Kinetic study of water transport in layered graphene oxide membranes

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**ABSTRACT**

Layered graphene oxide (GO) membranes are considered promising separation membranes because of their lamellar structure, strong mechanical strength and strong hydrophilic properties. However, there are a limited number of studies on the mechanism of water transport through a layered GO membrane. In this paper, by using a self-synthesized layered GO membrane, we developed and validated the first dynamic equation to describe water transport through a layered GO membrane. The results showed that the relationship between membrane flux and GO loading is an exponential function and that the water flux decreases with increasing GO loading. Then, the dynamic equation was applied to previously reported layered GO membranes to further verify its applicability. This kinetic study on layered GO membranes provides insight into the development of high-performance membranes with high permeability and selectivity.

1. Introduction

Graphene oxide (GO), a two-dimensional carbon material, has received an extensive amount of attention for its potential application in membranes owing to its numerous hydrophilic groups, exceptional mechanical strength, and superior structural and thermal properties [1–13]. Layered GO membranes composed of stacked GO nanosheets can be fabricated by vacuum filtration or layer-by-layer assembly methods. The vacuum filtration method filters a certain amount of a GO solution through the support layer \cite{14,15}, while the LBL method covalently links GO sheets and chips through the oxygen-containing functional groups on the GO surface \cite{16,17}. We have found that layered GO membranes have great potential for use in filtration and separation due to their unimpeded water permeation and excellent separation performance \cite{18–20}.

Currently, there are multiple views regarding the water transport mechanism in layered GO membranes. Boukhalov et al. \cite{17,21} believe that there are two main regions in GO membranes, the oxidized region and the non-oxidized region. The oxidized region causes adjacent microcrystals to separate and inhibits their reunion, allowing water to flow between the GO layers, while the non-oxidized region provides a capillary network that creates high capillary pressure, and water flows smoothly under this pressure. Moreover, the smooth surface of the non-oxidizing region enables water to flow without friction. Boukhalov has demonstrated that the water permeability of a GO membrane is mainly caused by the heterogeneous migration of water in the non-oxidized region. On the other hand, Wei et al. \cite{22} believed that the high water flux through a GO film is caused by the microstructure of GO, which includes valleys, folds, cavitations and voids.

Currently, the main research on the kinetic mechanism of water transport in layered GO membranes is as follows: Huang et al. \cite{23} have fabricated ultrathin graphene nanofiltration membranes (uGNMs) by filtering an extremely dilute solution of base-refluxed reduced GO (brGO) through a microporous substrate. They regarded the flow of water through the graphene oxide membrane as being a classical viscous flow and used the modified Hagen-Poiseuille equation to describe the flow of water in the graphene oxide membrane. The details can be seen in formula (1):

\[ J = \frac{h^4 \Delta p}{12L^2 \eta \Delta x} \]  

(1)

where J refers to the water permeability, h is the vertical space between adjacent graphene sheets, \( \Delta p \) is 1 bar, L is the average lateral length of the brGO sheets (1.0 \( \mu \)m in their study), \( \eta \) refers to the viscosity of water (0.001 Pa·s at 20°C) and \( \Delta x \) refers to the thickness of the graphene oxide membrane.

