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Indicator of percolation transition in graphite oxide suspension containing cations

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Abstract−The percolation transition behavior occurs in the solid-liquid separation of graphite oxide (GO), which changes the system from suspension to colloid state and terminates the separation process. An indicator of percolation transition is necessary to help control the status of GO suspension to finish the solid-liquid separation process. The swell ratio, macroscopic appearance TEM, and rheological behavior of GO suspension were tested before and after the percolation transition occurred in a specific interval of K^+ concentration. Then, the physical properties of 1.00 g/L GO suspension containing 0.40-0.80 g/L K^+, including conductivity, thermal conductivity, viscosity, surface tension, and absorbance were characterized. The values of these physical properties showed a sharp change in the specific interval of K^+ concentration. We calculated the first and second slope of two adjacent points of each physical property to obtain a proper percolation transition indicator. The conductivity with the second slope up to 500% was the most significant change among these physical parameters, which can be used as an indicator of percolation transition in GO suspension. To verify the availability of the indicator, we explored the percolation transition behavior of Ca^{2+} and Al^{3+} in GO suspension and K^+ with different GO solid content, found that the conductivity is still the most significant percolation transition indicator. The indicator obtained in this paper is reliable under varying content of GO and types of cations in suspension, which can be used to determine the percolation transition threshold during the solid-liquid separation of GO suspension containing cations.

Keywords: Graphite Oxide, Solid-liquid Separation, Percolation Transition, Indicator Of Percolation Transition

INTRODUCTION

Graphite oxide (GO) is an intermediate product of graphene prepared by oxidation-reduction method, containing rich oxygen-containing functional groups (epoxide, hydroxyl, and carboxylic groups) on the surface and maintaining sp^2-bonded carbon structural framework as graphene [1]. The presence of oxygenated groups gives the material a hydrophilic character, providing a stable suspension in water, organic solvent, and polymer systems [2].

K^+, Na^+, Mn^{2+} are the main metallic impurities introduced into graphite oxide prepared by modified Hummers method [3]. Metallic impurities have a negative impact on the potential applications of GO-based materials [4]. With continuous washing with deionized water to remove impurity ions, the percolation transition of graphite oxide suspension containing metal ions occurs in a specific concentration range, so it is difficult to remove impurity ions through centrifugation operation [5]. Thus, a deep understanding of GO sheet-to-sheet interaction in chemical aqueous will contribute to the improvement of solid-liquid separation process [6].

The graphite oxide suspension is transformed from suspension to colloid at a specific concentration of metal ions, and the increase of layer spacing makes the metal ions insert between layers, which brings difficulties for solid-liquid separation. This sharp phase transition accompanied by the sudden appearance of long-range connectivity in the process of solid-liquid separation is called the percolation transition phenomenon. Percolation theory shows that the transformation of internal connectivity leads to a significant change of some physical properties in a disordered system [7]. Many studies have been done to determine the percolation transition threshold related to physical properties, such as electrical conductivity, thermal conductivity and surface tension. Lux [8] determined the volume fraction of conductive filler reaching the percolation transition threshold based on electrical conductivity. Li [9] studied that the initial state affected the formation of percolation conductive channels in unipolar switching elements. Chen [10] found that the formation of particle clusters in nanomaterials is reflected by a significant increase in thermal conductivity. The brittleness - toughness transition of rigid inorganic particle toughened polymer system is related to its critical substrate layer thickness, which can be characterized by surface tension [11]. The interaction between the particles in the suspension and the solvent molecules affects the absorbance, which can also be a criterion for the percolation transition of the system [12]. These results show that sudden change in physical property tends to indicate the percolation transition behavior of the system. Therefore, characterizing physical properties can be used to study the percolation transition phenomenon of the solid-liquid system.

The stability of aqueous GO suspension results from interactions between long-range electrostatic repulsion and weak dispersive attractive force [13]. Ionic strength in the suspension and volume fraction of GO will impact the interaction among sheets according to DLVO theory [14]. The minimum concentration of electrolyte in-

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ducing the aggregation is related to the type and valence of the ions. Four kinds of aggregation modes of GO under different aqueous chemistries were revealed: face-to-face, edge-to-edge, partial face-to-face, and partial edge-to-edge [15].

Rheology studies fluid flow and deformation; thus, the rheological behavior of suspension will also change with GO suspension microstructure. Previous studies have reported the rheological behavior related to the volume fraction of aqueous GO suspension. The concentrated aqueous GO suspension is a typical yield stress fluid, while dilute suspension indicates a Newtonian flow behavior [16]. High shear rate imposed on concentrated aqueous GO suspension will disintegrate clusters formed; however, clusters are rebuilt again after flow is arrested sufficiently [17]. The rheological characterization of GO-electrolyte reveals the relationship between flow behavior and GO sheets interaction in the suspension [18]. The strength of GO gels can be modulated by changing the valence, types, and concentration of cations added [19]. Therefore, it is necessary to investigate the rheological behavior of GO suspension before and after percolation transition.

This paper focused on how to indicate and predict the percolation transition threshold of GO suspension with different concentrations and types of metal ions. Through testing the macroscopic and microscopic morphology, rheological behavior was to determine if the percolation transition occurred in the specific concentration range of K^+. Then the physical properties such as conductivity, thermal conductivity, viscosity, surface tension and absorbance of 1.00 g/L GO suspension containing 0.40-0.80 g/L K^+ were characterized. We calculated the first and second slope of physical properties in each concentration interval of K^+ to determine the indicator of percolation transition threshold. Finally, the universal verification of the selected percolation transition indicator was conducted in 1.00 g/L GO suspension under the addition of Ca^{2+} and Al^{3+} as well as K^+ in 0.50 g/L and 2.00 g/L GO suspension. The obtained indicator will help to characterize the percolation transition in the solid-liquid separation process, which contributes to the further application of functionalized and purified GO.

