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Dear Dr Husein,

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Your manuscript is now at our initial Technical Check stage, where we look for adherence to the journal's submission guidelines, including any relevant editorial and publishing policies. If there are any points that need to be addressed prior to progressing we will send you a detailed email. Otherwise, your manuscript will proceed into peer review.

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Regards,
f.zahednezhad, PHD student,

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Subject: Ionics: Decision on your manuscript

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Dear Dr Husein,
Your manuscript, "Bismuth oxides nanostructure supported on Cu-foam as efficient electrocatalyst toward carbon dioxide electroreduction", has now been assessed.

We invite you to revise your paper, taking into account the points raised and the general guidelines below. When your revision is ready, please submit it via:

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Kind regards,

Werner Weppner
Editor
Ionics

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Once you have revised your paper, the submitter Ismail Husein can use the following link to submit it:

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REVIEWER REPORTS

Reviewer Comments:

Reviewer 1

Attachments:
• https://reviewer-feedback.springernature.com/download/attachment/72d4d31d-940b-4ab0-8830-e738637f81e0

Reviewer 2
In this manuscript, Bi nanostructures on Cu foam through in-situ chemical oxidation reaction are synthesized, which are applied to CO2 electrochemical reduction reaction. The prepared Bi electrode has large accessible surface area, high porosity, and high conductivity and presents good formate production. The catalyst showed a high FE (>90%) for formate production during CO2ER. However, the preparation mechanism of this catalyst, the true active site of activity, and the electrochemical test conditions need to be further explained. In addition, the language should be further polished since there are many mistakes. Therefore, this manuscript needs a major revision before the publication.

1. The abstract contains few valid information and has logical errors. For example, the first sentence of the abstract “such as carbon dioxide conversion reaction and photocatalytic reaction”, the two reactions are not comparable.
2. The English of this manuscript needs to be polished thoroughly.
3. More relevant references are needed and the format should be uniform.
4. In the first paragraph of result, what is“GE process”?If it is the abbreviation of galvanic exchange, it should be indicated at its first appearance.
5. In Fig. 3b, “a small charge transfer obtained for CO2RR process with lower impedance value (~8Ω.cm2) than one for HER (~38 Ω.cm2)”, the resistance value here needs to be recalculated, and the test conditions for EIS need to be clearly written.
6. There are many discussions which lack necessary data support. For example, in line 2-5 of page 7, the author simply judged from LSV that after cyclic voltammetry pretreatment, most Bi sites exposed on the surface of copper foam have been transformed into metallic states. Such a judgment lacks relevant data support. So I suggest the author conduct some necessary characterization of the treated samples.
7. As for the section of results and discussion in the manuscript, there are some discussions that do not correspond to the data presented or not presented in the text. It should be noted that when discussing the relevant data, show it to readers at an appropriate position. As shown in lines 18 to 21 of page 8 of the manuscript, the catalytic activity of CO2RR for formate production at different potentials (-1.4 V to -3 V vs. Ag/AgCl) described by the authors is lacking.
8. There are some problems in the manuscript such as sample processing and unclear data expression. I suggest the author indicate the sample processing method corresponding to the data and replace the ‘ optimized Bi growth on Cu support’ expression in the text.
9. For lines 17 to 20 on page 9 of the manuscript, the authors state that the effect of copper foam on CO2RR is negligible because it is covered by thick Bi nanomedles, implying that high performance is only related to the Bi nanostructure. However, the conclusion presented here lacks evidence. In fact, CO2 diffuses to the copper foam

