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Electrokinetic transport of nanoparticles in functional group modified nanopores

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ABSTRACT

Nanopore detection is a hot issue in current research. One of the challenges is how to slow down the transport velocity of nanoparticles in nanopores. In this paper, we propose a functional group modified nanopore. That means a polyelectrolyte brush layer is grafted on the surface of the nanopore to change the surface charge properties. The existing studies generally set the charge density of the brush layer to a fixed value. On the contrary, in this paper, we consider an essential property of the brush layer: the volume charge density is adjustable with pH. Thus, the charge property of the brush layer will change with the local H^+ concentration. Based on this, we established a mathematical model to study the transport of nanoparticles in polyelectrolyte brush layer modified nanopores. We found that pH can effectively adjust the charge density and even the polarity of the brush layer. A larger pH can reduce the transport velocity of nanoparticles and improve the blockade degree of ion current. The grafting density does not change the polarity of the brush charge. The larger the grafting density, the greater the charge density of the brush layer, and the blockade degree of ion current is also more obvious. The polyelectrolyte brush layer modified nanopores in this paper can effectively reduce the nanoparticle transport velocity and retain the essential ion current characteristics, such as ion current blockade and enhancement.

Organisms need continuous metabolic activities to maintain the regular operation of the body, which depends on the ion channel responsible for material exchange in the body's cells. Inspired by the phenomenon, various biological and bionic nanopores have been developed to meet different needs [1–5]. With the continuous development of nanotechnology, the research of nanopores includes the detection of metal ions [6–9], proteins [10–12], DNA [13–16] and other targets, ion rectification [17–22], and energy conversion [23–27], among which DNA sequencing is undoubtedly the most representative [28]. The basic principle of detection is that when the object passes through the nanopore, a detectable current signal is generated. Since the surface charges of biomolecules with different components is different, the blockade degree is different when the biomolecules passing through the nanopores, resulting in different current signals. Therefore, the shape, size and other parameters of the object can be detected [29]. However, how to reduce the transport velocity of nanoparticles in nanopores to improve the detection accuracy is still a challenge to be solved [30,31].

For overcoming the above problems, many attempts have been made to slow down the transport velocity of nanoparticles in nanopores, including embedding gate electrodes [32–37] and floating electrodes [38] outside nanopores, changing the temperature and viscosity of solution [39], applying additional forces, such as optical tweezers [40], nanopores modified by polyelectrolyte brush layer (PEBL) [30,31,41], *etc.* The research on the PEBL is a hotspot. The PEBL can change the surface properties of materials, so it is widely used in ion rectification [42–45], particle modification [46–49], material transport [50,51] and material detection [30,31,52,53]. Many researchers combine it with nanopore detection. That is, nanopores modified by the PEBL are used to detect nanoparticles. It can be known from the existing research that the nanopores modified by the PEBL can effectively slow down the transport velocity of nanoparticles [31]. Related research has also been realized in some experiments: the surface of nanopores is modified by the lipid layer to improve the detection accuracy of single molecules [54]. These all illustrate the possibility of functionalized nanopores in applications, and Cairns-Gibson *et al.* have further introduced the chemical and biological modification of functionalized

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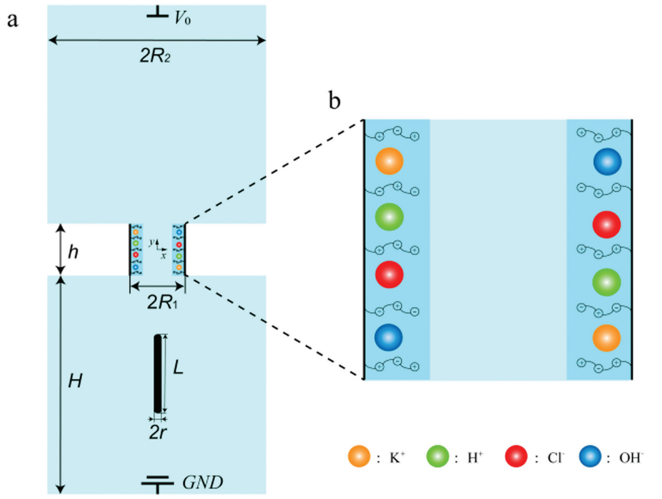

Fig. 1. (a) The diagram of electrokinetic transport of nanoparticles in functional group modified nanopores. (b) The diagram of inner nanopores.

nanopores [55]. This modification makes solid-state nanopores have physicochemical properties similar to biological nanopores. However, many existing studies set the charge density of the PEBL as a fixed value, ignoring an essential property of the PEBL: the charge property is adjustable with pH [30,31]. When nanoparticles move upward, they will inevitably change the ion concentration in the nanopores. Then leading to changes in charge density and even the polarity of the PEBL, which will affect the transport process of nanoparticles in the nanopores.

