nature portfolio

Peer Review File

Electrokinetic Janus micromotors moving on topographically flat chemical patterns

Web links to the author's journal account have been redacted from the decision letters as indicated to maintain confidentiality.

15th Jun 22

Dear Dr Baraban,

Thank you for submitting your manuscript, "Electrokinetic Janus micromotors moving on topographically flat chemical patterns", to Communications Materials. It has now been seen by 2 referees, whose comments are appended below. You will see that while they find your work of interest, some important points are raised. We are interested in the possibility of publishing your study in Communications Materials, but would like to consider your response to these concerns in the form of a revised manuscript before we make a decision on publication.

We therefore invite you to revise and resubmit your manuscript, taking into account the points raised.

We are committed to providing a fair and constructive peer-review process. Please don't hesitate to contact us if you wish to discuss the revision in more detail.

When submitting your revised manuscript, please include the following:

-A response letter with a point-by-point reply to each of the referee comments and a description of changes made. Please include the complete referee report in the response letter. Please note that the response letter must be separate to the cover letter to the editors.

-A marked-up version of the manuscript with all changes to the text in a different colored font. Please do not include tracked changes or comments. Please select the file type ‘Revised Manuscript - Marked Up’ when uploading the manuscript file to our online system.

-A clean version of the manuscript. Please select the file type ‘Article File’.

-An updated Editorial Policy checklist, uploaded as a ‘Related Manuscript File’ type. This checklist is to ensure your paper complies with all relevant editorial policies. If needed, please revise your manuscript in response to these points. Please note that this form is a dynamic ‘smart pdf’ and must therefore be downloaded and completed in Adobe Reader. Clicking this link will download a zip file containing the pdf.

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citations.pdf">http://www.nature.com/authors/policies/data/data-availability-statements-data-citations.pdf.

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DATA SOURCES: We strongly encourage authors to deposit all new data associated with the paper in a persistent repository where they can be freely and enduringly accessed. We recommend submitting the data to discipline-specific, community-recognized repositories, where possible and a list of recommended repositories is provided at http://www.nature.com/sdata/policies/repositories.

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We hope to receive your revised paper within three months; please let us know if you aren't able to submit it within this time so that we can discuss how best to proceed. If we don't hear from you, and the revision process takes significantly longer, we will close your file. In this event, we will still be happy to reconsider your paper at a later date, as long as nothing similar has been accepted for publication at Communications Materials or published elsewhere in the meantime.

We understand that due to the current global situation, the time required for revision may be longer than usual. We would appreciate it if you could keep us informed about an estimated timescale for resubmission, to facilitate our planning. Of course, if you are unable to estimate, we are happy to

accommodate necessary extensions nevertheless.

Please do not hesitate to contact me if you have any questions or would like to discuss these revisions further. We look forward to seeing the revised manuscript and thank you for the opportunity to review your work.

Best regards,

Larisa Florea, PhD
Editorial Board Member
Communications Materials
orcid.org/0000-0002-4704-2393

Reviewers' comments:

Reviewer #1 (Remarks to the Author):

The present manuscript (COMMSMAT-22-0105-T) is an experimental and numerical study of active colloids moving in the vicinity of a chemically patterned substrate. In contrast with previous experimental studies, the authors prepare “topographically flat” substrates. The authors find that step-like changes in the chemical composition of the substrate can guide the motion of active colloids. For instance, the authors find that a “constriction” patterned can focus particle trajectories and align particle orientations. The authors hypothesize that active particle-induced electro-osmotic flows on the substrate are responsible for these behaviors. This hypothesis is supported by continuum calculations in Comsol. Additionally, the authors reproduce the phenomenology in simulations of active Brownian particles with soft potentials.

The experimental system under consideration is novel, and the results are interesting. Most experimental studies on active particle motion in patterned environments have considered topographical patterning. To my knowledge, this is the first to consider a chemically patterned “flat” substrate. The manuscript may be suitable for publication after significant revision, as detailed below.

