



Energy-saving process development for the purification of propylene glycol based on MVR heat pump distillation combined with thermally coupled technology

Jing Ma, Xiejun Wang, Yamei Zhou, Yaxuan Du, Baohe Wang^{*}

Collaborative Innovation Center of Chemical Science and Engineering, Tianjin University, Tianjin 300072, China

Key Laboratory for Green Chemical Technology of Ministry of Education, R&D Center for Petrochemical Technology, Tianjin University, Tianjin 300072, China

ARTICLE INFO

Keywords:

Wastewater
Propylene glycol
MVR
Thermally coupled

ABSTRACT

In the process of recovering organic compounds from wastewater generated by propylene oxide (PO) production plant, the low concentration of propylene glycol (PG) in the wastewater makes it difficult and energy-intensive to achieve the high concentrations purify of PG using conventional distillation technology. Additionally, there is no publicly available commercial technology for this purpose. Therefore, the aim of this work is to develop an energy-efficient PG purification process based on mechanical vapor recompression (MVR) heat pump distillation combined with thermally coupled technology. Several energy-saving methods were implemented as follows. Firstly, MVR distillation technology was used to purify PG from 2.22% to 33.3% in the PG concentration tower, and the gas at the top of the tower was compressed and pressurized to serve as the heating medium for the reboiler, which significantly reduced the consumption of fresh steam. Secondly, in order to achieve heat matching, the dehydration tower was divided into two parts. The surplus steam compressed by the compressor was then served as the heat source for the reboiler of the first dehydration tower to reduce steam consumption, while achieving 98.7% water removal. Thirdly, the condenser of the de-heavy tower and the scraping film evaporator were thermally coupled to reduce the consumption of heating and cooling media. Based on the aforementioned energy-saving measures, the developed optimization process can achieve PG purification of over 90%, while significantly reducing costs. The total annual cost (TAC) is reduced by 34.89% compared to the traditional distillation process.

1. Introduction

Propylene oxide (PO), one of the prominent downstream product derived from propylene [1,2], functions as a principal precursor for the production of propylene glycol (PG), unsaturated resins, nonionic surfactants, and carbonates within the manufacturing realm [3–6].

A wide array of industrial process has been investigated to produce PO, which can be divided into the traditional technology and modern cleaner production technology.

The former encompasses processes such as the chlorohydrin (CH) process, the propylene oxide/styrene monomer (PO/SM) process, and the propylene oxide/methyl tertiary butyl ether (PO/MTBE) process, etc. On the other hand, the latter includes the cumene hydroperoxide propylene oxide (CHPPO) process and hydrogen peroxide-propylene oxide (HPPO) process, etc. [7].

Among these methods, the PO/SM approach has achieved the largest production scale worldwide, reaching 50%. However, in recent years, this method has encountered economic challenges due to an oversupply of its byproduct. Additionally, this method poses certain environmental hazards. As a result, attention is gradually shifting towards the more environmentally friendly and economically advantageous HPPO process. It has been reported that the HPPO process yields noteworthy advantages in terms of reducing the volume of wastewater (a reduction of about 80%) and in terms of reducing the energy use (a reduction of 35%) when compared to preeminent traditional techniques [8,9].

From a structural perspective, the HPPO process is fundamentally comprised of two integral components: the epoxidation reaction unit and the PO refining unit [2]. In the epoxidation reaction unit, the catalytic agent TS-1 molecular sieve is employed, with methanol as the solvent, operating under suitable temperature and low-pressure liquid

^{*} Corresponding author.

E-mail address: bhwang_tju@sina.com (B. Wang).

<https://doi.org/10.1016/j.seppur.2023.125064>

Received 28 June 2023; Received in revised form 30 August 2023; Accepted 8 September 2023

Available online 18 September 2023

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phase conditions. This enables the reaction between propylene and hydrogen peroxide, resulting in the formation of crude PO products. These products are then sent to the PO refining unit. Here, the final PO products are produced through a refining process, while concurrently, a series of operations are carried out to recover the methanol solvent for subsequent reuse [8,10].

It's worth noting that although the pollution caused by the HPPO process is relatively diminished, during the PO refining process, alongside the production of PO, industrial wastewater (approximately 1 to 2 tons of wastewater per ton of produced PO) containing organic compounds such as PG and Propylene glycol monomethyl ether (PGME) is also generated [11]. Given the dual difficulties of environmental and economic considerations, it is necessary to recover these organic compounds from the large quantities of discharged wastewater.

Numerous methodologies exist for the separation of organic compounds from wastewater, such as extraction[12], adsorption[13], and distillation[14]. However, extraction techniques encounter difficulties in achieving efficient separation of polyols from aqueous streams due to their pronounced affinity for water, which hinders the identification of an organic solvent with favorable distribution coefficients and low miscibility with water[15]. Similarly, adsorption techniques are generally unsuitable for high-volume treatment and pose challenges in terms of selecting and acquiring suitable adsorbents, necessitating significant investments of time and financial resources[16]. Consequently, neither of these methods aligns with the specific requirements outlined in this study concerning the treatment of wastewater discharged by PO production facilities. In the context of the wastewater discharged by PO production facilities discussed in this study, the treatment process faces the characteristics of low concentration and high volume. Hence, distillation, being a common and effective method, is widely considered in the industry due to its ability to address such challenges.

The recovery of PGME from wastewater discharged by PO production facilities has been a subject of much discussion and exploration, and there have been numerous reports on its recovery have been proposed. For example, Wang et al.[17] used the optimized thermal coupling mode to achieve the treatment of HPPO process wastewater with low energy consumption. Moreover, the product purity of PGME could reach more than 99.8% and the recovery rate reached 95%. HU et al.[11] employed multiple-effect evaporation to separate PGME from the wastewater, achieving a recovery rate of 97%. Tang et al.[18] provided a process comprising a methanol removal device, a concentration device, an extraction device, an extraction recovery device, a dehydration device, and a separate device connected in sequence to recover PGME, achieving a recovery rate of 99% and a product purity of 99.5%. Additionally, other methods, such as benzene azeotropic distillation and PDMS membrane separation, also have been employed to recover PGME from wastewater generated by PO production facilities[19].

As another high value-added organic in the effluent from PO production plant, PG has a wide application prospect, including as an antifreeze agent, engine cooling, utilization in food products, and liquid detergent, etc.[20] Recently, the recovery of PG from wastewater has become an urgent problem to be solved in the production of PO by HPPO process. Otherwise, it will not only lead to non-compliance with wastewater discharge standards, but also cause material losses. Therefore, the development of an effective separation process to recover PG from wastewater is essential for achieving both the standardized wastewater discharge and the comprehensive utilization of waste materials present in the wastewater. Such efforts can further contribute to the economic competitiveness of the HPPO process by reducing costs.

As the HPPO process inevitably produces a large amount of wastewater[21,22], and the content of PG in wastewater is low, traditional methods such as conventional distillation or multiple effect evaporation, the energy consumption is quite large. Therefore, it is necessary to develop a new green recovery process of PG with low energy consumption. Unfortunately, so far, there has been limited research on separation processes for recovering PG from wastewater produced by

HPPO process, and only a few patents filed by HU[11], Zhang[23], and others. Both of these patents describe a two-effect evaporation method for recovering PG from wastewater, which has improved the efficiency of steam utilization and saved energy to some extent. However, this method also has obvious drawbacks in terms of energy efficiency. On the one hand, the first-effect evaporator requires fresh steam as a heat source, and the secondary steam from the second-effect separator also needs to be condensed, which consumes cooling water. On the other hand, during the evaporation process, it has been found that PG is easily carried away by the evaporated water in the separator, resulting in losses. Therefore, more efforts should be made to optimize the process so as to reduce energy consumption.

