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Forming concentric double-emulsion droplets using electric fields

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ABSTRACT

Double-emulsion droplets may be assembled into highly concentric shells using a uniform AC electric field to induce dipole/dipole interactions. The resulting force centers the inner droplet with respect to the outer shell if the outer droplet has a higher dielectric constant than the ambient, suspending liquid. The dielectric constant of the inner droplet does not influence this condition. Applying an electric field $>10^4\,V_{rms}/m$ achieves centering of approximately 3–6 mm diameter droplets suspended in ~10 centipoise liquids within ~60 s. If the outer shell is electrically conductive, the effect depends strongly on frequency. In the case of the monomer-containing liquids requisite to forming foam shells for laser target fabrication, the electrical field frequency must be ~10 MHz or higher. Because of very stringent requirements imposed on the concentricity and sphericity of laser targets, electric field induced droplet distortion must be minimized. Consequently, the liquid constituents must be matched in density to ~0.1%.

1. Introduction

Many chemical and materials technologies are based on processes that employ double-emulsion droplets, that is, droplets of one fluid contained within another immiscible fluid, see Fig. 1. One technically challenging application for such droplets is in the formation of polymer foam shells for laser fusion. The challenge stems from extraordinarily stringent concentricity requirements imposed upon the very delicate foam shells (diameter ~3 mm and thickness $d \sim 25 \pm 1\ \mu m$) [1]. In particular, fusion-initiating inertial confinement can work only if the fabricated shells are concentric within ≤5%. Larger deviations introduce asymmetry and hydrodynamic instability, disrupting the ablation-driven implosion before sufficient temperature and density can be achieved. Batch processes now used to produce laser fusion targets lack effective process control other than crude adjustment of agitation and polymerization rates, which results in unacceptably low yields of useable shells. Because of these poor yields, present schemes cannot be scaled up to produce the $\sim 10^4$ targets per hour needed for even a laser fusion-based power plant of modest size [2,3].

An ideal solution to meet the concentricity requirement would be a scalable, self-assembly mechanism that avoids or minimizes the need for intervention or macroscale process control [4]. The objective of the research reported in this paper is to use an externally applied, uniform AC electric field – inducing dipole moments and enlisting resultant dipole/dipole forces on multiphase assemblies of dielectric liquids – to form highly concentric, spherical liquid shells, which can serve as mandrels for polymerization.

2. Theory

Refer to Fig. 1, showing a uniform AC electric field imposed on a double-emulsion droplet suspended in an ambient liquid. The dielectric constants of the three liquids, starting from the outside, are κ_1, κ_2, and κ_3. The necessary and sufficient condition for achieving stability is $\kappa_2 > \kappa_1$; the inner droplet's dielectric constant, κ_3, has no effect on this condition. The almost counterintuitive simplicity of this condition may be explained by the theory of electrical images.

There is no closed-form image theory for dielectric spheres [5]; nevertheless, any charge distribution located near or within such shapes may be approximated using the lead image term. Consider a sphere of radius R_{outer} and dielectric constant κ_2 suspended in an infinite medium of dielectric constant κ_1. Locate within this sphere a point dipole $p_o\,\hat{z}$ with either axial (δz) or equatorial (δx) displacement from the center, as shown in Fig. 2a and b, respectively. The dipole induces an infinite set of electrical images in the medium κ_1, with steadily increasing distance from the spherical dielectric boundary. The signs of the lead image terms depicted in Fig. 2a and b represent the case of $\kappa_2 > \kappa_1$. From inspection, it is evident that the dipole/dipole interaction force pushes p_o back

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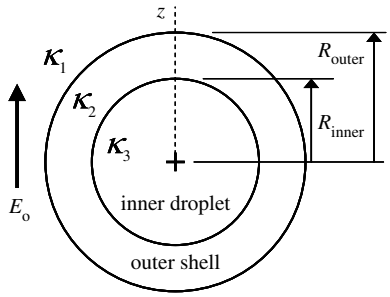

Fig. 1. Concentric double-emulsion droplet with uniform applied AC electric field E_o plus definitions of radii and dielectric constants.

toward the origin for both δz and δx, thereby assuring stability. In fact, the interaction is a stabilizing force for displacements in any direction as long as $\kappa_2 > \kappa_1$.

