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ac/dc Magnetic Fields for Enhanced Translation of Colloidal Microwheels

Dante Disharoon, Keith B. Neeves*, and David W. M. Marr*

Department of Chemical and Biological Engineering, Colorado School of Mines, Golden, Colorado 80401, United States

Abstract

Microscale devices must overcome fluid reversibility to propel themselves in environments where viscous forces dominate. One approach, used by colloidal microwheels (μ wheels) consisting of superparamagnetic particles assembled and powered by rotating ac magnetic fields, is to employ a nearby surface to provide friction. Here, we used total internal reflection microscopy to show that individual 8.3 μ m particles roll inefficiently with significant slip because of a particle–surface fluid gap of 20–80 nm. We determined that both gap width and slip increase with the increasing particle rotation rate when the load force is provided by gravity alone, thus providing an upper bound on translational velocity. By imposing an additional load force with a dc magnetic field gradient superimposed on the ac field, we were able to decrease the gap width and thereby enhance translation velocities. For example, an additional load force of 0.2 F_g provided by a dc field gradient increased the translational velocity from 40 to 80 μ m/s for a 40 Hz rotation rate. The translation velocity increases with the decreasing gap width whether the gap is varied by dc field gradient-induced load forces or by reducing the Debye length with salt. These results present a strategy to accelerate surface-enabled rolling of microscale particles and open the possibility of high-speed μ wheel rolling independent of the gravitational field.

Graphical Abstract

*Corresponding Authors kneeves@mines.edu (K.B.N.). dmarr@mines.edu (D.W.M.M.).

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ASSOCIATED CONTENT

Supporting Information

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Schematic of coils used to produce ac and dc magnetic fields and experimental apparatus; electron micrographs of Ag Janus particles, where red crosses indicate regions with Ag and representative EDX results for Ag and normal hemispheres; and particle rotation rate measured on Ag-coated Janus particles as a function of applied ac field frequency (PDF)

INTRODUCTION

Colloidal materials can be used as microscale bots capable of translation^{1–8} and cargo delivery.^{9–14} Methods to power and direct them include electric,¹⁵ optical,¹⁶ magnetic,^{17–19} or chemical^{20,21} gradients, self-propelling particles,^{22–24} various motile structures including rotors,²⁵ microworms,²⁶ and swarms,^{27,28} and particle geometries that break symmetry.^{29–31} Our previous work has focused on the use of colloidal building blocks that can be assembled in situ using alternating current (ac) magnetic fields to create rotating microwheels (μ wheels) that translate by rolling on nearby surfaces.^{32,33} While we have shown that μ wheels can move at speeds up to $\sim 100 \mu\text{m/s}$, they are inefficient rollers. Their translational velocity is $\sim 10\%$ of their maximum because of the slip caused by the fluid-filled gap between rotating μ wheels and the associated surface.³² In such small gaps under translation, lubrication forces, in addition to colloidal forces, play an important role in controlling the gap width.³⁴ It is this gap that determines the wet friction coefficient μ_k and the resulting friction force $F_f = \mu_k L$, with L load, propelling rotating μ wheels forward.³² For particles under a load force provided by gravity alone, this force can be expressed as $F_f = \frac{\eta}{hP} \Omega a(\rho - \rho_f)Vg$, where a is the particle radius, h is the gap width, Ω is the rotation rate, η is the viscosity of the medium, P is the pressure in the gap, ρ is the particle density, ρ_f is the fluid density, V is the particle volume, and g is the gravitational acceleration.³² From this, we expect that reducing the gap width will increase wet friction and seek here to develop strategies to manipulate the gap for enhanced μ wheel translation.

While wet friction and slip determine the efficiency of movement, a load force is required to provide contact between the μ wheel and surface. Relying solely on gravity is limiting because it is fixed in magnitude and direction, reducing the effective load L when the rolling surface is not normal to gravity. To address this, an alternative or supplemental load force can be provided with direct current (dc) magnetic field gradients. In this study, we use dc field gradients superimposed on ac fields to provide forces of order 60 fN or $\sim 1/5$ th the load for single particles provided by gravity and then use total internal reflection microscopy (TIRM) to measure the gap width because of its sensitivity and nanometer-scale resolution.^{35–37} The accuracy of TIRM for slightly absorbing colloids with magnetic domains has been previously verified,³⁸ and we apply it here to characterize μ wheels during translation where models based on diffusion such as Stokes–Einstein cannot be applied.³⁹ We then show that

gradient-based load forces can vary the distance between the μ wheel and surface and investigate the role of the gap width on the translation speed.

