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Dehydration of Ethanol by Adsorptive Distillation

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Abstract: This The production of anhydrous ethanol is of significant importance in the fuel, pharmaceutical, and chemical industries. However, conventional distillation is restricted by the formation of an ethanol–water azeotrope, which limits ethanol purity to approximately 95.6%. Adsorption-based dehydration techniques have emerged as promising alternatives for overcoming this limitation due to their high selectivity, low energy requirements, and operational simplicity. In the present study, ethanol dehydration was investigated using adsorption and adsorptive distillation techniques employing molecular sieves (3A), coconut shell activated carbon, and silica gel as adsorbents.

Four experimental trials were conducted using ethanol–water mixtures containing 90% ethanol and 10% water by volume. In the first two trials, liquid-phase adsorption was performed by initially distilling the feed mixture and subsequently passing the condensate through adsorption columns packed with molecular sieves (3A), coconut shell activated carbon, and silica gel arranged in series. Ethanol purities of 91.10% and 90.63% were obtained for feed compositions of 900 mL ethanol + 100 mL water and 1800 mL ethanol + 200 mL water, respectively.

In the third and fourth trials, a vapor-phase adsorptive distillation approach was employed. Ethanol–water vapours generated during distillation were passed directly through adsorption columns containing 50 g molecular sieves (3A) and 50 g silica gel before condensation. Ethanol purities of 93.29% and 97.43% were achieved for feed compositions of 450 mL ethanol + 50 mL water and 90 mL ethanol + 10 mL water, respectively. Ethanol concentration was determined using an Anton Paar analyser.

The results demonstrate that vapor-phase adsorptive distillation provides superior dehydration performance compared to liquid-phase adsorption. Furthermore, the incorporation of silica gel and activated carbon reduced the dependence on costly molecular sieves, thereby improving the economic viability of the process. The highest ethanol purity obtained was 97.43%, indicating that the combined adsorption system is an effective approach for ethanol dehydration. The study highlights the potential of integrating molecular sieves with low-cost adsorbents for the development of efficient and economically sustainable ethanol purification processes.

Keywords: Ethanol dehydration, Adsorptive distillation, Molecular sieves (3A), Silica gel, Activated carbon, Adsorption, Ethanol purification, Bioethanol.

I. INTRODUCTION

Ethanol is one of the most widely used chemicals in the pharmaceutical, chemical, food, cosmetic, and fuel industries. The growing demand for renewable and environmentally friendly fuels has increased the importance of bioethanol production. Ethanol is commonly produced through the fermentation of biomass feedstocks such as sugarcane, molasses, corn, and agricultural residues. However, fermentation produces ethanol containing a significant amount of water, which must be removed before industrial utilization.

The purification of ethanol is challenging because ethanol and water form a minimum-boiling azeotrope at approximately 95.6% ethanol. Due to this azeotropic behaviour, conventional distillation cannot produce completely anhydrous ethanol. Therefore, alternative dehydration techniques are required to achieve higher ethanol purity [1].

Various separation techniques such as azeotropic distillation, extractive distillation, membrane separation, adsorption, and adsorptive distillation have been developed to overcome the azeotropic limitation [1,2]. Among these methods, adsorption has emerged as an attractive dehydration technique because of its simplicity, lower energy requirement, and high selectivity for water removal [2].

Molecular sieves, particularly 3A zeolites, are widely used for ethanol dehydration because of their ability to selectively adsorb water molecules while excluding larger ethanol molecules [2,8]. Studies have reported that molecular sieve

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adsorption can effectively produce ethanol with purity greater than 99%, making it suitable for industrial applications [2,8].

Several researchers have investigated alternative low-cost adsorbents to reduce the dependence on expensive molecular sieves. Adsorbents such as activated carbon, silica gel, natural zeolites, and agricultural waste-derived materials have demonstrated promising water adsorption capabilities [4,5,6]. The use of such materials can significantly reduce operating costs while maintaining acceptable dehydration performance.

Recent studies have shown that combining adsorption with distillation can further improve ethanol dehydration efficiency. In adsorptive distillation, ethanol–water vapours are directly contacted with adsorbents before condensation, resulting in enhanced water removal and improved ethanol purity [3,6,7]. Experimental investigations have demonstrated that adsorptive distillation can produce ethanol concentrations beyond the azeotropic limit and, in some cases, achieve purities greater than 99% [3,7].