As an important means of energy-saving, mechanical vapor recompression (MVR) technology can increase the temperature, pressure and enthalpy of steam by compressing the low-temperature steam through the compressor, so as to make full use of the latent heat of steam to achieve the effect of energy saving[24–26]. As far as steam compression is concerned, MVR is already a mature technology in distillation-related industries[27]. It has been widely employed in various fields, such as seawater desalination[28], traditional Chinese medicine concentration [29], and industrial wastewater treatment[30,31], etc. Furthermore, the MVR heat pump distillation technology has also been applied for the recovery of organic compounds such as alcohols/esters, trichlorobenzene and ethylene, etc. For instance, MVR technology was added to the basic extractive distillation process by Li et al.[32] for separating ester/alcohol/water systems, resulting in a 36.03% reduction in total annual cost (TAC). Yang et al.[33] utilized MVR coupled with ORC technology to collect trichlorobenzene, which reduced energy consumption by 83.5% compared with the four-column conventional distillation process, and resulted in a 22.9% reduction in TAC. It is worth noting that, Yang et al.[34] also incorporated the MVR heat pump technology into thermally coupled distillation, thereby introducing the MVR thermal coupled distillation process for the separation of ternary mixture alkane of n-hexane, cyclohexane and isooctane. Comparative analysis against traditional thermal coupled distillation revealed significant energy savings of 38% and a reduction of 48% TAC through the implementation of this process. Thus it can be seen that the energy saving effect of MVR technology is remarkable in various separation processes. However, to the best of our knowledge, the use of MVR heat pump distillation technology to recover PG from PO production wastewater has not been reported.

In this work, in view of the characteristics of low content of PG in PO production wastewater, energy-saving technologies such as MVR heat pump distillation and thermally coupled technology are used to develop a separation and recovery process for PG. And it is expected to explore an efficient and energy-saving technology for the recovery of high value-added organic compounds from wastewater.

2. Problem statement

The raw materials employed in this study ultimately stem from the wastewater discharged from the PO refining unit within the PO production process. Following distillation for the separation and removal of PGME, the resultant wastewater is directed from the column pot of the upstream distillation. The residual wastewater continues to contain water (96.70%), PG (2.22%), dipropylene glycol methyl ether (DPGE, 0.23%), salt (0.45%) and others. Additionally, the temperature generally exceeds 90 °C. In order to meet the standard discharge of wastewater and achieve the recovery of high-value organic compounds, it is necessary to concentrate the PG content in the wastewater to over 90%. This study centers on the purification of this residual wastewater subsequent to the removal of PGME.

However, there are three key challenges associated with obtaining such high concentrations of PG from the aforementioned feed. First of all, because of the azeotrope of PG and DPGE, it is difficult to separate them. Secondly, the content of PG in feed is generally low (2 ~ 5%),

which undoubtedly further increases the difficulty of PG recovery. Third, the feed contains inorganic salts, which are easy to cause salt precipitation and blockage of pipes or equipment in the treatment process.

3. Results and discussion

3.1. Thermodynamic model

The choice of thermodynamic method plays a decisive role in the accuracy of the simulation results, because the system is mainly composed of polar substances such as PG, water, ethylene glycol (EG), and condensed ethers, including DPGE and tripropylene glycol methyl ether (TPGE), the Universal Quasi-Chemical (UNIQUAC) activity coefficient method combined with the Redlich-Kwong (RK) equation of state was selected for the simulation of PG purification. To our knowledge, the UNIQUAC-RK model has proven to be a mature approach for modeling the vapor-liquid equilibrium (VLE) relationship in previous simulations of alcohol-water and ether-water systems[35,36].

The UNIQUAC activity coefficient model, as represented by equations (1)-(5), was utilized in the analysis, with Table 1 presenting the binary interaction parameters for the primary components. From Fig. 1, it is observed that PG ($T_b = 187^\circ\text{C}$), EG ($T_b = 197^\circ\text{C}$), and DPGE ($T_b = 203^\circ\text{C}$) exhibit substantial differences with H_2O in their boiling points, and there is no azeotropic behavior between the components and H_2O , which implies that separation can be readily accomplished. Nevertheless, Fig. 1(d) reveals that when the mole fraction of PG is greater than 0.8, it is difficult to separate DPGE from PG due to the presence of azeotropic behavior between them. Consequently, the final PG product obtained contains DPGE.

The UNIQUAC model is:

$$\ln \gamma_i = \ln \frac{\Phi_i}{x_i} + \left(\frac{Z}{2}\right) q_i \ln \frac{\theta_i}{\Phi_i} + l_i - \frac{\Phi_i}{x_i} \sum_j x_j l_j - q_i \ln \left(\sum_j \theta_j \tau_{ji} \right) + q_i - q_i \sum_k \frac{\theta_k \tau_{ik}}{\sum_k \theta_k \tau_{ki}} \quad (1)$$

$$l_i = \frac{Z}{2} (r_i - q_i) - (r_i - 1) \quad (2)$$

$$\Phi_i = \frac{r_i x_i}{\sum_j r_j x_j} \quad (3)$$

$$\theta_i = \frac{q_i x_i}{\sum_j q_j x_j} \quad (4)$$

$$\tau_{ij} = \exp \left(a_{ij} + \frac{b_{ij}}{T} + \alpha_{ij} \ln T \right) \quad (5)$$

Where Φ_i and θ_i are defined as the mean volume fraction and area fraction of substance i or j , respectively, along with the molecular van der Waals surface area (q_i), volume (r_i) and the parameters of a_{ij} and b_{ij} which are calculated based on the Aspen Plus database or UNIFAC. The lattice coordination number is assumed to be $Z = 10$. The α_{ij} is also considered to be a non-random parameter, the value of which is related to the properties of the solution.

To verify the accuracy of the thermodynamic model, a comparative

analysis between the simulated and experimental values of VLE for binary systems (PG-water(a), EG-water(b), DPGE-water(c) and PG-DPGE (d)) were performed, and the result is presented in Fig. 1. It is worth noting that for the water-DPGE system, only its PXY data have been reported in the literature, so a comparison of its PXY simulated and experimental data is included in Fig. 1(c). It can be seen that the simulated values are in good agreement with the experimental values, which proves that the thermodynamic model has high prediction accuracy. However, in the case of the PG-DPGE system, due to the absence of available VLE data, the azeotropic composition under various temperatures and pressures was computed as a substitute. As listed in Table S1 of the Supporting Information, it can be seen that the average deviation between the simulated and experimental values is less than 2.4%. This result also reveals the accuracy of the binary interaction parameters used.

In addition to organic compounds, the feed also contains inorganic salt Na_2SO_4 , which exists mainly in the form of Na^+ and SO_4^{2-} due to its low concentration (less than 0.5%) in water. In our simulation, Na_2SO_4 was defined as an electrolyte, and the Electrolyte-NRTL thermodynamic model (ENRTL) coupled with the RK state equation was used to investigate the impact of salt on water evaporation. The local interaction energy parameters between molecule-electrolyte pairs in ENRTL were calculated using equations (6)-(7), and the interaction parameters between $\text{Na}^+/\text{SO}_4^{2-}$ and H_2O were proposed as shown in Table 2. Furthermore, in order to ensure the reliability of the data, the density, viscosity and specific heat of Na_2SO_4 aqueous solution were calculated and compared with the experimental data. As shown in Table S2 of the Supporting Information, it can be seen that the mean value of the relative deviation between the simulated and experimental values is about 0.62%, which verifies that the ENRTL interaction parameters have high prediction accuracy.

$$\tau_{m,ca} = C_{m,ca} + \frac{D_{m,ca}}{T} + E_{m,ca} \left[\frac{T^{\text{ref}} - T}{T} + \ln \left(\frac{T}{T^{\text{ref}}} \right) \right] \quad (6)$$

$$\tau_{ca,m} = C_{ca,m} + \frac{D_{ca,m}}{T} + E_{ca,m} \left[\frac{T^{\text{ref}} - T}{T} + \ln \left(\frac{T}{T^{\text{ref}}} \right) \right] \quad (7)$$

Where, the “m” denotes the molecule, the “ca” represents the ion pair, $T^{\text{ref}} = 298.15 \text{ K}$, “C”, “D”, and “E” correspond to the interaction constants.