MATERIAL AND METHOD

1. Material

GO aqueous suspension with a solid content of 10 g/L was provided by the Institute of Coal Chemical, Chinese Academy of Sciences. The GO suspension with concentration of 2.00 g/L, 1.00 g/L, and 0.50 g/L was obtained by diluting the raw GO suspension with deionized water. All the chemicals (KCl, $CaCl_2$ and $AlCl_3$) used in this experiment were analytical grade and purchased from Tianjin Yuxiang Technology Co., Ltd. The KCl, $CaCl_2$ and $AlCl_3$ reagents weighed by BS 224 S electronic balance (Sartorius Scientific Instrument Co., Ltd) were separately added to the diluted GO suspension to prepare samples containing various content and types of electrolytes.

2. Characterization

The electrical conductivity of GO-electrolyte was measured by Mettler Toledo Instrument, conductivity meter (model: S230). Measurements were performed using 50 mL of the suspension in a beaker to ensure full immersion of the probe. The thermal conductivity of GO-electrolyte suspension was measured based on the transient hot-wire technique using a DRE-2A thermal properties analyzer (Xiangtan Instrument Co., Ltd). All samples were kept in a temperature-controlled bath for enough time to reach the desired set temperature. The viscosity of suspension was measured by NDJ-1B rotation viscometer (Shanghai Changji Instrument Co., Ltd). Data were recorded until the viscosity reached a steady state. The surface tension was measured by QBZY series automatic surface tension meter (Shanghai Fangrui Instrument Co., Ltd). The absorbance of suspension was measured by Mapada UV-Visible spectrophotometer. All the above tests were performed at room temperature and ambient pressure. Each group of experiments was repeated three times and the average value was taken. We got the relationships of these parameters with the metal ions concentration, then calculated the slope change rate of the adjacent points. These values were used to judge the concentration range of the percolation transition.

The rheological behavior of GO-KCl suspension around percolation transition was measured using a stress-controlled TA Rheometer (DHR-2). A cone and plate geometry (diameter of 40 mm and truncation angle of 2°) with a truncation gap of 54 μm was used at 25 °C and ambient pressure for the following rheological measurements. Before each test, the sample was kept at rest for 10 min after loading, so that the fluid microstructure was allowed to rebuild properly. The rheological behavior of the suspension was analyzed by steady-state and oscillatory flow tests. Flow curves were obtained with shear rates ranging from 0.01 to 10 s^{-1}. The strain amplitude ranged from 0.01 to 100% at 1 Hz to determine the linear viscoelasticity region. We introduced small amplitude oscillatory sweep (SAOS) to explore GO-KCl suspension microstructure variation.

RESULTS AND DISCUSSION

1. Determine the Indicator of Percolation Transition Threshold

1-1. Phenomenon of Percolation Transition

The added cations compact the double electric layer, which changes the suspension behavior of GO sheets in water. According to our previous study, the swelling ratio of graphite oxide changed significantly before and after percolation transition in the solid-liquid separation process [5]. 20 mL raw material and GO suspension containing K^+ were centrifuged for 20 min under the condition of separation factor 1500 to observe the changing trend of swelling ratio, and the equation of swelling ratio is shown as follows:

$$\beta = \frac{V - V_2}{V - V_1} \tag{1}$$

where, V is the GO suspension raw material volume, V_1 and V_2 represent the supernatant volume obtained by centrifuging the raw material and after the corresponding operation, respectively.

In Fig. 1, the graphite oxide swelling ratio has a critical value with the concentration of K^+, and tends to be flat after 0.6 g/L. This may be because the addition of electrolyte makes the functional groups on the hydrophilic surface of GO gradually occupy, and the aggregation of sheets inhibits the swelling of GO suspension [20]. The suspension varies from colloid to suspension, and the TEM image in Fig. 2 shows the internal clusters increase rapidly and form a

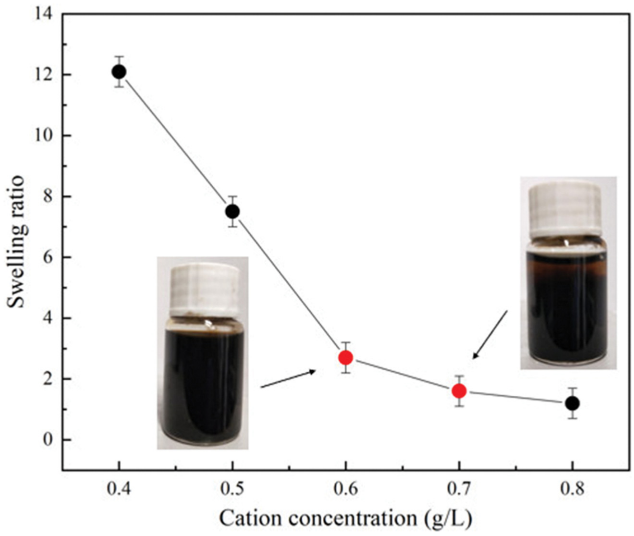

Fig. 1. Swelling ratio appearance of GO suspension in the specific concentration of K^+.

partially connected network at the 0.7 g/L. That means the percolation transition occurs in this concentration range, which is schematically demonstrated in Fig. 3. When the percolation transition occurs, the GO suspension status changes and the transform of the internal network structure of the suspension is macroscopic reflected by the sudden change of physical parameters [21,22].