A major oversight here is that the authors fail to cite the extensive record of theoretical papers on this very idea. For instance, Chiang and Velegol (Langmuir 2014) considered localized electro-osmosis as a possible explanation for the size-dependence of the speed of Au/Pt micromotors. Although that work considered uniform substrates, Uspal et al. (Phys. Rev. Lett., 2016) proposed controlling particle motion via chemical patterning and particle-induced chemi-osmotic flows. This idea was thoroughly investigated in subsequent works, e.g., Uspal et al., J. Chem. Phys, 2019, and behaviors like reflection and crossing (at a step-like interface) and focusing (to the center of a stripe) were predicted. Although those works assumed self-diffusiophoretic motion, the governing equations are identical to the governing equations for a linearized model of self-eletrophoresis (e.g., Ref. 17 in the present manuscript); the only necessary modifications are a suitable relabeling of variables (e.g., from concentration “c” to potential \phi) and adjustment of the boundary condition on the particle surface. Overall, the present manuscript should not be published without incorporation of these citations and appropriate discussion in the Introduction.

In the Introduction, the authors motivate the study by making an extended analogy between micromotors moving near a patterned substrate (a microscale phenomenon) and ions moving in ion channels (nano/molecular scale). I find this framing odd and rather strained. What is a motile / self-propelled ion, i.e., the molecular equivalent to active colloids? (Nevertheless, if the authors want to proceed with this framing, I will not insist on revising it.)

More detailed comments and questions follow:

• The authors write: "..microspheres exhibit positive Zeta potentials (\zeta = +27 mV)." Since the Janus particle surface comprises different materials, I would expect a zeta potential contrast between the two "faces." Therefore, is the quantity given here a surface-averaged measurement? What method is used to measure the zeta potential? Have the authors tried measuring the zeta potentials of PS and the coating material separately? I infer that the Comsol simulations assume uniform zeta potential on the JP. This may affect the results.
• What are the units in Fig. 1D?
• Fig. 1F is misleading and needs to be revised. Do these curves represent actual data, or is this a schematic illustration of a concept? Misleadingly, it looks like crossing probability is a discontinuous function of height.
• Fig. 1G needs error bars.
• In Fig. 3, how do the authors infer three-dimensional motion? Do they observe the particles going out of focus?
• What are the solid curves in Fig. 3G-I? Are these to guide the eye?
• Fig. 4F and Fig. 6C need error bars. If the error bars are smaller than the marker size, that should be noted in the caption.
• If the dashed line in Fig. 6C is only there to guide the eye, it should not be called a "fitting," as fitting implies that there is some function that is fitted to the data.
• The substrates are not perfectly flat, due to the 5 nm thickness of the gold layer. The authors argue that this finite thickness is irrelevant, due to the substantially larger (200 nm) Debye length in this system. This is plausible, but this argument needs to be moved to the main text, not buried in Methods.
• Eq. 1, line 178, seems to have gone rogue in the active colloids literature. I checked the references given here (Refs. 26 and 27), and neither has an actual derivation of this equation. In fact, I believe this equation comes from Keh & Anderson, J. Fluid Mech., 1985; see Eq. 4.7c there. Notably, this equation assumes a colloid with a uniform zeta potential, and the electric field E_inf is the strength of an externally maintained uniform field (i.e., the field asymptotes to E_inf far from the particle.) Is this equation still valid for a Janus colloid (which may have non-uniform zeta potential) and a self-generated electric field? Please find suitable citations, if possible.
• In the SI, remove the "Type or paste text here" paragraph.
• In the SI, the electric field is the positive gradient of the potential, contrary to the usual convention. Please confirm that this is not a typo.
• In the SI, the dot product is missing in the incompressibility condition.
• In the SI, please provide a complete specification of the governing equations and boundary conditions solved in Comsol. In particular, I note that the boundary conditions for the potential and the fluid velocity are not specified. The paper should not be published without this information, as the calculations cannot be reproduced without it. For instance, is the particle assumed to have zero translational and angular velocity?
• In Fig. S1b, the particles take unusually long time (from a hydrodynamics standpoint) to reach steady velocity. This is most likely due to some timescale of the light-activated reaction (compare

