Designing and Constructing an Optimization Operating Model for a Bioethanol Production System

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Abstract: Around the world, there are efforts underway to develop alternative sources of energy. One area in which advancements are being made is that of car fuel, and a new alternative from traditional fuels is oil with bioethanol which helps to reduce air pollution. Anhydrous ethanol is commonly produced with the use of molecular sieves to adsorb the water in ethanol samples. Regeneration, which is instigated after molecular sieves become saturated, involves the use of continuous heating with high-temperature nitrogen. The course of this study involved the construction of sufficient equipment to produce ethanol on a medium-scale, at a rate of approximately 600 L of 95wt% ethanol per day. The motivation for doing this was to enable analysis of a bioethanol production optimization model. The results of the experiments undertaken in the course of this study, which could be used as reference for mass production in large factories to shorten the ethanol production trial process, indicated that the application of 3A-type molecular sieves yields more anhydrous ethanol, and also demonstrated that 95.08wt% ethanol yielded 61 L of anhydrous ethanol at most. The regeneration of the molecular-sieve (the unit energy yield was 0.281 L/kWh) was lowest cost when there was a heating time of 8 hours at a temperature of 200 °C.
[W.C. Chen, C.T. Sheng. **Designing and Constructing an Optimization Operating Model for a Bioethanol Production System.** *Life Sci J* 2013;10(2):1787-1793] (ISSN: 1097-8135). http://www.lifesciencesite.com. 252

Keywords: molecular sieves, adsorption, desorption, regeneration, anhydrous ethanol

1. Introduction

At present, many countries are seeking means to develop alternative energy sources (ex., solar energy, winds energy [6, 8, 11, 17]). The most widely adopted power source for vehicles is fossil fuels. However, the price of oil is increasing at all times in correlation to the continual drop in oil stocks. As a result, in order to reduce dependence on fossil fuels. One type of alternative energy that is being actively developed in many countries is bioenergy. The term bioenergy refers to the conversion of various energy crops into bioethanol. There is a biofuel process that involves maintaining minimal parameter adjustments in gasoline or diesel engines, while blending 25% or less anhydrous ethanol into oil to serve as car fuel [4, 13]. The use of this blend as fuel effectively decreases the demand for fossil fuels and also effectively reduces the air pollutants generated in engine exhaust emissions [2, 10, 15]. These attributes could potentially help to overcome air pollution issues in major metropolitan areas in Asia. The process of producing ethanol typically involves the saccharification and fermentation of starches, sugars, or cellulose. The last, it gets referred to as mash, which is composed of large amounts of water and can be distilled to yield aqueous ethanol of 95 wt% which is an azeotropic mixture of ethanol and water [3, 16], which must then be dehydrated in order to yield anhydrous ethanol with a purity of 99.3 wt% or higher for use as a gasoline additive [7].

Techniques of ethanol dehydration currently in using include the use of extractive distillation, azeotropic distillation, molecular sieve adsorption-separation etc [14]. Azeotropic and extractive distillation processes are consumed substantial amounts of time and energy. An absorption-separation technique that has been developed more recently is better at saving energy [12], as a consequence, a lot of attention has been given to this method, leading to its application in ethanol dehydration.

The fundamental principle behind the use of molecular sieves to adsorb and separate ethanol-water relies on regularly arranged pores and the use of high surface area inside molecular sieves. This allows the absorption into the pores of water molecules (with a diameter of approximately 2.8 Å). Subsequently, the water is separated from the ethanol (which possesses molecules that are larger with a diameter of approximately 4.46 Å) allowing dehydration to be achieved [22]. Molecular sieves of the 3A-type and the 4A- types can be employed as the dehydration adsorbent for ethanol, which regular arranged pores is similar to 3A-type and 4A-type. Additionally, molecular sieves are hydrophilic and their dehydration efficiency is enhanced by their affinity toward water rather than ethanol. Studies have shown that optimal efficiency in the separation of ethanol-water can be achieved by applying 3A-type or 4A-type molecular sieves [1, 5] since it leads to easy adsorption of water molecules. Pressure

swing adsorption (PSA) can be applied to allow for reuse once they are saturated with water. PSA was firstly hypothesized by Skarstrom in 1960 at which point its main application was air drying [21]. Improvements were subsequently made through numerous developments and the advancement of molecular sieves, leading PSA to be widely used in gas and liquid separation and purification. PSA involves a series of steps involving applied pressure and thermal desorption. It also requires reduced energy input and short-duration cycles [18, 19].

