RESPONSES TO THE EDITOR'S/REFEREE'S COMMENTS

Ref: Submission ID ce271aa2-338e-4714-b76f-5a88c45d470e

Formation mechanism of mesoporous Cu/CuSe and its application as electrocatalyst for methanol oxidation reaction

Dear Editor/ Reviewer

We are highly thankful to the reviewer for showing significant interest in our research work. We appreciate the reviewer's valuable contributions in providing best possible guidance and recommendations. The highlighted incorrect statements have been corrected as red colored text enclosed in quoted marks. All the mentioned points have been corrected with best alternatives and literature agreement. Our responses to reviewers' comments are listed as follows.

Reviewer 1

General Comment:

In this manuscript, single step solvothermal method is used to prepare Cu/CuSe as an electrocatalyst for methanol electro-oxidation reaction (MOR). The catalyst has a mesoporous structure, which is composed of nanoballs with a high purity, crystallinity, and uniform morphology. The electrocatalyst is good for MOR, as it delivers current density of 37.28 mA/mg at potential of 0.6 V (vs Ag/AgCl) in the electrolyte of 1 M KOH and 0.75 M methanol at a 50 mV/s scan rate under conditions of cyclic voltammetry. My detailed suggestions are followings:

Response: Thank you for your kind words and appreciation of our work. Your positive feedback means a lot to us and inspires us to continue our research with enthusiasm. We are grateful for your valuable contribution in reviewing our manuscript.

Comment 1: The format of this manuscript needs to be confirmed. And the quality of figures are not high, you'd improve these.

Response: Thank you for your concern and positive feedback, high quality of Fig. are also attached in PPT format**.**

Comment 1: I could not find Fig,3d, how to explain the EIS in Fig.4c-d.

Response : Thank you for your comment. We have attached Fig. 3D in the Fig. file (PPT) within the manuscript, and we are also providing it here for your convenience.

Regarding the EIS analysis, our focus was primarily on the high-frequency range, where the presence of a very small and almost negligible semicircle confirms the low charge transfer (Rct) resistance.

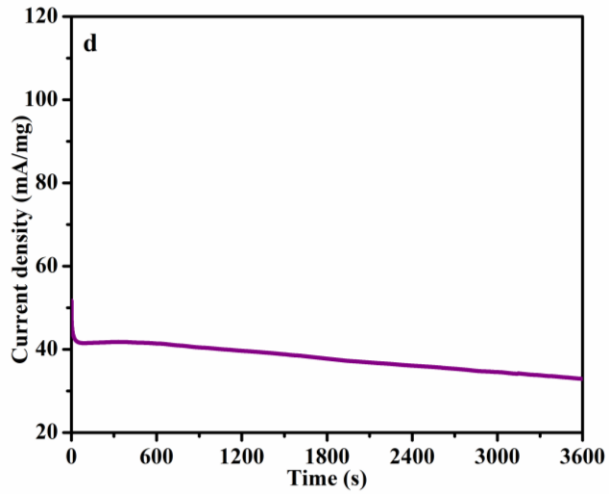

Fig. 3d, Chronoamperometric test for stability

Reviewer 2

This manuscript reports a single step solvothermal method to prepare Cu/CuSe as an electrocatalyst for methanol electro-oxidation reaction (MOR). There are a lot of research focused on MOR. However, no obvious innovativeness could be found here. The characterization of materials is also simple and inadequate. The author emphasized the mesoporous structure of the catalyst but didn't give the evidence of nitrogen adsorption. It's not suggested to be accepted for publication in this journal.

Response: Thank you for your valuable comment. We agree with the reviewer; however, this article is a short communication/letter intended to demonstrate the formation of Cu-CuSe as a catalyst for MOR, which is being studied for the first time. The novelty of this work lies in the unique morphology and composition for MOR. While we acknowledge the reviewer's suggestion regarding the BET study, the mesoporous appearance is clearly evident from the SEM and TEM images. Our primary focus is on the electrochemical application and the electrochemical active surface area (ECSA), as indicated in Figure 4a. It is important to note that the specific surface area obtained from BET may not always be equal to ECSA, which has been confirmed in our previous study. (https://doi.org/10.1016/j.electacta.2018.12.053).

