

Short communication

A preliminary study: Distillation of isopropanol–water mixture using fixed adsorptive distillation method

Muhammad Mujiburohman^{a,*}, Wahyudi Budi Sediawan^b, Hary Sulisty^b^a Chemical Engineering Department of Muhammadiyah University of Surakarta (UMS), Surakarta, Indonesia^b Chemical Engineering Department of Gadjah Mada University (UGM), Yogyakarta, Indonesia

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Abstract

2-Propanol (isopropyl alcohol; IPA)–water mixture reflects a homogeneous minimum-boiling azeotrope (at 80.3–80.4 °C, 87.4–87.7 wt.%). Some methods of enhanced distillation of this mixture have been developed, such as azeotropic distillation, reactive and extractive distillation, and membrane separation either in the form of membrane distillation or pervaporation. This work is a preliminary exploratory study and denotes a state of the art of technology, called fixed adsorptive distillation, in which distillation and adsorption are applied simultaneously. Fixed adsorptive distillation consists of two Raschig ring-packing distillation columns, which are equipped with an inter-bed of adsorbent filled of silica gel. First column (I) distills the mixture to produce distillate at slightly below the azeotropic composition. Silica then adsorbs selectively one of the components (water), so that IPA rises in purity passing the azeotropic point. Finally, the second column (II) distills it to achieve higher composition or pure. This research has aims to investigate the possibility of fixed adsorptive distillation in application of enhancement of azeotropic IPA–water solution; the influence of feed composition of column I and the ratio of adsorptive and bypass flow on the purity of the second column product. The result shows that this method can pass the azeotropic point; the higher the feed composition of column I as well as the higher the ratio of flow is, the higher the purity of the second column product is.

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1. Introduction

At atmospheric condition, a binary mixture of 2-propanol (isopropyl alcohol, IPA)–water forms a homogeneous minimum-boiling azeotrope at 87.4–87.7 mass% and 80.3–80.4 °C [1]. Consequently, it is required in a particular way to break this point in behalf of obtaining very high purity or pure IPA. An azeotropic distillation with an entrainer of cyclohexane (CyH) has been done [2], in particular, to investigate the steady-state and dynamic behavior of IPA–CyH–water heterogeneous azeotropic column both theoretically and experimentally. The results also showed that the addition of CyH be able to break azeotropic point of IPA–water and yielded IPA with the purity of 99–100 wt.%

Some investigations have reported that IPA–water azeotrope can also be broken with other azeotropic distillations to form heterogeneous azeotropic systems by adding one of the following entrainers: isopropyl ether, benzene, methyl ethyl ketone, and isopropyl acetate. Sometimes, ethyl ether is used as entrainer at pressures substantially above atmospheric [1].

A modification technique, which is a combination of reactive and extractive distillation by using the mixture of ethylene glycol (C₂H₆O₂) and glycolic potassium (C₂H₅O₂K) as additional components has been investigated [3]. By this way, with a volume ratio of feed and solvent of 1:1, IPA produced has purity over 96.0 mass%. A novel method, sweep gas membrane distillation, which is similar to sweeping gas pervaporation has also been examined to purify the same system [4]. Another type of membrane separations, pervaporation, which is originally applied in dehydration of high concentration of organic compounds, was found potential to break IPA–water azeotropic as well and can be applied by

* Corresponding author.

E-mail addresses: mmujiburohman@yahoo.com,
mmujibur@engmail.uwaterloo.ca (M. Mujiburohman).

combining with distillation [5]. However, pervaporation itself still encounters some challenges in particular to the membrane productivity and membrane stability [6]. In addition, investigations of general azeotropic systems have also been studied by some researchers by applying the currently developed methods, such as azeotropic distillation [7], extractive distillation [7–10], reactive distillation [11,12], and adsorptive distillation slurry system [13,14]. In principle, those common methods require other specific components to be mixed directly together with the solution that will be separated.

