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Controlling the concentration of gold nanorods during their dielectrophoresis-assisted deposition

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Abstract

Gold nanorods (AuNRs) have attracted great interest due to their excellent plasmonic properties which makes them a promising candidate for many applications. However, most of the applications require control over the position and concentration of nanorods (NRs) by processes that are fast, reliable and scalable. The focus of this work is to study the effects of variation of various parameters, such as applied voltage, frequency, solvents and drying time on the concentration of dielectrophoresis (DEP) -assisted deposition of AuNRs. We have seen that the concentration of AuNRs within the electrodes can be considerably increased by increasing the applied voltage and deposition time, and by choosing a more volatile solvent. Furthermore, the applied frequency also strongly influences the deposition of AuNRs. At lower frequencies, the electro-osmotic flows drag AuNRs to the top of electrodes, while at higher frequencies NRs are deposited close to the electrode edge where the electric field and the field gradient are maximum. We have also carried out simulations using the finite element method to generate the electric field patterns for coplanar electrodes. Our results provide insight into ways in which the concentration of AuNRs can be controlled during DEP-assisted deposition.

1. Introduction

Optical properties of anisotropic AuNRs make them interesting for numerous applications such as optoelectronics devices, sensing, single-molecule detection and therapeutics [1–4]. Especially, the enhanced electrical and optical properties originating from the precisely aligned NRs assemblies have been demonstrated for electronics, sensing, and photonic applications [5–7]. Most of the applications of NRs require control over their concentration, position, orientation and inter-particle separation on the substrate. For this assembly, tools are needed that should be rapid, scalable and compatible with the existing technology. The assembly of one-dimensional rod-shaped arrays has been demonstrated using polymeric linked colloidal NPs [8], lithographical channels [9] and templates [10], which are limited by yield, resolution and substrate complexity. Hierarchical electric field assisted deposition, termed as DEP is a concept put forward in 1951 by Herbert A. Pohl [11], which is now used to manipulate position and orientation of DNA [12, 13], polymeric structures [14], viruses [15], bacteria [16] and particles both on micro and nano-scale [17–19].

Considerable progress has been made in DEP of NPs during the last two decades due to development in technology enabling electrodes fabrication with gap size within micron and sub-micron range [20]. Various researchers have manipulated voltage, gap size and solvent effects to trap particles by the electric field [21–23]. Moreover, the frequency dependence of DEP, which gives rise to other processes such as electro-osmotic and electro-thermal flows, has been shown to affect the deposition of NPs between the electrodes [24–26]. Such advances in DEP resulted in the successful bridging of NPs in the electrodes gap [27–29].
Herein, we report a comprehensive study of the effect of various parameters on the concentration of AuNRs, deposited within the coplanar electrodes by DEP. Our results provide the basis for controlling the concentration of AuNRs within the electrode gap. Various experimental parameters, such as voltage, frequency, solvents and drying time have been varied to see their effect on the NRs deposition. Increasing voltage considerably extends the critical field and dielectrophoretic force (DEPF) above and fills the electrode gap, which in turn increases the concentration of AuNRs in the electrode gap. The influence of frequency on the deposition of AuNRs has been carried out. Drying time significantly increases the concentration of AuNRs towards the electrodes. Alternatively, the enhanced concentration of AuNRs towards the electrodes can also be achieved by using a more volatile solvent, such as ethanol. We have also carried out a detailed simulation study of the electric field and dielectrophoretic force (DEPF) profile generated from the coplanar electrodes, which support our experimental observations.