For the case of interest here, we replace the point dipole by the inner spherical droplet of Fig. 1. The applied AC electric field E_o induces an effective dipole moment p_{eff} within the inner droplet, the sign of which can be positive or negative depending on the relative magnitude of κ_2 and κ_3; however, the sign relationship of p_{eff} to its lead image term p'_{eff} (and all other higher-order terms) is unchanged. Therefore, the condition $\kappa_2 > \kappa_1$ still guarantees the existence of a stable equilibrium at the center of the shell, and the dielectric constant of the inner droplet, κ_3, has no effect on the centering stability. An estimate for the centering force, accurate for small displacements, may be obtained using multipolar expansion (see Appendix) [6].

$$f_{\delta z} = -(\kappa_2-\kappa_1)\frac{432\pi\varepsilon_o\kappa_1^2\kappa_2 R_{inner}^6 R_{outer}^6(\kappa_2-\kappa_3)^2(3\kappa_2+2\kappa_3)E_o^2}{A^2B}\delta z \tag{1a}$$

$$f_{\delta x} = -(\kappa_2-\kappa_1)\frac{324\pi\varepsilon_o\kappa_1^2\kappa_2 R_{inner}^6 R_{outer}^6(\kappa_2-\kappa_3)^2(3\kappa_2+2\kappa_3)E_o^2}{A^2B}\delta x \tag{1b}$$

where

$$A = 2R_{inner}^3(\kappa_1-\kappa_2)(\kappa_2-\kappa_3)+R_{outer}^3(2\kappa_1+\kappa_2)(2\kappa_2+\kappa_3) \tag{2a}$$

$$B = 6R_{inner}^5(\kappa_1-\kappa_2)(\kappa_2-\kappa_3)+R_{outer}^5(3\kappa_1+2\kappa_2)(3\kappa_2+2\kappa_3) \tag{2b}$$

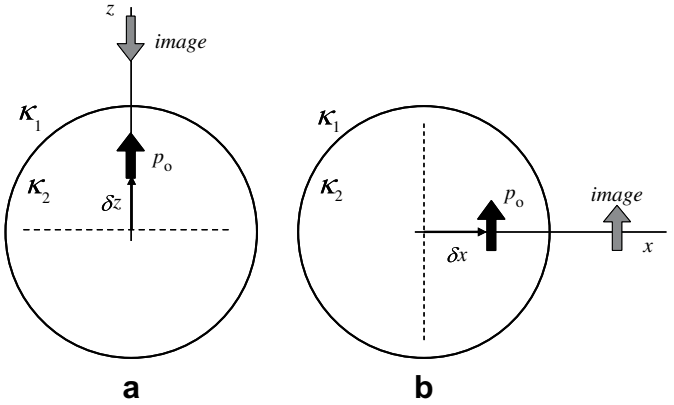

Fig. 2. Dipole p_o located inside sphere of radius R_{outer} and dielectric constant κ_2 showing lead image term for the case of $\kappa_2 > \kappa_1$. (a) Axial displacement δz. (b) Equatorial displacement δx.

We extensively tested the $\kappa_2 > \kappa_1$ stability condition using the Comsol Multiphysics® finite element software. Numerical calculations, as presented in a previous paper [7], show excellent agreement with Eqs. (1a) and (1b) at small displacements.

3. Frequency dependence of centering

To fabricate foam shells, the outer shell liquid will probably have to be an aqueous solution containing foam-forming monomer chemicals, surfactants, and possibly other constituents. Because these additives invariably increase the liquid conductivity, the shell will electrostatically shield the inner droplet, decreasing the induced dipole moment and diminishing the effective centering force. The effective dipole moment phasor of the inner droplet is [8]:

$$\underline{p}_{eff} = 12\pi\varepsilon_2 R_{inner}^3 R_{outer}^3 \times\frac{\underline{\varepsilon}_1(\underline{\varepsilon}_2-\underline{\varepsilon}_3)}{2R_{inner}^3(\underline{\varepsilon}_1-\underline{\varepsilon}_2)(\underline{\varepsilon}_2-\underline{\varepsilon}_3)+R_{outer}^3(2\underline{\varepsilon}_1+\underline{\varepsilon}_2)(2\underline{\varepsilon}_2+\underline{\varepsilon}_3)}E_o \tag{3}$$