However, most investigations regarding anhydrous ethanol production that uses molecular sieve equipment have employed small-scale experimental equipment [18, 20]. Moreover, the operational conditions and obtained parameters have not been fit for mass production. Simultaneously, water desorption operations and reuse capabilities for molecular sieves require further examination to accommodate mass production models. Therefore, in the course of this study, construction was undertaken on equipment for medium-scale ethanol mass production, which can yield approximately 600 L of 95 wt% ethanol per day. Also examined in the course of this study were the optimized operational model using molecular sieve desorption and regeneration. These methods could possibly serve as a reference for mass production in large factories and thus shorten the ethanol production trial process. This could in turn accelerate the use of biofuel in Asian regions, and in the long run contribute to the improvement of air quality in large Asian cities.

2. Material and Methods

2.1. Design Experimental Equipment

This study involved the construction of equipment for anhydrous ethanol production. Initially the raw material which was used to distill mash from a distillation tower obtained highly concentrated ethanol. After this, molecular sieves (3A-type or 4A-type) were employed as an adsorbent with the aim of obtaining anhydrous ethanol. This product was required to achieve a concentration higher than 99.3 wt% purity in order for it to be used as biofuel. Following this, an investigation of the regeneration conditions of the molecular sieves in the system was carried out so as to clarify their statuses in terms of water adsorption and desorption. These were derived from the ethanol through the distillation tower and separately passed as samples through completely dry molecular sieves. This process was carried out in order to evaluate the efficiency of the ethanol water adsorption and also to identify the most appropriate ethanol concentration. These findings could help form the basis for subsequent water desorption experiments and related research. After the molecular sieves had adsorbed the water in the ethanol samples until they reached saturation, a high-temperature gas (i.e., nitrogen) was used to perform water desorption to achieve desorption and regeneration [18]. Once regeneration of the molecular sieves is complete, a suitable ethanol concentration be passed through the sieves again and testing relevant regeneration results and determining the yield of the anhydrous ethanol.

2.2. System Introduction and Operational Processes

Equipment used in this study included a distillation tower, a temporary ethanol storage tank, a nitrogen storage tank, a heater, a condenser, and a molecular sieve system, as illustrated in Fig. 1. In addition, the system using the boiler as the heat source of the distillation. The distillation tower's body had a cylinder with a diameter of 0.8 m. This tower stood at 7 m tall and was operated with the purpose of distilling mash. As a result of keeping the temperature at the top of the tower at 78 °C, the ethanol concentration could reach at least 90 wt%. Polyethylene was used to construct the temporary ethanol storage tank, which was primarily used to store low-concentration ethanol samples for dehydration. The capacity of the nitrogen storage tank was 5 kg/cm^2 of 3 m^3 of nitrogen. The system transported ethanol using a diaphragm dosing pump with an operational flow range of 150 to 1400 mL/min. This dosing pump would not be damaged despite sustaining long periods of continuous idling. It could also be safely used in an explosive environment. The molecular sieve system consisted of three double-layer tanks. The outer layer of these tanks could allow gas or steam to pass through for the sake of providing heating support. 22 kg of molecular sieves could be placed in each of the tanks, yielding 66 kg in total. Additionally, this study employed a 3A-type and a 4A-type molecular sieve. Furthermore, within each tank a thermocouple was installed to record the temperature changes of the molecular sieves. In the system, the ethanol, gas inlets and the gas outlet flow were controlled by the (A), (D), and (E) ball valves respectively. Temperatures were reduced by the condenser after heat energy was produced as a result of desorption of water from ethanol through the molecular sieves. This made sure of the substantial constancy of operational conditions and lowered the amount of ethanol evaporated into the air. The heater, which had a power of 5 kW, was used to heat nitrogen medium during the regeneration of molecular sieves. An electricity meter was installed in the system to record the energy consumption during anhydrous ethanol production and molecular-sieve regeneration. This system also made use of central controller to manage the valves,

pump, heater, and condenser, which was a programmable logic controller (PLC; Mitsubishi, Q01UCPU series). The PLC was also used to store collected data for subsequent organization.