Sridhar et al., PNAS, 2020). When a particle crosses a chemical step, does the adjustment of particle speed take comparably long?
• It is curious that the ABP model can recover the behavior, since there is no a priori reason that the substrate-driven flows should be the gradient of a potential. For instance, one can easily imagine a vortex (ring-like) flow with closed streamlines driven by electro-osmotic flow. Obviously, such a flow cannot be the gradient of a potential. Therefore, I was somewhat bemused by all the references to "potential" in the text (before the ABP model was even mentioned!) Nevertheless, I suppose this language is acceptable for a materials journal.

Minor / typographical issues:
• "tuning the direction" on p. 7. Should this be "turning the direction"?
• Line 469, "trajectory shoes" should be "trajectory shows"

Reviewer #2 (Remarks to the Author):

Inspired by the long-range potentials established in nanofluidic systems for control over the ion-selective transport, the authors demonstrate that topographically complex patterns containing regions of positive and negative Z potentials can affect the motion of charged Janus colloids. This is an original approach that will allow a higher control over the trajectories of chemically propelled micro- and nano swimmers for microfluidic applications. The paper is outstanding and well written. Experiments have been carefully designed and the conclusions are well supported by the experimental results. The focusing experiment is also impressive. I highly recommend the publication of this article after a minor clarification:

- What happens when the surface exhibits a close-to-zero Z-potential? An interesting experiment could be done (although it is not necessary) by functionalizing the surface with an amphiphilic surface.

Response to the reviewers' comments

We thank the Reviewers for providing valuable remarks which we have used to refine the manuscript. All the revisions are indicated in blue in the revised manuscript. Our itemized responses to all the reviewers' comments are enclosed below.

Reviewer #1 (Remarks to the Author):

The present manuscript (COMMSMAT-22-0105-T) is an experimental and numerical study of active colloids moving in the vicinity of a chemically patterned substrate. In contrast with previous experimental studies, the authors prepare "topographically flat" substrates. The authors find that step-like changes in the chemical composition of the substrate can guide the motion of active colloids. For instance, the authors find that a "constriction" patterned can focus particle trajectories and align particle orientations. The authors hypothesize that active particle-induced electro-osmotic flows on the substrate are responsible for these behaviors. This hypothesis is supported by continuum calculations in Comsol. Additionally, the authors reproduce the phenomenology in simulations of active Brownian particles with soft potentials.

The experimental system under consideration is novel, and the results are interesting. Most experimental studies on active particle motion in patterned environments have considered topographical patterning. To my knowledge, this is the first to consider a chemically patterned "flat" substrate. The manuscript may be suitable for publication after significant revision, as detailed below.

> **Reply:** We thank the Reviewer for his/her positive assessment of our work and recognizing that the system under consideration is novel, the results are interesting, and that the work is suitable for publication after a revision. The manuscript has been revised, carefully taking in account all the comments and suggestions of the Reviewers.

1. A major oversight here is that the authors fail to cite the extensive record of theoretical papers on this very idea. For instance, Chiang and Velegol (Langmuir 2014) considered localized electro-osmosis as a possible explanation for the size-dependence of the speed of Au/Pt micromotors. Although that work considered uniform substrates, Uspal et al. (Phys. Rev. Lett., 2016) proposed controlling particle motion via chemical patterning and particle-induced chemi-osmotic flows. This idea was thoroughly investigated in subsequent works, e.g., Uspal et al., J. Chem. Phys, 2019, and behaviors like reflection and crossing (at a step-like interface) and focusing (to the center of a stripe) were predicted. Although those works assumed self-diffusiophoretic motion, the governing equations are identical to the governing equations for a linearized model of self-eletrophoresis (e.g., Ref. 17 in the present manuscript); the only necessary modifications are a suitable relabeling of variables (e.g., from concentration "c" to potential \phi) and adjustment of the boundary condition on the particle

surface. Overall, the present manuscript should not be published without incorporation of these citations and appropriate discussion in the Introduction.