When the thickness of the graphene oxide sheets is 2.03 nm and their interlayer spacing is 0.7 nm, the theoretical water permeability is calculated to be 10\(^{-6}\) L/m\(^2\)-h-bar. However, the experimental value was 71 L/m\(^2\)-h-bar, which is 5 to 6 orders of magnitude higher than that of the theoretical value \cite{15}. Similarly, Han et al. \cite{14} used this formula to calculate the flux of
a modified graphene oxide membrane. When the thickness is 53 nm, the theoretical membrane permeability is $1.4 \times 10^{-4}$ m$^2$/h-bar, while the experimental water permeability was 3.3 L/m$^2$h-bar. The difference between the theoretical and experimental results can be explained by the increased interlayer spacing of the graphene oxide membrane under wet conditions compared with that of the dry graphene oxide membrane [15]. Additionally, the structural defects on the surface of graphene oxide can form surface micro-pores and large folds, resulting in a smaller $L$ value [14]. The high permeability can also be explained by the slip flow theory of the Hagen-Poiseuille equation. The basic assumptions of the equation are the presence of a flow and no slipping at the boundary layer; however, the departure of the experimental flux from that calculated by the classical equation suggests that the velocity of the liquid flow at the graphene wall is not zero [14]. However, there are only a limited number of studies on the kinetic mechanism of water transport through a layered GO membrane [24,25]. Furthermore, the applicability of this kinetic equation is not good. Therefore, studies on the water transport kinetics of layered graphene oxide membranes are urgently needed.

In this paper, based on the transport mechanism of graphene oxide films, a transport kinetics equation for layered graphene oxide membranes was developed. The equation was applied to a synthesized brominated poly(phenylene oxide)/graphene oxide (BPPO/GO) membrane to verify the accuracy of the kinetic equation, and the corresponding parameters were obtained. The kinetic equation was also applied to reported layered graphene oxide films to verify its applicability. This work provides a theoretical foundation for the application of graphene oxide membranes in water treatment.

2. Experimental section

2.1 Materials

Natural graphite powder, sodium nitrate (NaNO$_3$), potassium permanganate (KMnO$_4$), sulfuric acid (H$_2$SO$_4$), hydrogen peroxide (H$_2$O$_2$), ethylenediamine (EDA), hydrochloric acid (HCl; 32%) and 1-methyl-2-pyrrolidone (NMP, 99.5%) were purchased from Sigma-Aldrich. Brominated poly(phenylene oxide) (BPPO, Mw = 60,500) with a benzyl substitution ratio of 0.57 was provided by Tianwei Membrane Co., Ltd. China.

2.2 Preparation of the BPPO/GO membranes

The layered GO membrane used in this study was a BPPO/GO composite membrane. The preparation and properties of the membrane were described in detail in a previous report [26]. Briefly, the BPPO membrane substrate was fabricated by a phase inversion method, and GO/EDA mixtures were prepared by adding different amounts of GO into an EDA solution. Subsequently, the mixture was filtered through the BPPO substrate. Then, the hybrid membrane was placed in a water bath and heated for 4 h at 80°C. The obtained BPPO/GO membranes were labeled based on their GO loading.

2.3 Membrane permeability evaluation

The permeabilities of the BPPO/GO membranes were evaluated by measuring the membrane flux using DI water as the feed solution. Membrane performance tests were conducted using a dead-end filtration cell (Sterlitech. HP 4750). The filtration system contained a feed tank, digital balance, and filtration chamber. The filtration chamber had a capacity of 300 mL and an effective membrane area of 14.2 cm$^2$. The details of the filtration process can be seen in a previous report [26]. For the flux tests, five samples were analyzed for each membrane, and the standard deviation of the water flux ranged from 0.079 to 3.68.

2.4 Calculation method of the dynamics equations

All of the fitting calculations in this paper were performed using MATLAB software, as shown in the Appendix. The transportation dynamic equation was regressively obtained through the least squares method in the in-house toolbox (cftool) of MATLAB. Both the pre-exponential factor and the change coefficient of the membrane flux were assessed, with statistical significance denoted at the 5% level. The sum of squares error (SSE) and the root mean squared error (RMSE) were also calculated for each test (Appendix A and Appendix B).

3. Results and discussion

3.1 Dynamics model development

For the dynamic model in this study, it is assumed that the high water flux of the GO film is caused by the microstructure of GO, including its valleys, folds, cavitations and voids. We first prepare two support membranes: membrane A and membrane B. It is assumed that membrane A and membrane B have the same structures (individual pore size, hydrophilicity, roughness, thickness, etc.) except for the number of pores on the membrane surface. Thus, the membrane water flux is only determined by the number of surface pores. The more pores there are on the surface, the higher the water flux is. Thus, membrane A, which has a greater number of pores, has a higher flux, while membrane B has a lower water flux. In this study, it is assumed that
all pores can be collected and assembled into a single large pore, as described in Figure 1. In this case, the assembled pore of membrane A is larger than that of membrane B.