The raw material used to produce ethanol in this study was molasses, which were sourced from the Taiwan Sugar Corporation. These were subsequently fermented into mash and contained an ethanol concentration of approximately 12 wt%. The distillation tower was then used to ferment the mash and produce a 95wt% ethanol sample. Subsequently, a diaphragm dosing pump was adopted to extract ethanol from the temporary storage tank at an outlet rate of 1400 mL/min. The (A) ball valves below the double-layer tanks were fully opened to allow ethanol to flow into the first tank; when the first tank was filled, ethanol flowed into the second tank from the bottom via a connecting pipeline. This process was repeated until the third tank was overflowing with ethanol. Ethanol is affected exothermically when it passes through molecular sieves. As a result, after flowing through the tanks, the ethanol was filtered through the condenser to produce anhydrous ethanol. When the ethanol concentration of the product flowing from the condenser was the same as that of the ethanol in the temporary storage tank, molecular sieves adsorbed the water in the ethanol until they reached saturation. At this point, the pump was stopped. Subsequently, the (A) ball valves were closed to allow the regeneration of molecular sieves. Before regeneration, the remaining ethanol in each of the tanks was recycled by fully opening the (B) ball valves, and the (C) ball valves were opened to fill each of the tanks with nitrogen, which employed gas pressure to allow ethanol to flow back into the temporary storage tank. Once all the ethanol was recycled, the (B) and (C) ball valves were closed and the heater was activated for nitrogen heating. The (D) ball valves at the top of the tanks were then opened to inject hot nitrogen. In the end the (E) ball valves at the bottom were opened to emit the hot nitrogen and prevent excessive pressure inside the tanks. The tanks inside were heated individually during molecular-sieve regeneration in order to avoid the danger caused by insufficient nitrogen storage in the nitrogen storage tank. From the top of the double-layer tanks, one could see molecular sieves regeneration.

2.3. Adsorption Experiment of Molecular Sieve

During the course of the study, the six samples were prepared. Each sample is 200 L with ethanol respectively concentrations of 90, 93, and 95 wt%, which were prepared two groups. The six samples were used to product anhydrous ethanol operations. Each time selecting type 3A-type or 4A-type molecular sieve is as an adsorbent and using a single kind concentration of alcohol conduct operations. Before operations, molecular sieves underwent a 24-h drying treatment at 200 °C to ensure that no humidity remained in the activated materials [1]. The anhydrous ethanol operational model described in the previous section was then employed. Anhydrous ethanol samples were collected at the condenser outlet using 1 L volumetric flasks, after which 2 cc of the sample was extracted from the flasks and analyzed using gas chromatography (GC; Hitachi G-3000) to identify the adsorption ability of molecular sieves and the anhydrous ethanol yield. During this process, GC was performed with a flame ionization detector. The capillary column was a stainless column [200 cm × 3 min (id)] which was set at a temperature of 120 °C. The carrier gas was nitrogen at a flow rate of 15 mL/min. The inlet temperature was 150 °C, and the liquid sample injection volume was 1 µl. The experimental results were using Sigma Plot plotting software (Scientific Graph System, Version 7.0, SPSS Inc., 2001, USA) to plot the result and analysis.