3.2. The first stage of purification

3.2.1. Different purification techniques

Due to the low content of PG in the feed, direct purification to a purity of over 90% would require higher operating conditions for the distillation tower, resulting in less energy efficiency. To avoid this, an alternative method is to first increase the concentration to above 30% as the first stage of purification. In this work, three different processes were designed and simulated to enrich PG: triple-effect evaporation technology (Fig. 2), conventional distillation (Fig. 3), and MVR heat pump distillation (Fig. 4). The assessment is based on product purity, yield, and energy consumption, aimed at formulating a process route that is both economically efficient and energy-saving.

For the Multi-effect evaporation technology, it has emerged as a

Table 1
Binary UNIQUAC interaction parameters among main components in the systems.

Component i	Water	Water	PG	PG	Water	Water
Component j	PG	EG	DPGE	EG	DPGE	TPGE
a_{ij}	–	–0.4787	0.3037	0.4147	0.0	0.0
a_{ji}	–	0.4661	–0.6170	0.2677	0.0	0.0
b_{ij}	–273.3928	78.2432	–79.2428	–477.3730	–1311.0419	–1078.0489
b_{ji}	250.7139	27.3610	116.4651	96.4942	477.4028	412.9969
Source	APV VLE-RK	APV VLE-RK	APV VLE-RK	APV VLE-RK	UNIFAC	UNIFAC

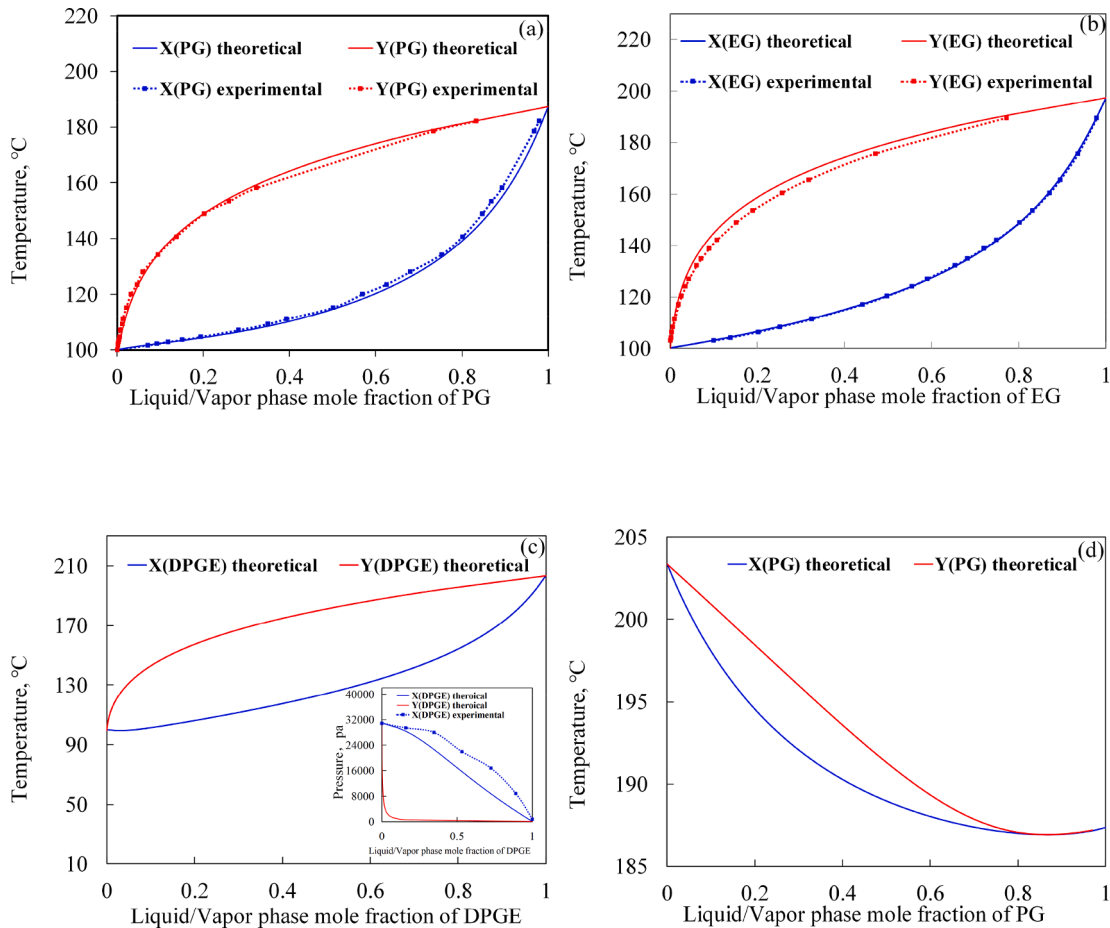


Fig. 1. Comparison between simulated and experimental value of TXY for binary systems of PG-water(a), EG-water(b), DPGE-water(c) and PG- DPGE(d)[37–39].

Table 2
Binary ENRTL interaction parameters of H₂O molecule and Na⁺/SO₄²⁻ electrolyte pair.

Component i	Component j	C	D	E
H ₂ O	Na ⁺ /SO ₄ ²⁻	1.9545	1762.1850	7.5524
Na ⁺ /SO ₄ ²⁻	H ₂ O	−2.0332	−537.9684	6.9197 × 10 ^{−3}

commonly used approach for achieving energy conservation in a diverse range of applications, including the purification of organic compounds [40,41], the desalination of seawater [42,43], and more. In order to comprehensively assess the effectiveness of this technology, the

evaporation process of PG [44] was taken into account. The double-, triple-, and quadruple-effect evaporations were scrutinized with regards to yield, heat load, and pressure variations, respectively. The corresponding research findings are documented in Table S3 in the Supporting Information. As shown in the data, the yield of PG for the double-effect system is 80.90% with a heating load of 8873kw. For the triple-effect system, the yield of PG is 83.95% with a heating load of 5481kw. The quadruple-effect system achieves a yield of PG of 84.30% with a heating load of 4460kw. These results indicate that increasing the number of effects leads to higher PG yield and lower heating load. However, the transition from triple-effect to quadruple-effect evaporation shows a relatively modest increase in yield. Considering the relevant equipment investment concerns, a triple-effect evaporation system

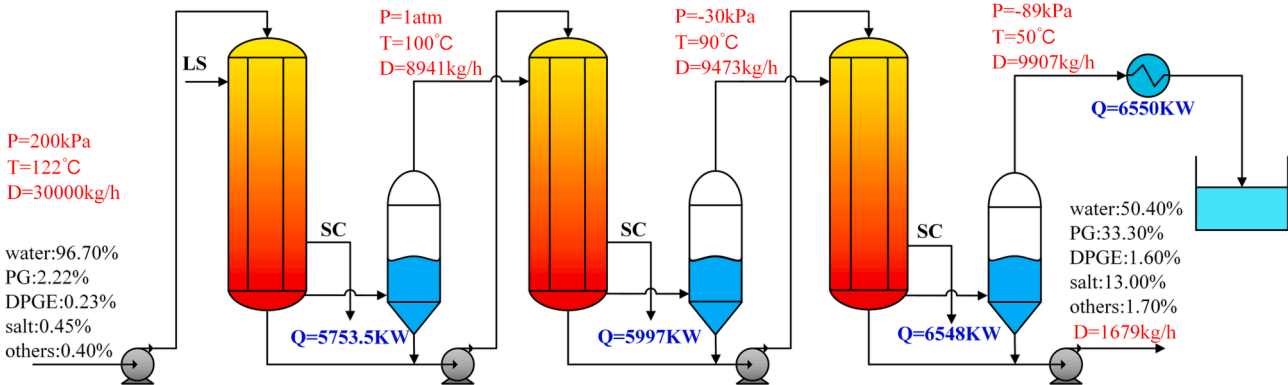


Fig. 2. Triple effect evaporation technology for PG purification.