base during the reaction process and may be reduced. I suggest that the author compare the efficiency of all products in the Bi nanoneedles CO2RR of different thicknesses, and provide the corresponding characterization of samples before and after the test.
10. For lines 16 to 23 on page 12 of the manuscript, the authors indicate that the catalyst has an ultra-fine nanoneedle shape, with surface morphology will be converted over time after 50 hours of stability testing. I suggest that the author take samples of different test time for approximate in-situ characterization to finally determine the reliability of this conclusion. In addition, the hypothesis that the formation of Bi2O2CO3 is a possible reason for surface reconstruction.
11. For lines 3 to 5 on page 15 of the manuscript, the authors show that the highly efficient active sites on the Bi-Cu catalyst increase the electrode surface charge transfer phenomena, while the strong contact between substrate and catalyst layer enhances the electron transfer effects, and largely available active surface area. The discussion here lacks relevant data support, so I suggest that the author provide the ECSA measurement results of different samples.
12. As for the efficiency of the CO2RR main product of the Bi-Cu electrode material shown by the authors, in fact, a small amount of CH4 products will be produced for the pure Bi catalyst under normal test conditions, and even multi-carbon products will be produced for the pure Cu-based catalyst. It is suggested that the authors confirm other products of the Bi-Cu catalyst or provide the GC and NMR data in the manuscript.
13. For the SEM images of several samples shown by the author in Figure1, I suggest that the author compare the results at the same magnification scale, especially for the SEM images of Bi growth on Cu foam.
14. As for the CO2RR performance reported by different literatures compared in manuscript Table1, there was a mistake in the Formate selectivity (%) column of Bi Nps line. It is suggested that the author change ‘t0’ to ‘to’.
15. As for the durability test in manuscript Figure4, (a) and (b), the author believes that FE at the beginning of the test is about 92%, and there is basically no change within 50 hours, indicating that selectivity has not decreased over a long period of time. However, the FE of Format dropped below 88% after 50 hours of reaction and further decreased after 70 hours of reaction. In fact, the drop in yield from 92% to 88% does not seem very significant, and I recommend that the authors extend the test time to a drop of about 10% to determine the durability of the sample. The same is also recommended for stability tests at high current densities of 50 mA.cm-2. In addition, for the morphology of samples before and after the reaction in (c) and (d), I suggest that the author use the results of the same SEM scale for comparison.

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Regards,
f.zahednezhad, PHD student,
Islamic Azad University.

Response letter to reviewer comments.docx.pdf
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Dear Dr Husein,

Re: "Bismuth oxides nanostructure supported on Cu-foam as efficient electrocatalyst toward carbon dioxide electroreduction"

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Werner Weppner
Editor
Ionics

Reviewer Comments:

Reviewer 1
All comments have been addressed

Reviewer 2
The authors add more data to improve the quality of manuscript, now it can be accepted.

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Regards,
f.zahednezhad, PHD student,
Islamic Azad University.

Response letter to referees' comments on manuscript ID: c7e18cad-94ba-4c0d-9c3e-766d86a5dcaa

Bismuth oxides nanostructure supported on Cu-foam as efficient electrocatalyst toward carbon dioxide electroreduction

Dear Prof. Werner Weppner,

The authors would like to thank you and the referees for their comments and suggestions to improve the quality of the work. The manuscript was carefully revised to fulfil the expectation of the referees. A point-to-point response is explained below.

We hope that this revision fully addresses your concerns. I am looking to receive your positive response.

Sincerely yours,

Ismail Husein

a.l2022@yahoo.com

Reviewer #1:

This manuscript reports the galvanic exchange methods to synthesize Bi nanostructure on a Cu substrate for CO_2 reduction. The electrode shows good catalytic performances toward CO_2 reduction reaction and formate production. And using the synthetic method for Bi nanostructure is interesting. However, there is misleading descriptions on material properties and electrochemical aspects. In this regard, the reviewer would like to recommend the publication of this manuscript after minor revision. The detailed comments are as follows.

Response: Thank you for taking the time to read through the manuscript and for providing your valuable feedback. Your positive input is greatly appreciated.

1. In the Abstract, authors described CO_2 reduction in a neutral environment with high stability is challenging. In general, basic environment can help to reduce overpotentials for CO_2 reduction, while catalysts are likely to be more stable in neutral conditions because hydroxide ions can act as a ligand for metal complexation. Authors need to describe the stability issue well.

- ✓ Thanks for your precise points. We do agree with you completely. Based on your explanation we have tried to provide more explanation on Bi stability issue to point it out and discuss it better. The changes have highlighted with yellow mark in the MS.