In this paper, we construct a continuum model coupled with the Navier-Stokes equations, the Poisson equation and Nernst-Planck equation. The motion of nanoparticles and fluid is described by the arbitrary Lagrangian-Eulerian (ALE) method, and study the transport process of nanoparticles in pH adjustable PEBL modified nanopores. The PNP equations have been verified to successfully describe the process of the particle pass through the nanopore [30,56,57]. Therefore, using this method to solve the mathematical model is reliable. Based on this mathematical model, we theoretically study the effects of pH, solution concentration and grafting density of the PEBL on the transport velocity of nanoparticles, ion current and total charge density of the PEBL. By accurately describing the process of nanoparticles passing through nanopores, the influence of the PEBL modified nanopores on the nanoparticle transport velocity and ion current can be directly reflected.

Fig. 1 describes the basic mathematical model of this paper: a nanopore with thickness h and radius R_1 connects two reservoirs with radius R_2 and height H. A cylindrical nanoparticle with radius r and length L is placed directly below the nanopore. Under the action of applied potential V_0, the nanoparticle will move upward through the nanopore. The electrolyte solution is filled with the nanopore and reservoirs. A rectangular coordinate system (x, y) is established at the center of the nanopore for the subsequent description of the nanoparticle position. A PEBL (such as lysine) composed of biomimetic zwitterionic functional groups with ion penetrability and pH adjustable is grafted on the surface of nanopores. The thickness of the PEBL is d (ranges from 3 nm to 5 nm), and the grafting density is σ (ranges from 0.1 chains/nm^2 to 0.6 chains/nm^2) [45,58].

The finite element simulation software COMSOL Multiphysics (5.0) is used to solve the above model, and the free triangle mesh is used to divide the model. The total number of units at the initial position of the nanoparticle is 19524, the number of units in the PEBL is 291, the number of mesh vertices on the particle surface is 98. The parameters used in the model are as follows: $\sigma_1 = -0.01$ C/m^2, $L = 10$ nm, $r = 1$ nm, $R_1 = 6$ nm, $h = 3$ nm, $R_2 = 50$ nm, $H = 100$ nm, $c_0 = 20$ mol/m^3, $V_0 = 0.5$ V, $F = 96490$ C/mol, $D_i = 9.31 \times 10^{-9}$, 1.96×10^{-9}, 2.03×10^{-9}, and 5.3×10^{-9} m^2/s ($i = 1, 2, 3, 4$), $T = 300$ K, $\varepsilon = 80$, $\mu = 10^{-3}$ Pa·s, $\rho = 10^3$ kg/m^3, $R = 8.31$ J/(K·mol). The parameters of the PEBL are as follows: $d = 3$ nm, $N = 20$, $\lambda = 1$ nm, $\sigma = 0.15$ chains/nm^{2,} p$K_c = -\log K_c = 2.2$ (α-carboxyl), p$K_n = -\log K_n = 8.8$ (α-amino) [45]. No matter where the nanoparticle is, it is always move in the same direction as the electric field and move to the central axis of the nanopore until it passes through the nanopore, it can be seen in Movies S1-S4 (Supporting information). Therefore, to decrease the calculation, the nanoparticle is placed directly below the nanopores. Thus, the initial position of the nanoparticles is (0, −50). The governing equations are provided in Supporting information.

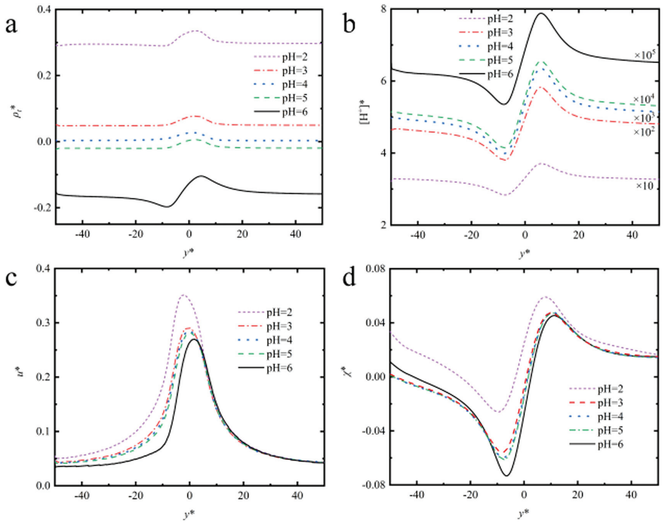

Fig. 2. At different pH, (a) the total charge density of the PEBL, (b) H$^+$ concentration, (c) the transport velocity and (d) ion current deviation vary with nanoparticle position. For comparison, multiply the H$^+$ concentration by the corresponding multiples. The solution concentration is 20 mol/m^3, the grafting density is 0.15 chains/nm^2.