For the layered GO membrane, GO loading can affect the water flux of the composite membrane. If the initial GO loading is assumed to be m, then the pores on membrane A are assumed to be approximately 10% covered, and the smaller number of pores on membrane B will be approximately 70% covered. When GO loading is increased from m to 4m, the coverage of the pores on membrane A will increase to approximately 40%, and the coverage of the pores on membrane B will increase to approximately 80%, as shown in Figure 2. Correspondingly, the change in the flux of water through membrane A increases from a 10% to a 40% reduction and that of membrane B increases from a 70% to an 80% reduction. Therefore, we can conclude that the effect of GO loading on the flux of the composite membrane is related to the initial water flux of the composite membrane. Moreover, GO loading has a larger effect on a composite membrane with a larger flux and little effect on a composite membrane with a smaller flux.

The membrane flux is represented by Q, and the GO loading is represented by m. With a change in GO loading (Δm), the membrane flux of water will also change (ΔQ). The change in water flux (ΔQ) is related to the initial membrane water flux (Q). Thus, formula (2) can be obtained in this study:

\[ \frac{dQ}{dm} = kQ \]  

(2)

where k is the change coefficient of the membrane flux. Equation (2) can be rewritten as formula (3).
Then, formula (4) can be obtained by integrating both sides. Formulas (4), (5) and (6) can be obtained by integration. By solving the equation, Equation (7) for the membrane flux can be obtained.

\[-\frac{dQ}{Q} = kdm\]  \hspace{1cm} (3)

Combining the experimental results in Figure 3 and dynamics Equation (7), the simulated results are obtained and shown in Figure 4. The blue markers are the experimental values of the water flux, and the red curve is the simulated results.

The simulation results (Figure 4) show that \(a = 557\), \(k = -0.05854\) and the water transport dynamics equation in the BPPO/GO composite membrane in this study can be expressed as:

\[Q = 557 \cdot e^{-0.05854m}\]

where \(k\), \(a_1\), \(a_2\) and \(a\) are constants.

The \(k\) is the change rate of the membrane flux onto per mass of oxidized graphene. The \(m\) is the pre-exponential factor, which depends on the inner reaction properties and independent of the temperatures and the concentrations.

When \(m\) is equal to 0, \(e^{-km}\) is equal to 1 and \(Q = Q_{\text{max}}\).

As \(m\) approaches infinity, \(e^{-km}\) approaches 0 and \(Q = Q_{\text{min}}\) approaches 0.

3.2 Verification of the water transfer dynamics equation in the BPPO/GO composite membrane

Figure 3 shows the effect of GO loading on the water flux of the prepared BPPO/GO membrane. As shown in Figure 3, with increasing GO loading from 0 mg/m² to 39 mg/m², the BPPO/GO membrane flux decreases linearly from 550 L/m²·h·bar to 44.89 L/m²·h·bar. Further increasing the GO loading from 39 mg/m² to 78 mg/m² reverses the reduction of the BPPO/GO membrane flux. The results show that the water flux is related to the loading of GO. The higher the GO loading is, the thicker the formed GO membrane, and the higher the resistance to water transport.

The obtained equation was analyzed by regression analysis, and the results are shown in Tables 1, 2 and 3.

The degree of freedom is equal to the number of samples minus the number of variables minus 1; here, the degree of freedom is 5. For a significance level of 0.05, the correlation coefficient for the critical value of a test is 0.75994. In this study, the correlation coefficient is 0.9982 > 0.75449, suggesting that the regression analysis is significant.