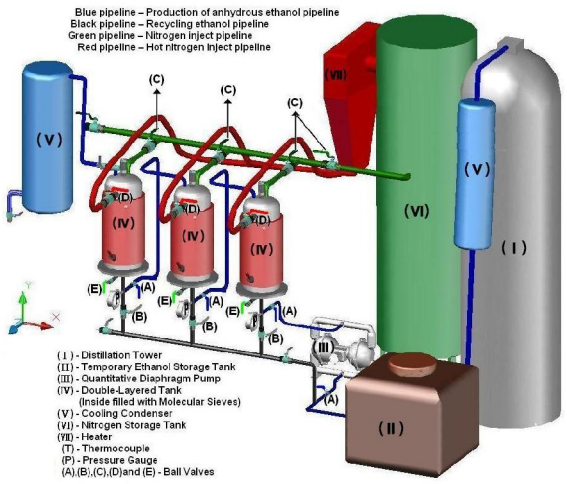

Figure 1. System equipment for anhydrous ethanol production

2.4. Desorption Experiment of Molecular Sieve

The inlet gas was considered an ideal gas. Because of during desorption and regeneration of the molecular sieves, inlet gas pressure was less than 5 km/cm^2, which was not substantial. In the system was not equipped with a flow meter; consequently, relatively precise control of gas flow was executed by adjusting the opening and closing sizes of the (E) ball valves at the bottom of the double-layer tanks, with gas flow controlled based on the pressure gauge reading. This study adopted this method to set the required temperature for molecular-sieve regeneration. Molecular-sieve regeneration removes

water molecules inside molecular sieves, and water molecules evaporate into gas at above 100 °C. Consequently, molecular-sieve regeneration temperatures of 120, 140, 160, 180, 200, and 220 °C were applied for this experiment, and an appropriate operational pressure condition was chosen based on observations of the correlation between pressure and temperature. All subsequent experimental procedures were based on this appropriate pressure and the following experiments of molecular sieve desorption-regeneration perform with operational conditions of required temperature.

The experimental factors of molecular sieves regeneration set in this study included the temperature (T) and the heating time (t) for molecular-sieve regeneration: 120, 140, 160, 180, 200, and 220 °C and 4, 5, 6, 7, 8, and 9 h, respectively. In this way, this study identified the lowest energy consumption for regeneration. After regeneration, ethanol passed through molecular sieves for dehydration and the found required for the maximum anhydrous ethanol yield. The experimental results were analyzed to identify an optimization operating model for the system.

3. Results

3.1. Adsorption Experimental Results

The initial concentration of ethanol samples were 90.05, 93.07 and 95.08 wt% by GC analysis of the results. The analysis results of the samples obtained at the condenser outlet are shown in Fig. 2. The ethanol samples were passed through dry molecular sieves for adsorption. Different initial ethanol concentrations produced at the condenser outlet during the first 20 min can get the higher concentration (up to 99.3 wt%, which can be added to gasoline). After the 20 min, the alcohol concentration decreased significantly. The ethanol concentration at the condenser outlet decreased as the ethanol treatment amount increased. The experimental results are shown in Figure 2(A). The concentration variation trend that applied the 3A-type molecular sieves serving as adsorbent to treat with an initial ethanol concentration of 90.05 wt% changed significantly, in which ethanol concentration declined from 99.61 wt% to 99.30 wt% when the ethanol yield at the condenser outlet reached 30 L. The ethanol concentration further decreased to 95.98 wt% at a 60 L output and nearly returned to the initial concentration when the output amount reached 100 L. Molecular sieves treated with an initial ethanol concentration of 95.08 wt% reached saturation for ethanol adsorption at 150L and ceased to possess adsorption functions. The initial ethanol concentrations of 90.05, 93.07, and 95.08 wt% absorbed by the molecular sieves respectively yielded concentrations of 99.3 wt% was 30L, 41L, and 60L. After 150 min adsorption, the final ethanol concentrations of the three samples were elevated to 93.62, 96.29, and 97.71 wt%, respectively, indicating that the molecular sieves removed 7.14, 6.44, and 5.26L of water.