2. Experimental

2.1. Synthesis of AuNRs
The synthesis of AuNRs was carried out by modifying a three-stepped seeded-protocol developed by Murphy et al.\textsuperscript{[30]}; see figure 1 (a) for a schematic illustration of the synthesis process. In order to synthesize seeds, 25 $\mu$l (0.1 M) HAuCl\textsubscript{4} (Sigma-Aldrich) was introduced into the aqueous solution of 10 ml of 0.1 M CTAB (Calbiochem). Then, 450 $\mu$l of 0.02 M ice-cold NaBH\textsubscript{4} (Fluka Analytical) was added to the solution, resulting in brownish color. The solution was then left undisturbed at 25 °C for 2 h to decompose the excess NaBH\textsubscript{4}. The growth solution was prepared by adding 136 $\mu$l of 0.1 M HAuCl\textsubscript{4} to 53 ml of 0.1 M CTAB solution. Afterward, 18 $\mu$l of HNO\textsubscript{3} (63%, EMSURE) and 300 $\mu$l of 0.1 M ascorbic acid (Sigma) was added to the mixture. The addition of ascorbic acid to the growth solution changed the color of the solution from yellowish to colorless, indicating the partial reduction of Au(III) ions. Three vials were labeled as A, B, and C; 4.5 ml of the growth solution was added to A and B, and the remaining solution was added to the vial C. 200 $\mu$l of seed solution was added to vial A under vigorous stirring. After 8–10 s, 200 $\mu$l of solution from vial A was added to vial B, which was shaken for 8–10 s. Finally, 3.75 ml of solution from B was transferred to C. The solution was mixed briefly and then left undisturbed overnight at 30 °C. The top solution from vial C was removed carefully, leaving brownish precipitates at the bottom. These precipitates were re-dispersed in 15 ml DI water and centrifuged.
twice at 6000 rpm for 10 min in order to remove the excess CTAB molecules and supernatants. The resultant solution was again re-dispersed in an equal amount of DI water.

Before DEP experiments, characterization tests for the synthesized AuNRs were performed. Figures (1(b), (c)) depicts the scanning electron microscopy (SEM) image of AuNRs and their optical absorption spectra, respectively. The average length of NRs is about 1.1 μm and the average width is 50 nm. The optical spectra of AuNRs show a peak at around 510 nm, which is the transverse plasmon peak of NRs. The rise in the absorption after that indicates the presence of a second plasmon peak, which is the longitudinal plasmon peak of NRs. Due to the high aspect ratio of NRs, the longitudinal peak lies at a longer wavelength, beyond the measuring range of our spectrometer.

2.2. Fabrication of electrodes and deposition experiments

Shadow mask technique was used to fabricate two coplanar Au-electrodes on a glass substrate (see figure 2). The electrode gap was defined by sticking a 50 μm thick copper wire in the middle of the glass substrate, and thermal evaporation was used to deposit 15 nm of Cr (to provide better adhesion) and, subsequently, 100 nm of the Au on the glass substrate. After the evaporation process, removing the copper wire from the substrate gives the final geometry of the electrodes, the resultant electrode gap of our device was 50 μm, whereas the length of the electrode was about 1.5 cm.

Agilent 33220 A function generator was used to apply the AC potential where the corresponding potential and frequency across the electrodes, which were monitored by an Agilent DSO 3062 A oscilloscope. In order to perform the deposition experiments, we put a colloidal droplet of 40 μl on the substrate in the presence of an applied voltage, covering both the electrodes. After 5 min the droplet was carefully removed and afterward the voltage was turned off.

2.3. Simulations

The finite element method (FEM) was used to simulate the electric field patterns between the electrodes. The length of the electrode is much larger than its thickness and the electrode gap, therefore, the electric field can be assumed to be invariant along the length (z-axis) of the electrodes. It varies only along the electrode gap (x-axis) and perpendicular to the substrate (y-axis). Therefore, we carried out 2D simulation only along the latter two axes. The mesh size ‘x’ used for our simulation is in the range 0.0025 < x < 2 μm with the maximum element growth rate of 1.2 and a resolution of 2.

Furthermore, we calculated the Re(CMF) and fluid flow velocities as a function of AC frequency using MATLAB software. Table 1 depicts the values of various parameters used in the simulation.
where, $\delta$ is the depolarization factors along long and short axes respectively, $\varepsilon_{p,m}$ is the permittivity of the liquid medium surrounding the particle, $E$ is the electric field strength, and $K_\delta$ is the CMF given by,

$$K_\delta = \left(\frac{\varepsilon'_p - \varepsilon'_m}{(\varepsilon'_p - \varepsilon'_m)A_{major} + \varepsilon'_m}\right)$$