where $\underline{\varepsilon}_n = \varepsilon_n + \sigma_n/j\omega$ are complex permittivities, ε_n = dielectric permittivities, σ_n = electrical conductivities, ω = radian frequency, and E_o = external AC field. The centering effect depends even more strongly on frequency than Eq. (3) suggests [7], because the dipole/dipole interaction force is proportional to the product of p_{eff} and p'_{eff}. To overcome shielding, the frequency of the electric field must exceed a critical value, ω_{crit}. For a typical foam target, (i) the shell thickness is very thin, that is, $d = R_{outer} - R_{inner} << R_{outer}$, and (ii) the outer shell liquid is aqueous based, while the other two phases will be dielectric oils, so that $\kappa_2 >> \kappa_1, \kappa_3$. Then, the critical frequency is [8]

$$\omega_{crit} \approx 2\sigma_2 d/\varepsilon_3 R_{outer} \tag{4}$$

4. Dynamic model for centering

The centering time is a useful figure of merit for electric field induced centering. We developed a simple, reduced-order, dynamic model to estimate it by ignoring inertia. When the voltage is applied, the centering force starts to move the inner droplet. This motion is impeded only by viscous drag [9,10], as long as the various densities are closely matched. If we assume that $\delta z, \delta x << d$, the dipole/dipole centering force is linear in terms of the displacement. Furthermore, in the limits of small Reynolds and capillary numbers for double emulsions with $R_{inner}/R_{outer} \sim 1$, a simple power law expression accurately represents the viscous drag. Consequently, the reduced-order equation of motion is

$$\alpha\cdot\delta z - \left(\beta + \zeta\cdot\delta z^{\lambda}\right)\frac{d\delta z}{dt} = 0 \tag{5}$$

The equation for δx is very similar. The coefficient for the dipole/dipole force, α, comes directly from Eq. (1a), while the coefficients β, ζ and exponent λ, algebraically complex expressions representing the viscous drag, were presented by Sadhal and Oguz [9]. Eq. (5) has an implicit analytical solution:

$$t = \frac{\beta}{\alpha}\ln(\delta z) + \frac{\zeta}{\alpha\cdot\lambda}(\delta z)^{\lambda} + \text{const.} \tag{6}$$

Using material properties and process parameters typical for foam shell formation, the characteristic time constants of centering are found to range from ~10 to ~100 s.

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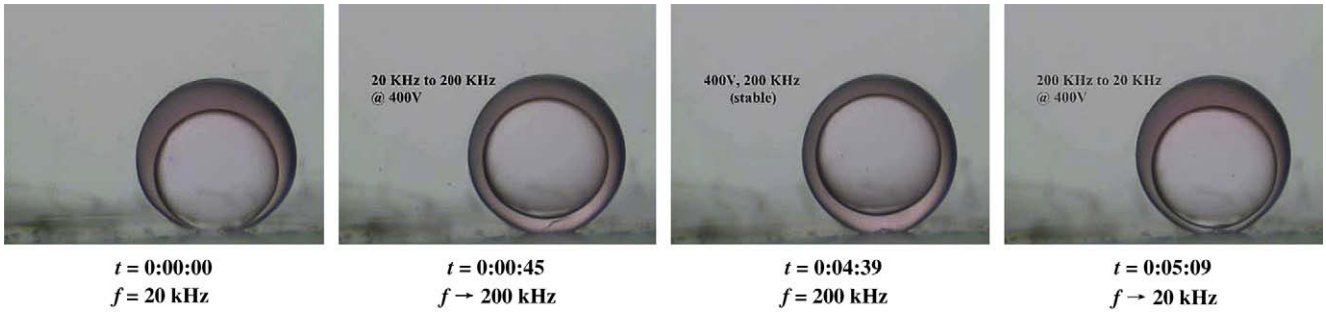

Fig. 3. Video frames revealing frequency dependence of the centering effect for a silicone oil droplet in DMA containing a dye (Mordant Blue 9, Acros Organics) to improve visibility. The radii of the outer shell and inner droplet are, respectively, ~2.9 mm and ~2.2 mm. The fixed electric field strength is 1.3×10^4 V_{rms}/m.