This work was an exploratory study and was done with respect to develop a state of the art of technology, namely fixed adsorptive distillation. Referring to its name, the method applies simultaneously distillation and adsorption in which the regular form of adsorbent used in the adsorption process is placed in a separated fixed bed. The application of hybrid method which combines together distillation and adsorption has actually been studied [15], and a pressure swing adsorptive distillation has been enforced in industrial scale as well, but both used a molecular sieve adsorbent, a more complicated form of adsorbent. One thing that distinguishes the fixed adsorptive distillation from other common methods of azeotropic distillation is that the additional component is not mixed directly together with the solution to form a well-mixed solution or a slurry system, but is placed separately. By this circumstance, further separation between additional component and solution is not needed. This work utilized two raschig ring-packing distillation columns, which were equipped with an inter-bed of adsorbent filled of silica gel. The first column distilled the mixture of IPA–water to achieve the purity slightly below the azeotropic point, after that the condensate was split into two streams, one was passed through the bed of adsorbent (silica gel) and the other was bypassed. Because of its surface's polarity, silica then adsorbed selectively the more polar existing component, in this case, water. As a result, IPA underwent rise in purity passing the azeotropic composition. This higher purity mixture was then fed to the second column and be distilled to obtain very high purity or pure product. As a minimum-boiling homogeneous azeotrope, over the azeotropic point IPA became less volatile than water so that the main product was taken out from the bottom. The distillate of the second column, which its concentration has been above azeotropic point was recycled to the first column to maintain feed composition of the first column.

As an exploratory study, this research has primary aim to investigate the possibility of fixed adsorptive distillation method in application of enhanced distillation of azeotropic IPA–water mixture. The effect of two process conditions including a common variable, feed composition, and a specific variable, ratio of flow around adsorbent bed, on the purity of product were also studied. There is no specific reason to pick feed composition instead of other common variables as the investigated variable, because any process conditions will eventually be restricted by the existence of azeotropic point. Ratio of flow represents a comparison of volumetric

rate between stream passed through adsorbent and stream, which is bypassed. Split of condensate flow into two streams was done regarding to optimization of adsorption operation. Other common process conditions in distillation operation including number of equilibrium stage or height of packing, reflux ratio, and operating pressure were not investigated specifically.

2. Exploratory study

2.1. Basic concept and approach

Fixed adsorptive distillation is an integration of conventional techniques to overcome the separation problem in azeotropic solution. In theory, conventional distillation cannot pass azeotropic point even using infinite number of equilibrium stage or height of packing because of the existence of pinch point. This study tried to apply a hybrid separation technique to break or to pass the azeotropic point. One of conventional techniques that is considered to be able to handle this problem is adsorption. This technique changes only a little bit difference in concentration of component in the mixture. Hence, it is suitable to carry out this purpose, breaking azeotropic point. Therefore, in simply, fixed adsorptive distillation comprises three-zone operations: distillation–adsorption–distillation, as described in VLE diagram of Fig. 1.

Schematically, fixed adsorptive distillation can be simply figured as shown in Fig. 2.

Fig. 2 is, especially to illustrate this method in behalf of enhancement of minimum-boiling homogeneous azeotrope. However, it can also be modified to treat the maximum-boiling one by recycling the bottom product of the second column to the first column and take out the distillate as the main product. Based on Figs. 1 and 2, there are essentially three approaches used in the fixed adsorptive distillation method:

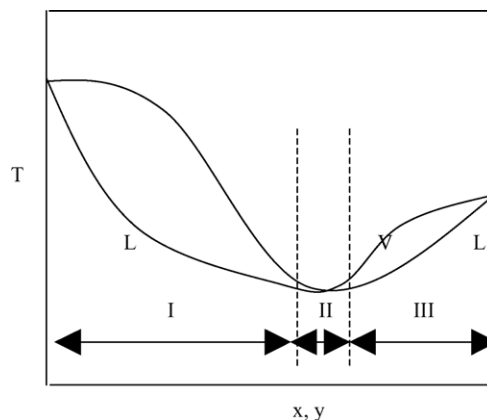


Fig. 1. Three-zone operations of fixed adsorptive distillation in minimum-boiling azeotrope: distillation (I), adsorption (II), and distillation (III).

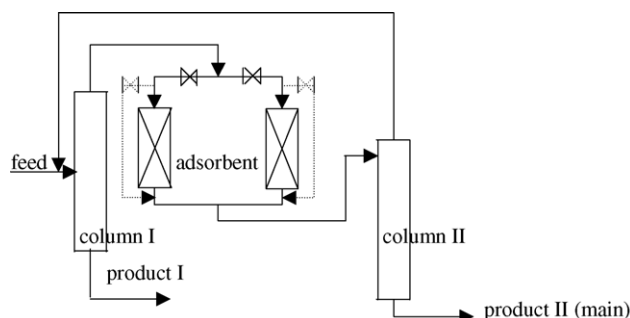


Fig. 2. Schema of enhanced distillation of azeotropic solution using fixed adsorptive distillation.