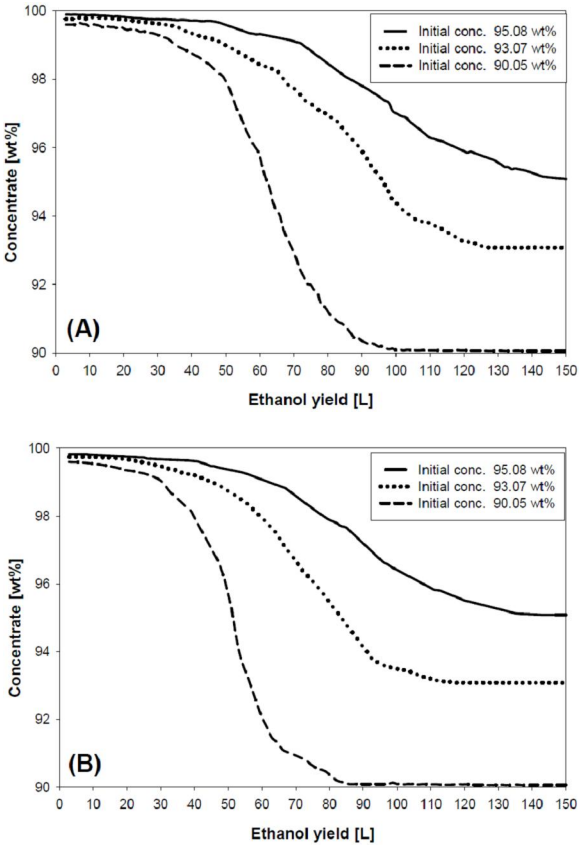

Figure 2. Adsorption ability of different molecular sieves for different initial ethanol concentrations: (A) is 3A-tpye; (B) is 4A-tpye.

In figure 2(B) shows the experimental results of the 4A-type molecular sieve used as the adsorbent. The trend of the change in the concentration was similar to the results of 3A-type molecular sieve. The molecular sieves treated with the initial ethanol concentration of 90.05 wt% reached 85L, which was saturation; the initial ethanol concentration of 95.08 wt% reached 135L. The ethanol yielded concentration of 99.3 wt% reached 23L, 35L and 54L, and the final ethanol concentration of the three samples were elevated to 92.98, 95.78 and 97.49 wt%, indicating that molecular sieves removed 5.86, 5.42 and 4.82L of water. The results showed that using 3A-type molecular sieve separated ethanol-water achieved

optimum efficiency. The results for molecular sieve water adsorption were converted into a ratio of the water content in the produced ethanol sample to the water content of the initial ethanol sample, it was similar to the results obtained by Al-Asheh (2004). In samples with lower initial concentrations of ethanol, molecular sieves to get a higher adsorb more water, however, to achieve a higher yield, a higher initial concentration was necessary. Thus, the sample selected concentration of 95.08 wt% for the subsequent experiments.

3.2. Desorption Experimental Results

The nitrogen in the storage tank was guided into the molecular sieves after it was heated using the heater. As a consequence, the (E) ball valves located at the bottom of the double-layer tanks then vented some gas to adjust the internal pressure of the tanks, as well as the internal temperature of the molecular sieves. The correlation between the internal temperature and pressure of the tanks is shown in Table 3. When the (E) ball valves were not sufficiently opened, pressure accumulated in the nitrogen storage tank and double-layer tanks. Subsequently, the temperature could not easily reach the set value, resulting in energy waste. As is displayed in Table 1, when the internal pressure of the tanks was 0.5 kg/cm^2, the difference between the set and actual temperature inside the double-layer tanks was minimal. The temperature also dropped significantly once the pressure dropped, indicating that the obtained 0.5 kg/cm^2 internal pressure was the most appropriate for subsequent research. Consequently, when regenerating the molecular sieves, this pressure control condition was employed.

Table 1. Correlation between the temperature and pressure inside the double-layer tanks

Double-layer tanks: internal pressure (kg/cm^2)	3.5	2.5	1.5	0.5	0.2
Required temperature inside the double-layer tanks (°C)	Actual temperature inside the double-layer tanks (°C)				
220	196	207	215	220	205
200	175	188	195	200	186
180	156	169	176	181	167
160	134	150	156	161	148
140	113	128	136	140	129
120	93	106	114	120	104

Desorption of the molecular sieves, as well as regeneration, was carried out with continuous heating at high temperatures. Once regeneration was completed, the molecular sieves could be seen as opalescent from the top of the double-tank layers. As shown in Figure 3, molecular sieves regeneration operated at different heating temperatures and times were employed by the 95.08 wt% ethanol to measure its absorption capacity again and find out the concentration yield (99.3 wt%, which can be applied to gasoline additive).