The presented electrolytes impact the suspension status of GO and change the rheological behavior. We explored the microstructure of GO suspension around percolation transition threshold via characterizing the rheological behavior.

The flow behavior of aqueous GO suspensions with various concentration of KCl was investigated by flow sweep test, as shown in Fig. 4. The flow curves of stress versus shear rate indicate the GO suspension with KCl is a typical yield stress fluid, and shear stress is independent in low shear rate while increasing monotonically in the high shear rate region. High cation strength decreases the electrostatic repulsive forces between GO particles, which contributes to the aggregation of sheets [23]. The increase of aggregation degree leads to the formation of a more stable structure, which presents

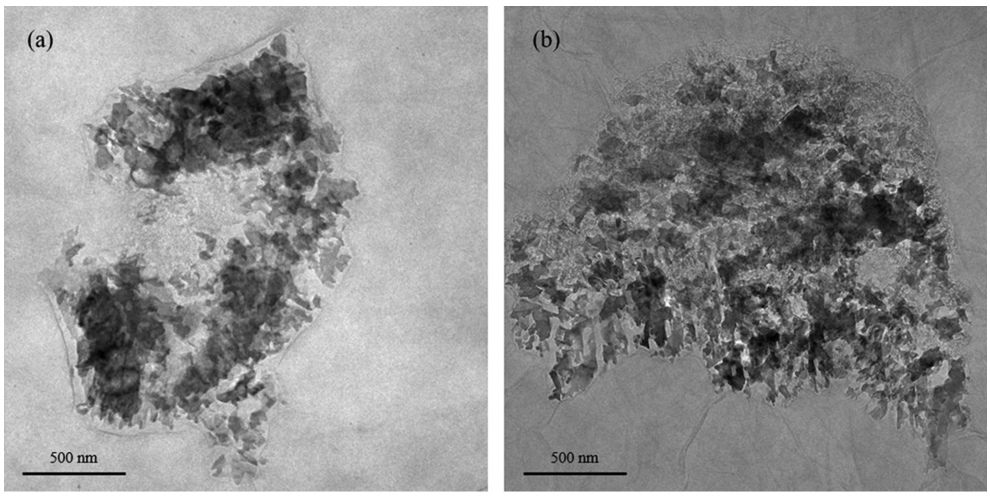

Fig. 2. TEM images of GO suspension before (a) and after (b) percolation transition.

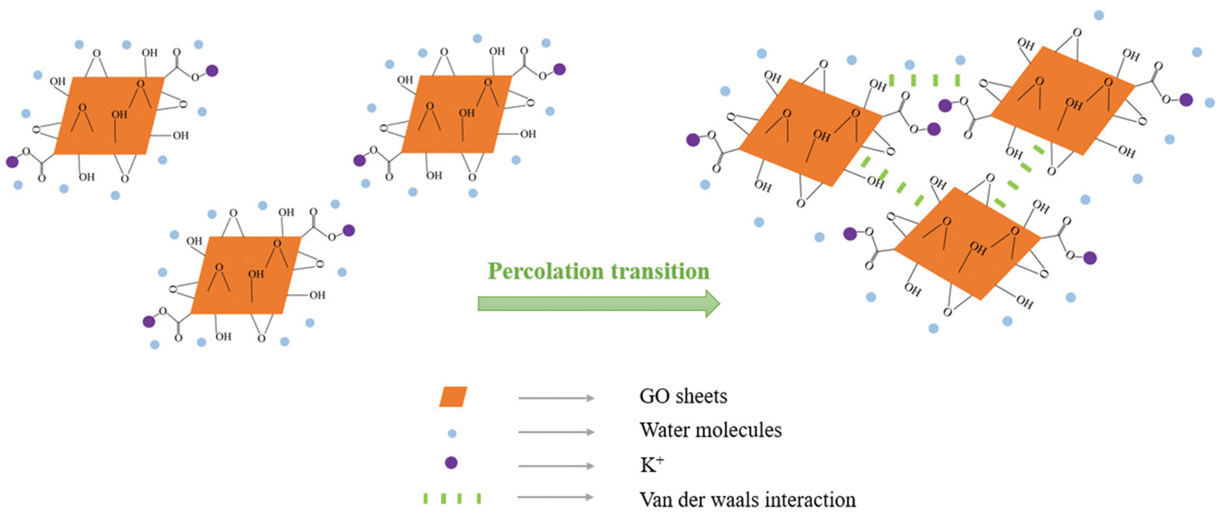

Fig. 3. Schematic drawing of percolation transition behavior in GO suspension containing K^+.

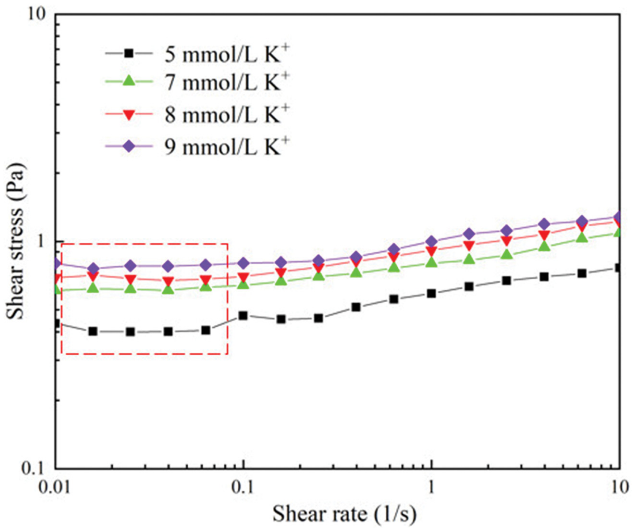

Fig. 4. Flow curves of stress of GO-KCl suspension around percolation transition threshold.

