IMPERIAL

Fabrication of Graphene Oxide Membranes with Controlled Graphitic Domain for Liquid Separation

Ande Fudja Rafryanto
Department of Chemical Engineering, Imperial College London

1 Introduction

Amidst the growing emphasis on sustainable energy, bioethanol has emerged as a promising solution. However, the **azeotropic point** presents **a significant challenge in its purification**. **Conventional methods, such as distillation**, are known to be **inefficient and energy-intensive** [1, 2]. Recent research highlights **graphene oxide (GO)** membranes as a potential alternative for ethanol upgrading due **to their tunable pore sizes**. However, **their hydrophilic nature makes them highly susceptible to swelling**, compromising performance [3]. Therefore, controlling hydrophobicity is critical to prevent swelling. In this study, **reduced graphene oxide (rGO),** with **enhanced stability and selectivity** due to its **hydrophobic properties,** is being explored as a solution [4].

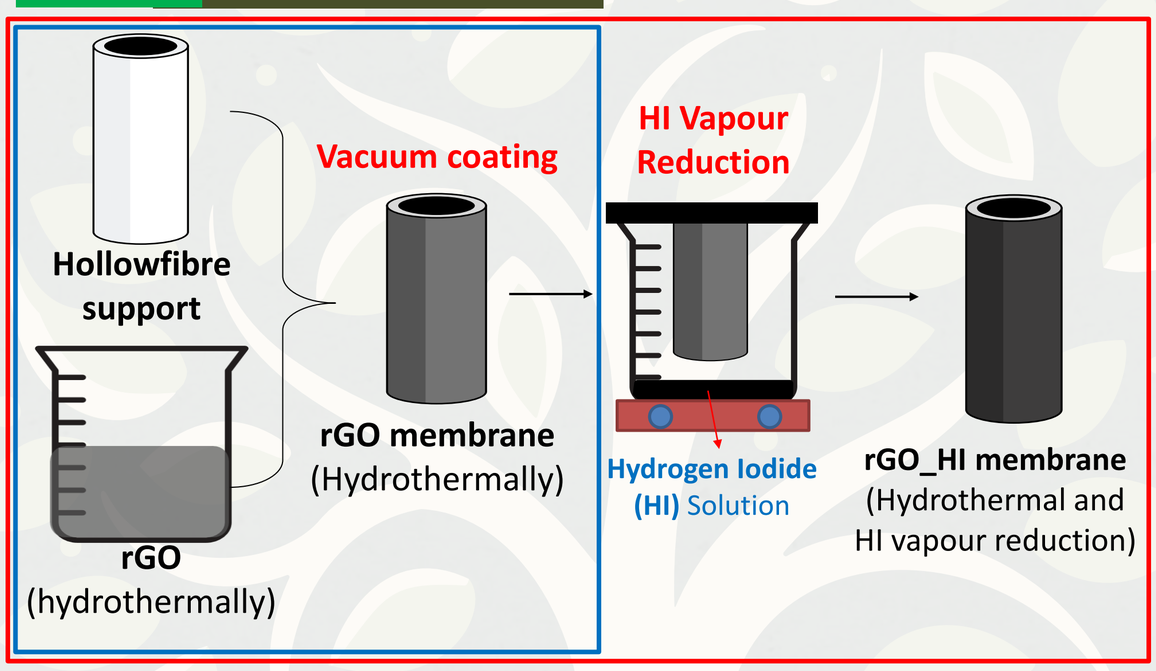
2 Objective

In this study, we aim **to fabricate graphene oxide membranes** with **controlled graphitic domains** through an **optimized reduction process**, resulting in both reasonable permeate flux and effective rejection. The following are proposed reduction approaches:

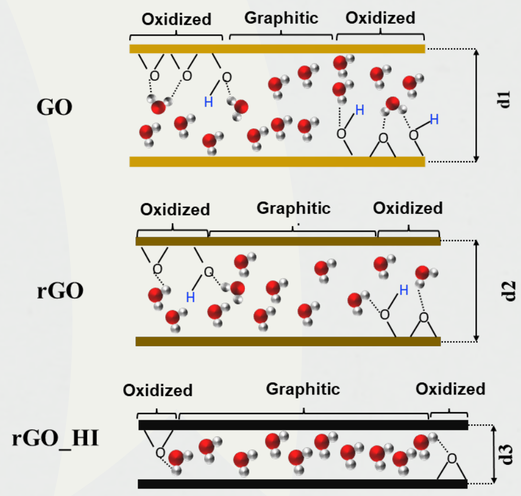
1. **Single Hydrothermal**
2. **Hydrothermal with HI**

3 Method

Membrane Coating with controlled graphitic domain



Hypothesis:
Combining two reduction processes could **increase the C/O ratio**, leading to **a decrease in interlayer d-spacing**



4 Result and Discussion

4.1.Structure and Properties

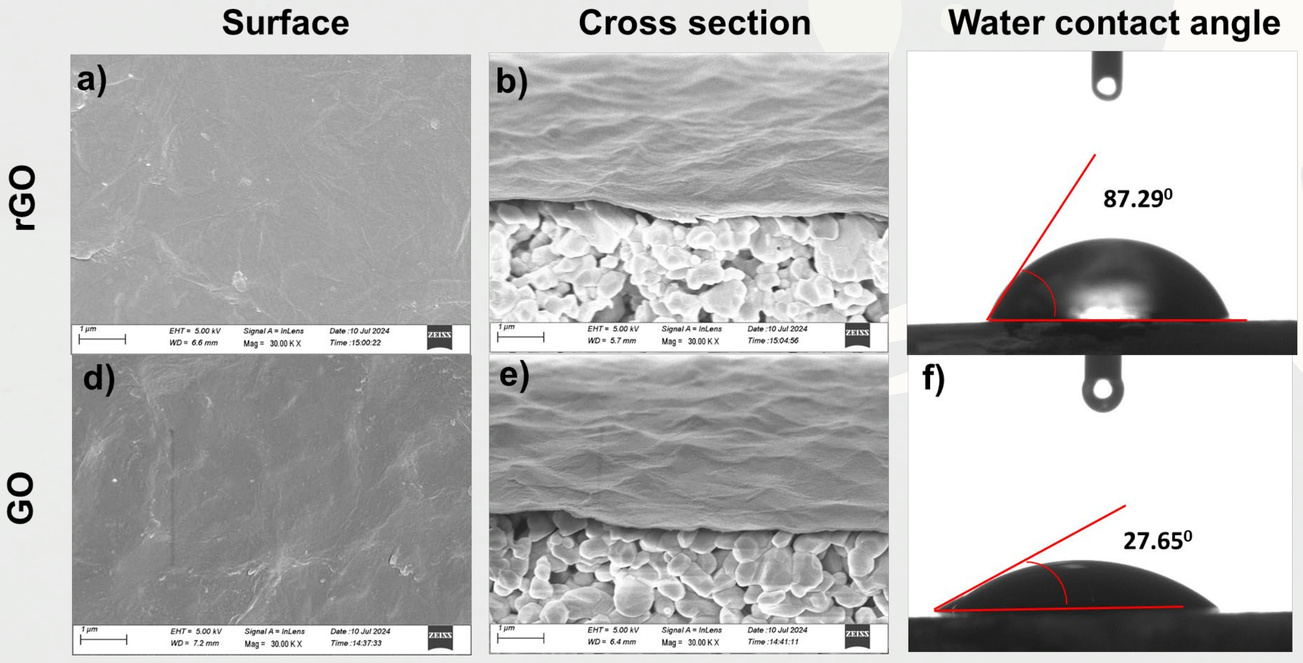


Figure 1. SEM images of (a) rGO surface area, (b) rGO cross-section, (d) GO surface area, (e) GO cross-section, and water contact angles (WCA) of (c) rGO and (f) GO.