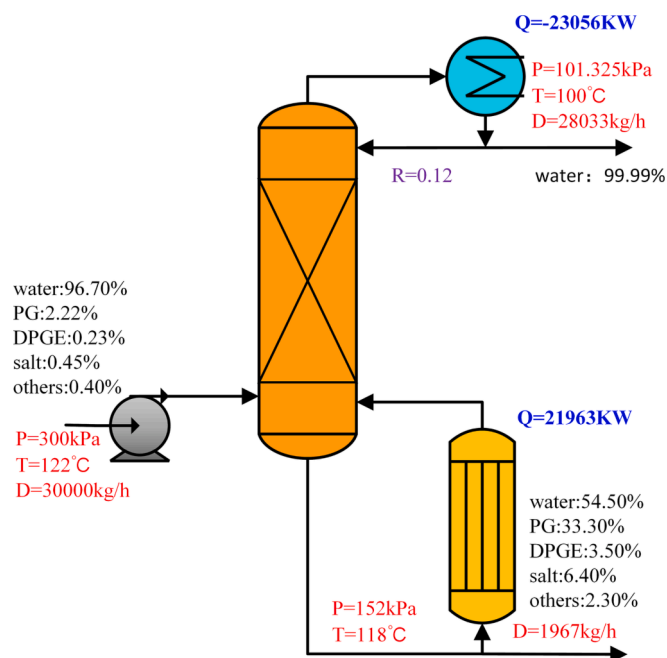


Fig. 3. Conventional distillation technique for PG purification.

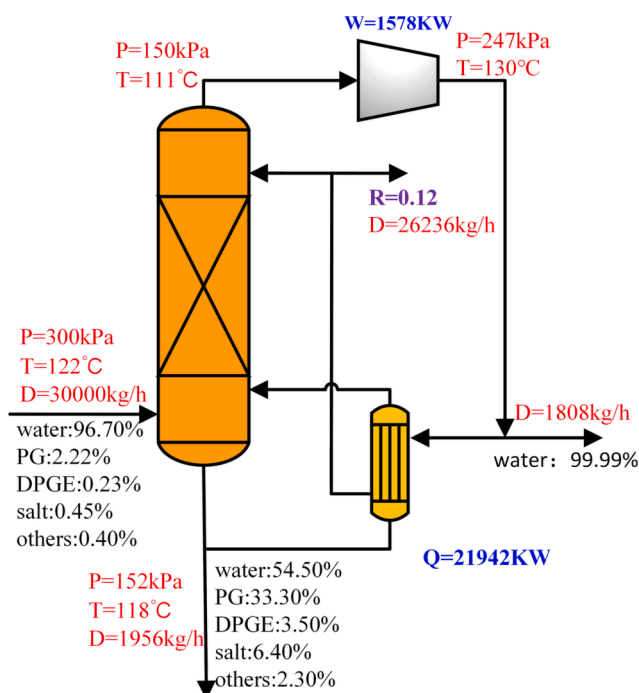


Fig. 4. MVR distillation technology for PG purification.

was ultimately chosen as a representative case of multi-effect evaporation.

Fig. 2 illustrates the process of PG purification using a three-effect evaporation technology. As shown in the figure, the feed liquid is pumped to the uppermost section of the first-effect evaporator and introduced into the heat exchanger tube where it undergoes heat exchange with fresh steam outside through the liquid distributor, and then evaporates in a falling film manner. The gas-liquid mixture generated during the evaporation process at atmospheric pressure is subjected to gas-liquid separation in a separator and produces secondary vapors and concentrated liquid. The secondary steam released from the separator is

served as a heat source in the second-effect evaporator with a pressure of -30 kPa, while the concentrated liquid leaving the separator is pumped by a circulation pump into the second-effect evaporator for further concentration. The secondary steam separated from the second-effect evaporator is likewise utilized as a heat source in the third-effect evaporator with a pressure of -89 kPa, and the concentrated liquid leaving the separator is transported by a circulation pump into the third-effect evaporator and progressively concentrated until reaching the specified concentration, after which it is discharged from the system through a discharge pump. The secondary steam emanating from the third-effect separator is entirely conveyed to the condenser for condensation.

In summary, the multi-effect evaporation system has the capacity to utilize the secondary steam generated in the preceding effect as the heat source for the subsequent effect to improve energy efficiency. Nevertheless, the first effect evaporator requires considerable fresh steam from the external environment as a heat source for heating, and the secondary steam from the last effect separator also needs to be condensed, which results in the consumption of cooling water.

In addition, during the evaporation process, it has been found that PG would be easily entrained in the evaporated water, which would make some PG to enter the steam condensate and cause loss. To avoid this phenomenon, a purification process utilizing conventional distillation as shown in Fig. 3 was designed. Additionally, in order to examine the influence of tower pressure on separation, a systematic assessment spanning operating pressures ranging from 50 kPaA to 200 kPaA was conducted. To ensure that the PG content in the tower kettle is consistent under the uniform standard, which is 33.30%. The computational results are presented in the Supporting Information Table S4. It can be seen that with the decrease of pressure (When the pressure is positive), there is a gradual reduction in TAC accompanied by an increase in PG yield. It's worth noting that as the pressure reaches atmospheric levels, the yield remains basically the same. However, when the operational pressure falls below 101.325 kPaA (taking 50 kPaA as an example), the influence of vacuum device leads to an increase of TAC. Based on the PG yield and TAC calculation results, the operational pressure was conclusively determined to be 101.325 kPaA.

It can be seen that the feed is fed into the distillation tower from the bottom, where they undergo a series of evaporation and separation that result in the high-purity PG being withdrawn from the bottom of the tower. Meanwhile, a considerable amount of water is collected by condensing the gas at the top of the tower. In addition, it should be noted that the pressure listed in the subsequent process refers to the absolute pressure.

The precise calculation showed that only 10 theoretical stages were needed to achieve the desired separation of PG and water, effectively reducing the loss of PG. However, due to the low concentration of PG in the feed, it is necessary to evaporate 93.5% of the water in order to achieve the target concentration in this way. The aforementioned process requires a significant amount of energy consumption, as both the steam utilized by the distillation kettle and the cooling water used in the condenser at the top of the tower are energy-intensive.

On the basis of the conventional distillation process in Fig. 3, a process of recovering PG using MVR distillation technology was designed as depicted in Fig. 4. The feed is introduced into the distillation tower from the bottom, subsequently undergoes complex series of processes in the tower. The resulting top gas of the tower is directly fed into the compressor (the inputs of simulations are the compression ratio and the efficiency is 85%), where it is compressed to generate high-temperature and high-pressure steam. Much of this steam is then utilized as the primary heat source for the reboiler of the tower. Following the heat exchange process, a portion of the resulting condensate is recycled back into the tower as reflux, while the remaining portion is extracted. This process is repeated until the desired level of purity is achieved. Ultimately, the kettle of the distillation tower yields a purified PG solution (composed of water (54.5%), PG (33.3%), DPGE (3.5%), salt

(6.4%), and others), which can be further processed or utilized for various applications. Meanwhile, the top of the tower produces high-purity steam with a composition of 99.99% water vapor.

Although the MVR distillation technology has been reported for seawater desalination, to the best of our knowledge, there is currently no published report on the application of this technology for the purification of PG. It should be noted that there exist some differences in this technology between seawater desalination and PG purification. In this work, the feed used in the purification process of PG are sourced from the wastewater discharged by the synthesis unit in the PO production facility. These materials are sent out from the tower kettle of the upstream distillation unit and are at a temperature higher than 90°C. In contrast, seawater desalination is typically obtained at ambient temperature, which presents higher requirements for heat matching and compressor operation. Additionally, the temperature difference between the top and bottom of the distillation tower in our study is small ($\Delta T = 7^\circ\text{C}$), which has higher design requirements for the reboiler of the tower kettle. Through the calculations, it is found that in order to guarantee that the temperature difference between the hot side and cold side of the reboiler is higher than 10°C , the compression ratio of the compressor needs to exceed 1.6. This results in a surplus of steam after the compressed steam matches the heat required by the reboiler, which eliminates the need for fresh steam from outside during operation. Consequently, this procedure greatly saves the consumption of steam and cooling water.