Table 2 shows the results as analyzed by GC. They indicate the yield of ethanol concentration increased with temperature and operational time. The greatest yield of the ethanol with concentration of 99.3 wt% collected at the condenser outlet was 61L. Taking the process cost into consideration, the study converted the results into the obtained results for the unit energy yield to find out operational conditions for the maximum yield, as shown in Table 3. At the temperature 200C and with a continuous heating time at 8 h, the process cost was the lowest with the maximum unit energy yield of 0.281. Assuming that the unit ethanol cost was calculated at NT$5.28 per kWh as industrial electricity in Taiwan, the dehydration cost for each liter of anhydrous ethanol produced was NT$18.8.

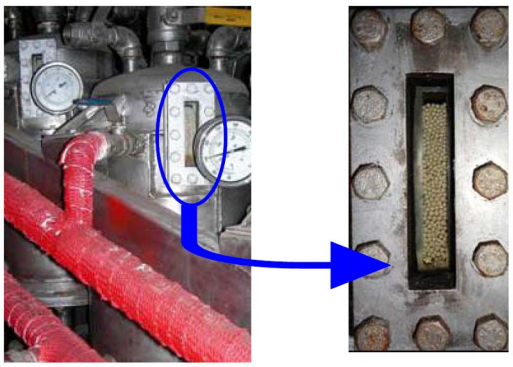

Figure 3. The status of molecular sieves regeneration

Table 2. The unit energy yield of the ethanol production process

Temp. (℃) / Time (h)	220	200	180	160	140	120
3	0.238	–	–	–	–	–
4	0.241	0.216	–	–	–	–
5	0.245	0.230	0.251	–	–	–
6	0.245	0.275	0.267	0.257	–	–
7	0.243	0.271	0.271	0.255	0.201	–
8	0.247	0.281	0.268	0.259	0.221	0.183
9	0.232	0.257	0.263	0.252	0.242	0.182

Table 3. Ethanol concentration of 99.3 wt% yields after regeneration of the molecular sieves under different operational conditions

Temp. (℃) / Time (h)	220	200	180	160	140	120
3	33	–	–	–	–	–
4	38	30	–	–	–	–
5	44	34	35	–	–	–
6	49	49	41	38	–	–
7	54	53	46	42	33	–
8	60	60	50	47	40	32
9	61	60	54	50	48	35

4. Discussions

This study constructed medium-scale ethanol mass-production equipment and analyzed its optimized operational model to shorten the procedure for the examination of mass ethanol production in large factories. The ultimate purpose is expectation accelerate provide ethanol for motor fuel additive. These could potentially help to overcome air pollution issues in major metropolitan areas in Asia. Molasses was fermented and became to mash, using a distillation column distil and got highly-concentrated ethanol in this research. At last, using the 3A-type and 4A-type molecular sieve was as the adsorbent to adsorb the water in the ethanol. When the molecular sieves saturated with water, ethanol requires reclaim form double-layer tanks to avoid wasting. Continuous heating with high temperature, nitrogen was carried out to regenerate the molecular sieves for reuse. The experimental results showed that the use of 3A type molecular sieves for the separation of ethanol-water gives optimum efficiency and produce more of anhydrous ethanol. Higher initial concentrations of ethanol produced higher yields of anhydrous ethanol. In addition, the temperature required for regeneration of a molecular sieve could be achieved when nitrogen was passed through the heater and was guided into the tanks. The pressure of tanks internal was set at 0.5 kg/cm^2 for the best operating conditions. By applying the conditions and considering the cost of production, the regeneration of molecular sieves were operated at 200 ° C with a heating time of 8 h. Using initial ethanol concentration of 95.08 wt% to be performed adsorption experiments, which yield 60L of anhydrous ethanol. Under these operating conditions, the cost of the molecular sieve regeneration was at a minimum (the unit energy yield was 0.281L / kWh). The calculations were performed using the industrial electricity prices in Taiwan, which produces a price of NT $ 18.8 per liter of anhydrous ethanol for using molecular sieve dehydration and regeneration.

Acknowledgements:

We express our gratitude for the support provided by the National Science Council research project fund and the cooperation and generous help of relevant testing agencies. We also thank Ming-Shi Huang for assistance and suggestions, which were extremely helpful for the progress and completion of this study.

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5/11/2013