- (1) Application of two separation techniques in three-zone operations: (a) distillation from range of feed concentration of zero to slightly below azeotropic point; (b) adsorption from slightly below to slightly above azeotropic point; (c) distillation from slightly above azeotropic point to 100%.
- (2) Application of two streams in adsorption operation, one passed through adsorbent and the other is bypassed to optimize the adsorption process.
- (3) Application of recycle of side product to maintain the feed concentration.

The approach (2) in this method is considered as the most distinctive application from other adsorptive distillation methods.

Fixed adsorptive distillation uses a specified adsorbent located between the two distillation columns to apply separation based on adsorption. Adsorption is separation of components in fluid phase (liquid or gas) in which the component will be adsorbed onto the active surface of solid [16]. Because one of components is adsorbed selectively or more strongly, the unadsorbed component will increase in its composition in fluid phase. This is basically the principle of fixed adsorptive distillation, an attempt to pass the azeotropic point by adsorbing the undesired component. Even though adsorbent has ability to capture all components existing in the mixture, the adsorbates are adsorbed in different proportion due to their different polarities. In this case, the desired component in the main product is hopefully the least captured component.

The adsorbent is set fixedly in a bed, so, as the process runs the adsorbent will be dense with the adsorbed component, and in a certain time it will be saturated. In this condition, the adsorption process does not work, and will disturb the whole process. To avoid the adsorbent undergoing saturated in short time, it is needed to minimize the flow passed through the adsorbent, or in other word to maximize the bypass flow. As consequently, fixed adsorptive distillation utilizes two streams around the condenser and bed of adsorbent. The ratio of flow has significant role to optimize the adsorption process. If the flow flown through the adsorbent is too high, it will produce better mixture composition, or in other word, adequate far above azeotropic point. However,

the adsorbent will be fast saturated or non-durable use so that regeneration must be frequently done and more energy is required to activate the saturated adsorbent. In contrast, if the flow is low enough, the adsorbent will be durable in use but the composition of the mixture entering the second column might be still below the azeotropic point. As abovementioned, the use of two streams around adsorbent bed mainly deals with an effort to optimize the operation of adsorption. It is not urgent to flow all condensates of the first column through the adsorbent because the major purpose of this approach is to attain a mixture entering the second column with composition at least slightly above the azeotropic point. The application of this approach, however, cannot eliminate the saturation of adsorbent. As a result, to ensure the continuity of the whole process, the utilization of two parallel adsorbent beds is still needed as the common adsorption operation. When one adsorbent part undergoes saturation, the other one is then used, while the saturated one is being regenerated. In principle, some methods of regeneration including thermal and pressure are able to be applied due to no strict requirements in this regular adsorbent re-activation which is placed separately from the mixture system. Thermal regeneration will probably be more interesting to be used because at the same time the vapor mixture from the top of the first column releases amount of heat to condense. This mixture's latent heat can cover partially or completely the heat required to desorb the adsorbates from the surface of adsorbent. A combination of thermal and pressure swing will be more effective, but it will of course be more costly.

This laboratory study was run in a batch operation. However, basically fixed adsorptive distillation can also be operated continuously as many applications in industries. The feed of the first column and the bottom products of both the first and the second columns can be fed and put out continuously. The saturation of adsorbent that disturbs the whole process can be anticipated by the application of two parallel adsorbent bed as above mentioned.

In comparison with the currently applied methods, fixed adsorptive distillation has some advantages as follows:

- (a) *Selection of additional component (adsorbent)*: The selection of the type of adsorbent is easier than that of the entrainer, solvent, catalyst, or inert. In general, there are two kinds of adsorbents, i.e. hydrophobic and hydrophilic adsorbents [17]. Hydrophobic, or non-polar surface adsorbent including active carbon and polymer materials, will prefer to adsorb the non-polar component than the polar one, whereas hydrophilic including silica gel and active alumina is in vice versa. The selection of adsorbent will strongly depend on the polarity of component that will be adsorbed. The strength of polarity of some components can be easily known from the functional chemical compounds existing in the component. Even though adsorbent has capacity to adsorb all components existing in the mixture, the components will be captured in different proportion due to their various polarity.