To facilitate a comprehensive comparison of the three processes, a detailed cost analysis was performed, as presented in Table 3, which provides an overview of quality indicators, energy consumption and TAC associated with various technologies employed in the purification process of PG. It is evident that compared to the conventional distillation process, both the three-effect evaporation process and the MVR heat pump distillation process significantly reduce energy consumption, with respective cost reductions of 75.04% and 83.12%. Additionally, the TAC is correspondingly reduced by 36.80% and 49.22% for these processes. However, it is noteworthy that when a substantial concentration of PG solution is obtained, the yield of the conventional distillation process and the MVR heat pump distillation process (97.80%) surpasses that of the three-effect evaporation process (83.95%), primarily due to the entrainment of PG during the water evaporation stage in the three-effect evaporation process. Consequently, while ensuring a satisfactory yield, the MVR heat pump distillation process proves to be more energy-efficient and more economics-efficient compared to the other two processes. This is attributed to the fact that the MVR heat pump distillation process eliminates the need for external steam and cooling water, relying solely on the electrical consumption generated by the compressor. Based on this information, it is evident that the MVR heat pump distillation process emerges as the optimal choice for the first stage of this process.

Table 3
Comparison of quality indicators and energy consumption of different processes.

Item	Triple-effect evaporation	Conventional distillation	MVR heat pump distillation
Product purity, wt%	33.30	33.30	33.30
Product yield, %	83.95	98.50	97.80
Cooling load, kw	6550	23,056	–
Heating load, kw	5481	21,963	–
Electricity, kw	–	–	1578
Cost of Steam or Electricity, USD/h	266.74	1068.88	180.46
TAC, USD/year	3566332.88	5642598.02	2865349.03

Note: the steam (0.4 MPa) price is set at 28.60 USD/hr, while the industrial electricity cost is assumed to be 0.11 USD/kWh.

3.2.2. MVR heat pump distillation technology

In order to optimize parameters for achieving efficient system heat matching and thereby harness the full potential of MVR heat pump technology, it is necessary to investigate the factors influencing its system energy matching. Hence, the effect of compression ratio, feed temperature, and tower pressure have been included in the scope of consideration.

3.2.2.1. Effect of compression ratio. To clarify the effect of compression ratio on the system energy matching, and thus to find the suitable compression ratio for the energy matching, it is necessary to investigate the correlation between the compression ratio (ϵ) of the compressor, the power consumption (P) of the compressor, the TAC, as well as the logarithmic mean temperature difference (ΔT) between the hot and cold sides of the reboiler in the column. The heating medium of the reboiler is provided by compressed steam from the compressor. Therefore, the ΔT between the hot and cold sides of the reboiler is restricted by the temperature of the compressed steam, which is related to the compression ratio of the compressor. In addition, in terms of energy consumption, the compression ratio also determines the power consumption of the compressor. Based on the above, the relationship between the ϵ , P , ΔT and TAC was investigated when the absolute pressure at the top of the PG concentration tower is 50 kPa and the vaporization rate of the reboiler is 13.5%, with ΔT calculated using equation (8).

$$\Delta T = \frac{\Delta t_2 - \Delta t_1}{\ln \frac{\Delta t_2}{\Delta t_1}} \quad (8)$$

Where, Δt_2 , Δt_1 is the larger and smaller of the temperature difference between the hot and cold sides of the reboiler, respectively, $^\circ\text{C}$.

From the Fig. 5 representations, it can be seen that with the increase of the ϵ , there is a noticeable linear growth in compressor power. This phenomenon coincides with the rise in ΔT . Moreover, the TAC exhibits a gradual upward trend, especially pronounced when the compression ratio surpasses 1.65. This phenomenon can be attributed to the fact that when the tower top pressure and reboiler vaporization rate are constant, the fixed pressure exerted at the top of the tower and the rate of gasification exhibited by the reboiler give rise to a consistent flow rate of gas at the tower top, which leads to a constant volume of gas being compressed by the compressor. Thus, as the compression ratio increases, the power of the compressor will cause a proportional increase. Moreover, the enthalpy of the top gas of the PG concentration tower increases after compression, and thus increasing the temperature of the saturated steam provided after compression. Therefore, when the vaporization rate of the reboiler and the cold side inlet and outlet temperature remain constant, an increase in compression ratio leads to a gradual rise in the logarithmic average temperature difference between the hot and cold sides of the reboiler. Additionally, it should be noted that the lower the compression ratio the larger the heat exchange area of the reboiler. According to the calculation analysis, when the compression ratio is lower than 1.55, the required heat exchange area of the reboiler is more than 5600 square meters, which poses a serious challenge to the design of the reboiler. By considering the practicality and cost factors, a compression ratio of 1.65 was chosen wisely.

3.2.2.2. Effect of feed temperature. During the process of MVR heat pump distillation, the matching of heat is constrained by the temperature of the feed. Specifically, the temperature of the feed entering the tower, the enthalpy of the steam after compression by the compressor, the vaporization rate and the reflux temperature in the reboiler restrict each other. Only when an optimized balance is achieved among these four factors, the compressed steam can quantity be exactly sufficient for the reboiler. Otherwise, there will be a mismatch between the compressed steam produced and the amount required for the reboiler. To investigate the influence of the feed temperature on the system heat, the relationship between the two under the condition of a certain

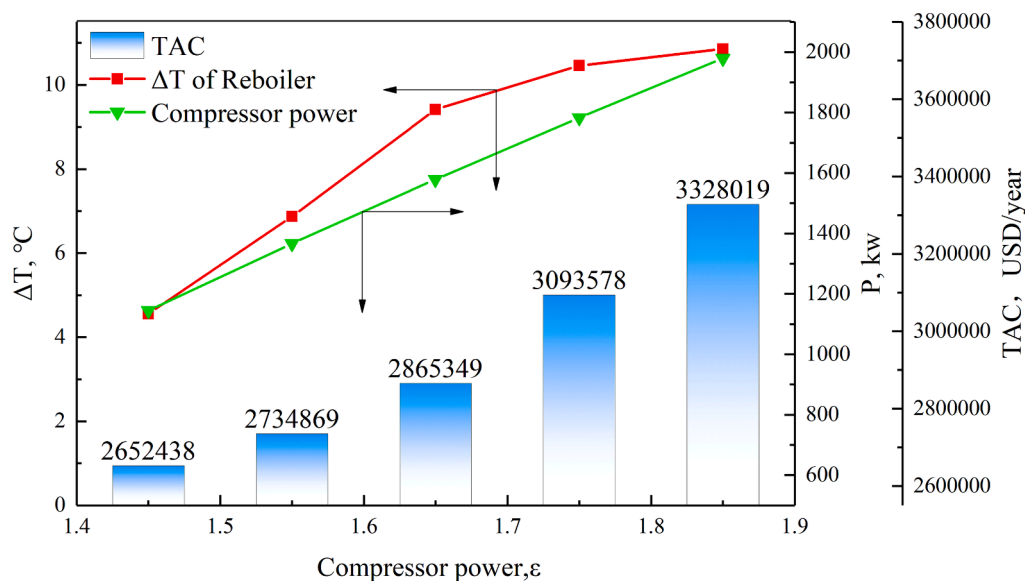


Fig. 5. The relationship among compression ratio (ϵ), compressor power (P), TAC and logarithmic mean temperature difference between the hot and cold sides of the reboiler (ΔT).

compression ratio of the compressor ($\epsilon = 1.65$) was examined.