Reply: We thank the Reviewer for mentioning these important references. We implemented the explanatory sentences into the introduction of the revised version of the manuscript.

The new text is introduced on page 3, line 66-68 of the revised manuscript, as follows: In this respect, Uspal et al. theoretically proposed to considered chemically patterned surfaces for catalytic Janus particle and predicted a variety of interaction scenarios, including reflection of particles from the boundary, hovering, and sliding along the boundary (*15-17*).

2. In the Introduction, the authors motivate the study by making an extended analogy between micromotors moving near a patterned substrate (a microscale phenomenon) and ions moving in ion channels (nano/molecular scale). I find this framing odd and rather strained. What is a motile / self-propelled ion, i.e., the molecular equivalent to active colloids? (Nevertheless, if the authors want to proceed with this framing, I will not insist on revising it.)

Reply: charged species, e.g., ions and molecules are motile due to the intense Brownian diffusion, when the typical displacements exceed their linear dimensions. This resembles the behavior of active particles (governed by active diffusion). Further, transport of the ions/molecules is governed (i.e., in this case directed) by the transmembrane electric potentials. Here we see certain similarity between the ionic and colloidal systems.

3. The authors write: "..microspheres exhibit positive Zeta potentials (\zeta = +27 mV)." Since the Janus particle surface comprises different materials, I would expect a zeta potential contrast between the two "faces." Therefore, is the quantity given here a surface-averaged measurement? What method is used to measure the zeta potential? Have the authors tried measuring the zeta potentials of PS and the coating material separately? I infer that the Comsol simulations assume uniform zeta potential on the JP. This may affect the results.

Reply: A Malvern Zetasizer Nano ZSP was used to measure the Zeta Potential of the Janus particles based on electrophoretic effect of charged particles in an applied electric field. It is a surface-average measurement. The Janus particle was based on polystyrene sphere, and one side was covered Ag/AgCl complexes. Indeed, we measured the zeta potential of the PS particles separately, as control. Adding a Ag/AgCl layer shifts the value of the Zeta potential towards more neutral values. Finally, when a very thin layer iron oxide-hydroxide crystals (β-FeOOH) covers the whole surface of Janus particle (See Figure 2A), the particles appear to be positively charged (*Zeta = +27 mV*). Thus, we assume the zeta potential of fresh Janus particle is uniform. After some time under light illumination, an Ag ($4AgCl+2H_2O = 4Ag+4H^+ +4Cl^- +O_2$) layer was deposit back on the surface of the Janus particle, then the Zeta potential of the Janus particle becomes non-uniform.

The Zeta potential of pure iron oxide-hydroxide crystals (β-FeOOH), Ag/AgCl complex and PS particle are +40.3 *mV*, -23 mV,-17.5mV, respectively.