EXPERIMENTAL SECTION

Superparamagnetic Particles.

A double-sided gasket cut from 3M Tape (467MP with 200MP adhesive, Maple, MN) of 0.5 cm inner diameter was affixed to the top surface of a Dove prism (Thorlabs, PS991, Newton, NJ). A 10 μ L sample of 8.3 μ m superparamagnetic spheres (COMPEL COOH Glacial Blue, UMGB003-UMC4f, Fishers, IN) of density 1.1–1.2 g/mL suspended in deionized (DI) water, resistivity = 18.2 m Ω /cm, was injected into the gasket well and sealed with a coverslip. These particles were chosen for their large size, which minimizes the contribution of Brownian fluctuations on the measured gap width. To determine particle surface roughness, particle suspensions were dried on glass slides, and an atomic force microscopy (AFM) (Asylum Research, Santa Barbara, CA) was used in air tapping mode with a silicon probe with a resonant frequency of 190 kHz and a spring constant of 48 N/m (Tap190E-G, BudgetSensors, Sofia, Bulgaria). Zeta potential was measured with a ZetaPALS zeta potential analyzer (Brookhaven Instruments, Long Island, NY).

Magnetic Field Application.

A rotating ac magnetic field was generated using five 50 mm i.d. air-cored solenoids with 400 turns. An analog output card (National Instruments (NI), NI-9623, Austin, TX) controlled by MATLAB generated a current that was subsequently amplified (Behringer, EP2000, Willich, Germany). The final current passed through each coil, measured using an analog input card (NI-USB-6009), was 2 A, resulting in a field strength of 4.5 mT at the sample. The frequency of the rotating field was varied between 4 and 100 Hz. Additional coils positioned above or below the sample were used to generate dc load forces of magnitude $F_x = V\chi_p(B_x dB_x/dx)/\mu_0$, where $\chi_p = 0.96$ is the particle susceptibility and μ_0 is the permeability of free space. The fields were determined along the coil axis via $B_x = \mu_0 R^2 I / [2(x^2 + R^2)]^{3/2}$, where R is the coil radius and I is the current, summed over the coil windings, including a 50 mm i.d. 400 turn coil, a 25 mm i.d. 200 turn coil, and a 100 mm i.d. 400 turn coil (Figure S1). Videos were recorded using custom-developed BFCam software using a camera (EPIX, SV643M, New York City, NY) at 200 fps through a 40 $\times$ objective (Olympus, Plan Ach NA = 0.65, Center Valley, PA). Video processing and data analysis were performed using custom MATLAB code (MATLAB file exchange ID# 69646).

Total Internal Reflection Microscopy.

In TIRM, colloids scatter an evanescent wave with an intensity that depends on the location of the colloid relative to the surface: $I = I_0 e^{-\beta h}$. Here, I is the scattered light intensity, I_0 is the scattered intensity at the glass-water interface, and β^{-1} is the evanescent wave penetration depth. TIRM experiments were performed on an Olympus BX40 microscope using a 532 nm laser (Spectra Physics, Millennia V, Mountain View, CA) emitting at 0.25 W.

The laser beam passed through the 10 mm Dove prism at a 42° incident angle (Figure 1). To calibrate the measured scattered intensity, we identified a stuck particle in intimate surface contact and used the scattered intensity as our baseline, I_0 . Prior to each experiment, the prism was washed in 1:1 12 M HCl and MeOH for 45 min before being rinsed in filtered DI water for 5 min in triplicate. The samples and the scattered light were imaged using an EPIX SV 643M camera calibrated for intensity measurements with an optical power meter (Newport, model 835, Irvine, CA) equipped with a photosensor assembly (Newport, model 818-SL) with a detection range of gap widths between 0 h 400 nm (Figure S1).

Preparation of Janus Spheres.