Fig. 2 depicts the changes of total charge density, H$^+$ concentration, particle transport velocity and ion current deviation with nanoparticle position at different pH. The volume charge density of the PEBL is closely related to the H$^+$ concentration according to the Eq. S3 (Supporting information). With the increase or decrease of pH, the charge density of the PEBL will also change. It can be known from previous studies that the isoelectric point (IEP) of the PEBL is 5.5 [45,47]. It means that the protonation and deprotonation reactions in the PEBL reach a balance when the pH is 5.5, and the volume charge density of the PEBL is 0. At this time, the total charge density of the PEBL is equal to the charge density in the electrolyte solution. When the pH is less than 5.5, the PEBL shows a positive charge, and the charge density of the PEBL decreases with the increase of pH. When the pH is greater than 5.5, the PEBL shows a negative charge, and the charge density increases with the increase of pH [47]. It can be seen from Fig. 2a that when the nanoparticles are far away from the nanopores, the total charge density of the PEBL is hardly affected. When the nanoparticles move towards the nanopore, there is no doubt that the total charge density of the PEBL will change. This phenomenon is different from fixing the charge density of the PEBL to a certain fixed value [30,31,59].

When the nanoparticles continue to move until they are far away from the nanopore, the total charge density of the PEBL returns to the initial state. Since the negatively charged nanoparticles move up close to the nanopores, they will attract part of the cations in the PEBL, and the cation concentration inside the PEBL decreases (Fig. 2b). When the nanoparticles continue to move until they pass through the nanopores, due to the large number of cations carried by the nanoparticles, some of the cations is adsorbed by the PEBL, and the cation concentration in the PEBL is increases. When the nanoparticles leave the nanopores, the cations in the PEBL decrease and return to the initial equilibrium state. When the pH is 6, the H^+ concentration in the PEBL first decreases (the local pH increases), then increases (the local pH decreases) and finally remains stable. Eventually, this change is reflected in the process that the total charge density of the PEBL first increases, then decreases and finally maintains a stable value. When the pH is 5, the initial total charge density of the PEBL is negative. When the nanoparticles move towards the nanopores, as the H^+ concentration inside the PEBL changes, the polarity of the charge properties of the PEBL is reversed. The total charge density is negative and then positive, and a local maximum appears. When the pH is 3, 4 and 5, the total charge density of the PEBL does not decrease when the nanoparticles enter the nanopores. Since the total charge density is determined by the charge density of the PEBL and the solution. Combining with Figs. S2 and S3 (Supporting information), it shows that the charge density of the PEBL is very small. That means when the pH is 4 or 5, the charge density of the solution is dominant. When the pH is 3, the charge density of the PEBL first decreases and then increases, but the decrease of the charge density of the PEBL is less than the increase of the solution charge density in the PEBL. Therefore, there is a process in which the total charge density of the PEBL first increases and then decreases.

