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Modeling the co-assembly of binary nanoparticles

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Abstract

In this work, we present a binary assembly model that can predict the co-assembly structure and spatial frequency spectra of monodispersed nanoparticles with two different particle sizes. The approach relies on an iterative algorithm based on geometric constraints, which can simulate the assembly patterns of particles with two distinct diameters, size distributions, and at various mixture ratios on a planar surface. The two-dimensional spatial-frequency spectra of the modeled assembles can be analyzed using fast Fourier transform analysis to examine their frequency content. The simulated co-assembly structures and spectra are compared with assembled nanoparticles fabricated using transfer coating method are in qualitative agreement with the experimental results. The co-assembly model can also be used to predict the peak spatial frequency and the full-width at half-maximum bandwidth, which can lead to the design of the structure spectra by selection of different monodispersed particles. This work can find applications in fabrication of non-periodic nanostructures for functional surfaces, light extraction structures, and broadband nanophotonics.

Supplementary material for this article is available online

Keywords: self-assembly, colloids, nanoparticles, nanolithography, nanostructures

(Some figures may appear in colour only in the online journal)

1. Introduction

Colloidal nanosphere lithography (NSL) has attracted significant interest as a bottom-up fabrication method in the recent years [1–9]. In this approach, monodispersed spherical nanoparticles are assembled on a planar surface to form an ordered, hexagonal close-packed lattice. Early works on NSL have used a single layer of nanoparticles as an etching mask

Original content from this work may be used under the terms of the Creative Commons Attribution 4.0 licence. Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI. to fabricate micro/nanostructures, which can find applications for antireflection structures, superhydrophobic surfaces, photonic crystals, plasmonics and photovoltaics [3–12]. In addition to subtractive processes, NSL can also be used to make two-dimensional (2D) nanostructures by using a particle array as a lift-off mask for additive processes such as physical vapor deposition [9, 13]. The popularity of NSL can be largely attributed to its low cost, high throughput, and large area compared to other conventional lithography processes. In the past, gradient-index antireflection diffraction optics have been successfully fabricated using NSL, where diffracted orders in diffraction gratings can be suppressed by two orders of magnitude [14]. NSL has also shown





Figure 1. (a) Assembly model schematic of single crystal and (b) predicted 2D spectra with six distinct peaks. (c) Assembly model schematic of binary assembly and (d) predicted 2D spectra with ring-like pattern.

applications in nanochemistry, where surface plasmonics in nanostructured materials play a role in determining the spatial and temporal control of chemical reactions [15]. Additionally, recent works have also demonstrated variations of nanosphere lithography that make use of nanobridges to have a controlled template assembly of nanoparticles [16].

There are various methods to implement the controlled assembly of colloidal particles into hexagonal closed packed arrays, including drop coating, spin-coating electrophoretic deposition, and Langmuir-Blodgett assembly [17-22]. NSL can be performed using monodispersed silicon dioxide, polystyrene, or PMMA (poly-methyl methacrylate) nanoparticles having narrow size distribution that are obtained using various polymerization methods [23]. Recent work in colloidal assembly has also demonstrated more complex geometries such as binary, non-close packed, and disordered colloidal crystals [24–29]. Specifically, these processes have been used to create surface structures that have a lattice order similar to the particle arrays. More complex patterns can also be patterned by harnessing the near-field light diffraction from colloidal assemblies to create 3D nanostructures in photoresist [10, 30]. While these advances are exciting, most existing approaches are more focused on using periodic array of nanospheres to create the initial pattern.

Traditional self-assembly of colloidal nanoparticles have many applications and have been extensively studied in plasmonics and nanophotonics [31]. One potential area is light extraction in solid-state lighting, where photonic crystals with single periodicity have been studied [32]. Periodic nanostructures in light emitting diode (LED) devices can diffract light that is typically trapped due to waveguide or surface plasmon polariton mode and increases the outcoupling efficiency of light [33, 34]. However, structures with a single period exhibits wavelength and angle-sensitive behavior, which are not ideal for broadband or wide-angle applications. On the other hand, random surface roughening is known to exhibit a broad periodic range, but is difficult to precisely fabricate [33, 35]. Nanostructures with dual periodicity have been studied to realize broadband light extraction in OLEDs with high efficiency enhancement, but these nanostructures are expensive to fabricate [36]. The main challenge for this approach is that there are presently no scalable methods for modeling and fabricating of nanostructures that are non-periodic.