2. Based on standard reduction potentials for Cu and Bi metals, it seems that the reduction of Cu is easier than that of Bi, as authors mentioned. However, the reduction potential also depends on the activity of metal ions (e.g. concentration). Could you explain how the Bi reduction occurs spontaneously based on the Nernst equation.

- ✓ Yes, you are correct that the standard reduction potential alone does not fully determine the spontaneity of a redox reaction. The Nernst equation takes into account the concentration of the reactants and products and allows us to calculate the actual cell potential, which determines the spontaneity of the reaction.
  The Nernst equation is:

  $E = E° - (RT/nF) * \ln(Q)$

  where E is the cell potential, E° is the standard cell potential, R is the gas constant, T is the temperature in kelvin, n is the number of electrons transferred, F is Faraday's constant, and Q is the reaction quotient.

  In the case of the reduction of Bi, we can write the half-reaction as follows:

  $Bi^{3+} + 3e^- \rightarrow Bi(s)$

  The standard reduction potential for this reaction is -0.279 V. However, the actual cell potential depends on the concentrations of Bi^{3+} and Bi in the reaction mixture. If the concentration of Bi^{3+} is high enough, the reaction can occur spontaneously.

  For example, let's assume that the concentration of Bi^{3+} in the reaction mixture is 1 M and the concentration of Bi is 1×10^{-10} M. The reaction quotient, Q, is then:

  $Q = [Bi(s)] / [Bi^{3+}]^3$
  $Q = (1 \times 10\text{-}^{10}) / (1)^3$
  $Q = 1 \times 10^{-10}$

  Substituting the values into the Nernst equation, we get:

  $E = -0.279\ V - (0.0257\ V/K) * \ln(1 \times 10^{-10})$
  $E = -0.279\ V - (-70.8\ V)$
  $E = 70.521\ V$

  The positive value for E indicates that the reaction is spontaneous and Bi will be reduced to Bi metal. Therefore, even though the standard reduction potential for Bi is more positive than that of Cu, Bi can still be reduced spontaneously under

certain conditions, as determined by the Nernst equation. We added this part of our explanation in the MS, as it is highlighted in yellow mark.

3. Discuss the oxidation state of Bi based material during CO2 reduction. As authors mentioned, Bi is vulnerable to oxidation reaction by outer environments. For this, I would like to recommend recent articles: https://doi.org/10.1021/acscatal.7b03242

✓ Thanks for your suggestion. We have compared our discussion to the findings presented in this paper, and there appears to be a good level of agreement between the two.

4. At the OCP condition, it is difficult to drive charge transfer. As a result, the charge transfer resistance should be very high. It is assumed that the single circle observed in Figure 3a originates from charge transfer behavior. To clearly discuss mass transfer process from the Nyquist plot, this should be fitted well with semicircles. For this, I would like to recommend recent articles: https://doi.org/10.1016/j.apcatb.2022.122095

✓ Thanks for your concise point. We have added the suggested paper as a potential reference list and included some the discussion of that paper in our MS. We also would like to thank you for suggesting of fitting the data, however, the aim of providing EIS at this condition was to compare the two different condition and get some clear idea what happens when we have the main reaction is HER or CO_2 electroreduction. And the effect of Bi on top of Cu surface for CO_2 and HER reactions. Which is quite observable from our tested results.

5. In this manuscript, there are many grammatical mistakes which have to be improved.

✓ Thanks, revised. We have further checked the MS and polished its English writing.

Reviewer #2:

In this manuscript, Bi nanostructures on Cu foam through in-situ chemical oxidation reaction are synthesized, which are applied to CO2 electrochemical reduction reaction. The prepared Bi electrode has large accessible surface area, high porosity, and high conductivity and presents good formate production. The catalyst showed a high FE (>90%) for formate production during

CO2ER. However, the preparation mechanism of this catalyst, the true active site of activity, and the electrochemical test conditions need to be further explained. In addition, the language should be further polished since there are many mistakes. Therefore, this manuscript needs a major revision before the publication.

