solution is prepared. The silicate gel is mixed with aluminate solution and agitated vigorously, formation of homogenous gel take place. It consist of  $\text{SiO}_2/\text{Al}_2\text{O}_3=2.0$ ,  $\text{Na}_2\text{O}/\text{SiO}_2=1.0$  and  $\text{H}_2\text{O}/\text{Na}_2\text{O}=60$ . The membrane is coated with seed crystal and dried for 3-4 hours at 100°C. The analysis were done by using X-ray diffraction (XRD), scanning electron microscopy (SEM) for surface morphology and electron probe microanalysis (EPMA) for compositions of the membrane.

### C. Synthesis process used by Naotsugu Itoh, Jun Ishida [11].

A support of alumina tube with length of 11.5cm, outer diameter and inner diameter of 11.5mm and 2.5mm respectively were used. Here secondary growth method is used for the synthesis and the support were coated with polycrystalline NaY zeolite layer. The solution is prepared by mixing sodium oxide, sodium aluminate, water glass and distilled water with a molar ratio of 12.8 SiO<sub>2</sub>:1 Al<sub>2</sub>O<sub>3</sub>:17 Na<sub>2</sub>O:975 H<sub>2</sub>O. Before synthesis NaY type zeolite powder were coated on support tube. The polycrystalline NaY type zeolite layer will form on the support tube by placing it horizontally in a glass vessel which is pressure tight, filled with synthesis solution and should be kept in oven for 12 hours at 358 K. The membrane is washed with water and dried in oven at 333k after synthesis. The analysis were done by X-ray diffraction and scanning electron microscope.

### III. CONCLUSION

Azeotropic distillation is important separation process used in many chemical industry. Product purity is limited in azeotropic distillation which can be obtain using batch distillation. Adsorptive distillation has a potential to enhanced azeotropic distillation of IPA–water solution. More purity can be obtain in adsorptive distillation by increasing the height of packing and reflux ratio. It is found from the VLE experiment and process that adsorptive distillation cannot replace extractive distillation. In extractive and azeotropic distillation, entrainer selection play a very important role. . It is seen that extractive distillation is challenging and promising method than azeotropic and adsorption distillation method. In extractive and azeotropic distillation, entrainer selection play a very important role. There is a need of improving the distillation process in terms of cost and energy. For azeotropic and extractive distillation process require two distillation column so as a replacement zeolite membrane can be used. The cost of synthesis of zeolite membrane is more. From the literature analysis it is seen that by using vapour permeation process 9.7% reduction in operating cost and 48% saving in energy cost can be done. This process does not require entrainer.

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