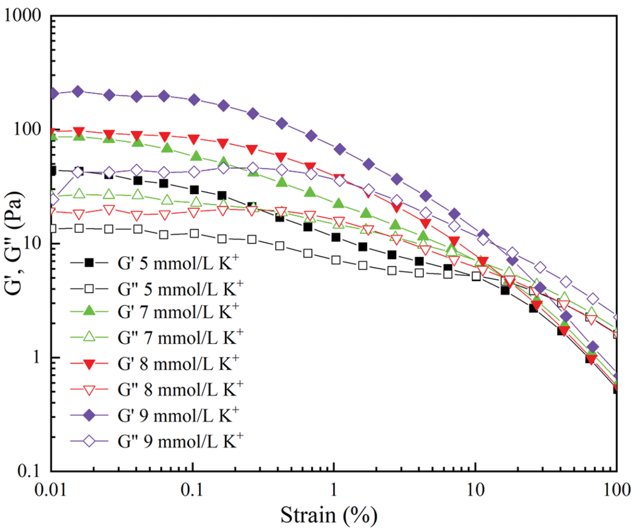

Fig. 5. G′ (filled legend) and G″ (unfilled legend) under oscillatory strain sweep of GO-KCl suspension around percolation transition threshold.

the increase of yield stress value. Additionally, metal ions increase pi-pi interaction, also responsible for the accelerated aggregation of GO particles [24].

For the GO suspension with different KCl content, evolution of storage modulus G′ and loss modulus G″ under strain sweep at 1 Hz are shown in Fig. 5. All suspensions show linear viscoelastic behavior under a certain strain depending on the KCl content. The G′ and G″ are independent of strain, defined as linear viscoelastic region (LVR) in the range of low oscillation strain. The applied strain in LVR is so small that the microstructure of GO suspension has not been broken properly. The "at rest" modulus (as the average of G′ within the LVR) differs with the loading of the concentration of GO suspension, and higher loading tends to have a larger modulus. Besides, large clusters are easily formed under high loading of

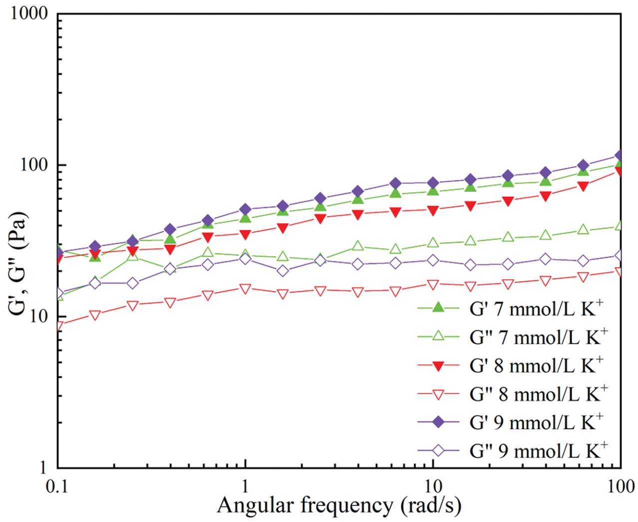

Fig. 6. G′ (filled legend) and G″ (unfilled legend) of GO-KCl suspension around percolation transition threshold at varying angular frequency.

electrolytes, thus with a larger critical strain in LVR. The storage modulus G′ is greater than the loss modulus G″ in the LVR, indicating that the suspensions still maintain solid-like behavior in the presence of electrolytes, which is similar to pure GO [25]. Discontinuous deformation that occurs to the internal structure weakens the interaction between the GO sheets which is responsible for the decrease of G′ and G″.

The small amplitude frequency sweep test results of aqueous GO suspensions with KCl at 7.0, 8.0, 9.0 mmol/L are shown in Fig. 6. We imposed a strain in the LVR to keep the suspension structure, and the experimental data demonstrate G′ is almost independent of angular frequency in the range of 0.1-100 rad/s. G′ is always larger than G″ in all samples, exhibiting solid-like characteristics in the region of tested angular frequency. The clusters formed under low concentration of GO still exhibited remarkable elastic behavior. Similar behavior in GO suspension has been reported by Vasu [17].

Rheological experimental results indicate that the addition of electrolyte increases the yield stress of GO-KCl suspension. Higher concentration of KCl in GO suspension tends to have a wider range of LVR, and all GO-KCl suspension under small amplitude frequency sweep test shows G′>G″ with applied angular frequency, indicating the signature of solid-like fluid.