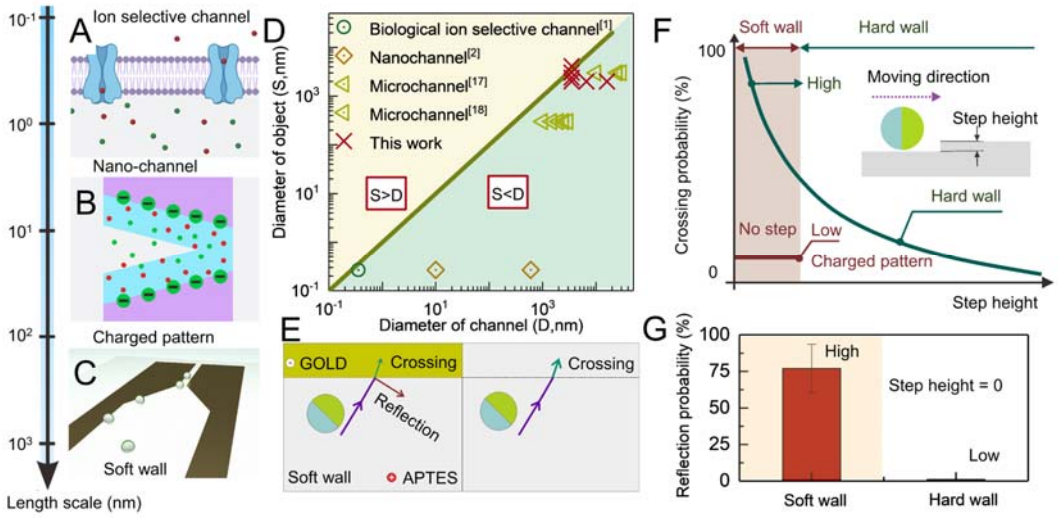
In the COMSOL model, Janus particle is fixed, it does not move, and the Janus particle surface is set as no slip boundary. The charged pattern substrate is set as electroosmotic velocity boundary condition. We only model the electroosmotic flow on the APTES/Gold charged pattern. The electroosmotic flow on the APTES/Gold charged pattern is affected by electric field generated by a Janus particle and the Zeta potential of the charged pattern.

4. What are the units in Fig. 1D?

Reply: We thank the Reviewer for this observation. We have revised Figure 1D. The unit is nanometer (nm).

5. Fig. 1F is misleading and needs to be revised. Do these curves represent actual data, or is this a schematic illustration of a concept? Misleadingly, it looks like crossing probability is a discontinuous function of height. Fig. 1G needs error bars.

Reply*:* We update the Figure 1F and add the error bars in Figure 1G. The update Figure 1 is shown as follows:



6. In Fig. 3, how do the authors infer three-dimensional motion? Do they observe the particles going out of focus?

Reply: We observed Janus particles moving out of focus, indeed. We refer to Movie S2. Figure 3E schematically describes the motion of Janus particles near the charged pattern interface.

7. What are the solid curves in Fig. 3G-I? Are these to guide the eye?

Reply: The blue solid line in Fig.3G-1 are guide to the eye. We clarify this in Figure 3 caption of the revised manuscript.

8. Fig. 4F and Fig. 6C need error bars. If the error bars are smaller than the marker size, that should be noted in the caption.

> **Reply**: We thank the Reviewer for pointing this out. We update Figure 4F and revised Figure 6C accordingly.

In Figure 4F, there are three Janus particles. Each Janus particle has a different initial speed and distance to the charged interface. Therefore, we did not plot the average speed plus error bar of these three particles, rather, we plotted three individual curves of these three Janus particles.

9. If the dashed line in Fig. 6C is only there to guide the eye, it should not be called a "fitting," as fitting implies that there is some function that is fitted to the data.

> **Reply:** The corresponding correction was introduced in the figure caption to make these statements clear.

10. The substrates are not perfectly flat, due to the 5 nm thickness of the gold layer. The authors argue that this finite thickness is irrelevant, due to the substantially larger (200 nm) Debye length in this system. This is plausible, but this argument needs to be moved to the main text, not buried in Methods.

> **Reply:** We agree with the Reviewer (actually, we initially mentioned this in the main text), and we added a sentence in the revised manuscript. The changes are introduced on page 5, line 118 to line 123.

11. Eq. 1, line 178, seems to have gone rogue in the active colloids literature. I checked the references given here (Refs. 26 and 27), and neither has an actual derivation of this equation. In fact, I believe this equation comes from Keh & Anderson, J. Fluid Mech., 1985; see Eq. 4.7c there. Notably, this equation assumes a colloid with a uniform zeta potential, and the electric field E_inf is the strength of an externally maintained uniform field (i.e., the field asymptotes to E_inf far from the particle.) Is this equation still valid for a Janus colloid (which may have non-uniform zeta potential) and a self-generated electric field? Please find suitable citations, if possible.