Both **GO** and **rGO** membranes exhibit **smooth and dense surface structures**, as shown in **Fig. 1**. In terms of **wettability**, **GO is more hydrophilic** with a **water contact angle (WCA) of 27.65°**, while **rGO is more hydrophobic** with a **WCA of 87.29°**. Additionally, the combined hydrothermal and 1-minute HI vapor reduction resulted in a WCA of approximately **101.33°**.

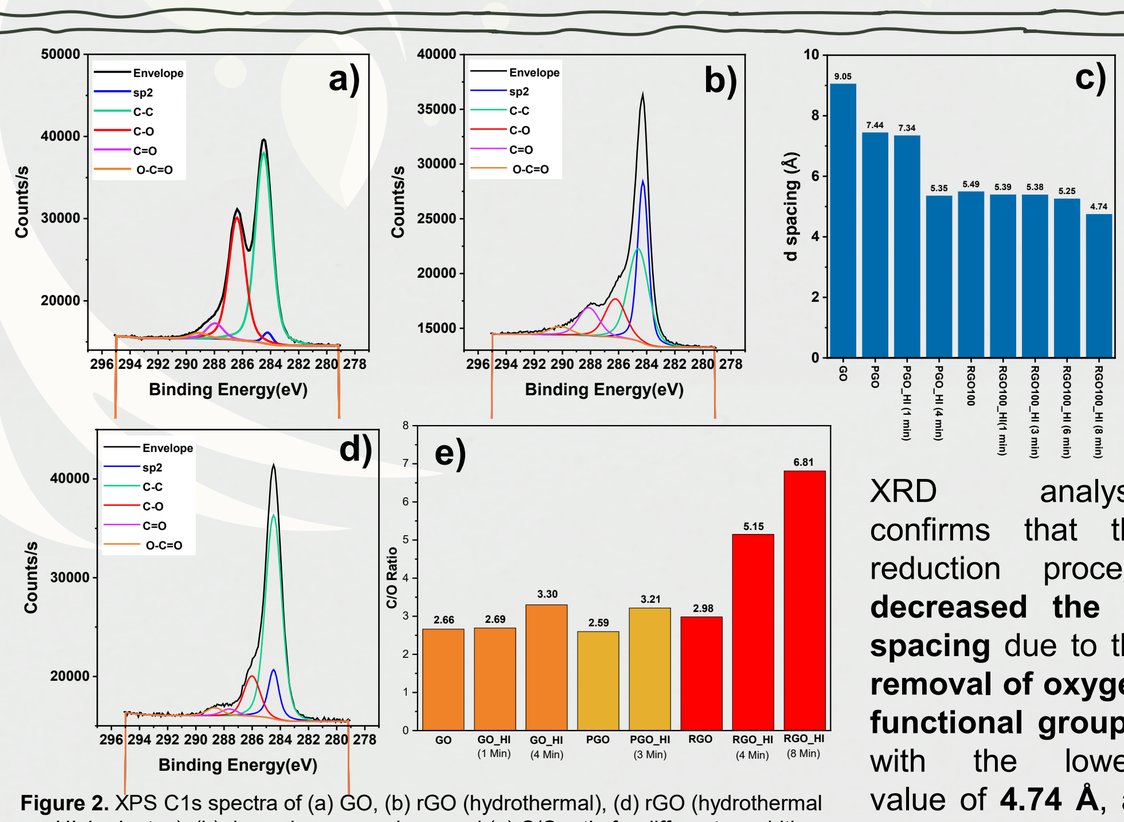


Figure 2. XPS C1s spectra of (a) GO, (b) rGO (hydrothermal), (d) rGO (hydrothermal + HI 4 minutes), (b) d-spacing comparison, and (e) C/O ratio for different graphitic controls.