Measuring the rotation frequency of individual particles requires labeling and, to do so, we sputtered 49 ± 3 nm thick silver hemispheres onto particles for contrast (Figure S2 and Video S1). A solution (10 μL) of 8.3 μm beads (Glacial Blue, COMPEL, Fishers, IN) at a concentration of 3.3×10^6 beads/ μL was deposited onto a glass coverslip. A permanent magnet was used to spread the solution evenly across the coverslip before the sample was dried. Sputtering was performed in an AJA sputter system using a 99.99% purity silver target (Kurt J. Lesker, Jefferson Hills, PA). The bead sample was brought to a base pressure of 0.38 mTorr before argon was injected to a working pressure of 5 mTorr, and a 50 W radio frequency power supply was used to apply 46 V dc for 4.5 min. The thickness of the silver film was measured with profilometry (Dektak 3030, Veeco, Plainview, NY). The composition of the silver caps was verified using an energy-dispersive X-ray spectroscopy (EDAX, Draper, UT) system. Janus spheres were recovered by washing the glass slide with DI water while physically wiping the coverslip with a swab. Janus spheres were used to build standard curves (Figure S3) of rotation rate versus applied field frequency but, because of additional evanescent-wave scattering induced by the metal coating, were not used in measurements of the gap width.

RESULTS AND DISCUSSION

Gap Width Measurement for Rolling Spheres.

We measured that spheres with a zeta potential of -68 ± 10 mV under Brownian motion centered on average 21 ± 6 nm from the wall ($n > 100$, where n is the number of measurements), in accord with other studies using TIRM experiments under similar conditions.^{40–42} Note that this gap is much smaller than the particle diameter, indicating that the spheres are in a regime where wall effects, including increased viscous drag and hindered mobility on rotating and translating spheres, are significant.^{43,44}

Upon application of an ac magnetic field, particles rotate, translate, and move away from the surface (Figure 2A). Particle rotation rate and ac field frequency are equivalent up to a field frequency of 40 Hz (Figure S3). The observed movement of the particles away from the wall is attributed to lubrication forces that arise during translation. We note that the gap width and translation velocity v_t increase with increasing particle rotational frequency from 10 to 30 Hz (Figure 2B); however, for rotation rates greater than 30 Hz, there is no observable increase in velocity or gap width.

As wet friction is inversely proportional to gap, increasing the load force should result in enhanced translation. To test this, we applied a dc field gradient load force equal to $\pm 0.2 F_g$ ($F_g = 0.3$ pN); a dc load in the direction of gravity (+) pulls spheres closer to the surface, whereas a force applied against gravity (−) pushes them farther away. The gap width decreases with increasing load and increases with decreasing load (Figure 3A; Video S3). We measured a corresponding behavior in translational velocity; as the gap width decreases, velocity increases. A 22% increase in gap width reduces velocity by 27%, but a corresponding decrease in gap width doubles it; therefore, velocity is very sensitive to particle proximity to the wall. In addition, the frequency dependence of both gap width and translational velocity is preserved in the presence of a dc load force (Figure 3A). An alternative approach to reducing the gap width is to manipulate the Debye length λ_D by adding salt (Figure 3B inset) as described by the Derjaguin–Landau–Verwey–Overbeek (DLVO) theory.^{45,46} At these separations, DLVO interactions are dominated by charge–charge repulsions between the carboxyl groups on the particle surface and the hydroxyl groups on the glass slide. Addition of NaCl resulted in expected trends: the gap width decreases at higher salt concentrations⁴² while velocity increases (Figure 3B). It is important to note that these data represent values for those particles that remained mobile. At higher salt concentrations, most particles stuck to the wall with ~50% stuck at a salt concentration of 0.001 M and >95% at 0.1 M. While added salt is an effective method for reducing the gap width, controlling gap via magnetic forces avoids most sticking in our system and provides enhanced velocities compared to load forces by gravitational fields alone (Figure 3C).

To describe the relationship between the velocity and gap width, we equate the frictional force $F_f = \frac{\eta}{hP} \Omega a(\rho - \rho_f)Vg$ with the drag force $F_D = 6\pi\eta a v_t$. Assuming constant pressure in the gap under steady-state translation, we expect $v_t \propto \Omega L/h$ (Figure 3D) and the velocity to increase as the gap width decreases because of increased wet friction. To quantify the rolling efficiency, we define slip as $s = 1 - \frac{v_t}{v_r}$, where $v_r = 2\pi \Omega a$ is the rotation velocity.⁴⁷ At $s=1$, there is complete slip and no translation; at $s=0$, the translational velocity is equal to the rotation velocity. With this, a corresponding scaling for slip is $(1-s) \propto L/h$ as provided in Figure 4A.