1-2. Physical Properties of Suspension around Percolation Transition Threshold

Percolation transition existed in GO suspension containing 0.6-0.7 g/L K^+ during the process of solid-liquid separation, as shown in Fig. 1. The percolation transition significantly reduced the efficiency of solid-liquid separation. In this section, the GO suspension percolation transition behavior under the corresponding concentration of K^+ in Fig. 1 was revealed by monitoring the change of physical properties. The results of electrical conductivity, thermal conductivity, viscosity, surface tension, and absorbance of GO aqueous suspension with the various concentration of K^+ are shown in Fig. 7. The physical properties of suspension change linearly when

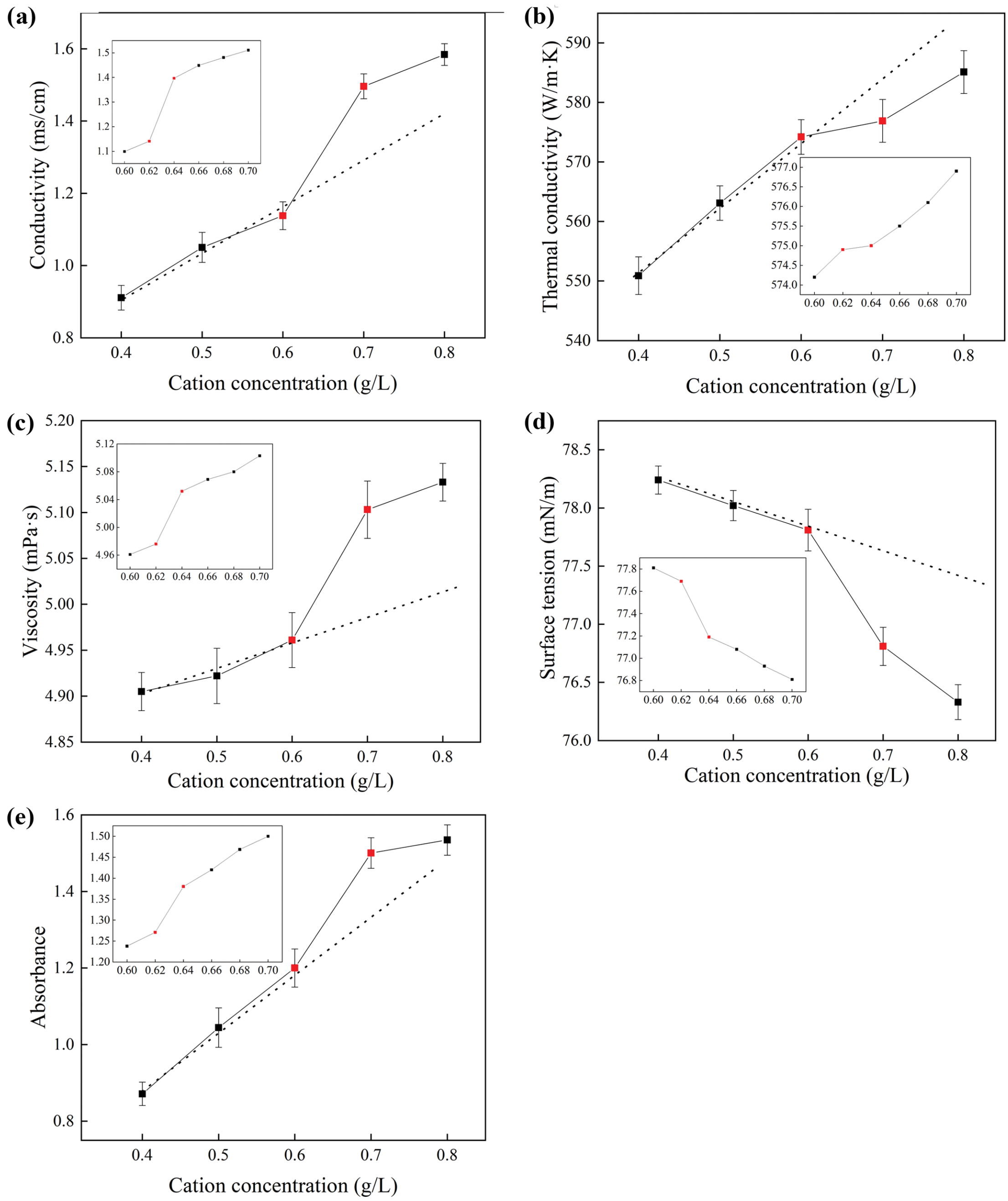

Fig. 7. Physical parameters of GO suspension with K^+ concentration, (a) conductivity, (b) thermal conductivity, (c) viscosity, (d) surface tension, (e) absorbance.

the concentration of K^+ is 0.40 to 0.60 g/L. The slope of physical properties differs from previous point when the concentration of K^+ is more than 0.70 g/L. The change trend of physical property indicates that the percolation transition occurs in 0.6-0.7 g/L (marked in red dotted lines), which is consistent with the corresponding interval in Fig. 1. Therefore, it can be considered that there are significant changes in physical properties before and after percolation transition. Furthermore, we conducted more detailed experiments by decreasing the increment of cation concentration between 0.60 and 0.70 g/L to determine a more accurate percolation transition threshold.

The five physical parameters have a sudden change when the concentration of K^+ is located in the range of 0.62-0.64 g/L in the zoom view of Fig. 7. The viscosity, conductivity and absorbance are positively correlated with the concentration of K^+, and the slope at this interval increases significantly; while surface tension is inversely related, and the slope at this interval reduces abruptly. The interaction between GO sheets and metal ions makes the physical parameters of the suspension change nonlinearly with the increase of electrolyte concentration. The added cations change the aggregation pattern of the GO sheets, which also affects the hydrogen bonding interaction between GO sheets and liquid phase [26].