Although molecular sieves provide excellent dehydration performance, their relatively high cost remains a challenge. Consequently, there is increasing interest in combining molecular sieves with economical adsorbents such as silica gel and activated carbon to develop cost-effective dehydration systems [5,7,9].

In the present work, ethanol dehydration was carried out using adsorption and adsorptive distillation techniques. Molecular sieves (3A), coconut shell activated carbon, and silica gel were employed as adsorbents. The performance of liquid-phase adsorption and vapor-phase adsorptive distillation was evaluated and compared. The study aims to develop an efficient and economical process for ethanol dehydration while minimizing the consumption of expensive molecular sieves.

II. EXPERIMENTAL METHOD

A. Materials and Methods

1. Materials:

Commercial ethanol and distilled water were used to prepare ethanol–water mixtures for dehydration experiments. Molecular sieves (3A), silica gel, and coconut shell activated carbon were selected as adsorbents. Molecular sieves (3A) are widely employed for ethanol dehydration because their pore size selectively adsorbs water molecules while excluding ethanol molecules, resulting in effective moisture removal from ethanol–water mixtures [1,2]. Silica gel was used as a secondary adsorbent due to its high affinity for water and low cost, while coconut shell activated carbon was incorporated to provide additional adsorption capacity and reduce the consumption of expensive molecular sieves [5,7].

2. Experimental Setup:

The experimental setup consisted of a batch distillation unit, adsorption columns, a condenser, and a product collection vessel. For liquid-phase adsorption experiments, three adsorption columns were connected in series and packed with molecular sieves (3A), coconut shell activated carbon, and silica gel, respectively. Each adsorption column contained 100 g of adsorbent.

For vapor-phase adsorptive distillation experiments, the distillation unit was connected directly to two adsorption columns arranged in series. The first column contained 50 g of molecular sieves (3A), while the second column contained 50 g of silica gel. Ethanol–water vapours generated in the distillation flask were passed through the adsorption columns before entering a water-cooled condenser. The condensed product was collected in a receiving flask for analysis. The experimental arrangement was based on the adsorptive distillation concepts reported in previous studies on ethanol dehydration [3,6,7].

3. Preparation of Feed Mixture:

The feed mixture was prepared by mixing ethanol and distilled water in a volumetric ratio of 90:10. Four different feed quantities were investigated:

TABLE I FEED QUANTITIES FOR EACH TRIAL

Trial	Quantity (Ethanol + Water)
Trial 1	900 mL ethanol + 100 mL water
Trial 2	1800 mL ethanol + 200 mL water
Trial 3	450 mL ethanol + 50 mL water
Trial 4	90 mL ethanol + 10 mL water

The selected feed composition represents a typical ethanol–water mixture requiring dehydration beyond conventional distillation limits [1,3].

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4. Liquid-Phase Adsorption Procedure:

In Trials 1 and 2, the prepared ethanol–water mixture was first subjected to simple batch distillation under atmospheric pressure. The condensed distillate was subsequently passed through three adsorption columns packed alternately with molecular sieves (3A), coconut shell activated carbon, and silica gel. Each adsorption bed contained 100 g of adsorbent. The treated ethanol was collected after passing through all adsorption columns and stored for purity analysis.

The arrangement of adsorbents was selected to utilize the high water selectivity of molecular sieves while reducing overall process cost through the incorporation of activated carbon and silica gel [2,5].