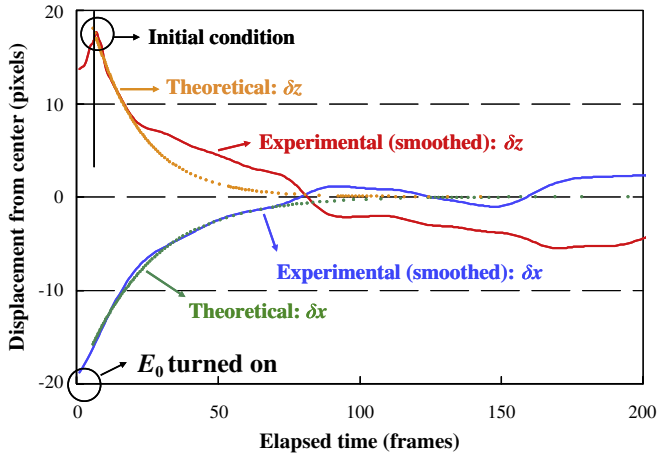

Fig. 4. Axial (δz) and equatorial (δx) dynamic centering data versus time t with comparison to predictions of the reduced-order dynamic model, Eq. (6). These curves are plotted using, as initial values, data points at a time shortly after transients associated with voltage turn-on have died out. Optical resolution = 9 μm/pixel; frame rate = 1.5 fps. Original data points from MatLab program are shown. To suppress unavoidable noise due to resolution limits of the camera, experimental data are smoothed using a FFT low-pass filter.

5. Experiments

We conduct droplet centering experiments using a small, liquid-filled chamber fitted at top and bottom with parallel metal electrodes to impose a vertical applied electric field. Typical electrode spacing is ~14 mm, so a voltage of ~1 kV_{rms} can achieve a field strength of $E_o \sim 7 \times 10^4$ V_{rms}/m. Such voltages at frequencies ≥1 MHz are generated using resonant matching networks.

The three liquids – suspension, outer shell, and inner droplet – are closely matched in density, with the shell liquid intermediate between the inner droplet (most dense) and the suspension (least dense). Other liquid selection criteria include transparency, immiscibility, and $\kappa_2 > \kappa_1$. The suspending liquid and the inner droplet are mixtures of 10 and 50 centistokes silicone oils (DC-200: Dow Corning), which have mass densities 0.934 and 0.960 g/cm^3, respectively. For initial investigations, we selected the organic solvent N,N-dimethylacetamide or DMA (Alfa Aesar) as the shell liquid. DMA has density 0.941 g/cm^3, which is bracketed by the silicone oils. We dope the DMA with surfactant Span 80 (Sigma–Aldrich) to prevent coalescence of the double emulsions.

The droplets are gently supported on a 1 mm thick glass plate, treated with ITO (Indium-tin-oxide) and coated with Teflon-AF™, positioned halfway between the electrodes. A DMA droplet is dispensed onto the ITO glass holder with a micropipette. Then, silicone oil is carefully injected into it using a micro-tip syringe mounted on a micromanipulator.

For a 670 μm thick DMA shell of radius 2.88 mm and conductivity $\sigma \sim 9 \times 10^{-4}$ S/m, the critical frequency, calculated from Eq. (4), is $f_{crit} \approx 400$ kHz. In one preliminary experiment to demonstrate the influence of frequency upon the centering effect, we fixed the field strength at $\sim 1.3 \times 10^4$ V_{rms}/m and varied frequency, see Fig. 3. When the frequency was raised from 20 kHz to 200 kHz, the inner silicone oil droplet immediately rose to the center where it resided stably. Reducing the frequency back to 20 kHz caused the inner droplet to sink back to the bottom.

We use a video camera with 1024 × 1024 pixel resolution and a 6× objective lens to capture the dynamics of centering and droplet distortion. Droplets are back illuminated by the collimated, focused beam of a blue LED. A Matlab® program analyzes video frames, using edge detection on the outer shell and the bright ring (caustic) of the inner droplet. It fits the data using a least-squares routine to ellipsoids, locating their centers and determining the ratio of the semi-major (a) to semi-minor (b) axes. The ellipsoidal distortion ($\gamma = a/b$) is typically 1.00–1.01 for the outer shell and 0.99– 1.00 for the inner droplet.

Fig. 4 plots smoothed experimental data for center-to-center displacements (δz and δx) of a double-emulsion droplet having