XRD analysis confirms that the reduction process **decreased the d-spacing** due to the **removal of oxygen functional groups**, with the lowest value of **4.74 Å**, as shown in **Fig. 2 (e).**

The degree of reduction, indicated by the **C/O ratio** depicted in **Fig. 2 (c)**, confirms that rGO produced by combining hydrothermal treatment and an 8-minute HI reduction achieved the **highest value of 6.81**, compared to **2.98 for the single hydrothermal process.**

4.2. Performance

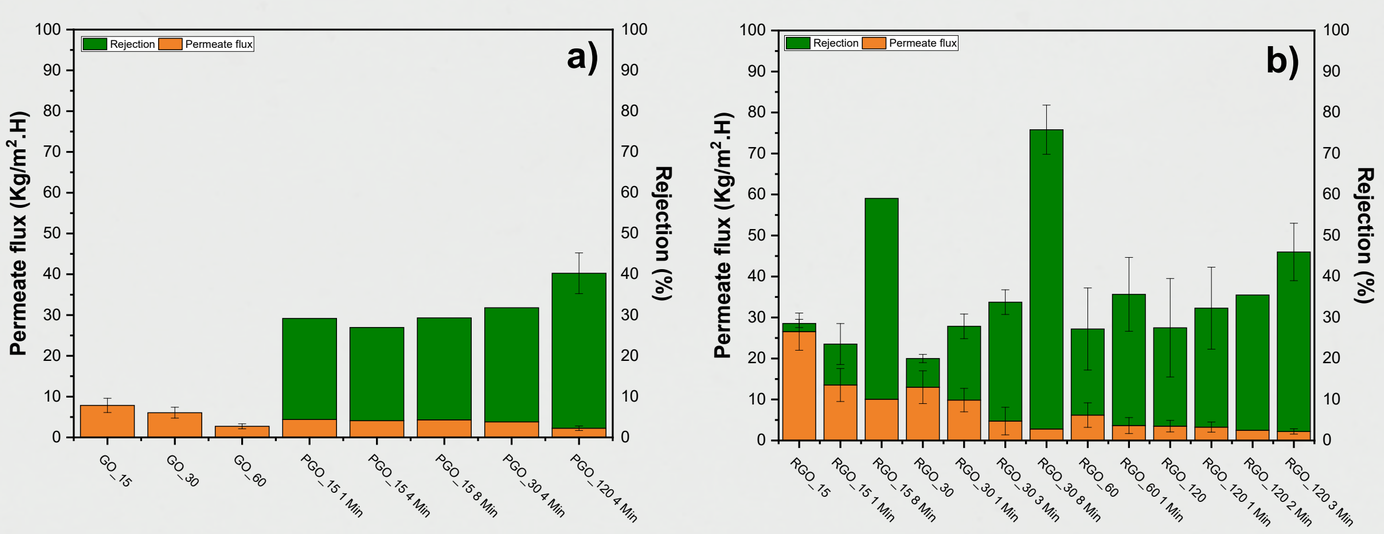


Figure 3. Rejection and permeate flux in pervaporation tests for (a) GO and PGO membranes, and (b) rGO membranes with various reduction methods.

The combined hydrothermal process and 8-minute HI vapor reduction resulted in an impressive rejection rate of approximately **73%**. HI vapor effectively penetrates thinner membranes, further reducing oxygen groups. This creates a more hydrophobic rGO surface, enabling a smoother, frictionless flow of water vapor molecules through the membrane channels.

5 Conclusion

GO-based membranes with regulated graphitic structures were successfully fabricated using a combined reduction approach. The combination of hydrothermal treatment and 8 minutes of HI vapor reduction increased the C/O ratio to 6.81, supported by water contact angle measurements indicating a highly hydrophobic surface (101.33°). The d-spacing was reduced to 4.78 Å. Further research is needed to enhance performance, particularly improving the separation factor of the membranes while maintaining permeate flux.

6 Acknowledgement

I would like to express my sincere gratitude to my daily supervisor, Dr. Farhad Moghadam, for his unwavering support and guidance throughout this project. Finally, I extend my heartfelt thanks to Professor Kang Li for his invaluable support and insightful advice.

REFERENCES:

[1] Castro-Muñoz, 2023. Separation and Purification Technology 308, 122919.
[2] Huang, H.-J., 2013. John Wiley & Sons, Ltd, pp. 1–36.
[3] Zheng, S., 2017. ACS Nano 11, 6440–6450.
[4] Yu, 2017. 2D Mater. 4, 045006.