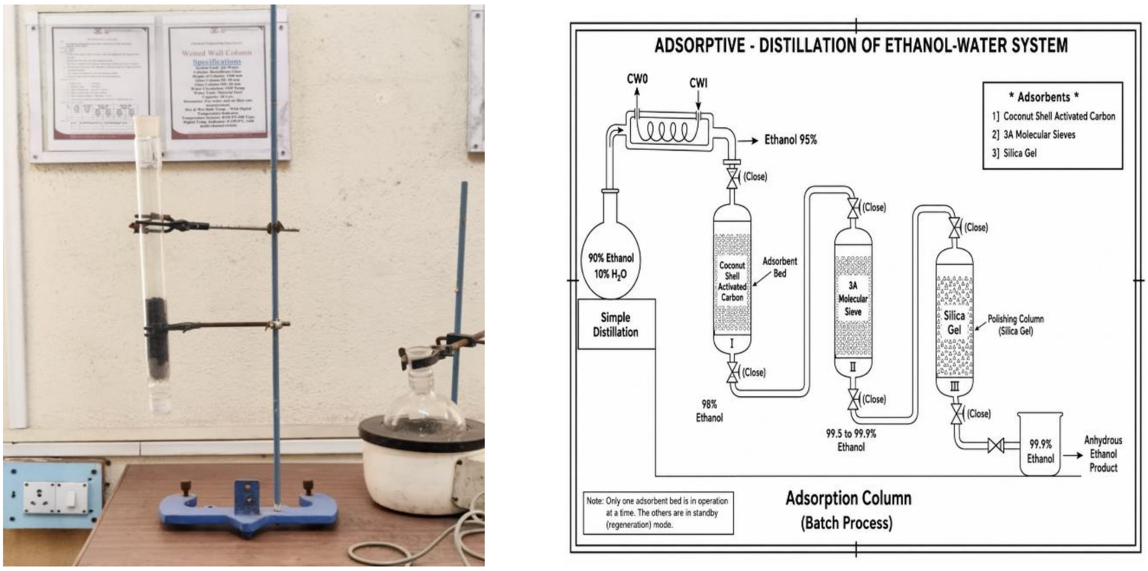

Fig. 1 Trial 1&2 Setup for Batch Process

5. Vapor-Phase Adsorptive Distillation Procedure:

In Trials 3 and 4, adsorptive distillation was performed. The ethanol–water feed was introduced into the distillation flask and heated under atmospheric pressure to generate vapours. The resulting vapours were passed directly through adsorption columns containing 50 g of molecular sieves (3A) followed by 50 g of silica gel connected in series. Water molecules were selectively adsorbed from the vapor stream before condensation.

The dehydrated vapours were then condensed using a water-cooled condenser, and the product ethanol was collected for analysis. Vapor-phase adsorption has been reported to provide superior dehydration efficiency compared to liquid-phase adsorption because of improved mass transfer and enhanced contact between vapor molecules and adsorbent surfaces [3,6,7].

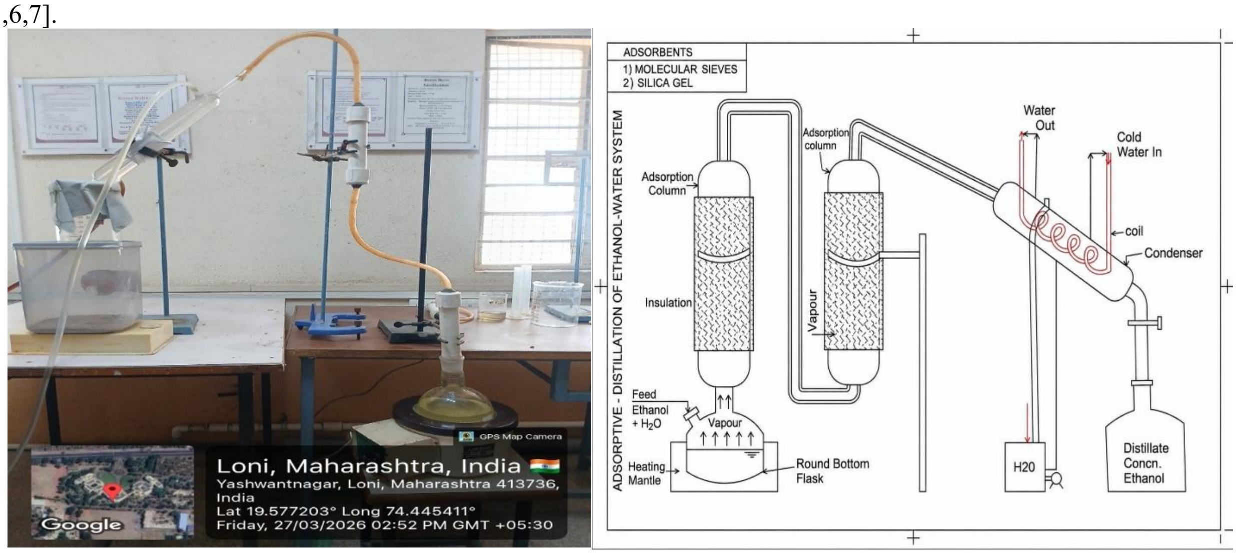

Fig. 2 Trial 3&4 Setup for continuous process

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6. Product Analysis:

The ethanol concentration of the collected samples was determined using an Density Meter (Anton Paar). The instrument was used to measure ethanol purity after each experimental trial. The obtained results were used to compare the effectiveness of liquid-phase adsorption and vapor-phase adsorptive distillation.

Fig. 3 Density Meter (Anton Paar)

7. Experimental Conditions:

All experiments were conducted under atmospheric pressure and batch operating conditions. The feed composition was maintained at 90% ethanol and 10% water for all trials. Adsorbent loading was fixed at 100 g per adsorbent bed for liquid-phase adsorption experiments and 50 g per adsorbent bed for vapor-phase adsorptive distillation experiments. The effect of adsorption mode and adsorbent configuration on ethanol purity was evaluated through comparative analysis.

III. RESULTS AND DISCUSSION

A. Ethanol Purity Obtained in Different Experimental Trials

The performance of the adsorption and adsorptive distillation processes was evaluated based on the final ethanol purity obtained after treatment. The ethanol concentration was measured using an Anton Paar analyser. Table 1 summarizes the experimental results.

TABLEs. ETHANOL PURITY OBTAINED IN DIFFERENT TRIALS

Results of Setup 1 for Batch process

TABLE II TRIAL 1 RESULTS

Step	Process Stage	Ethanol Purity %
1	After Simple Distillation	89.42%
2	Activated charcoal	90.17%
3	Molecular sieve	90.56%
4	Silica Gel	91.1%

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TABLE III TRIAL 2 RESULTS

Step	Process Stage	Ethanol purity %
1	After Simple Distillation	89.42%
2	Silica Gel	89.67%
3	Activated Charcoal	90.12%
4	Molecular sieve	90.63%

Results of Setup 2 for continuous process

TABLE IV TRIAL 3 RESULTS

Step	Process Stage	Ethanol Purity %
1	After Simple Distillation	89.42%
2	Molecular sieve	92.94%
3	Silica Gel	93.29%

TABLE V TRIAL 4 RESULTS

Step	Process Stage	Ethanol Purity %
1	After Simple Distillation	89.42%
2	Molecular sieve	96.72%
3	Silica Gel	97.43%

The results indicate that ethanol purity varied depending on the dehydration technique and operating conditions employed. The highest ethanol purity of 97.43% was achieved in Trial 4 using vapor-phase adsorptive distillation, while the lowest purity of 90.63% was obtained in Trial 2 using liquid-phase adsorption.

B. Performance of Liquid-Phase Adsorption

Trials 1 and 2 were conducted using liquid-phase adsorption in which the ethanol–water mixture was initially distilled and the condensed distillate was subsequently passed through adsorption columns packed with molecular sieves (3A), coconut shell activated carbon, and silica gel.

Trial 1 achieved an ethanol purity of 91.10%, indicating that the combination of adsorbents was capable of removing a significant portion of water from the ethanol–water mixture. Molecular sieves selectively adsorbed water molecules, while activated carbon and silica gel contributed to additional moisture removal. Similar observations have been reported Similar observations have been reported in previous studies [2,5], who demonstrated the effectiveness of molecular sieves in ethanol dehydration, and by [5], who reported the usefulness of activated carbon in water adsorption.

In Trial 2, ethanol purity decreased slightly to 90.63% despite maintaining the same feed composition. This reduction may be attributed to the larger feed volume processed through the adsorption beds. The increased liquid loading could have led to partial saturation of the adsorbents, reducing the overall water adsorption capacity and consequently lowering dehydration efficiency.

The results suggest that while liquid-phase adsorption is capable of improving ethanol purity, its effectiveness is limited by mass-transfer resistance and adsorbent saturation effects.

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C. Performance of Vapor-Phase Adsorptive Distillation

Trials 3 and 4 employed vapor-phase adsorptive distillation, where ethanol–water vapours generated during distillation were passed directly through adsorption columns containing molecular sieves and silica gel before condensation.