(2)

here, $\delta = \text{major, minor axes}$. Also, the complex primitives of the particle and medium are given by $\varepsilon'_p = \varepsilon_{p,m} + \sigma_{p,m}/i\omega$, where, $\varepsilon_{p,m}$ and $\sigma_{p,m}$ are the permittivity and conductivity of the particle and suspended medium, respectively, $\omega$ represents the frequency of the applied electric field, and, $A_\delta$ designates the depolarization factors along long ($A_{major}$) and short ($A_{minor}$) axes, respectively given by,

$$A_{major} = \frac{1 - e^2}{2e^2} \left(\ln\left(\frac{1 + e}{1 - e}\right) - 2e\right)$$

(3)

and

$$A_{minor} = \frac{(1 - A_{major})}{2}$$

(4)

where,

$$e^2 = 1 - \frac{b^2}{a^2}$$

(5)

As evident from equation (1), DEP for ellipsoidal particle depends on the gradient of the electric field strength, the volume of the particle and the real part of the frequency-dependent CMF, $Re(CMF)$, which also determines the direction of the DEP. When the $Re(CMF)$ is positive, NRs are attracted to the higher field regions, while the negative value of the $Re(CMF)$ leads to the repulsion of NRs from the higher field region.

Figure 3, depicts the variation of $Re(CMF)$ with frequency for AuNRs. For AuNRs used for this study, depolarization factors of $A_\delta = 0.00395$ and $A_\delta = 0.4980$ are calculated by using equations (3) and (4). This yields the value of about 253 for $Re(CMF)$ up to $10^{14}$ Hz (see figure 3(a)) when the long-axis is aligned parallel to the electric field. At frequencies higher than $10^{14}$ Hz, the value of $Re(CMF)$ starts decreasing, crosses over to the negative region and then becomes stable at $-0.9$. For AuNRs, weak negative DEP is only observed at frequencies higher than $10^{17}$ Hz, due to the much higher conductivity of Au compared to water. For the short-axis, the maximum value of $Re(CMF)$ is only 2 (See figure 3(b)), which means that the DEP is much weaker when the short-axis is oriented along the electric field. As the switch-over in both cases is at much higher frequencies, we can assume that for AuNRs dispersed in water would experience positive DEP at all practical experimental frequencies.

3.2. Brownian motions and buoyancy force

In solution, DEP induced motion encounters antagonistic Brownian motion, which becomes more prominent when the particle size decreases down to submicron or nano-scale. The strength of the electric field required to overcome the Brownian motion can be approximated by comparing the electrostatic energy with the thermal energy [32]. The critical field, $E_{critical}$ needed to overcome the Brownian motion is given by the following equation.
where $K_B$ is the Boltzmann constant and $T$ is the temperature. Using the dimension of AuNRs and the corresponding CMF, the critical field strength is $1.42 \times 10^4 \text{ V m}^{-1}$ and $1.59 \times 10^5 \text{ V m}^{-1}$, when the long and short axes of NRs are aligned along the field direction, respectively.

Another important factor dictating the motion of nanorods is the Buoyancy force. For prolate ellipsoids, its value is given by the following equation \[ F_{\text{buoyancy}} = \frac{(4/3)\pi ab^2}{\rho_p} (\rho_p - \rho_m) g \]

where $\rho_m$ and $\rho_p$ is the density of medium and particle, $g$ is acceleration due to gravity. For AuNRs used in this work, the buoyancy force has a magnitude of $2.34 \times 10^{-15} \text{ N}$.

3.3. Electric field and DEPF pattern

Figure 4(a) depicts the electric field pattern (in xy plane) between the electrodes having a gap of 50 $\mu\text{m}$, generated for an applied potential of 5 V. As evident from the figure, there is strong confinement of the electric field close to the electrode tips. This is expected as the field lines converge at the tip of the electrode. There is also a very strong field gradient close to the electrode tips. As we move away from the tip, in any direction, the field strength decreases sharply. A strong field gradient close to the electrode tip means that NRs will be attracted to the electrode tip due to the DEPF. Figure 4(b) depicts the line plot of the fields within the electrode gap, 3 nm above the substrate surface. The plot shows that the maximum field strength is about $2.4 \times 10^6 \text{ V m}^{-1}$ close to the electrode tip and decays within the electrode gap. However, there is still a minimum field strength of about $6 \times 10^4 \text{ V m}^{-1}$ in the middle of the electrode gap close to the substrate. These field strengths are significantly larger than the critical field strength along the long axis of NRs. However, as we move perpendicular to the substrate along the y-axis, the field strength decreases sharply. In order to see the extension of the region having electric field strength greater than the critical field strength, we repeated the simulation by choosing the maxima.
of the contour scale, which is the maximum range of the color bar, equal to the critical field along the long-axis \((1.42 \times 10^4 \text{ Vm}^{-1})\). Therefore, the field strength in the region having a dark red color is greater than the critical field strength for NRs oriented along the electric field direction; the region is subsequently referred to as critical field region. The region has a semicircular profile having a radius of approximately 110 \(\mu\text{m}\), for an applied voltage of 5 V (see figure 4(e)).