- (b) *Addition of component*: Both in batch and continuous processes, the addition of adsorbent is enforced only once in the early stage of process. The saturation of adsorbent can be anticipated by the use of two parallel adsorbent beds in which adsorption and regeneration are operated simultaneously.
- (c) *Design of equipment*: The design of equipment is simpler because there are no changes in equilibrium-phase curve due to the addition of adsorbent. In other word, the addition of adsorbent does not convert a binary system into multi-system distillation. Distillation process at the two columns of fixed adsorptive distillation is intrinsically similar to the conventional distillation and the performance as well.
- (d) *Cost requirement*: In general, total annual cost of a process is affected by two main cost components, i.e. investment and operating expenses. In case of a large and long-life production, operating cost usually gives more contribution to the total annual cost rather than investment cost. Based on rough estimation and assumption that the latent heat released by the vapor mixture of the first column could provide energy for thermal regeneration, using adsorbents rather than other additional chemicals (entrainer, solvent, or catalyst) is a potential way to reduce operating cost because of the cheapness of adsorbents.

3. Experimental method

Materials used in this experiment involve: IPA 99.7%, water, silica gel of which average size is 1.19 mm. The set of equipment consists of two packing columns with the diameters of 4.5 cm and 4 cm, respectively, and the height of packing of 32.1 cm and 25 cm; two bowls with capacity of 1 L act as the reboiler I and II; two condensers; an accumulator; a bed of adsorbent with diameter of 2.4 cm and height of 10.1 cm. The columns are packed with glass raschig ring of which measurement is 6–6.5 mm of height, 4.5 mm of inside diameter, and 6.5 mm of outside diameter.

This experiment is an exploratory study, so the first aim that will be achieved is to know the possibility of fixed adsorptive distillation as an alternative method in enhanced distillation of azeotropic IPA–water solution. Moreover, this research also investigated two variables, which affect the purity of product in this process, i.e. feed composition of column I and the ratio of adsorptive flow and bypass flow. Adsorptive flow is distillate of column I, which is passed through the bed of adsorbent, whereas bypass flow is that which is not passed through the bed. The composition of feed is varied as follows: 87.2%, 85.1%, 80.4%, 70.1%, and 60.0%, and the variations of the ratio of flow are 8.80, 4.52, 2.67, and 2.07.

The experiment is started by filling 278 mL of IPA–water with various compositions into column I and then be distilled. The distillate of column I is divided into two

streams with various ratio, one to bed of adsorbent and the other to bypass. The two flows are remixed again before entering column II. The distillate of column II is condensed and recycled to column I. The samples of product are taken from the reboiler II at a certain interval of time, and stopped after a definite time of operation.

4. Results and discussion

4.1. Effect of feed composition of column I

To investigate the effect of feed composition of column I on purity of the second column product, other variables that can also influence the purity are kept constant, i.e. height of packing columns I and II, reflux ratio, height of adsorbent bed, and the ratio of adsorptive flow:bypass flow (4.4 mL/min:0.5 mL/min). The result is tabulated in Table 1.

Table 1 and Fig. 3 show that fixed adsorptive distillation method is proved being able to break the azeotropic point. This is referring to the fact that most of the second product's purity has passed the azeotropic composition (87.4 wt.%). Those data also justify the possibility of this method as an alternative technique in enhanced distillation of azeotropic solution. From the same data, it can be seen that at the same operating time, the higher the composition of IPA in the feed of column I is, the higher the purity of the second product is. It can be explained that theoretically, distillation of a minimum-boiling azeotrope solution with the feed composition under the azeotropic point will yield distillate whose purity is higher than the initial composition. This is because of that vapor phase contains more concentration of component than that in liquid phase. The purity of distillate increases up to slightly below the azeotropic point as the feed composition rises. This distillate is then fed into the second column after being passed through the bed of adsorbent. The higher the purity of distillate of the first column is, the higher the feed composition of the second column is, and causes the purity of the second column product more pure.