> **Reply:** We thank the Reviewer for this comment. We changed the text accordingly. The changes are introduced on page 7, line 183 to line 187.
>
> We thank the Reviewer for this comment. We changed the text, as follows: The speed of a charged Janus particle is determined by a superposition of electrophoresis of charged particle itself and the electro-osmotic flow caused by the charged wall. The speed of colloid micromotors undergoing ionic diffusiophoresis is proportional to the difference in Zeta potential between the colloid and the wall: $U \propto (\zeta_c - \zeta_w)$, where ζ_c and ζ_w are Zeta-potentials of colloidal micromotor and nearby charged wall (12, 31-33).
>
> The changes are introduced on page 7 line 182 to line 184.
>
> As mentioned by the Reviewer, the equation from Keh & Anderson et al (Journal of Fluid Mechanics 153 (1985): 417-439. Eq. 4.7c) based on the assumption of uniform particles Zeta potential. Velegol et al. (Langmuir 30.10 (2014): 2600-2607.) and Zhang et al.(Phys. Rev. Lett. 117, 198001) simply chose zeta potential to be uniform over

the entire surface of catalytic micromotor and calculated the electrophoretic speed of the micromotor and electroosmotic flow near charged surface.

12. In the SI, remove the “Type or paste text here” paragraph. In the SI, the electric field is the positive gradient of the potential, contrary to the usual convention. Please confirm that this is not a typo.

Reply: Thank you for pointing out the incorrect wording. We addressed this issue in the updated supporting information.

13. In the SI, the dot product is missing in the incompressibility condition.

Reply: Thank you for pointing this out. We addressed these issues in the revised manuscript.

14. In the SI, please provide a complete specification of the governing equations and boundary conditions solved in COMSOL. In particular, I note that the boundary conditions for the potential and the fluid velocity are not specified. The paper should not be published without this information, as the calculations cannot be reproduced without it. For instance, is the particle assumed to have zero translational and angular velocity?

Reply: We thank the Reviewer for this comment. We added all the boundary conditions and parameters used for COMSOL simulation. The Janus particle position is fixed in the COMSOL model. Thus, the Janus particle is assumed to have zero translational and angular velocity.

15. In Fig. S1b, the particles take unusually long time (from a hydrodynamics standpoint) to reach steady velocity. This is most likely due to some timescale of the light-activated reaction (compare Sridhar et al., PNAS, 2020). When a particle crosses a chemical step, does the adjustment of particle speed take comparably long?

Reply: The Janus particle moves due to the decomposition of AgCl. The consumption of AgCl reduces the amount of available AgCl at the particle surface. On the one hand, the speed decay is due to the limited amount of AgCl at the surface. One the other hand, AgCl deposition reaction leads to some Ag formation ($4AgCl+2H_2O = 4Ag+4H^+ +4Cl^- +O_2$) and grew into larger crystals and blocked AgCl underneath from further reactions.

When a Janus particle crosses a charged pattern interface, it goes out of focus (see Figure 3, C, F, I), then it is hard to long-time track and analyze the speed of the Janus particle. However, when silver chloride is about to be consumed, Janus particles are less active anymore and stick to the APTES/ Gold charged pattern interface.

16. It is curious that the ABP model can recover the behavior, since there is no a priori reason that the substrate-driven flows should be the gradient of a potential. For instance, one can easily imagine a vortex (ring-like) flow with closed streamlines driven by electro-osmotic flow. Obviously, such a flow cannot be the gradient of a potential. Therefore, I was somewhat bemused by all the references to “potential” in the text (before the ABP model was even mentioned!) Nevertheless, I suppose this language is acceptable for a materials journal.