Response: Thank you for allocating time to read out the manuscript and for your positive input.

1. The abstract contains few valid information and has logical errors. For example, the first sentence of the abstract “such as carbon dioxide conversion reaction and photocatalytic reaction”, the two reactions are not comparable.

   ✓ Thanks, revised. The sentence is rewrite and highlighted.

2. The English of this manuscript needs to be polished thoroughly.

   ✓ We have thoroughly polished the paper and changed or rewrite some part of it. Which all are highlighted.

3. More relevant references are needed and the format should be uniform.

   ✓ Thanks, revised. Some other references added to the list, and modified cited bibliography.

4. In the first paragraph of result, what is “GE process”? If it is the abbreviation of galvanic exchange, it should be indicated at its first appearance.

   ✓ Thanks, revised.

5. In Fig. 3b, “a small charge transfer obtained for CO2RR process with lower impedance value (~8Ω.cm2) than one for HER (~38 Ω.cm2)”, the resistance value here needs to be recalculated, and the test conditions for EIS need to be clearly written.

   ✓ Thanks, revised. There were minor mistakes in calculations, we have rechecked it and modified it. Also, the way we did EIS and its details are provided in the MS.

6. There are many discussions which lack necessary data support. For example, in line 2-5 of page 7, the author simply judged from LSV that after cyclic voltammetry pretreatment, most Bi sites exposed on the surface of copper foam have been transformed into metallic states. Such a judgment lacks relevant data support. So I suggest the author conduct some necessary characterization of the treated samples.

   ✓ To provide further evidence, it is necessary to perform in situ characterization. However, we do not currently have access to this type of characterization. But instead we have done a quick check of XPS to provide further information on Bi state inside structure. We hope this could address this issue. We have tried to do XPS after reaction however, we got the same XPS spectrum, we believe that this is due to the susceptibility of the Bi to oxidation and that is why we only observe Bi oxide. So to further ignore any misleading discussion we have changed our claim and made it more reasonable.

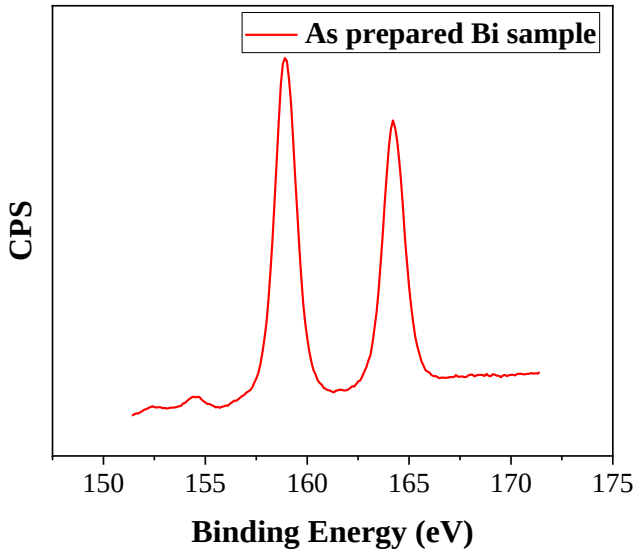

Figure S1: The XPS spectrum of Bi sample.

7. As for the section of results and discussion in the manuscript, there are some discussions that do not correspond to the data presented or not presented in the text. It should be noted that when discussing the relevant data, show it to readers at an appropriate position. As shown in lines 18 to 21 of page 8 of the manuscript, the catalytic activity of CO2RR for formate production at different potentials (-1.4 V to -3 V vs. Ag/AgCl) described by the authors is lacking.

   ✓ Thanks, revised. I think this mistake was made during sending to our other colleagues for their review and comments. Thanks for your preciseness. We have corrected it.

8. There are some problems in the manuscript such as sample processing and unclear data expression. I suggest the author indicate the sample processing method corresponding to the data and replace the ‘optimized Bi growth on Cu support’ expression in the text.

   ✓ Revised. To make everything consistent we have changed the naming of our samples.