At a given feed composition, from a certain time there is fluctuate decline in the purity of the second column. This

Table 1
The effect of feed composition of column I to the purity of second column product in the fixed adsorptive distillation

<i>t</i> (min)	The purity of second column product (mass%)				
	87.2% ^a	85.1% ^a	80.4% ^a	70.1% ^a	60.0% ^a
25	92.7	93.3	91.3	84.7	63.1
30	94.5	92.4	–	85.0	66.7
35	97.8	92.5	89.1	83.5	65.2
40	95.8	91.4	91.2	86.8	65.7
45	93.7	92.4	90.8	85.3	58.4
50	95.8	90.9	89.6	86.1	64.6
55	99.5	92.5	90.2	85.8	69.0
60	–	92.6	88.9	82.7	65.8

Note: %, weight percent.

^a Feed composition.

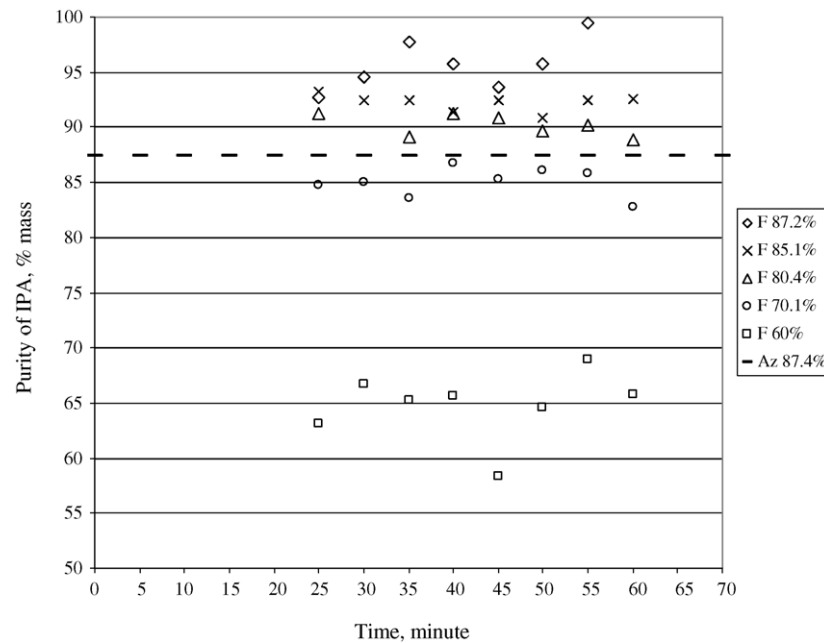


Fig. 3. The effect of feed composition of column I to the purity of product of column II at various times.

phenomenon indicates the surface of adsorbent be denser of solute, the driving force of adsorption becomes less as well as the rate of mass transfer. The fluctuation occurs due to recycling of distillate from the second column to the first one. Moreover, this experiment is a batch process in which at a constant reflux both top and bottom products of the distillation column will change in composition every time.

At composition of 87.2%, 85.1%, and 80.4%, this method shows good performance and success in passing the azeotropic point. At those feed compositions, the purity of the second product is always above the azeotropic composition during the operation. The time is restricted by 60 min because most silica gel becomes saturated known from the change of its color. At the feed composition of 70.1% or below it, fixed adsorptive distillation fails to break the azeotropic point. This failure is mainly caused by the limited height of the packing. The height of packing is not sufficient to provide mass contact between vapor and liquid to achieve the purity of distillate enabling the adsorption process to pass the azeotropic point. The value of HETP of a raschig ring with the measurement of 1/4 in. \times 1/4 in. (6.35 mm \times 6.35 mm \approx 6.5 mm \times 6.5 mm) is 0.1–0.6 ft (or average value of 0.35 ft = 10.67 cm) [18]. In this experiment, the height of packing is 32.1 cm, based on the HETP concept, the number of equilibrium stage will be three stages only (32.1 cm: 10.67 cm/stage = 3.01 \approx 3). The restricted ability of adsorbent in adsorbing causes the distillate, which is still under the azeotropic point cannot pass it. Because its composition is still below the azeotropic point, the second conventional distillation will also be unable to obtain the purity above its azeotrope. By comparing the results of feed composition of 70.1% and 80.1%, it can be inferred

that to ensure the investigated method success in enhanced distillation of azeotropic solution based on this experimental condition, the feed composition should be set up at least a little bit above the composition of 70.1%.