The cations promote the aggregation behavior of the GO sheets, and the formation of particle clusters increases the flow resistance between the fluid layers, which is manifested by the increase of viscosity. The linear relationship of Lambert-Beer law is no longer applicable due to change of internal structure. The extension of cluster area and the decrease of total number of clusters lead to the decrease of thermal resistance, which greatly improves the thermal conductivity of the system and causes an abrupt change of the GO suspension. Meanwhile, the presence of long-range connectivity results in a sudden increase in conductivity [27,28]. Above all, we denoted the interval of physical properties sudden change as percolation transition threshold in GO suspension, corresponding to 8.322-8.591 mmol/L.

1-3. Indicator of Percolation Transition

Here we introduced the first and second slope of physical paraments in section 3.1.2 to select a proper indicator to illustrate the percolation transition behavior in GO-KCl suspension. We calculated the change rate of physical parameters in each incremental interval, denoted as "first slope". Similarly, we calculated the change rate of "first slope" of physical parameters in percolation transition interval, denoted as "second slope", to obtain the most obvious indicator of percolation transition.

The first and second slope of electrical conductivity, thermal conductivity, viscosity, surface tension and absorbance with the concentration of K^+ are listed in Fig. 8. To further analyze the changes of physical properties before and after percolation transition, we calculated the first and second slope of various physical properties of GO under the increment of 0.10 g/L K^+.

The second slope of absorbance is only 89.47%, which is not evident enough to represent percolation transition of GO suspension. While thermal conductivity, viscosity and surface tension show more obvious change in second slope of 340.74%, 264.10% and 257.14%, respectively. The second slope of electrical conductivity reaches 645.31%, which is the most obvious change trend compared to other parameters. The interaction between metal ions and GO sheets in water makes the conductivity of the suspension vary significantly [29]. Hence, we selected conductivity as the indicator to characterize the phenomenon of percolation transition of GO suspension. The percolation transition threshold of KCl in 1.00 g/L GO suspension is 0.60-0.70 g/L, corresponding to 8.322-8.591 mmol/L, which is consistent with the abrupt change interval of each physical property tested previously.

2. Universal Verification of the Indicator

2-1. Percolation Transition with Differ Cation Types in Suspension

The cation type and the GO concentration have great influence on the percolation transition process; thus, the indicator of percolation transition needs to be further verified in different suspensions. In this section, we investigated the percolation transition threshold with different solid content and suspension containing various types of cations.

Fig. 9(a) and Fig. 10(a) show the GO suspension physical properties vary with divalent and trivalent cations, Ca^{2+} and Al^{3+}, respectively. We marked red dotted lines to indicate the intervals of significant changes in physical properties in Fig. 9(a) and Fig. 10(a). Similarly, we calculated the first slope of each physical parameter under the increment of 0.10 g/L Ca^{2+} and 0.01 g/L Al^{3+} (in Fig. 9(b) and Fig. 10(b)). The electrical conductivity has the highest second slope up to 500% among other parameters in the interval marked by red vertical dotted lines, which can be selected as a significant indicator to characterize the percolation transition behavior of GO

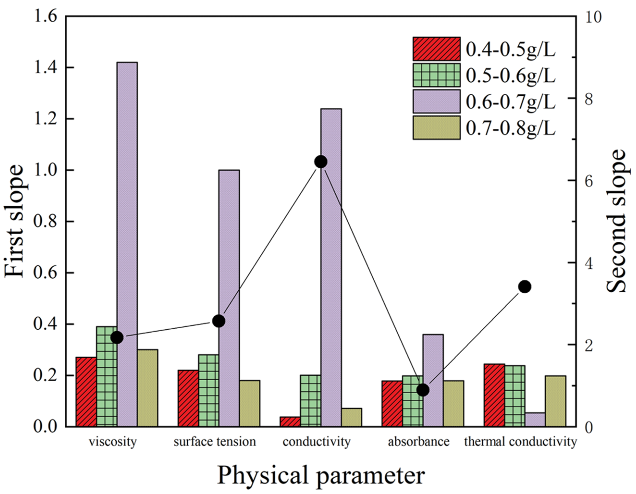

Fig. 8. First (bar symbol) and second (line symbol) slope of physical parameters at different K^+ concentration interval.