9. For lines 17 to 20 on page 9 of the manuscript, the authors state that the effect of copper foam on CO2RR is negligible because it is covered by thick Bi nanomedles, implying that high performance is only related to the Bi nanostructure. However, the conclusion presented here lacks evidence. In fact, CO2 diffuses to the copper foam base during the reaction process and may be reduced. I suggest that the author compare the efficiency of all products in the Bi nanoneedles CO2RR of different thicknesses, and provide the corresponding characterization of samples before and after the test.

   ✓ You are completely correct. That is why we have tried to provide the result for different growth time of Bi. We have put the product distribution graph for the other samples in SI for more information.

10. For lines 16 to 23 on page 12 of the manuscript, the authors indicate that the catalyst has an ultra-fine nanoneedle shape, with surface morphology will be converted over time after 50 hours of stability testing. I suggest that the author take samples of different test time for approximate in-situ characterization to finally determine the reliability of this conclusion. In addition, the hypothesis that the formation of Bi2O2CO3 is a possible reason for surface reconstruction.

    ✓ In situ characterization is one of the important thing that we should accompany our work with. But unfortunately as we stated above, we do not have access to this type of characterization. However, to make our discussion reliable we have cited to the most recently published papers which they have discussed a similar trend. Also the formation of Bi2O2CO3 is a possible explanation, however, Bi oxidation and migration is the proven mechanism for the surface reconstruction. So, that is why we also believe that Bi migration besides oxidation is the most possible explanation for the surface reconstruction after long time reaction.

11. For lines 3 to 5 on page 15 of the manuscript, the authors show that the highly efficient active sites on the Bi-Cu catalyst increase the electrode surface charge transfer phenomena, while the strong contact between substrate and catalyst layer enhances the electron transfer effects, and largely available active surface area. The discussion here lacks relevant data support, so I suggest that the author provide the ECSA measurement results of different samples.

    ✓ We have applied ECSA for three conditions of Cu/Bi_x samples and compared their CV curve at a single scan rate for better comparison.

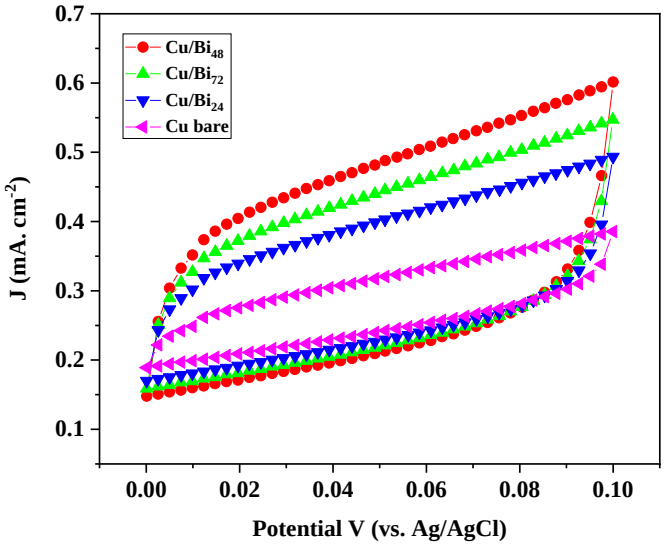

Figure S3: CV demonstration for the different samples at the same scan rate (50 mV/s) to demonstrate their surface area and capacity of the samples for electrocatalysis process.

12. As for the efficiency of the CO2RR main product of the Bi-Cu electrode material shown by the authors, in fact, a small amount of CH4 products will be produced for the pure Bi catalyst under normal test conditions, and even multi-carbon products will be produced for the pure Cu-based catalyst. It is suggested that the authors confirm other products of the Bi-Cu catalyst or provide the GC and NMR data in the manuscript.

✓ Yes, you are completely right. We have provided the minor product distribution in Figure S2 of the SI section. For the Cu/Bi sample there a small amount of CH_4 and C_2H_4 however, for bare Cu sample we observed some of them. Their amount was negligible that is why we did not provide them in our initial submission.

13. For the SEM images of several samples shown by the author in Figure1, I suggest that the author compare the results at the same magnification scale, especially for the SEM images of Bi growth on Cu foam.

