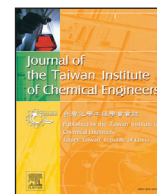




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## Dehydration of waste cutting oil using a pervaporation process

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## ABSTRACT

A commercial polyvinyl alcohol (PVA)/polyacrylonitrile (PAN) hybrid membrane (B3 membrane) was tested to evaluate its ability to dehydrate waste cutting oil by pervaporation (PV). The major material and the water content of the waste cutting oil were analyzed by TGA, NMR, and Karl Fisher titrator. A preliminary PV experiment that involved treatment with 15 wt% aqueous diethylene glycol (DEG) was carried out at various temperatures, and 85 °C was found to be the optimal temperature. The separation factor increased from around 530 to 1140 and the total flux decreased from approximately 0.88 to 0.01 kg/m<sup>2</sup>/h as the water concentration in the feed solution decreased. Then, a PV experiment that involved treating the waste cutting oil was carried out at 85 °C and the obtained results were almost the same as those in the preliminary experiment, indicating that PV is an effective method for recycling waste cutting oil.

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## 1. Introduction

The solar energy and semiconductor industries are requiring more and better wafers. The general cutting process cannot satisfy these requirements, so the wire saw process was developed. In the wire saw process, a material called cutting oil is added to the wire saw instrument. The cutting oil is composed an abrasive, such as SiC particles, solvent, and additives. The solvent must have two features: it is easily washed away using water and viscosity must cause the abrasive to be suspended in the solvent. Ethylene glycol (EG), diethylene glycol (DEG), and polyethylene glycol (PEG) are frequently used as solvents for cutting oil.

DEG, (HOCH<sub>2</sub>CH<sub>2</sub>)<sub>2</sub>O, is a co-product with ethylene glycol and triethylene glycol. It is a colorless, sticky, and practically odorless liquid that is miscible in water, alcohol, and ether [1]. Therefore, DEG exhibits the necessary characteristics requirements of cutting oil and it is the most commonly used solvent in Taiwan. However, the boiling point of DEG is 245.3 °C. This fact and its miscibility with water increase difficulty of its recovery. Tons of waste cutting oil is generated by wafer slicing. The waste cutting oil consists of the original cutting oil, water, and Si particles. SiC and Si particles will be recovered as filter cake by solid-liquid separation. The filtrates cannot be returned as the solvent and can be burned as fuel because of their high water content. The only way to return them as the solvent is to dehydrate them. The most commonly used method of dehydration is distillation. However, even the use of vacuum distillation to dehydrate the filtrates requires heating of

the filtrates to 160 °C at a pressure of 700 mmHg to yield pure DEG. A new process to recover the filtrates must be developed. In our previous research [2], membrane distillation (MD) has been tested to dehydrate DEG solution and the water content of DEG can be reduced from 15 wt% to 2.26 wt%. Although the result is lower than 5 wt% which is the maximal water content to recycle as the cutting oil, MD still cannot make the water content less than 1 wt%. It still needs to find another process to make the water content less than 1 wt%. There are two papers mentioned with the dehydration of DEG by pervaporation [3,4]. Therefore, pervaporation will be tested to treat the waste cutting oil.

Pervaporation (PV) is a membrane method involves permeation and evaporation. It is an effective alternative process for the separation of aqueous-organic mixtures, and especially azeotropic mixtures, heat-sensitive products, and mixtures of substances with similar boiling points [5]. Unlike conventional membrane-based techniques, such as reverse osmosis (RO) and ultrafiltration (UF), PV uses a compact membrane or a hybrid membrane. PV exploits the affinity between the membrane and the liquids to separate the mixture [6]. The separation mechanism of PV mainly occurs on the surface of the membrane. The liquids are easily absorbed by the membrane if the affinity between the membrane and the liquids is very strong [7]. For example, the membrane used for the dehydration in PV is hydrophilicity for which the polar groups in the membrane matrix are responsible. The polar groups attract water and act as the fixed carriers to let water quickly diffuse through the membrane [6,8,9]. The other organic molecules diffuse through the membrane by the free volumes between the polymer chains due to the dense membrane. This phenomenon leads organic molecules to be almost blocked on the feed side. Therefore, the water and

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the organic solution can be separated on the surface of the membrane. The transport processes in the PV membrane can be described by solution-diffusion model [10,11]. In PV, the upstream, delimited by the membrane, is maintained at atmospheric pressure and the downstream is kept in a vacuum. The pressure drop across the membrane causes the liquids to diffuse through the membrane and separate. PV has been widely investigated. Kober [12] was the first one to observe the phenomenon of pervaporation, but the first known quantitative work on the topic was published by Heisler et al. [13], who separated water/ethanol mixtures using a cellulose membrane [14]. PV has three broad applications, which are the dehydration of organic solution [15–20], the removal of organic matter from dilute aqueous solution [21–23], and the separation of organic/organic mixtures (such as methyl tert-butyl ether (MTBE)/methanol and thiophenes/FCC gasoline) [24]. If the membrane is made of hydrophilic material, such as chitosan [25,26] or polyvinyl alcohol (PVA) [27–29], it can be used to dehydrate organic solutions, such as alcohols, ethers, esters, and acids. However, a hydrophobic membrane is suitable for removing organic matter from dilute aqueous solution, such as is carried out in the removal of volatile organic compounds. There are already two articles discussing the dehydration of DEG solution by pervaporation, the systems are different between this study and the other two papers. The membrane used in this study is flat polymer membrane and those used in the other two articles are hollow fiber membrane [3] and HybSi ceramic membrane [4]. Another different part is the target of treatment. The other two articles force on the DEG solution used in gas complex processing plants at gas fields and this study forces on the waste cutting oil.