4.2. Effect of ratio of adsorptive flow:bypass flow

This ratio is important to study because it will be close related to the operating cost of the fixed adsorptive distillation process. High ratio is beneficial to make sure the success of this method in passing the azeotropic point. However, it will have consequence that the adsorbent will be saturated rapidly. As a result, regeneration must be conducted more frequently, and will raise the operating cost. In contrast, if the ratio is too low, on the one hand, the adsorbent will be more durable usage, in the other hand, this condition might fail to break the azeotropic point. Therefore, it is urgent to determine the limit ratio to guarantee the process run well. To investigate the influence of this variable, other variables which basically also affect the result are maintained constant as the treatment of the first variable (feed composition of column I).

As the previous data, Table 2 and Fig. 4 also justify that fixed adsorptive distillation method can be utilized in enhanced distillation of azeotropic solution. At the same operating time, the higher the ratio is, the higher the purity of the second column product is. This trend occurs due to the fact that the distilled passed through the adsorbent is much more in proportion than that be bypassed. It enlarges in amount of the distillate, which passes the azeotropic point, and will automatically raise the composition of the second column feed. For the given height of packing and reflux ratio, the higher the feed composition of the second column is, the

Table 2

The effect of the ratio of adsorptive flow:bypass flow to the purity of second column product in the fixed adsorptive distillation

<i>t</i> (min)	The purity of second column product (mass%)			
	8.80 ^a	4.52 ^a	2.67 ^a	2.07 ^a
25	92.7	–	86.2	74.7
30	94.5	87.9	79.2	73.0
35	97.8	88.2	89.2	66.5
40	95.8	88.0	91.6	61.9
45	93.7	–	89.3	53.6
50	95.8	92.7	77.7	–
55	99.5	95.4	84.4	83.6
60	–	96.4	83.0	84.4

Note: ratio = comparison of adsorptive flow:bypass flow.

^a Ratio.

higher its product's purity is. At a value of ratio, after a definite time of operation the purity of the second column product fluctuates with time. As the preceding explanation, this phenomenon happens because of the changes of mass transfer rate in adsorption and the recycle of distillate from column II.

The result shows that at the ratio of 8.80 (4.4 mL/min:0.5 mL/min) and 4.52 (6.1 mL/min:1.35 mL/min), the purity of the second column product during the operation is always above the azeotropic composition. This indicates at those ratios fixed adsorptive distillation operates successfully. In other case, if the ratio is set at 2.67 (3.6 mL/min:1.35 mL/min), there will be variation in purity above and under the azeotropic point. So, to make this method work properly with this experimental condition, it is advised to set up the ratio slightly above 2.67. At the ratio below the value of 2.67, in this experiment was represented by 2.07 (2.8 mL/min:1.35 mL/min), this method fails to

break the azeotropic point. Initially, within 45 min, the purity of the product decreases gradually, then after around 50 min, it increases approach to azeotropic composition. This phenomenon happens due to the unsuccessful mixing between the flow passed through the adsorbent and the bypass flow. During 45 min in the early process, almost all the feed of the second column derives only from the bypass flow. The condensate, which is flown to adsorbent takes time to reach the mixing zone because it must pass through a porous media of adsorbent, moreover, the flow rate is not much enough. The decline of the product purity during this period of time is caused by the drop of the purity of condensate as in a batch distillation, and directly decreases the feed composition of the second column. Since the feed of the second column has composition still below azeotropic point, it is essentially similar to the first column that works under azeotropic point and cannot obtain a top product with the purity above its azeotrope. The product has purity below the feed composition of the first column, because it must be noticed that the product is taken out from the bottom site, which is less concentration than the top site. After around 50 min operation, the adsorptive flow and bypass flow mix well, however, the composition of this mixture does not exceed the azeotropic point, a pre-requirement of the success of this method.