This work presents a model that simulates the coassembly pattern of monodispersed nanospheres with different diameters, size distributions, and relative concentrations and their spatial frequency spectra. This model is based on an iterative approach, where the different particles are placed in sequence with geometric constraints to yield the final assembly structure. The algorithm is able to simulate the randomized close packing of the binary particles, which gives



Output image

Figure 2. Flowchart of binary particle co-assembly model based on geometric constraints.

rises to non-periodic colloidal assembly. The spatialfrequency spectra of the modeled assembly are analyzed based on their ring intensity distribution using Fourier analysis to evaluate the broadening of the spectra peaks. The simulated assembly pattern and spectra are in qualitative agreement with experimental assembly result, which can facilitate the design of structures with broadened spectra using co-assembly of colloids with multiple sizes. This work can facilitate the fabrication of non-periodic nanostructures, which can find applications in surface nanostructures [35, 36].

2. Modelling of binary assembly

The goal of this research is to create a model that can predict the co-assembly pattern of monodispersed nanoparticles with two distinct particle size distributions, as illustrated in

figure 1. For self-assembly of spherical particles with a single diameter, the assembly pattern results in an unary colloidal crystal in hexagonal close-packed lattice, as shown in figure 1(a). The 2D spatial-frequency spectra are shown in figure 1(b), where 6 distinct peaks are expected for the fundamental orders. For a co-assembly of particles with a designed size ratio, the smaller particles can assemble into the voids of the larger particles to obtain a binary crystal. However, since the periodicity is largely defined by the larger particle, the spatial-frequency spectra would contain distinct peaks similar to an unary colloidal crystals [24–26, 37]. This work focuses on the scenarios when the particle sizes are relatively similar so that the resultant assembly is nonperiodic, as illustrated in figure 1(c). Since the co-assembly contains features that are characteristic of the particle diameters but are randomly oriented, the frequency spectrum is expected to be circularly symmetric, as illustrated in figure 1(d). The average radial frequency f_o and the full width at half-maximum (FWHM) of the spectra is expected to be functions of the relative size distribution of the binary particles, which can be controlled to broaden the spectra. Note the structure is non-periodic but contains periodic features that are defined by the relative size of the two particle diameters used.

The co-assembly model developed in this work is based on geometric constraints, and the algorithm is illustrated in figure 2. Implemented in MATLAB, the input variables to the algorithm are the diameters of the particle (D_1, D_2) , standard deviation of the size distribution (σ_1 , σ_2), and the relative ratio of the particle concentration. A table of the particle diameter to assemble is then generated based on the input variables, assuming a normal distribution for the particle sizes and randomly selecting between the two particles based on the relative concentration. The particle sizes introduce randomness in the model as they are generated by a normal distribution function using the mean and standard deviation parameters as input. Assuming a one-dimensional drying front during the transfer coating process, the model places the tabulated nanoparticles in a straight line at the initiation stage by referencing the center and diameter of the particles. Subsequent particles are then placed in a line-by-line manner while ensuring they form direct contact with the assembled particles. The model assumes that the assembled particles in the previous line do not move, which is consistent with a linear evaporation front commonly observed in colloidal assembly. As the algorithm progresses, some particles cannot stay in contact with its immediate neighbors due to the variations in particle size, resulting in voids in the assembly pattern and thereby decreasing the packing fraction. To address this issue, a search algorithm was created to find the best possible contact solution by finding the shortest distance between the five neighboring particles. This function effectively reduces the surface area of the assembled pattern and prevents in overlapping of particles. The shortest distance is calculated by using the center-to-center distance of the particles and the diameter of the particles. The process continues until the predefined assembly area has been fully populated. The model does not consider kinetic pathways towards the particle



Figure 3. Calibration study for particle size distribution. Predicted assembly pattern with (a) 0%, (b) 5%, (c) 10%, and (d) 15% size variations. The corresponding 2D FFT of the pattern with (e) 0%, (f) 5%, (g) 10%, and (h) 15% size variations.

assembly structure, which can lead to discrepancy of the assembly pattern in the spatial domain. Therefore it is important to note that this model can serves as an approximate model for qualitative predictions of the spatial frequency spectra rather than an exact replica of experimental conditions.