- ✓ Yes, sure. We have added the same magnification of the SEM images for better comparison.

14. As for the CO2RR performance reported by different literatures compared in manuscript Table1, there was a mistake in the Formate selectivity (%) column of Bi Nps line. It is suggested that the author change ‘t0’ to ‘to’.

- ✓ Thanks, revised.

15. As for the durability test in manuscript Figure4, (a) and (b), the author believes that FE at the beginning of the test is about 92%, and there is basically no change within 50 hours, indicating that selectivity has not decreased over a long period of time. However, the FE of Format dropped below 88% after 50 hours of reaction and further decreased after 70 hours of reaction. In fact, the drop in yield from 92% to 88% does not seem very significant, and I recommend that the authors extend the test time to a drop of about 10% to determine the durability of the sample. The same is also recommended for stability tests at high current densities of 50 $mA.cm^{-2}$. In addition, for the morphology of samples before and after the reaction in (c) and (d), I suggest that the author use the results of the same SEM scale for comparison.

- ✓ Yes, you are right. We have tried to rerun the reaction for around 68 hours as we expected the FE for formate dropped to around 55%. Which is too low for Bi based catalysts to operate. That is why we have stopped the test at this point. One thing we have observed is the increasing of other gas products such as CH_4 and C_2H_4 which we believe this is due to the exposer of Cu under-layer. Also, the SEM images after long time reaction shows high destruction of Bi particles on surface and even some parts of the Bi particles have leached out. Since we have the proper conclusion from our experiment at 50 mA/cm^2 current density, we did not run reaction for other current density because our facility is highly occupied with other projects and actually we are working on this project to further improve the stability of Bi-based catalysts hopefully we want to reach a high stability more than 1000 hours though we are taking completely different approaches.

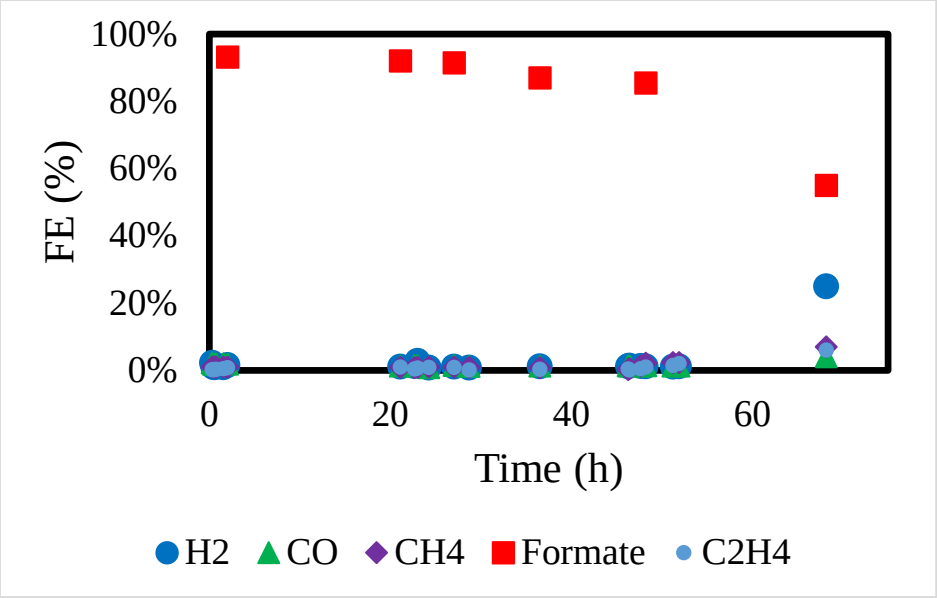

Figure S3: The FE product distribution for other gas products during whole reaction time.

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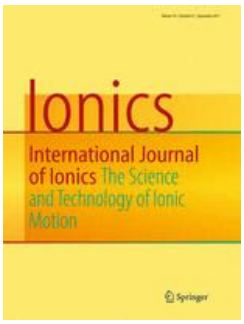

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