In this study, the target of treatment is the filtrates after solid-liquid separation. First, the filtrates are analyzed by NMR and TGA to confirm what the main component is. According to the result, a synthetic wastewater that comprises the main component and water (binary mixture) was tested to determine the optimal treatment temperature. Real waste cutting oil was tested under the optimal conditions and compared with synthetic wastewater to confirm that PV is an effective process for dehydrating waste cutting oil.

## 2. Material and methods

### 2.1. Material

In this study, a commercial hybrid PVA/PAN pervaporation membrane, crosslinked by maleic anhydride, is as described in previously published papers [16]. DEG with a purity of 99% was purchased from Alfa Aesar. Real waste cutting oil after solid-liquid separation was obtained from a recycling plant.

### 2.2. Characterization of waste cutting oil

The vaporization and solid content of the real waste cutting oil were measured using a thermal gravimetric analyzer (TGAQ50, Perkin Elmer). Nuclear magnetic resonance (Bruker Avance: 600 MHz NMR) was used to determine the composition of the real waste cutting oil.  $^1\text{H}$  NMR spectra of the real waste cutting oil was compared with those of pure DEG to prove that DEG was the main component of the real waste cutting oil. In the NMR analysis,  $\text{D}_2\text{O}$  was used as the solvent. The solid content was analyzed by TGA. In TGA, the sample was heated from 50 to 400 °C at a rate of 10 °C/min in  $\text{N}_2$  gas. The water content of the real waste cutting oil was measured using a Karl Fischer titrator (787 KF Titrino, Metrohm).

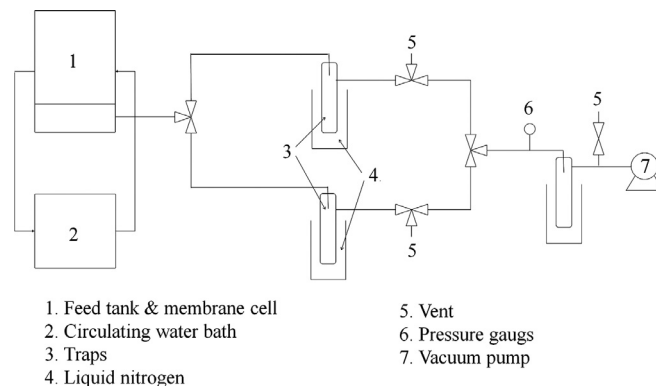


Fig. 1. PV apparatus.

### 2.3. Pervaporation experiment

Fig. 1 schematically depicts the PV apparatus. The effective area of the membrane was 19.6 cm<sup>2</sup> and the downstream pressure was 0.04 to 0.1 Torr. The system ran for one hour to establish a steady state. Both feed and permeate samples were obtained at 0, 1, 3, 5, 7, 11, 19 and 24 h. The permeate was collected in a tube that was cooled by liquid nitrogen. The water content of both the feed and the permeate solution were measured using a Karl Fischer titrator. The water contents of the permeates that were collected at 1, 19, and 24 h were not measured because their volumes were too small. The experiment was divided into two parts: the first involved synthetic wastewater and the second involved the real waste cutting oil. First, synthetic wastewater that was composed of water and DEG was used to determine the effects of temperature on the dehydration of aqueous DEG. The initial feed solution was 85 wt% aqueous DEG (75 g). The temperature was controlled between 55 °C and 95 °C. Afterwards, the real waste cutting oil was tested under the optimal temperature and compared with the synthetic wastewater. The membranes before and after experiment were detected using a high resolution field emission scanning electron microscopy (SU8010, Hitachi).

To determine the performance of the PV system, the total flux ( $F$ , kg/m<sup>2</sup>/h), separation factor ( $\alpha$ ), and pervaporation separation index (PSI), defined by Eqs. (1), (2), and (3), were used. Where  $W$  (kg) is the total amount of the permeate that was collected at experimental time  $\Delta t$  (h);  $A$  is the effective area of the membrane, and  $X_{\text{water}}$  and  $Y_{\text{water}}$  are the weight fractions of the water in the feed and permeate solutions, respectively.

$$F = W/A\Delta t \quad (1)$$

$$\alpha = [Y_{\text{water}}/(1 - Y_{\text{water}})]/[X_{\text{water}}/(1 - X_{\text{water}})] \quad (2)$$

$$\text{PSI} = F \times (\alpha - 1) \quad (3)$$

## 3. Results and discussion

### 3.1. Characterization of waste cutting oil

The waste cutting oil was a yellow, sticky and clear liquid. Fig. 2 shows the  $^1\text{H}$  NMR results. The peak at 4.7 ppm was the solvent peak. The two longer peaks at 3.6 and 3.7 ppm were attributable to the  $\text{CH}_2$  group. Not only the positions of the two peaks from the DEG and the waste cutting oil, but also the splitting of these two peaks were almost the same. The results demonstrate that the major component of waste cutting oil is DEG. In the waste cutting oil result, there was a little peak at about 3.35 ppm. It belonged to the

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