For each assembly simulation, a 10 μ m by 10 μ m area is used. The assembly patterns are then converted into 1001 pixel by 1001 pixel images, from which the 2D spatial-frequency spectra are generated using fast Fourier transform (FFT). For non-periodic assembly, the spectra have radial symmetry and a 1D radial frequency plot versus f_r is generated by spatial averaging in the angular direction. For each assembly simulation, the peak frequency, the spectra range evaluated as FWHM, and the packing fraction can be determined. The model is used to examine the co-assembly of particles with 350, 390, and 500 nm with 0%–10% size distributions and at various particle ratios.

3. Experimental method

An assembly of colloidal nanoparticles with various diameters on planar silicon substrates has been fabricated and analyzed to validate the co-assembly simulation model. Polystyrene (PS) nanoparticles with 350, 390, and 500 nm diameters are used in the experiments, which has a nominal 2.6% concentration in aqueous solution (PolyScience). The suspensions of PS particles are mixed with ethanol to achieve the appropriate relative particle concentration. The suspension is then sonicated for 20 min to reduce aggregation, then spincoated on a carrier silicon substrate. The particles are then immersed in deionized water to form the assembly on the liquid surface. Sodium dodecyl sulfate (SDS) is applied as a surfactant to enhance the close packing of the nanoparticles.



Figure 4. Simulated (a) peak frequency and (b) assembly packing fraction versus the size variation for self-assembly of 500 nm particles.

The particles are then lifted off the water surface using a silicon wafer substrate by following the procedures in transfer coating method [38]. The assembled structures are imaged using scanning electron microscopy (SEM) and the spatial-frequency spectra analyzed using FFT.



Figure 5. Simulated assembly image of binary nanosphere assembly model with 1:1 relative concentration for (a) 350–390 nm, (b) 350–500 nm, (c) 390–500 nm and corresponding experimental SEM images of the fabricated assembly for (d) 350–390 nm, (e) 350–500 nm, (f) 390–500 nm particle assembly.



Figure 6. 2D FFT of binary nanosphere assembly model simulation image with 1:1 relative concentration for (a) 350–390 nm, (b) 350–500 nm, (c) 390–500 nm nanoparticle assembly. 2D FFT of experimentally assembled binary assembly SEM image with 1:1 relative concentration for (d) 350–390 nm, (e) 350–500 nm, (f) 390–500 nm nanoparticle assembly.

4. Results and discussion

A calibration study has been performed using monodispersed particles with various size distribution to examine the size distribution of the particles used. In this study, the selfassemblies of particles with diameter D = 500 nm and size distrubtions of 0%, 5%, 10% and 15% are simulated, as shown in figures 3(a)–(d), respectively. It can be observed that the particles with 0% variation form a perfect colloidal crystal with period $\Lambda = D\sqrt{3}/2$ and theoretical frequency of $f_x = 1/\Lambda = 2.31 \ \mu m^{-1}$. The corresponding 2D spatialfrequency spectrum is shown in figure 3(e), and shows



Figure 7. Angularly averaged 1D radial spectra for (a) different particle combinations of 350, 390, and 500 nm diameter having 1:1 relative particle concentration and (b) different relative particle concentrations for co-assembly using 350 and 390 nm particles.

distinct peaks as expected from a periodic lattice. Six fundamental peaks can be observed with measured spatial frequency $f_x = 2.33 \ \mu m^{-1}$, comparable to the predicted value. The deviation can be attributed to discretization error. Note that higher order harmonics of the fundamental frequency can also be observed. As the particle size variation increases, the self-assembled structures become increasingly non-periodic, as shown in figures 3(b)-(d). The corresponding 2D spectra are shown are shown in figures 3(f)-(h), respectively. Here, it can be observed that particle size variation increases, the packing fraction reduces. The nonperiodic arrangement also causes the 2D FFT spectra to shift from a distinct spots to a ring-like spectra that are broadened, as illustrated in figures 3(f)-(h). Given the circular symmetry, the radius of the ring can be measured to define the radial frequency f_r . Note, the peaks are more pronounced in the y direction, which are artifacts that can be attributed to the lineby-line assembly of the particles.