Trial 3 produced ethanol with a purity of 93.29%, which was higher than the values obtained from liquid-phase adsorption. The improved performance can be attributed to enhanced vapor-solid contact and reduced diffusion resistance during adsorption. Water molecules present in the vapor phase were more readily adsorbed by the molecular sieves and silica gel, resulting in greater dehydration efficiency.

Trial 4 achieved the highest ethanol purity of 97.43%. The superior performance observed in this trial may be associated with the lower feed quantity, which allowed more effective utilization of the available adsorption sites. The molecular sieves efficiently removed water molecules, while silica gel acted as a secondary adsorbent, further reducing residual moisture content. These findings are consistent with These findings are consistent with previously reported studies on adsorptive distillation [3,6,7], who reported that vapor-phase adsorptive distillation provides higher ethanol purity than conventional adsorption methods.

D. Comparison Between Liquid-Phase Adsorption and Vapor-Phase Adsorptive Distillation

A comparison of the experimental results clearly indicates the superiority of vapor-phase adsorptive distillation over liquid-phase adsorption. The average ethanol purity obtained through liquid-phase adsorption was 90.87%, whereas vapor-phase adsorptive distillation achieved an average purity of 95.36%.

The enhanced performance of adsorptive distillation can be attributed to improved mass transfer characteristics and direct interaction between ethanol–water vapours and adsorbent surfaces. In vapor-phase systems, water molecules diffuse more readily into the pores of the adsorbent, resulting in greater adsorption efficiency. Furthermore, the integration of adsorption with distillation enables simultaneous separation and dehydration, thereby improving overall process effectiveness.

The results obtained in this study support previous findings reported in the literature, where molecular sieves have been identified as highly effective adsorbents for ethanol dehydration. The incorporation of silica gel and activated carbon also contributed to moisture removal while reducing dependence on expensive molecular sieves.

E. Effect of Adsorbent Combination on Dehydration Performance

The combination of molecular sieves, silica gel, and activated carbon proved beneficial for ethanol dehydration. Molecular sieves acted as the primary adsorbent due to their high selectivity toward water molecules. Silica gel provided supplementary moisture adsorption, while activated carbon contributed additional adsorption capacity and reduced the overall adsorbent cost.

The results demonstrate that the use of low-cost adsorbents in conjunction with molecular sieves can improve the economic feasibility of ethanol dehydration systems without significantly compromising performance. This finding is particularly important for small-scale and industrial applications where adsorbent cost is a major consideration.

IV. CONCLUSION

The present study investigated the dehydration of ethanol using liquid-phase adsorption and vapor-phase adsorptive distillation employing molecular sieves (3A), silica gel, and coconut shell activated carbon as adsorbents. Four experimental trials were conducted using ethanol–water mixtures containing 90% ethanol and 10% water by volume to evaluate the effectiveness of different dehydration approaches.

The results demonstrated that both adsorption techniques improved ethanol purity; however, vapor-phase adsorptive distillation exhibited superior performance compared to liquid-phase adsorption. The liquid-phase adsorption trials achieved ethanol purities of 91.10% and 90.63%, whereas vapor-phase adsorptive distillation produced ethanol purities of 93.29% and 97.43%. The highest purity of 97.43% was obtained in Trial 4, indicating the effectiveness of direct vapor-phase contact between the ethanol–water vapor stream and the adsorbent beds.

The study further demonstrated that the incorporation of silica gel and coconut shell activated carbon can reduce the dependence on expensive molecular sieves, thereby improving the economic feasibility of the dehydration process. Molecular sieves acted as the primary water-selective adsorbent, while silica gel and activated carbon provided supplementary moisture removal and additional adsorption capacity.

Overall, the findings confirm that adsorptive distillation is a promising and efficient technique for ethanol dehydration and can achieve higher ethanol purity than conventional adsorption methods. The combined use of molecular sieves with low-cost adsorbents offers a practical approach for developing cost-effective ethanol purification systems. Further studies involving adsorbent regeneration, optimization of adsorbent loading, and continuous operation are recommended to enhance process performance and facilitate industrial-scale implementation.

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