Fig. 6 illustrates the relationship between the feed temperature and the heat difference of the system, wherein the heat difference refers to the difference between the heat provided by the system and the actual heating load of the reboiler when the compression ratio is 1.65 and the heating load of the reboiler is constant. It is evident that when the feed temperature is 122 °C, the heat provided by the top gas of the PG concentration tower is exactly equal to the heating load (21942kw) required by the reboiler after being compressed by the compressor at a compression ratio of 1.65. However, if the feed temperature exceeds 122 °C, the heat provided by the system exceeds the heating load required by the reboiler. For example, when the feed temperatures is 127 °C and 132 °C, the heat difference is −279 kw and −485 kw, respectively. On the other hand, when the feed temperature is lower than 122 °C, the system provides less heat than the required heating load by the reboiler. For example, at feed temperatures of 112 °C and 117 °C, the heat difference is 345 kw and 143 kw, respectively. Thus, to achieve the same evaporation amount, an additional heat supply is necessary from the external environment. In a word, the feed temperature has a considerable impact

on the system's heat matching, primarily because of the high feed flow rate of 30000 kg/h. As the temperature rises, a considerable amount of heat is introduced into the system.

3.2.2.3. Effect of tower pressure. Due to the significant difference in boiling points between PG and water in the system, their separation is relatively easy, and just 10 theoretical plates are sufficient for separation. The change in relative volatility has minimal impact on the ease of separation. While the pressure variations affect the temperatures of the column's top and bottom, which are closely related to the matching of the reboiler heating load and the ϵ of the compressor, and finally affects the energy balance of the whole system. Consequently, the focus of this investigation centers on studying the influence of tower pressure. Specifically, this study increases the pressure of PG concentration tower from 50 kPa(A) to 200 kPa(A) under the same evaporation rate (vaporization rate of 13%) in the reboiler, and explores the matching relationship between the reboiler heating load and the compression ratio of the compressor. As observed from the results presented in Fig. 7, an increase in tower pressure leads to a corresponding increase in the

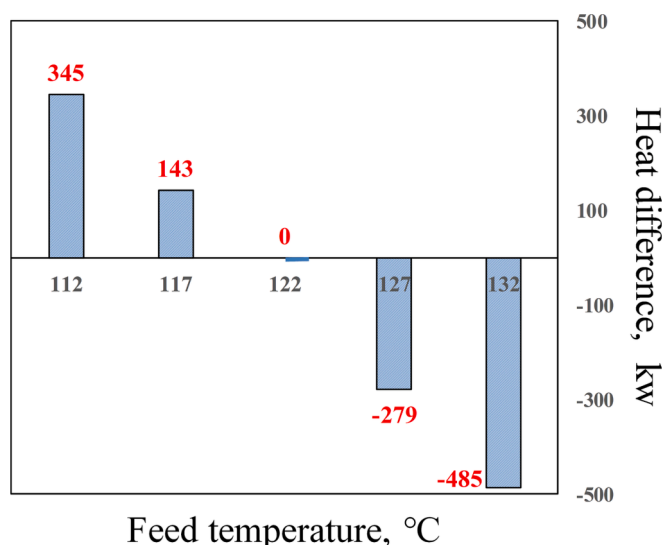


Fig. 6. The relationship between feed temperature and heat difference.

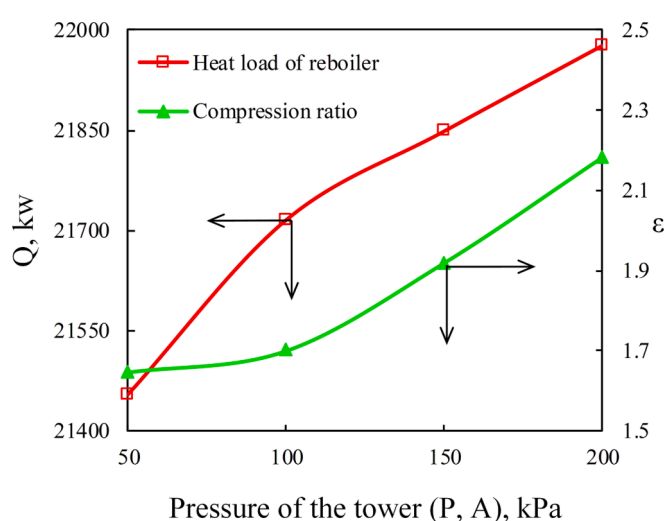


Fig. 7. Pressure of PG concentration tower as a function of reboiler heating load (Q) and compression ratio (ϵ).

reboiler heating load and compression ratio. This is attributed to the increase in pressure causes the temperature at the top and bottom of the tower to rise, thus reducing the amount of gasification. To achieve the same evaporation, more external heat is required, which increases the reboiler heating load. The heating source of the reboiler is the compressed steam from the top of the PG concentration tower, which is mainly water vapor. The enthalpy value of the steam increases after being compressed and pressurized by the compressor, thus rendering it a suitable heat source for the reboiler. Therefore, when the heating load of the reboiler increases from 21,454 kw to 21,976 kw, the compression ratio of the corresponding compressor must be increased by 2.180 from 1.647 to complete the heat matching.

3.3. The second stage of purification

Following the first stage of purification, the wastewater yields a 33.3% aqueous solution of PG, which primarily consists of the following components: water (54.5%), PG (33.3%), DPGE (3.5%), inorganic salts (6.4%), and other high boiling point organic compounds. In order to obtain a high concentration of PG product, a two-tower purification process flow diagram has been designed as shown in Fig. 8. The wastewater first passes through a scraping film evaporator, where PG, water, and organic compounds are evaporated and removed, and the high concentration salt solution is obtained at the bottom, thus the inorganic salt contained in the wastewater is separated from the system. The desalinated wastewater is subsequently introduced into the dehydration tower at its midpoint, where the lighter components, such as water, is separated from the heavier ones. Specifically, PG and high boiling point organic compounds are extracted from the bottom of the tower, while the purified water is recovered from the top of the tower. The material from the tower kettle is then fed into the de-heavy tower, where the high boiling point organic compounds are removed from the bottom of the tower and a PG product with a concentration higher than 90% is obtained from the top of the tower (the separation of PG and DPGE proves challenging).

Based on the results presented in Fig. 8, it can be observed that the heating load necessary for the scraping film evaporator is quite close to the cooling load required by the de-heavy tower. By increasing the

pressure of the de-heavy tower slightly, it is feasible to match both loads. This indicates that the overhead gas discharged from the de-heavy tower can serve directly as the heating medium for the scraping film evaporator, while the resulting condensed liquid from the heating process can be partially utilized as reflux for the de-heavy tower and partially extracted. The implementation of such an approach can significantly reduce the heat medium consumption of the scraping film evaporator and the cold medium consumption of the de-heavy tower condenser.

Moreover, through a comparative analysis of equipment energy consumption, it has been observed that the reboiler unit of the dehydration tower consumes the largest proportion of heat medium, accounting for 43% of the total. To mitigate external heat reliance and achieve a significant reduction in overall heat consumption, an optimized solution (as shown in Fig. 9) building upon the process outlined in Fig. 8 has been devised. This solution entails the division of the dehydration tower into two distinct towers: the first dehydration tower and the second dehydration tower. In the first dehydration tower, only 98.7% of the water is extracted, with complete elimination of the remaining water occurring in the second dehydration tower. By deliberately retaining a small amount of water (approximately 2.4% content) in the kettle of the first dehydration tower, the kettle temperature can be effectively reduced from 140 °C to 104 °C. As a result, the rich steam generated by the compressor in the first stage can be fully utilized in the subsequent stage of the process. Table 4 provides a comparison of the energy consumption between the two-tower process and the thermally coupled process. The results indicate that the heating load required by thermally coupled technology is from 2972 kW to 912 kW, representing a 69% reduction, while the cooling load is also reduced from 3067 kW to 2279 kW, a decrease of 25.6%.