The relationship between peak radial frequency and packing fraction is further examined, as shown in figure 4. Here it can be observed that the peak frequency decreases S Mohanty et al

variation, from of versus the size a maximum $f_x = 2.33 \ \mu \text{m}^{-1}$ for a periodic crystal with 0% variation to $f_o = 2.18 \ \mu m^{-1}$ for 15% size variation, as shown in figure 4(a). As expected, this trend strongly correlates with reduction in packing fraction, which was found to be 90.1%, 82.9%, 81.4% and 81.9% for 0, 5, 10, and 15% variations, respectively, as shown in figure 4(b). It was observed that packing fraction reduces dramatically by about \sim 8% between 0% and 5% particle size variation, although the change in packing fraction values above 5% particle size variation was found to be not very significant. Based on these simulations, a particle variation of 10% is used since an average packing fraction of around 80% was observed in experimental assembly of monodispersed particle, as discussed in the experiments.

To further validate the model, binary assembly experiments using two different particles with various combinations consisting of of 350, 390, and 500 nm particles were performed. The simulated co-assembly pattern with 1:1 particle size combination of 350-390 nm, 350-500 nm, and 390-500 nm, are shown in figures 5(a)–(c), respectively. This simulation assumes a 10% particle size variation for both particles, as informed by the calibration simulation. It can be observed that given the non-monodispersed nature of the assembly, the pattern is highly disordered. Similar behavior can be observed in the corresponding co-assembly patterns obtained experimentally for 350-390 nm, 350-500 nm, and 390-500 nm pairs, as shown in figures 5(d)-(e), respectively. The average packing fraction across all particle combinations calculated for the simulation and experiments are 82% and 77%, respectively. The lower packing density of the experimental assembly can be due to higher defect density since the model assumes all particles are in contact with neighboring particles. The model may also be overestimating the packing ratio since it is a two-dimentional model and assumes the contact point are all within a horizontal plane. Based on these results, the simulated model and experimental images match well and can be used to predict and the peak spatial frequency and the FWHM. Additional data on the simulated and experimental assembly results can be found in supplementary information A.

To analyze the accuracy of the spectral prediction, the 2D FFT spectra of the simulated and fabricated binary assembly are calculated, as shown in figure 6. As predicted and observed in figures 6(a)-(c), the 2D FFT spectra of the simulated assembly broaden with the relative increase in particle size ratio from 350-390 nm to 350-500 nm. It can be noted that the spectrum of the 350-390 nm assembly shows a slightly disconnected ring pattern, which can be attributed to the artifact introduced by the line-by-line assembly. The experimental SEM images are binarized and processed using a Hanning window function to reduce the edge effect, and the resulting 2D FFT spectra for the corresponding particle combinations are shown in figures 6(d)-(f). Here it can be observed that the experimental spectra also exhibit radial symmetry with broadened spectra and shows qualitative agreement to the simulation. It can be noted that lower frequency spectra from the experiments generally have higher



Figure 8. Extracted experimental and simulation (a) peak frequency and (b) FWHM values versus relative concentration for co-assembly of different nanoparticle combinations.

noise, which can be attributed to the image acquisition process. Additional data on the 2D FFT of the simulated and experimental assembly results can be found in supplementary information B.