When \(p < 0.05\), we can consider the model to be significant at \(\alpha = 0.05\), or the confidence level is 95%;
when \( p < 0.01 \), we can consider the model to be significant at \( \alpha = 0.01 \), or the confidence level is 99%; when \( p < 0.001 \), we can consider the model to be significant at \( \alpha = 0.001 \), or the confidence level is 99.9%. For the model in this study, \( p = 0.0000000542 < 0.0001 \), and we can consider the model to be significant at \( \alpha = 0.0001 \), or the confidence level reaches 99.99%. From the above fitting result, we confirm that the dynamic model is consistent with the experimental results, which proves the validity of the dynamic equation.

### Table 1. The regression analysis of the BPPO/GO membrane experimental results.

| Multiple R | R squared | Adjusted R squared | Standard error | Observation value |
|------------|-----------|-------------------|---------------|-------------------|
| 0.9982     | 0.9964    | 0.9957            | 13.0458       | 7                 |

### Table 2. The variance analysis of the BPPO/GO membrane experimental results.

| Variance analysis | df | SS     | MS      | F        | Significance of F |
|------------------|----|--------|---------|----------|------------------|
| Regression Analysis | 1  | 241,402.8077 | 241,402.8077 | 1418.4020 | 2.49E-07         |
| Residual Error    | 5  | 850.9675 | 170.1935 |          |                  |
| Total             | 6  | 242,253.7752 |          |          |                  |

### Table 3. The regression parameters of the BPPO/GO membrane results.

| Coefficients | Standard error | \( p \)-Value | Lower limit 95.0% | Upper limit 95.0% |
|--------------|---------------|---------------|-------------------|-------------------|
| Interception | 6.7726        | 6.2046        | 0.3248            | −9.1770           | 22.7222          |
| X Variable 1 | 0.9800        | 0.0260        | 2.49E-07          | 0.9131            | 1.0469           |

The obtained equation was further analyzed by regression analysis, and the results are shown in Table 4 and the Support Information (Table S1 and Table S2).

For a degree of freedom of 3 and a significance level of 0.05, the correlation coefficient for the critical value of a test is 0.87834, which can be obtained from a mathematical table. In this study, the correlation coefficient is 0.9760, which is larger than 0.87834, indicating that the regression analysis is significant.

When \( p < 0.05 \), we can consider the model to be significant at \( \alpha = 0.05 \), or the confidence level is 95%; when \( p < 0.01 \), we can consider the model to be significant at \( \alpha = 0.01 \), or the confidence level is 99%; when \( p < 0.001 \), we can consider the model to be significant at \( \alpha = 0.001 \), or the confidence level is 99.9%. For the results of Han et al., \( p = 0.0000000542 < 0.0001 \), and we can consider the model to be significant at \( \alpha = 0.0001 \), or the confidence level reaches 99.99%. From the above fitting result, we can conclude that the dynamic model is consistent with the experimental results. Additionally, the developed dynamics equation is proven to be applicable

\[
Q = 244.2 \cdot e^{-0.1715m}
\]

### 3.3 Verification of the water transport dynamics equation with previous scientific research

#### 3.3.1 Application of the developed dynamic equation to the results of Huang et al.

To further verify the validity and applicability of the dynamics equation developed in this study, the dynamics equations were applied to fit the results of previously reported research. Han et al. [14] fabricated an ultrathin layered GO membrane by filtering an extremely dilute solution of base-refluxed reduced GO (brGO) through a microporous substrate. The prepared ultrathin graphene nanofiltration membranes (uGNMs) showed well-packed layered structures. Figure 5 shows that the uGNMs that had the lowest loading of brGO (14.1 mg/m²) exhibited the highest water permeability (21.8 L/m²·h·bar). With increased brGO loading, the water flux decreased from 21.81 to 3.26 L/m²·h·bar. Figure 6 shows the results of the reported experimental results fit by the developed dynamics equations. The blue markers are the experimental values of the water flux, and the red curve is the simulated results. (The MATLAB calculation code is shown in Appendix B).