3.4. Optimization of PG purification process

Through the synergistic integration of the MVR technology applied in the first stage and the thermally coupled process employed in the second stage, followed by further optimization, a final, refined purification process (depicted in Fig. 10) has been devised. This process named MVR- thermally coupled technology exhibits the capability to achieve a PG concentration surpassing 90% in wastewater derived from

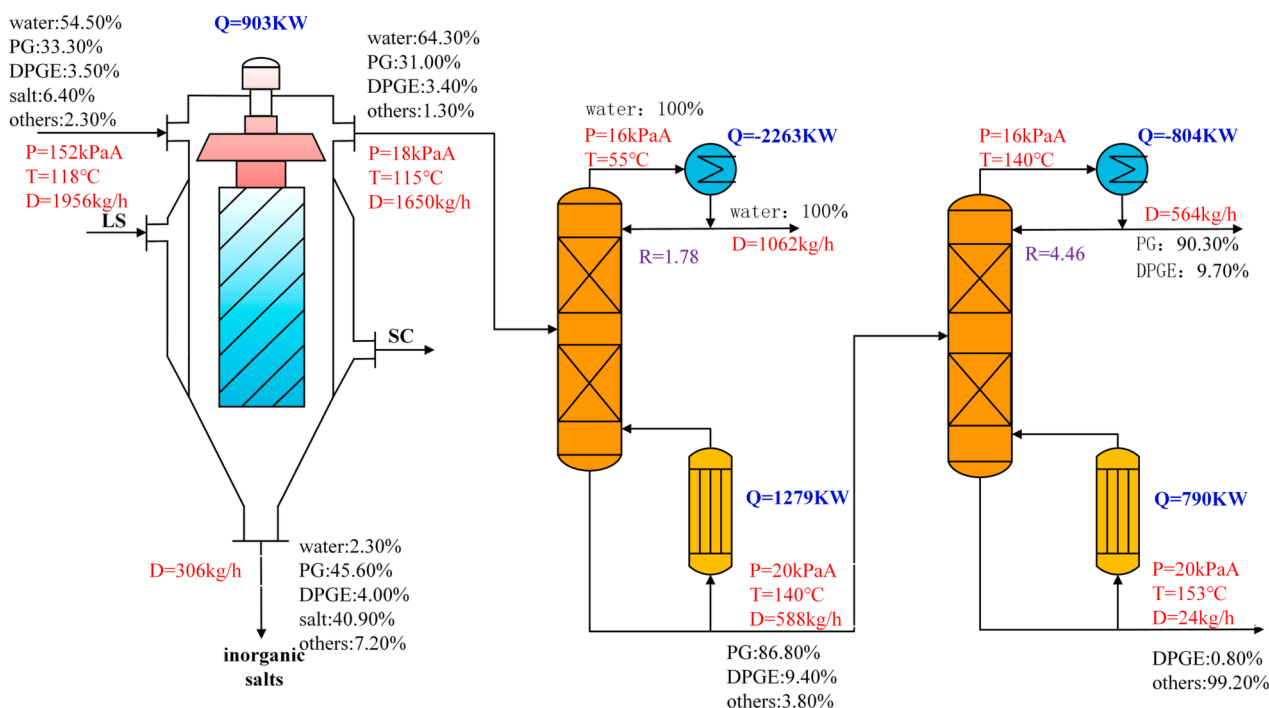


Fig. 8. The second stage of the PG purification process.

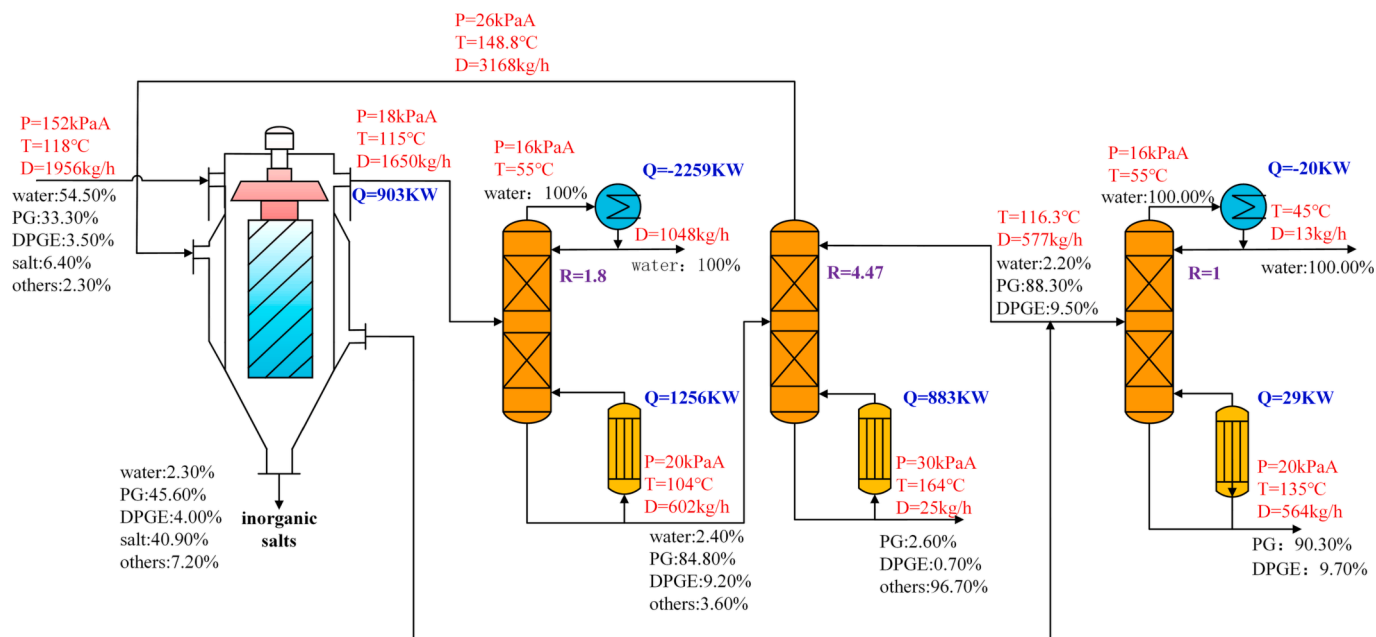


Fig. 9. Thermal coupling process for the second stage of PG purification.

Table 4

Comparison of energy consumption between the two-tower purification process and the thermally coupled process in the second stage.

Load, kw	Two-tower process	Thermally coupled process
Scraping film evaporator	903	—
Dehydration tower condenser	—2263	—2259
Dehydration tower reboiler	1279	—
De-heavy tower condenser	—804	—
De-heavy tower reboiler	790	883
Second Dehydration tower condenser	—	—20
Second Dehydration tower reboiler	—	29
Total cooling load	—3067	—2279
Total heating load	2972	912
PG purity, %	90.3	90.3
PG yield, %	78.2	78.2

PO production, even when the initial PG content is as low as 2.22%. With the objective of minimizing energy consumption, this process incorporates three key energy-saving measures, distinguishing it from conventional distillation technology (as shown in Fig. S1 of the Supporting Information). Firstly, the initial purification of PG was carried out using a MVR distillation process in the first stage, leading to a significant reduction in the consumption of fresh steam. Secondly, the second-stage dehydration tower is divided into two parts, and the surplus steam compressed by the first-stage compressor is served as the heat source for the reboiler of the first dehydration tower to reduce steam consumption. Finally, the condenser of the de-heavy tower is thermally coupled with the scraping film evaporator to reduce the consumption of heating and cooling media. Table 5 compares the energy consumption between conventional distillation technology and the MVR- thermally coupled technology.

As depicted in the [Table 5](#), the MVR-thermally coupled process, proposed in this study, exhibits notable improvements in terms of heating load reduction compared to the conventional distillation process. The heating load is reduced from 24,935 kw to 912 kw, reduced by 96.3%, accompanied by a decrease in cooling load from 26,123 kw to 2,279 kw, reduced by 91.3%. However, there is a modest increase in electrical load, rising by 1,578 kw. Considering the distinct cost

implications associated with these various loads, a thorough economic evaluation of both processes was conducted through TAC calculations to further compare their economic performance.