As shown in Fig. 2c, with the increase of pH, the maximum transport velocity gradually decreases, and the position where the maximum velocity is reached gradually backward. The position where the nanoparticle reaches the maximum velocity is always near the center of the nanopore. For example, when pH is 2, the position where the nanoparticle reaches the maximum velocity is below the nanopore center ($y^* = -2.06$). When pH is 6, the position where the nanoparticle reaches the maximum velocity is above the nanopore center ($y^* = 1.72$). With the increase of pH, the capture velocity of nanoparticles before entering the nanopore is also decreases, which is mainly related to the total charge density of the PEBL in the nanopore. As shown in Fig. 2a, when the pH is 4, the total charge density of the PEBL is approached to 0 when the nanoparticle is far away from the nanopore. When the pH is less than 4, the total charge density of the PEBL is positive, and when the pH is greater than 4, the total charge density of the PEBL is negative. Therefore, the nanoparticles move upward under the action of electrophoresis and generate electrostatic interaction with the PEBL. When the total charge density of the PEBL is positive, the electrostatic interaction with the particles is an attraction force. At the same time, an electroosmotic flow (EOF) is formed in the nanopores in the same direction as the nanoparticle movement, and the transport velocity of the nanoparticles increases. When the total charge density of PEBL is negative, the electrostatic interaction between particles and PEBL is a repulsive force, and an electroosmotic flow opposite to the direction of particle motion is formed in the nanopore, resulting in the transport velocity of particles before entering the nanopore in minimum under the same conditions. When the particle moves into the nanopore, the electrostatic interaction between the particle and PEBL mainly acts on both sides of the particle, so the repulsive force on the particle decreases and the particle transport speed increases. One point to note here is that when the pH is too high, under the conditions of this study, the pH is greater than 7, and the total charge density of the PEBL is negative and the value is large enough. The electrostatic repulsive force between the nanoparticles and the nanopore and the EOF in a contrary direction will cause nanoparticles to be trapped near the nanopore entrance. That means the nanoparticles cannot pass through the nanopore (Movie S5 in Supporting information).

It can be seen from Fig. 2d that when the nanoparticles enter the nanopores, ion current blockade occurs, and with the increase of pH, the degree of blockade increases. When the nanoparticles leave the nanopore, the ion current is increases. The smaller the pH is, the more pronounced the ion current increases. Before the nanoparticles enter the nanopores, the ion current deviations at pH 2 or 6 are significantly greater than other values. When the pH is 2 or 6, the total charge density carried by the PEBL is larger (Fig. 2a), and thus a larger electroosmotic flow is generated within the nanochannel, which promotes ion transport within the nanopore. The ion current in this paper is calculated by the ion flux (Eq. S8 in Supporting information), so the larger the ion flux, the larger the ion current is, the phenomenon occurs. When the nanoparticles enter the nanopore, they will occupy a part of the volume in the nanopore. Resulting in a reduction in the available volume for ion transmission since the volume inside the nanopore is limited, a decrease in ion current and a blockade phenomenon of ion current. Due to the nanoparticles being negatively charged, a large number of counterions will be adsorbed on the outside of the nanoparticle. When the nanoparticles move upward, these counter ions gradually accumulate in the lower part of the nanoparticles. When the nanoparticles pass through the nanopores, the large number of counterions carried at the bottom of the nanoparticles will increase the ion current. The increase of the cation concentration after the nanoparticles leave the nanopores in Fig. S4 (Supporting information) also reflects that the nanoparticles carry an enormous number of counterions, which causes the ion current to increase. Here, we compared the ionic currents with PEBL and without PEBL under the same conditions. For example, in the case of pH 6, the minimum ionic current deviations were −0.0734 and −0.0567, respectively, and the degree of current blockade increased by 29.45%. When the pH is 2, the minimum values of the ionic current deviations were −0.026 and −0.0449, respectively, and the degree of current blockage is reduced by 42.09%.

This part shows that the larger the pH is, the smaller the nanoparticle transport velocity is, and the ion current blockade is relatively large. That means the ion current signal is relatively strong. Therefore, by controlling the pH of the background salt solution, the transport velocity of the nanoparticle passing through the nanopore is slowed down, and the ion current blockade is increased, making the electrical signal more obvious.

Fig. 3 depicts the total charge density of the PEBL, H^+ concentration, the nanoparticle transport velocity and ion current deviation vary with nanoparticle position under different background salt concentrations. As shown in Fig. 3a, the total charge density of the PEBL increases as the nanoparticles move toward the nanopores. When the nanoparticles move and pass through the nanopores, the total charge density of the PEBL first decreases and then increases, and finally decrease to a stable value. The total charge density is increased with the increase of the background salt concentration, and the greater the difference between the maximum and minimum total charge density (the difference is 0.083, 0.093, 0.094, respectively). The negatively charged PEBL will absorb a large number of cations. When the concentration of the solution is increased, due to the existence of the counterions elimination effect, the H^+ concentration in the PEBL is decreases [31,46], as shown in Fig. 3b. This is the reason why the increase of solution concentration will lead to the decrease of H^+ concentration in the PEBL at the same pH. It can be seen from Fig. S5 (Supporting information) that the smaller the solution concentration is, the more obvious the K^+ concentration changes. When the nanoparticles approach the nanopores, the cations in the PEBL is adsorbed on the surface of the nanoparticles. The smaller the