From the two investigated variables, it can be inferred that fixed adsorptive distillation is able to be applied in enhancement of azeotropic IPA–water solution. The most important thing emphasized in this study is the ability of this method to make the azeotropic IPA–water solution has composition above its azeotropic point from below it. If it is required to produce more pure product, some efforts can be enforced to improve the performance of the second

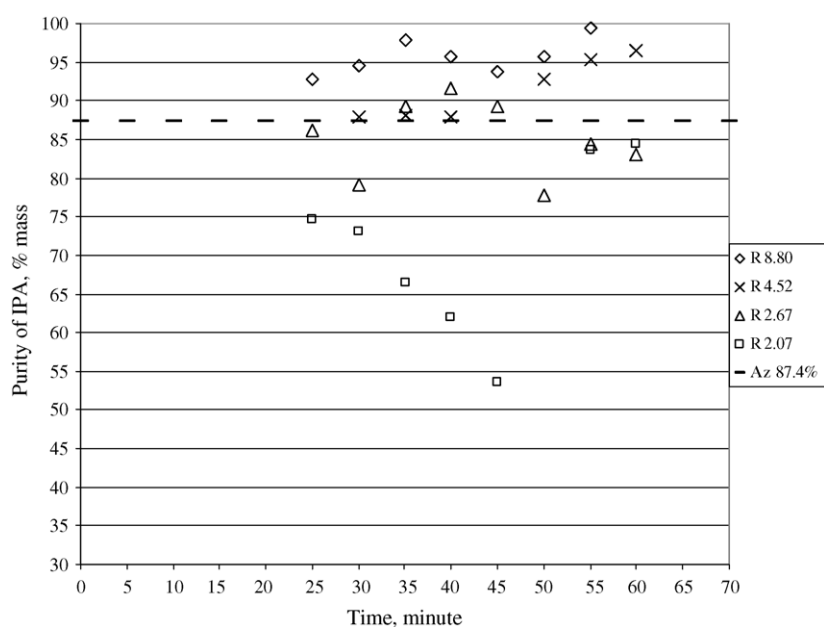


Fig. 4. The effect of ratio of adsorptive flow:bypass flow to the purity of product of column II at various time (R: ratio).

column, such as prolonging the height of packing and increasing the value of reflux ratio as well. Even though this study is an exploratory research, it might give some recommendations as follows: fixed adsorptive distillation has potential application in enhanced distillation of azeotropic IPA–water solution, the composition of the first column, and the value of ratio of adsorptive and bypass flow have roles in determining the purity of the second column product.

4.3. Future work

Intensive investigations will be preferably done related to the problems, which are inherent to adsorbent behavior, such as adsorption theory with two or more adsorbed components, and regeneration process in conjunction with integrated energy around condenser and bed adsorbent. The most common theory developed in adsorption phenomena is concerning a single adsorbed component. In this method, knowing the proportion of all components (water and IPA) which are adsorbed onto the surface of adsorbent (silica gel), is very important to develop a mathematical model that can predict some quantitative parameters including the amount of IPA and water adsorbed (water purity), the necessity of adsorbent, and the product purity for a given process condition. Finally, deep economical analysis by comparing this hybrid method from others then can justify whether this art of technology is feasible or not.

5. Conclusions

Some points can be concluded from this exploratory study. As the objective of this research, fixed adsorptive distillation method is proved having potential ability in enhanced distillation of azeotropic IPA–water solution. The most important thing emphasized in this hybrid method is an attempt to break or pass azeotropic point by applying an optimized adsorption process in which not all condensates from the first column are flown through the adsorbent bed. More efforts can be performed with respect to enhancement of the product purity by improving the performance the second distillation column, such as the addition of equilibrium stage or height of packing as well as the increase of reflux ratio. The two investigated variables, feed composition of column I and flow ratio, are able to influence the purity of the second column product. Within the range of feed concentration investigated in this study, the higher the feed composition of the first column is, the higher the purity of the second column product is. Based on these process conditions, packing height of columns I and II 32.1 cm and 25 cm, respectively, height of adsorbent 10.1 cm, and the ratio of flow 8.80, it is suggested to operate this method at the feed composition of the first column above 70.1 mass%. Likewise, the higher the ratio of adsorptive and bypass flow is, the higher the purity of the second column product is. Based on the similar process conditions with the feed composition of the first column 87.2%, it is advised to

operate this method at the ratio flow above 2.67. In addition, this method has opportunity to handle general homogeneous azeotropic system both minimum and maximum boiling by modifying type of adsorbents and recycle system. Finally, deeply economical analysis is urgently done to adjudge the feasibility of this method.

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