TAC is a key indicator for evaluating the economic performance of chemical processes. According to reports, TAC comprises the operating cost (OC) and annual capital investment (CI). The specific calculation formula for TAC is as follows:

$$TAC = \frac{CI}{Payback\ Period} + OC \quad (9)$$

Where, CI comprises shell of column, stages, condensers, reboilers, compressor, pump and other costs, while OC comprises cooling water, electricity and heating steam. Additionally, in this work, the payback period is set three years with the operating hours of 8000 h/year assumed.

Table 6 shows the cost of each specific component, and the details of TAC calculation are shown in Table S5 and Table S6 of the Supporting Information.

From the Table 6, it is evident that compared to the conventional distillation process, the MVR-thermally coupled process proposed by us results in a 34.89% reduction in the TAC. This reduction is attributed to the fact that although the MVR-thermally coupled process incurs higher CI due to the addition of a compressor and an extra tower, it significantly reduces steam consumption, thereby lowering OC and ultimately reducing the TAC.

4. 4. Conclusion

This study aims to investigate the treatment of wastewater from PO production units through a two-stage purification process that utilizes MVR heat pump technology and thermally coupled technology. The primary objective is to achieve purification of the low-concentration PG present in the wastewater.

In the first stage of the purification process, the content of PG was purified from 2.22% to 33.3% triple-effect evaporation, conventional distillation and MVR distillation, respectively. The findings demonstrate that although triple-effect evaporation technology saves energy compared to conventional distillation technology, it exhibits lower yield. Conversely, MVR technology ensures greater cost savings at the

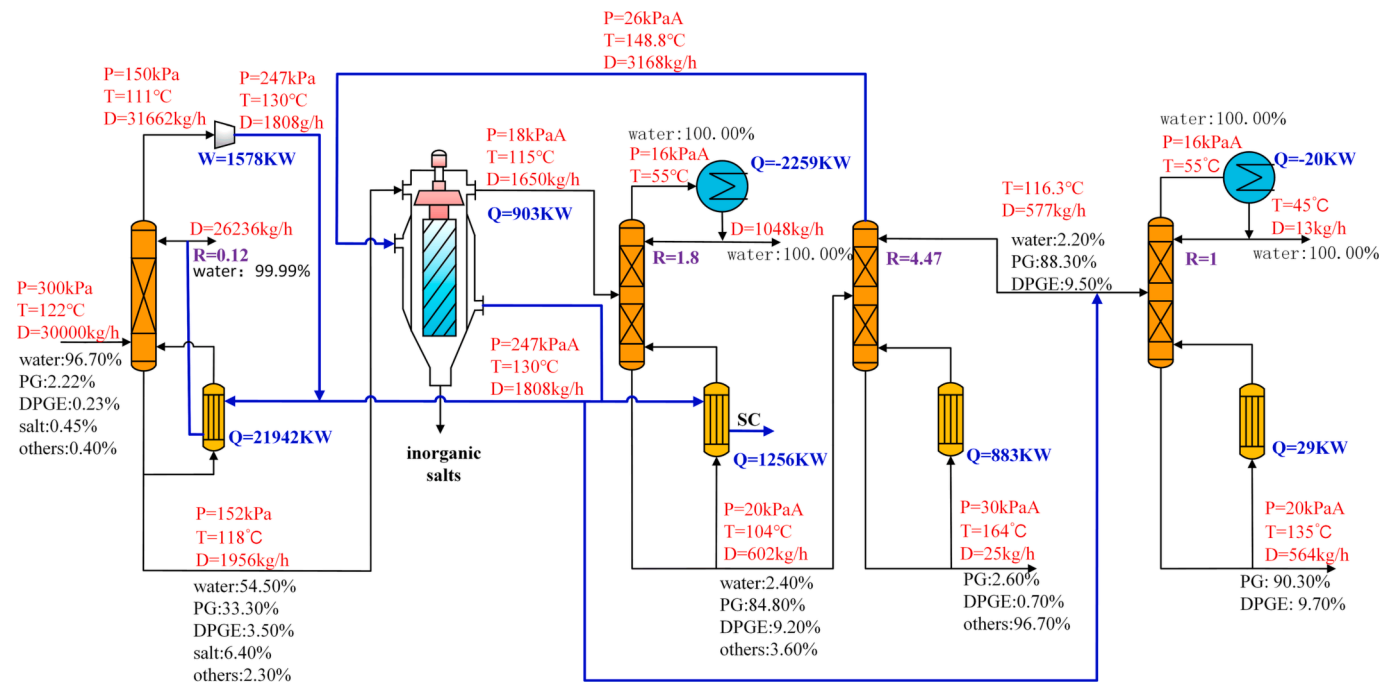


Fig. 10. Optimized process for purification of PG from PO production wastewater.

Table 5
Comparison of the conventional distillation process and MVR- thermally coupled process for the purification of PG.

Load, kw	Conventional distillation process	MVR- thermally coupled process
Heating load	24,935	912
Cooling load	26,123	2279
Electricity	–	1578

Table 6
Details of TAC and cost Accounting for the conventional distillation process and MVR- thermally coupled process.

Performance item	TAC of Conventional distillation process, USD/year	TAC of MVR- thermally coupled process, USD/year
Column shell1	470,869	470,869
Column shell2	2,761,108	2,761,108
Column shell3	2,265,188	2,265,188
Column shell4	–	1,054,077
Condenser 1	367,887	–
Condenser 2	239,708	239,433
Condenser 3	32,428	7,293
reboiler1	620,503	1,318,448
reboiler2	158,330	131,940
reboiler3	87,062	124,440
reboiler4	–	107,640
scrapping film evaporator	120,035	82,490
Compressor	–	4,501,549
CI	7,085,574	13,064,474
Electricity	2,171	770,216
water	266,329	23,235
steam	5,597,047	215,540
OC	5,865,547	1,008,991
TAC	8,237,748	5,363,815
TAC saving (%)	0	34.89%

same yield level. Its electricity cost is merely 67.65% of the steam cost associated with triple-effect evaporation, making it a more suitable choice for the first stage application of this technology. Moreover, the effects of compression ratio, feed temperature and pressure of the PG

concentration tower on the MVR distillation process were investigated, and the heat matching of the system was realized by optimizing parameters. In the second stage of the purification process, a distillation separation process of desalination, dehydration and removal of heavy components was designed. To save energy, the dehydration tower was divided into two parts, and the rich steam compressed by the compressor in the first stage was used as the heat source for the reboiler of the first dehydration tower. Additionally, the condenser of the de-heavy tower was thermally coupled with the scraping film evaporator, reducing the required heating load by 69% and the cooling load by 25.6% compared to the two-tower separation process.

The developed MVR-thermally coupled optimization process, composed of two stages, exhibits a significant reduction in the workload compared to conventional distillation process, resulting in substantial cost savings. Specifically, the heating load is remarkably reduced from 24963 kW to 912 kW, resulting in a reduction of 96.3%. Similarly, the cooling load experiences a substantial decrease from 26123 kW to 2279 kW, representing a reduction of 91.3%. However, there is a modest increase in the electrical load, which rises by 1,578 kW. Despite this, the TAC of the MVR-thermally coupled process is impressively reduced by 34.89% compared to the conventional distillation process. This process provides a new way of high efficiency and energy saving for the recovery of high value organic compounds from PO production wastewater.

CRediT authorship contribution statement

Jing Ma: Conceptualization, Methodology, Software, Data curation, Formal analysis, Validation, Visualization, Writing – original draft, Writing – review & editing. **Xiejun Wang:** Visualization, Writing – review & editing. **Yamei Zhou:** Formal analysis, Writing – review & editing. **Yaxuan Du:** Writing – review & editing. **Baohe Wang:** Conceptualization, Supervision, Resources, Project administration, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence

the work reported in this paper.

Data availability

No data was used for the research described in the article.

Acknowledgements

This work was supported by the Sinopec on the project of recovering alcohol ether from HPPO wastewater (416061)

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.seppur.2023.125064>.

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