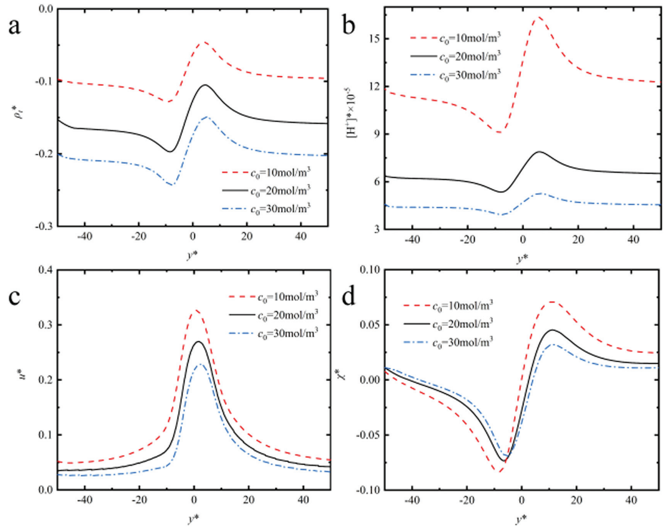

Fig. 3. Under different background salt concentrations, (a) the total charge density of the PEBL and (b) H^+ concentration, (c) the transport velocity and (d) ion current deviation vary with nanoparticle position. The pH is 6, and the grafting density is 0.15 chains/nm^2.

concentration of the solution, the more cations are required for the PEBL to adsorb ions to reach the saturation state. When the nanoparticles pass through the nanopores, a large number of counterions carried by the nanoparticles will enter the PEBL. Eventually appear as the cation concentration in the PEBL first decreases, then increases, and decreases until it maintains a stable value.

As shown in Fig. 3c that when the background salt concentration increases, the capture velocity and the maximum velocity both decrease. There are two main reasons for this. First, since the surface charge density of the nanoparticle is set as a constant, with the increase of background salt concentration, the electrophoretic force acting on the nanoparticle is decreases [60]. Secondly, when the pH is 6, the total charge density of the PEBL in the nanopore is negative. The total charge density is increased with the increase of the concentration of solution, as shown in Fig. 3a. Therefore, the electrostatic repulsive force between the nanoparticles and the nanopores also increases, which decreases the transport velocity of the nanoparticles.

It can be seen from Fig. 3d that when the solution concentration is increased, the current blockade and the degree of current enhancement when the nanoparticles pass through the nanopores are reduced, and the location of the current blockade is also different ($y^* = -8.3685$, -6.4559 and -5.5265). Combining Figs. 3b and S5, because the nanoparticles are negatively charged, they will attract the positive charges (H^+, K^+) in the PEBL when they approach the nanopore, leading to a reduction of cation concentration in the PEBL. The smaller the solution concentration, the greater the fluctuation of ion concentration. The degree of the current blockade caused by the elimination effect generated by the volume of the nanoparticles can be regarded as the same. Therefore, the greater the fluctuation of the ion concentration, the greater the impact on the ion current, and produce a more significant current signal. The different degree of current blockade is caused by the concentration of background salt solution. The electroosmotic flow intensity in nanopores decreases with the increase of background salt concentration [30], the ion flux in the nanopore decreases. The ion current in this paper is calculated by the ion flux through the nanopore, so the degree of current blockade is reduced. Therefore, the total charge density of the PEBL can be changed by controlling the concentration of the background salt solution, thereby achieving the purpose of controlling the nanoparticle transport velocity and the ion current.

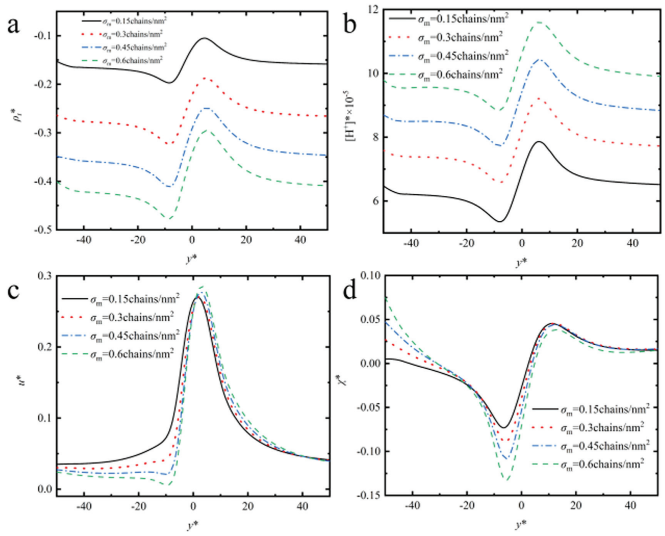

Fig. 4. Under different grafting densities, (a) the total charge density of the PEBL, (b) H^+ concentration, (c) the transport velocity and (d) ion current deviation vary with the position of the nanoparticles. The pH is 6, and the solution concentration is 20 mol/m^3.