From the fitting result in Figure 6, the constant \( a \) is calculated to be 244.2, while the constant \( k \) is −0.1715. Therefore, the transfer kinetics equation of the water flux through the composite membrane is:

![Figure 5](image-url)
to the research results of Han et al.

**3.3.2 Application of the developed dynamic equation to the results of Huang et al.**

Huang et al. reported nanostrand-channeled graphene oxide ultrafiltration membranes, which contained a network of nanochannels with a narrow size distribution (3–5 nm) and exhibited superior separation performance [27]. The layered GO membrane was prepared by filtering a mixed solution of GO and copper hydroxide nanostrands (CHNs) through a PC membrane, which was followed by hydrazine reduction and CHN removal. The dynamics equation developed in this study were used to simulate the results of Huang et al. (Figure 7). Figure 8 shows the results of fitting the data of Huang et al. with the kinetics equation developed in this study. The blue markers are the experimental values for the water flux, and the red curve is the simulated results. (The MATLAB calculation code is shown in Appendix C).

According to the above fitting results, a is equal to 1015, and k is equal to –0.7434. The obtained transfer kinetics equation of the water flux through the composite membrane is:

\[
Q = 1015 \cdot e^{\frac{-0.7434m}{C0}}
\]

The obtained equation was analyzed by regression analysis, and the results are shown in Table 5 and Support Information Tables S3 and S4.

**Table 4.** The regression analysis of the experimental results of Han et al.

| Multiple R | R squared | Adjusted R squared | Standard error | Observation value |
|------------|-----------|--------------------|----------------|-------------------|
| 0.9760     | 0.9527    | 0.9369             | 2.1847         | 5                 |

**Figure 6.** Simulated results based on Han’s experiment results.

**Figure 7.** Thickness-dependent separation performance of the graphene oxide membranes.

**Figure 8.** Simulated results based on the experimental results of Huang et al.
4. Conclusions
A water transport kinetics model for layered GO membranes was developed, and the kinetic equation is in good agreement with our experimental results. At the same time, the results of previously reported layered GO membranes were fit with the kinetics equation developed by us. The fitting results were highly consistent, which showed that the dynamics equation developed in this study is widely applicable. This theoretical kinetic study provides insight into the application of graphene oxide membranes in water treatment.

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Disclosure statement
No potential conflict of interest was reported by the authors.

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Table 5. The regression analysis of the experimental results of Huang et al.

| Multiple R | R squared | Adjusted R squared | Standard error | Observation value |
|------------|-----------|--------------------|----------------|-------------------|
| 0.9933     | 0.9868    | 0.9841             | 35.9466        | 7                 |

For a degree of freedom of 5 and a significance level of 0.05, the correlation coefficient for the critical value of a test is 0.75449. In this study, the correlation coefficient for these experimental values is 0.9933 > 0.75449. It can be seen that the results of the regression analysis are significant.

For this example, \( p = 6.79E-06 < 0.0001 \). Therefore, the results can be considered at the \( \alpha = 0.0001 \) level of significance, or the confidence coefficient is 99.99%. The above fitting results show that the developed dynamics model is in good agreement with the experimental results. It can be seen that the developed dynamics equation is also applicable to the research results of Huang et al.

In summary, the transfer dynamics equation developed in this study for the water flux through a BPPO/GO composite membrane is in good agreement with the experimental results. The kinetics equation is also suitable for previously reported layered GO membranes.

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Appendix A

x = [0 13 25 39 52 65 78];
y = [550 284 112 44.89 15 4.1 3.14];
cftool

Appendix B

x = [14.2 16.72 21.3 28.3 33.8];
y = [22.36 12.5 5.06 4.46 3.3];
cftool

Appendix C

x = [0.1 0.289402 0.58663 1.180243 1.77847 2.391629 2.979258 3.56];
y = [830.3896 706.1038 446.0721 200.2775 162.6748 124.5869 80];
cftool