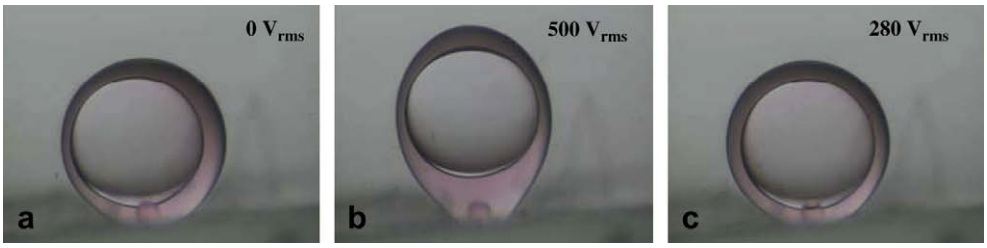

Fig. 5. Sequence of video frames showing accelerated centering of inner silicone oil droplet by intentionally inducing elongation of the outer shell. (a) Before application of voltage. (b) A strong electric field $E_o = 2.3 \times 10^4$ V_{rms}/m at 100 kHz is applied for ~15 s. (c) Field strength is reduced to 1.3×10^4 V_{rms}/m. The time required for the inner droplet to achieve centering is reduced from ~80 s to ~45 s and this lower field strength sustains the concentric condition indefinitely.

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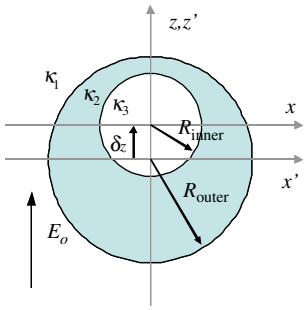

Fig. A1. For the multipolar re-expansion, independent spherical coordinate systems, (r, θ, φ) and (r', θ', φ'), are defined for the two spherical surfaces corresponding to the inner and outer droplets, respectively.

outer radius ~3.3 mm and shell thickness ~450 μm. Centering occurs within ~60 s after applying electric field $E_o = 3 \times 10^4$ V_{rms}/m at 200 kHz. Superimposed on the experimental data are predictions from the dynamic model, Eq. (6). We used the actual values of δz and δx after the initial transients as initial conditions, but no other adjustable parameters. The measured motion in the equatorial plane, δx, is reasonably consistent with the theoretical predictions. On the other hand, there are discrepancies between experiment and prediction for centering along the vertical axis. Chief among these is that the inner droplet seems to settle about 45 μm below the center, probably because of distortion of the electric field caused by the conductive ITO glass holder.

The elongation of the outer sphere for high field strengths is unacceptable for laser targets; however, this distortion might be exploited beneficially to reduce centering times simply by modulating the voltage. As shown in Fig. 5, ~500 V_{rms} at 100 kHz was applied to a double-emulsion droplet with ~2.9 mm outer radius and ~640 μm shell thickness. The droplet elongated very noticeably, but simultaneously the inner droplet moved quickly toward the center. After ~15 s, the voltage was reduced to ~280 V_{rms}, still at 100 kHz, allowing the outer shell to return to spherical. The inner droplet approached the center in ~45 s and remained there indefinitely, compared to ~80 s if the initial high voltage sequence is not used. The dominant centering mechanism here is certainly the strong viscous shear force enlisted when the droplet elongates.

6. Discussion and conclusions

We have shown that a uniform AC electric field $E_o \approx 3 \times 10^4$ V_{rms}/m can form highly concentric double-emulsion droplets of diameter approximately 3–6 mm suspended in ~10 centipoise liquids within ~60 s. The fundamental requirement to realize stable centering is $\kappa_2 > \kappa_1$. The effectiveness of centering also depends on frequency. For applications in fabricating the thin foam shells, the monomer-containing liquid comprising the outer shell will be conductive ($\geq 10^{-3}$ S/m), requiring frequencies >1 MHz. Another process requirement, dictated by the high concentricity and sphericity specifications, is that all liquid constituents be matched in density to ≤0.1%. This requirement stems from an unavoidable tradeoff between using a stronger field to overcome gravity and a weaker field to minimize elongation.

Our results show field induced droplet centering to be feasible, yet technical challenges remain to be overcome. For example, the glass holder used to suspend the droplet distorts the lower side of the droplet and slightly biases the equilibrium on the z axis. We hope to eliminate these problems using density gradient levitation. Another challenge is that we have not yet performed experiments using liquids with conductivity $> 10^{-2}$ S/m. From Eq. (4), we estimate that frequencies ≥10 MHz will be needed for such liquids. Some internal convection of the liquid is occasionally observed. It remains to be seen if ohmic heating and thermal convection will adversely affect droplet centering as conductivity is further raised. The biggest challenge of all is to identify sets of liquids that meet the rather disparate physical property requirements such as the dielectric constant condition ($\kappa_2 > \kappa_1$), excellent immiscibility, and good density-matching capability, while simultaneously accommodating the monomers and precursors essential to polymer foam-forming chemistries.