Fig. 4 describes the total charge density of the PEBL, H^+ concentration, the nanoparticle transport velocity and ion current deviation with the position of the nanoparticles under different grafting densities. Combined with Eq. S3, it can be known that the grafting density can directly affect the charge density of the PEBL. The greater the grafting density, the greater the charge density of the PEBL [45]. It can be seen from Fig. 4a that when the pH is 6, the total charge density of the PEBL is negative, and the greater the grafting density, the greater the total charge density. As the nanoparticles move upward through the nanopore, the total charge density of the PEBL first increases, and then decreases to a stable value. The total charge density of PEBL increases with the increase of grafting density. It can be seen from Fig. 4b and Fig. S6 (Supporting information) that as the grafting density increases, the changes in the cation concentration in the PEBL increase. Since the total charge density of the PEBL increases, the counterions that can be adsorbed is also increase, which means more H^+ and K^+ ions can be adsorbed [44]. The drastic change of H^+ ions in Fig. 4b is also explaining the change of the total charge density of the PEBL.

As shown in Fig. 4c that the greater the grafting density is, the smaller the capture velocity of the nanoparticles before entering the nanopore, and the higher the velocity when passing through the nanopore. When the grafting density is 0.45 or 0.6, the nanoparticle capture velocity decreases. Since the charge density of the PEBL is negative, and the greater the grafting density, the greater the charge density. Therefore, as the nanoparticles approach the nanopores, the electrostatic repulsive force between the nanoparticles and the nanopores increases, leading to a decrease in the transport velocity of the nanoparticles before entering the nanopores. When the particles continued to move into the nanopore, the electrostatic interaction between the particles and PEBL mainly acted on both sides of the particles, the repulsive force received by the particles decreased, and the velocity increased. The higher the charge density of the PEBL, the stronger the electroosmotic flow in the nanopore and the greater the hindering effect on particle transport. In the stage where y is less than 0, the larger the graft density, the smaller the transport velocity at the same position. When the particles pass through the nanopore, due to the existence of this electrostatic repulsive force, the greater the graft density, the greater the transport velocity of the particles at the same position, until the particles are far away from the nanopore.

It can be seen from Fig. 4d that the greater the grafting density, the more significant the change of ion current before the nanoparticles enter the nanopore, and the greater the current blockade generated. Due to the greater the grafting density, the stronger the electroosmotic flow generated in the nanopores, and result in a larger ion current is generated. When the nanoparticles enter the nanopores, the current blockade occurs due to the volume of the nanoparticles. The greater the grafting density of the PEBL, the greater the current blockade, and the smaller the ion current enhancement. This phenomenon is also similar to the existing research [52].

In this paper, a continuum model of the PEBL modified nanopores is constructed, and the electrokinetic transport of nanoparticles in PEBL modified nanopores is studied theoretically. The results reflect that the total charge density of the PEBL will change with the movement of the nanoparticles, and the pH can effectively adjust the polarity of the charge of the PEBL and slow down the transport velocity of the nanoparticles in the nanopores. When the pH is large enough, because the charge density of the PEBL is negative and the value is large enough, the nanoparticles cannot enter the nanopores. It means that they are captured at the entrance of the nanopores. The solution concentration and the grafting density will change the charge density of the PEBL without changing the polarity. Increasing the solution concentration or decreasing the grafting density can effectively slow down the transport velocity of nanoparticles in the nanopores. All these mean that pH adjustable PEBL modified nanopores can effectively adjust the nanoparticle transport velocity without changing the basic law of ion current. This provides a new theoretical explanation for slowing down the transport of nanoparticles in nanopores, and provides some theoretical guidance for the construction of PEBL modified nanopores and the design of single-molecule sensing devices. Functionalized nanopores also expand the scope of nanopore applications, from a sensing tool to a platform for biophysical research.

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.

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Supplementary materials

The mathematical model and governing equations, the code verification, the relationship between the volume charge density and the space charge density of the PEBL and the position of the nanoparticle with different pH; the changes of K^+ in the PEBL is described; the schematic process of particles passing through nanopores under different conditions are available in the online version of this article. Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2022.07.010.

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