Reply: We thank the Reviewer for this comment. Indeed, when talking of the interaction of particles with the interface we assumed the presence of “interaction

forces" that are gradients of "interaction potentials" whatever nature: electrostatic or electroosmotic. This is indeed the approach used in the ABP model. However, even in the above case of a circular flow, although induced by moving ions and not directly by any related potential, this flow can be considered as a "secondary effect" of a potential that drives the ions that in turn drive the flow (EOF). The online source provides the definition of EOF with a reference to a potential: *"Electroosmotic flow (or electro-osmotic flow, often abbreviated EOF; synonymous with electroosmosis or electroendosmosis) is the motion of liquid induced by an applied potential…"* https://en.wikipedia.org/wiki/Electro-osmosis

We agree with the comment of the Reviewer and we of course distinguish flows generated by a gradient of a potential and EOF, and we refer to "potential" as to a convenient "generalized" concept in the text, as explained above.

17. Minor / typographical issues:

• "tuning the direction" on p. 7. Should this be "turning the direction"?

• Line 469, "trajectory shoes" should be "trajectory shows"

Reply: Thank you for pointing out the incorrect wording. We addressed these issues in the revised manuscript.

Reviewer #2:

Inspired by the long-range potentials established in nanofluidic systems for control over the ion-selective transport, the authors demonstrate that topographically complex patterns containing regions of positive and negative Z potentials can affect the motion of charged Janus colloids. This is an original approach that will allow a higher control over the trajectories of chemically propelled micro- and nano swimmers for microfluidic applications. The paper is outstanding and well written. Experiments have been carefully designed and the conclusions are well supported by the experimental results. The focusing experiment is also impressive. I highly recommend the publication of this article after a minor clarification:

Reply: We thank the Reviewer for his/her very positive assessment of our work and recognizing the originality of the approach. The manuscript has been revised carefully taken in account all the comments and suggestions of the Reviewers.

1. What happens when the surface exhibits a close-to-zero Z-potential? An interesting experiment could be done (although it is not necessary) by functionalizing the surface with an amphiphilic surface.

Reply: We thank the Reviewer for this interesting idea. In our previous work (The European Physical Journal E 44.3 (2021): 1-11.), we investigated positively charged Janus particles moving on positively charged surface with different zeta potential. We found clear signs of a motor-substrate coupling in terms of speed, and a correlation with the Zeta potential of the substrate. That is, the ionic species released by the decomposition of AgCl form a gradient along the charged substrate, leading to a fluid flow along with it. This phenomenon is known from the work of Anderson (Rev. Fluid Mech. 21, 61 (1989), J. Fluid Mech. 153, 417 (1985), J. Fluid Mech. 194, 377 (1988)),

where it was shown that for externally imposed ion gradients speed of a colloid undergoing ionic diffusiophoresis is proportional to the difference in Zeta potential between the colloid and the wall: $U \propto (\zeta_c - \zeta_w)$, where ζ_c is the colloid's ζ-potential and ζ_w is that of the wall.

26th Jul 22

Dear Dr Baraban,

Your manuscript titled "Electrokinetic Janus micromotors moving on topographically flat chemical patterns" has now been seen again by Reviewer 1, whose comments appear below. In light of their advice I am delighted to say that we are happy, in principle, to publish a suitably revised version in Communications Materials under the open access CC BY license (Creative Commons Attribution v4.0 International License).

We therefore invite you to edit your manuscript to comply with our journal policies and formatting style in order to maximise the accessibility and therefore the impact of your work.

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We hope to hear from you within two weeks; please let us know if the process may take longer.

Best regards,

Larisa Florea, PhD
Editorial Board Member
Communications Materials
orcid.org/0000-0002-4704-2393

REVIEWERS' COMMENTS:

Reviewer #1 (Remarks to the Author):

The authors have answered all queries and made suitable revisions to the manuscript. I recommend publication with no additional changes.