Figure 4(c) depicts the DEPF pattern in the xy plane, experienced by AuNRs aligned along the field direction. The force is strongest close to the electrodes, where the electric field and field gradients are strong. The line plot of the normalized force strength between the electrodes, 3 nm above the substrate surface, is shown in figure 4(d). As evidence from the plot, in the middle of the electrode gap, the DEPF decreases sharply, which is due to the fact that here NRs are being attracted by both electrodes. However, the magnitude of the DEPF increases away from the center of the electrode gap. Close to the electrode tips, AuNRs experiences the strongest DEPF of about \(5 \times 10^{-8} \text{ N}\). This means that the buoyancy force is significantly lower than the DEPF close to the electrodes. Figure 4(f) shows the repetition of simulations of figure 4(d), by choosing the maximum value of the contour scale equal to the buoyancy force \((2.34 \times 10^{-15} \text{ N})\). Here the dark red area represents the regions (critical DEPF region) where DEPF is greater than the buoyancy force; the region is subsequently referred to as the critical DEPF region. The maximum height of this region is 35 \(\mu\text{m}\), which is about three times less than the maximum height of the region where the electric field was stronger than the critical field (figure 4(e)). This seems to suggest that the buoyancy force is effective even in the region where Brownian motion is dominated by the electric field. However, it should be noted that the y-component of the DEPF and buoyancy force is in the same direction. The net force on AuNRs in the critical field region is the sum of the Buoyance force and DEPF, which is still effectively directed towards the electrode edges.

3.4. Electro-osmotic flow
The electro-osmotic flow is the fluidic flow due to the presence of ions in the solution and is directed away from the electrode gap. Such flow can achieve velocities given by the following equation \([23]\)
where $V_0$ is peak-to-peak voltage, $d$ is half of the electrode gap, $\eta$ is solvent viscosity, $\Omega$ is the dimensionless frequency defined by,

$$\Omega = \frac{\omega \varepsilon_m \pi n_i \eta}{2 \sigma_m \lambda_D}$$

(8)

with,

$$\lambda_D = \sqrt{\frac{\varepsilon_m K_B T}{q^2 \sum n_i^2 z_i^2}}$$

(9)

here, $q$ is an elementary charge, $n_i^0$ and $z_i$ are the number concentration and ionic charge, $\lambda_D$ is the Debye-Huckel length, which gives the distance of counter ions away from the dielectric-electrolyte interface without being completely shielded by the ions in the electrolyte.
Figure 6. Simulation of the electric field strength for the long-axis for the applied potential of (a) 2 V and (b) 7 V (The region with dark red color are the regions where the electric field strength is greater than the critical field). Simulation of the DEPF strength for the long-axis for the applied potential of 2 V (c) and 7 V (d); the region with dark red color are the regions where DEPF is greater than the buoyancy force. Variation of the critical force region (e) and critical field region (f) with applied voltage. Optical and SEM images of AuNRs between electrodes at a frequency of 1 MHz and the potential of 2 V (g), (i), and 7 V (h), (j).
The magnitude of the electro-osmotic flow velocity for water is shown in figure 5(b). It has vanishing values both at low and high frequencies. This is due to the fact that at low frequencies ions completely screen the electrodes, while at high frequencies ions do not have sufficient time to respond to the changing polarities of the electrodes, resulting in no net flow towards the electrodes. However, in the frequency range from 5 Hz to 1 kHz, there is a certain magnitude of electro-osmotic flow velocities, with a peak at 30 Hz.