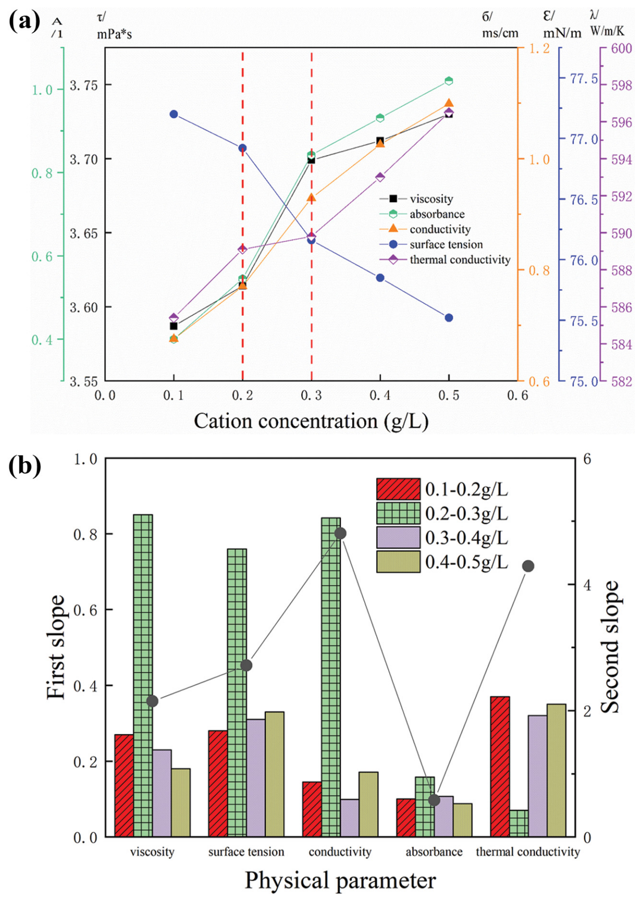

Fig. 9. (a)The physical parameters of GO with Ca^{2+} concentration, (b) first (bar symbol) and second (line symbol) slope of the physical parameters at different Ca^{2+} concentration interval.

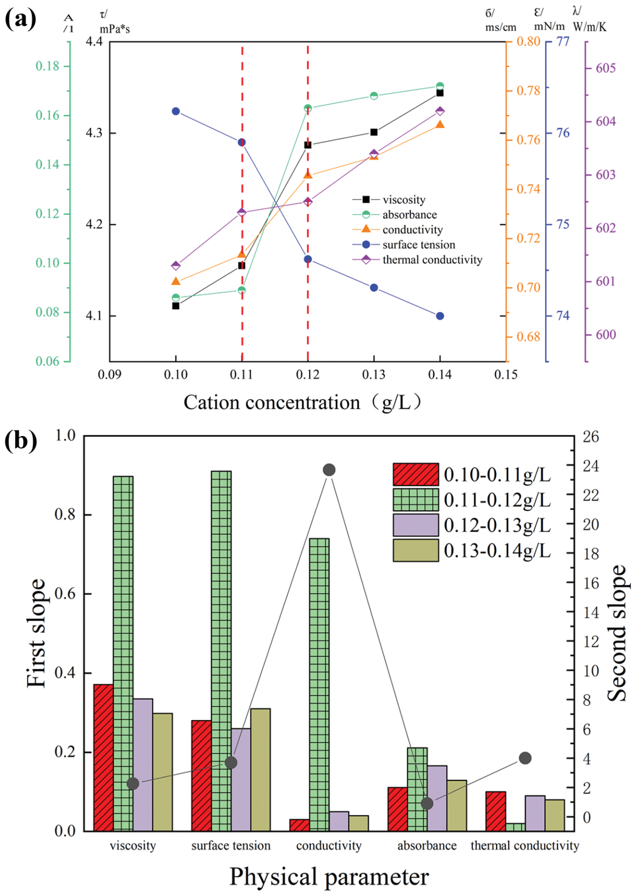

Fig. 10. (a) The physical parameters of GO suspension with Al^{3+} concentration, (b) first (bar symbol) and second (line symbol) slope of the physical parameter interval at different Al^{3+} concentration interval.

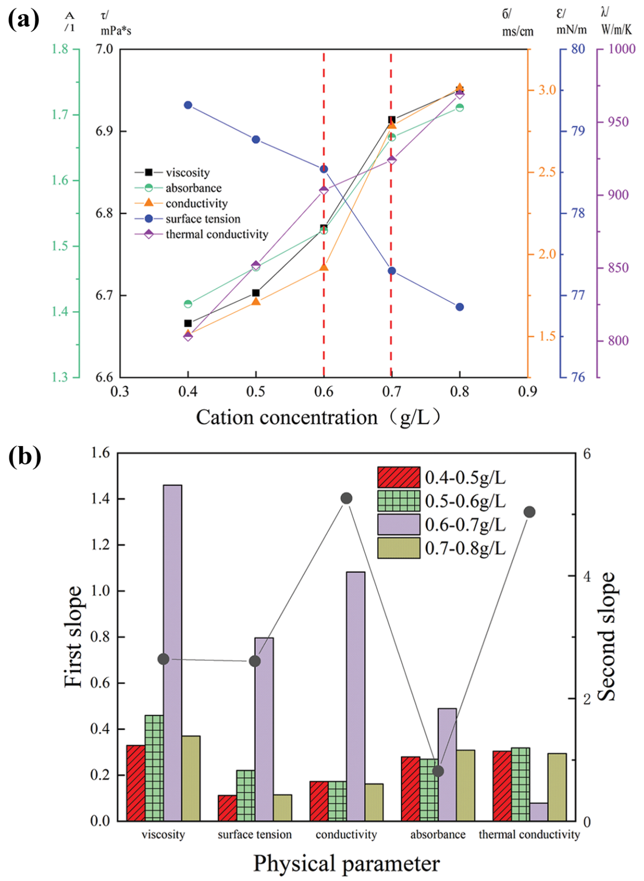

Fig. 11. (a) The physical parameters of 0.50 g/L GO suspension with K^+ concentration, (b) first (bar symbol) and second (line symbol) slope of the physical parameters at different K^+ concentration interval.