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Appendix. (adapted from M. Washizu)

Consider a double-emulsion droplet, with the inner droplet displaced by δz in the (vertical) z-direction as shown in Fig. A1. We define two spherical coordinate systems, (r, θ, φ) and (r', θ', φ'), one at the center of each sphere of radii R_{inner} and R_{outer}.

The potential outside and inside the outer sphere can be written in (r', θ', φ') system

$$\Phi'_{out} = \sum_{n,m,cs} E'_{n,m,cs}\phi'_{n,m,cs} + \sum_{n,m,cs} D'_{n,m,cs}\psi'_{n,m,cs} \text{ and}$$
$$\Phi'_{in} = \sum_{n,m,cs} A'_{n,m,cs}\phi'_{n,m,cs} + \sum_{n,m,cs} B'_{n,m,cs}\psi'_{n,m,cs} \quad \text{(A1)}$$

The potential outside and inside the inner sphere can be presented in (r, θ, φ) system

$$\Phi_{out} = \sum_{n,m,cs} A_{n,m,cs}\phi_{n,m,cs} + \sum_{n,m,cs} B_{n,m,cs}\psi_{n,m,cs} \text{ and}$$
$$\Phi_{in} = \sum_{n,m,cs} C_{n,m,cs}\phi_{n,m,cs} \quad \text{(A2)}$$

(ϕ, ϕ') and (ψ, ψ') are spherical harmonic functions with singularities at infinity and at zero, respectively. $E'_{n,m,cs}$ is the applied electric field E_0. In Eq. (A2), $\sum_{n,m,cs} B_{n,m,cs}\psi_{n,m,cs}$ is the electrostatic potential due to the effective multipolar moments of the inner droplet while $\sum_{n,m,cs} A_{n,m,cs}\phi_{n,m,cs}$ is the potential experienced by the inner droplet and due to the external applied field E_0 ($\sum_{n,m,cs} A'_{n,m,cs}\phi'_{n,m,cs}$ in Eq. (A1)). The force on the inner droplet results from the interaction between these two terms.

Using the boundary conditions on potential and electrical displacement:

$$[A'] + [K_A]\cdot[B'] = [K_E]\cdot[E'],\ [B] = [N]\cdot[A] \quad \text{(A3)}$$

Near the center of the coordinate system, $\phi'_{n,m,cs}$ can be re-expanded in terms of $\phi_{n,m,cs}$.

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$$\phi'_{n,m,cs} = \sum_{k=0}^{n} \delta z^k \frac{(n+m)!}{(n+m-k)!k!} \phi_{n-k,m,cs} \tag{A4}$$

Similar for $\psi'_{n,m,cs}$:

$$\psi'_{n,m,cs} = \sum_{k=0}^{\infty} (-\delta z)^k \frac{(n+k-m)!}{(n-m)!k!} \psi_{n+k,m,cs} \tag{A5}$$

Orthogonality dictates that there will be no interactions between different m or cs terms. Φ'_{in} in Eq. (A1) and Φ_{out} in Eq. (A2) represent the same potential, just expressed in the two different coordinate systems, so they are related by Eqs. (A4) and (A5):

$$[A] = [M_A] \cdot [A'], \; [B] = [M_B] \cdot [B'] \tag{A6}$$

From Eqs. (A3) and (A6), the coefficients $A_{n,m,cs}$ and $B_{n,m,cs}$ can be explicitly expressed in terms of $E'_{n,m,cs}$. We include terms to second order ($n = 2$), the minimum needed to calculate the DEP force. The equivalent dipole moment is $p_z = 4\pi\kappa_2\varepsilon_0 B_1$, so the z-directed DEP force is

$$\begin{aligned} f_{\delta z,\text{by } z\text{-field}} &= -p_z \frac{\partial^2 \left(\sum_{n,m,cs} A_{n,m,cs} \phi_{n,m,cs} \right)}{\partial z^2} = -4\pi\kappa_2\varepsilon_0 B_1 \cdot (2A_2) \\ &= -8\pi\kappa_2\varepsilon_0 B_1 A_2 \end{aligned} \tag{A7}$$

Substitute in expressions for A_2 and B_1, we have an expression (Eq. (1a)) for the electrostatic centering force that is proportional to δz and has a negative coefficient when $\kappa_2 > \kappa_1$. For the equatorial force proportional to δx, the coefficient is $^3/_4$ of the one in Eq. (1a).

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