The relationship between slip and gap width for all experimental conditions shows two regimes (Figure 4B). In the first, there is a significant increase in slip with the increasing gap width up to ~20 nm. In the second, there is a more modest increase in slip at larger gap widths. The transition between these two regions is equivalent to the average particle surface roughness (~20 nm) as measured by AFM (Figure 4C), suggesting that asperities on the particle may influence interactions with the rolling surface. This is congruent with the observation that particle motion appears to have a stick–slip-type motion and translate faster in the small gap regime (Figure 4D). At gap widths larger than 20 nm, the motion is smoother, suggesting that colloids roll primarily by a wet friction mechanism. Even with an increased load force of $1.2 F_g$, the minimum slip we observe is 0.79, suggesting that there is room for larger enhancements in translational velocity if sufficient torques are available to overcome increased friction.

CONCLUSIONS

Using TIRM, we have measured the gap width of ac magnetic field-induced rolling of colloidal spheres. Compared to resting spheres under the influence of Brownian motion, rolling spheres lift away from the surface to a gap width that dictates slip and translational efficiency. We have shown that adjusting the gap width with dc magnetic load forces on the order of $0.2 F_g$ facilitates enhanced translational velocity. Similarly, we showed that adjusting the gap by decreasing the double-layer thickness also yields enhanced translational velocity. Taken together, these observations suggest that the gap width directly impacts wet friction between μ wheels and the surface. Scaling arguments based on balancing frictional and drag forces demonstrate an inverse relationship between translation velocity and gap width. For small gaps, spheres translate via a stick–slip behavior where particle surface features increase the frictional force and enhance translation. Future studies could consider higher dc field gradients such that particles translate exclusively in the “stick” regime, where our data indicate that translation speeds are fivefold larger than in the “slip” regime. The technique of combining ac and dc fields to drive μ wheels could also be particularly useful when the gravitational load is reduced, for example, in the translation on nonhorizontal and curved surfaces.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

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Figure 1.

Experimental apparatus. Coils generate an ac or ac/dc magnetic field that cause μ wheels to roll near a prism surface, scattering an evanescent wave created by a laser passing through the prism. Laser power is measured continuously via power meter. Scattered intensity is measured using a calibrated camera and normalized by the laser output. Inset: $\Theta = 42^\circ$, $\Theta_1 = 70.1^\circ$, $\Theta_2 = 25.1^\circ$, $n_1 = 1.52$, and $n_2 = 1.33$.

Figure 2.

(A) Normalized intensity in TIRM and calculated gap width for a single sphere transitioning from Brownian to rolling. The dotted line indicates the time when the ac field is turned on. The field strength is 4.5 mT and $\Omega = 30$ Hz. The lines represent moving averages. (B) Gap width and velocity as a function of particle rotation rate (Video S2). Both velocity and gap width measurements at higher rotation rates are subject to a large experimental error. Sources of error include variable particle density, magnetic doping, surface roughness, sphericity, and size. Data represent the mean and standard deviation of $n = 25$ –450 particles.

Figure 3.

Varying gap with dc-induced load force or screening electrostatic repulsion. (A) Load force is varied $\pm 0.2 F_g$ using a 2.5 mT dc magnetic field. Velocity and gap width are shown at field frequencies of 10, 20, and 40 Hz. (B) NaCl concentration is varied to manipulate the Debye length (inset) for a field frequency of 40 Hz. Note that the data provided here are for nonsticking particles only. For (A,B), $n = 250$ –1250 measurements. (C) Translational velocity as a function of gap width for particle rotation rates of $10 < \Omega < 40$. For ac/dc samples (red triangles), the load force is $1.2 F_g$. Data represent averages and standard deviation of $n = 25$ –450 measurements. (D) Force balance between frictional force F_f and drag force F_D provides a linear relationship between the translational velocity and experimental parameters. Error bars are calculated from propagation of error in experimental measurements of v_t , Ω , and h .

Figure 4.

(A) Traction coefficient ($1 - s$) follows the predicted scaling (dotted line) for all experimental conditions. (B) Transition in slip is observed at a gap width of ~ 20 nm. Data summarize experiments across all rotation frequencies and field conditions. (C) Electron micrograph of a single colloidal particle and representative AFM image and surface roughness profile insets; the dashed line is the average height (all corrected for curvature). (D) Instantaneous velocity measurements from representative data in (B) show a stick–slip behavior at length scales less than or equal to the particle surface roughness (Video S4).