Table 1. Percolation transition concentration interval of K^+, Ca^{2+} and Al^{3+} in 1.00 g/L GO suspension

Cation types	Percolation transition interval (mmol/L)
K^+	8.322-8.591
Ca^{2+}	2.368-2.632
Al^{3+}	0.843-0.861

suspension. Thus, the percolation transition interval of K^+, Ca^{2+} and Al^{3+} in GO suspension is 0.60-0.70 g/L, 0.20-0.30 g/L and 0.11-0.12 g/L, respectively. The molar concentration corresponding to percolation transition threshold of K^+, Ca^{2+}, and Al^{3+} in 1.00 g/L GO suspension is listed in Table 1. Based on the Schulze-Hardly rule, high valent cations produce more aggressive charge screening effects and destabilizing capability. Thus, the percolation transition interval of GO containing cations followed the order of K^+>Ca^{2+}>Al^{3+}. The reduced concentration of percolation transition with the increase of cation valence suggested that energy-barrier repulsion is the main limitation for GO aggregation. Besides, the electronegativity of K^+, Ca^{2+}, and Al^{3+} and binding interaction with hydrophobic aromatic GO plane also resulted in the change of percolation transition interval [30].

2-2. Percolation Transition in Different Solid Content of Suspension

The physical properties of the suspension containing 2.00 g/L and 0.50 g/L GO with different concentration of K^+ are shown in Fig. 11(a) and Fig. 12(a). The interval marked by the red vertical dotted lines is the concentration of K^+ corresponding to percolation transition threshold. The first slope of each parameter with K^+ concentration is shown in Fig. 11(b) and Fig. 12(b). The first and second scope were calculated using the same method mentioned in section 3.1.3. As concluded in section 3.1.3, the conductivity of 0.50 g/L and 2.00 g/L GO suspension still has the highest second slope up to 500% in the interval of red dotted lines. The results show that the conductivity as percolation transition indicator was still effective in different concentrations of GO suspension.

CONCLUSION

Characterizing physical properties, including conductivity, thermal conductivity, viscosity, surface tension, and absorbance, can effectively reveal the percolation transition behavior in 1.00 g/L GO aqueous suspension. Rheological experiment results of steady-state shear test and frequency sweep demonstrate that cation loading has a significant impact on the linear or non-linear rheological behavior of GO suspension around percolation transition. The percola-

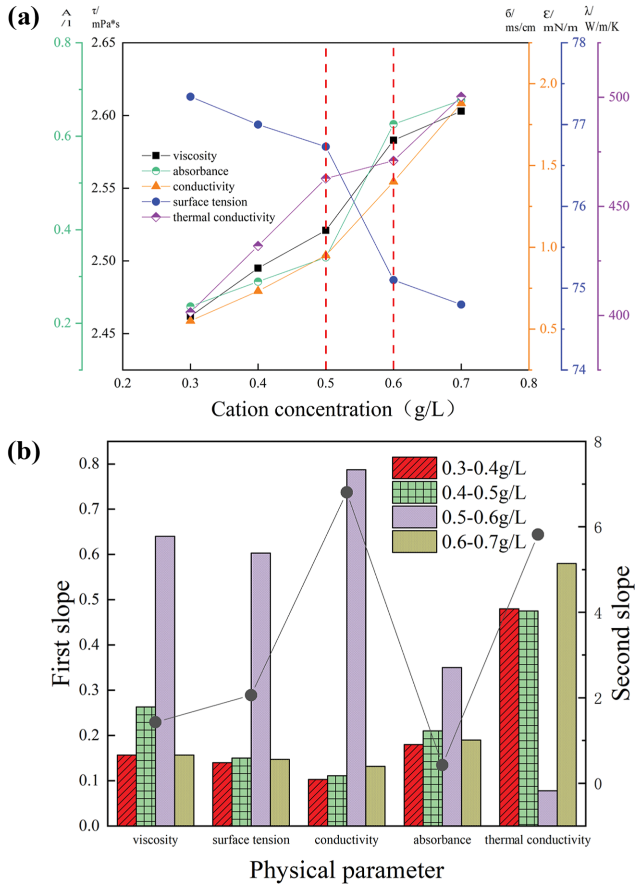

Fig. 12. (a) The physical parameters of 2.00 g/L GO suspension with K^+ concentration, (b) first (bar symbol) and second (line symbol) slope of the physical parameters at different K^+ concentration interval.

tion transition behavior indicator was selected by analyzing the physical property parameters, which can effectively indicate the percolation transition behavior Ca^{2+} and Al^{3+} in 1.00 g/L GO aqueous suspension and K^+ in 0.50 g/L and 2.00 g/L GO suspension. Solid-liquid separation is involved in the process of impurity removal and functional modification of GO suspension containing cations. Percolation transition of GO suspension at specific ion concentration has a negative effect on solid-liquid separation efficiency. In this case, traditional solid-liquid separation method is not suitable for GO suspension in percolation transition state, so other separation methods should be involved. The indicator selected in the study showed that conductivity changed most significantly with the second slope up to 500% in the interval of percolation transition, which can be used to choose the proper solid-liquid separation method for different state of GO suspension containing cations in the process of impurity removal and functional modification. In practical applications, we can determine whether percolation transition exists in certain ion concentration interval based on the indicator, further for choosing specific solid-liquid separation method. In addition, a general characterization indicator is provided in this study for the percolation transition of GO under varying content of GO and types of cations, avoiding repetitive work in the determination of multiple physical parameters. The indicator selected in this study can provide a basis for improving the solid-liquid separation process of GO suspension containing cations, thus further extending the application of GO.

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NOMENCLATURE

A	: absorbance of GO suspension
τ	: viscosity of GO suspension [$mPa \cdot s^{-1}$]
σ	: conductive of GO suspension [$mS \cdot cm^{-1}$]
ε	: surface tension of GO suspension [$mN \cdot m$]
λ	: thermal conductive of GO suspension [$W \cdot (m \cdot K)^{-1}$]

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