As a control experiment, we start by performing a deposition experiment simply by drying a 40 μl colloidal droplet on the electrode geometry, without the presence of any external electric fields. The resultant distribution of NRs is completely random (figure 5(b)). In order to see the influence of frequency on the deposition of AuNRs, we carried out the deposition experiments at 100 Hz and 200 kHz with the applied potential of 5 V. Figures 5(c)–(f) shows the optical and SEM images of the resultant deposition. At a frequency of 100 Hz, a large number of NRs can be seen near electrodes edges. However, a significant amount of particles can also be seen on top of the electrodes. Such NRs patterns arise because of the electro-osmotic flow which is prominent at a low frequency such as 100 Hz. Increasing the frequency to 200 kHz leads to the deposition of NRs within the electrode gap, mainly close to the electrode edges, as evident from figures 5(c), (e).

3.5. Effects of applied voltage
The strength of the electric field and field gradient for coplanar electrodes can be increased by increasing the applied voltage. Figures 6(a), (b) shows the simulation results of electric field patterns for a 50 μm electrode gap, for the applied voltages of 2 V and 7 V, respectively. Here again, we have chosen the maxima of the contour scale equal to the critical field along the long-axis (1.42 × 10^4 V m⁻¹), so that we can see the extent of the region where the electric field strength is greater than the critical field. Figures 6(c), (d) shows the DEPF pattern for 2 V and 7 V, respectively, with the counter scale maxima set equal to the buoyancy force. Although critical field region extends considerably above and fills the electrode gap, at lower voltages DEPF region is restricted closer to the electrode edges. The area of both regions increases with an increase in applied voltage. Figures 6(e), (f) quantitatively shows how the areas where critical electric field and critical DEPF dominate increase with the applied voltage. The areas were calculated using software image j. This is also verified from the experimental results as depicted by figures 6(g)–(j), showing the optical and SEM images, respectively. Clearly, the particles deposited within the electrode gap are much more for 7 V, compared to 2 V.
3.6. Effect of the deposition time
As discussed earlier, the concentration of NRs within the region where the NRs are trapped by the electric field increases as NRs from the outer regions of droplet enter there due to the Brownian motion as well as the buoyancy force. Therefore, the number of NRs trapped by the electric field is higher for higher deposition time. Figure 7 shows the compression of two different deposition times. Figures (a), (c) depict the images of the deposition experiments in which the AuNRs suspension was allowed to partially dry for 5 min; while Figures (b), (d) depict the deposition experiments in which the droplet was allowed to completely dry on the electrode in the presence of the applied field, which takes more than one hour. Both of the experiments were performed under the presence of the electric field generated by 5 V and 1 MHz. Clearly, there is a greater accumulation of AuNRs for higher deposition time. As during complete evaporation, to buoyancy force, the Brownian motions and solvent evaporation push the NRs in the regions where they are trapped by the electric field.

3.7. Effect of the solvent
Although complete drying of water droplet containing NRs leads to a higher deposition density of NRs, the experiment takes more than one hour due to the lower evaporation rate of water. The drying time can be reduced by dispersing NRs in a relatively more volatile liquid, such as ethanol. By changing medium from water to ethanol, the value of Re(CMF) does not change as the conductivity of Au is overwhelmingly greater than both water and ethanol. However, there is a slight shift in the crossover frequency (see figure 8(a)).
Figures 8(b)–(e) depicts the optical and SEM images of deposition experiments carried out by using NRs dispersed in water and ethanol. The deposition in both cases was done for 5 min. In case of ethanolic suspension, complete evaporation occurs while in case of water the suspension removed after 5 min. Clearly, there is a high concentration of NRs in figures 8(c), (e), where ethanolic suspension of NRs was used.

4. Conclusions

We have demonstrated the dielectrophoretic deposition of AuNRs by varying various deposition parameters. The concentration of deposited AuNRs can be controlled by controlling the frequency, applied voltage, deposition time and solvent in which AuNRs are dispersed. At lower frequencies, electro-osmotic flows affect the deposition and push AuNRs to the top of the electrodes. Choosing the frequencies where electro-osmotic flows are suppressed leads to the deposition of AuNRs close to the electrode edges where the electric field and field gradient are maximum. Increasing the applied voltage, deposition time and using a more volatile solvent can significantly increase the concentration of AuNRs in the electrodes gap.

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