To further reduce noise and given the radial symmetry, the 2D FFT spectra for both simulated and fabricated assemblies are angularly averaged to obtain the 1D spectra versus the radial frequency f_r , as shown in figure 7. The effect of different particle sizes is examined first, as the spectra for 1:1 ratio of 350-390 nm, 350-500 nm and 390-500 nm are shown in figure 7(a). It can be observed that the simulated spatial frequency shift higher as smaller particles are introduced. The corresponding experimental peaks are in qualitative agreement, with a slight shift to lower frequency. This shift can be attributed to the lower packing fraction that was observed in the experimental results when compared with the simulation. The lower packing fraction as the structure becomes less ordered is also observed in the single crystal assembly calibration simulation, as shown in figure 4. It is also important to note that the experimental spectrum for the 350-390 nm assembly are more noisy, which can be attributed to the relative similar size of the particles used. The effect of the relative particle concentrations is also examined, and the results for 1:3, 1:1:, and 3:1 for 350-390 nm assembly are shown in figure 7(b). Here it can be observed that as the particle concentration of the larger particle increases, the peak frequency shifts lower as expected.

The peak frequency f_o can be extracted from the spectra of the simulated and fabricated assembly versus the relative particle concentration, as plotted in figure 8(a). The predicted theoretical f_o values for a periodic colloidal crystal consisting of 350, 390, and 500 nm particles are 3.3, 3.0, and 2.3 μ m⁻¹, respectively. Based on the plots shown in figure 8(a), the simulated f_o values for 350, 390, and 500 nm are 3.1, 2.8, and 2.2μ m⁻¹, respectively. That represents a variation of about 4%–13% between experimental and simulated f_o values, which can be attributed to the lower packing density in the experimental nanoparticle assembly. It can be observed that both simulated and experimental frequencies shows a general decrease in frequency as the volume fraction of the larger particle increases. This result indicate that the frequency of the co-assembly to be tuned between the corresponding frequency of the particle used. The peak frequencies calculated from the co-assembly experiments qualitatively follow this trend, though a constant frequency offset can be observed. The shift to lower frequency can be attributed to the lower packing fraction observed in the experiments, as previously discussed. This effect can be mitigated by increasing the particle size variation or introducing more defects in the co-assembly model.

Another important property is the bandwidth of the spectra, and the FWHM values for the simulated and fabricated assemblies are shown in figure 8(b). Here it can be observed that the FWHM values for monodispersed particles are similar, ranging from 0.44 to 0.87 μ m⁻¹. However, the FWHM increases and peaks at around 50% volume fraction for all the samples, indicating the bandwidth of the spectra can be broadened by the binary assembly. This trend is qualitatively observed in the FWHM values from the fabricated sample, with all particle combinations showing a peak at 50% volume fraction. It is also interesting to observe that the 350-500 nm combination was observed to have the broadest FWHM as the relative size are most different. The respective simulated and experimental peak frequency and FHWM values have a variation of 6%-8%, which can be attributed to experimental SEM image noise and packing density uncertainty.

The result of the co-assembly experiments qualitatively follows and provides validation for the model developed in this work. One potential source of error is the deviation in packing fraction between simulated and experimental assembly, which leads to a constant peak shift to lower frequency in the calculated spectra. This effect is also expected to lead to a slight broadening of the FWHM do to more variations in the assembly density. This discrepancy can be attributed to the experimental assembly having more voids and not all particles are in direct contact, a key assumption in the co-assembly model. This error can be mitigated by introducing a void formation based on probability, which can further reduce the simulated packing fraction to the values measured in experiments. The model can also be improved by including the three dimentional packing effects to account for different particle heights. Co-assembly of particles with larger size distributions will also be explored in future work, including assembly systems with more than two distinct particle sizes.

5. Conclusion

This work demonstrates the proof-of-concept that the spatial frequency spectra of binary assembly can be designed and patterned using colloidal particles with different diameters. This work is enabled by a co-assembly model, which can accurately predict the assembly pattern and spectra of the assembled nanospheres with different combinations of 350, 390, and 500 nm diameters. The assembly models shows some edge effects due to the line-by-line particle placement, which can be mitigated by using a larger assembly area in the simulation. Future work will focus on finding the optimal particle parameters to demonstrate non-periodic nanostructures with desired spatial frequency spectra, as well as examine assembly systems with more than two particle sizes. Future work will also include a local annealing step in the model that will allow the assembled particles to reorganize themselves to improve the ordering and help address the longrange interaction limitations of the current model. This work can find applications for predictive design of nanostructures for light extraction in solid-state lighting, antireflection structures, and other nanophotonic elements.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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