Reviewer 3

General Comment:

In this work, mesoporous Cu/CuSe was synthszied by a hydrothermal approach, and used as MOR catalyst. Overall, the results are useful. However, several limitations need to be overcome.

Response: Thank you for your kind words and appreciation of our work. Your positive feedback means a lot to us and inspires us to continue our research with enthusiasm. We are grateful for your valuable contribution in reviewing our manuscript.

Comment 1: They should improve introduction and at the last paragraph, superiority of the paper should be defined, such as Rare Metals., 41 (2022):3069-3077; J. Colloid. Interf. Sci., 630 (2023) 888 doi.org/10.1016/j.jcis.2022.10.152; Rare Metals., 41 (2022):2237-2242

Response: Thank you so much for your suggestion, the introduction has been modified according to reviewer suggestion**.**

Statement change in manuscript

Platinum is an excellent catalyst for MOR but the high price, low abundance, and CO poisoning hinder its application [7, 8].

The electrocatalyst delivers current density of 37.28 mA/mg at potential of 0.6 V (vs Ag/AgCl) in the electrolyte of 1 M KOH and 0.75 M methanol at a 50 mV/s scan rate under conditions of cyclic voltammetry. The catalyst also shows good stability for 3600 s with negligible charge transfer resistance and high electrochemical active surface area (ECSA) value of 0.100 mF/cm^2.

Comment 2: The XRD and XPS analysis after MOR stability test.

Response: Thank you so much for such a valuable comment. We are agree with reviewer that such study can further confirm the reaction and help in reaction mechanism and the effect of reaction on the materials and their stability. However, we are extremely sorry to say, if we look our procedure, the materials drop on the surface was extremely low and it is not possible to recover that from the surface in such quantity to perform XRD and XPS analysis. Secondly the Aeris 1.2.0 PAN analytical setup need large amount of materials for characterization and same for XPS. Additionally, the we are unable to put the whole electrode for such study in the XPS and XRD. Hope the reviewer understands such situation.

Comment 3: The current density of LSV averaged by electrochemical surface area should be calculated, such as Nanoscale, 12 (2020) 4426; Int. J. Energy Res. 46 (2022) 15938.

Response: Thank you for your valuable suggestion, the manuscript has been modified according to reviewer suggestion.

Change in Manuscript

The current density was normalized by ECSA as shown in Fig. S1. The current density increased after normalization, which confirmed that MOR is not only related to ECSA, but it also related to the materials conductivity, synergism/interaction of Cu and CuSe, which enhanced its intrinsic activity for MOR [7, 12].

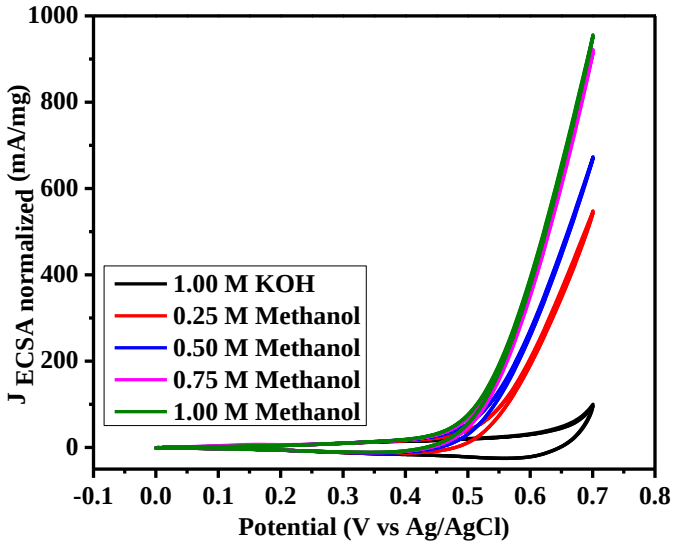

Fig. S1. CV plots of Cu-CuSe after averaged by ECSA

Comment 4: The Tafel plot in the whole range should be given.

Response: Thank you so much for this valuable suggestion. We agree with the reviewer that examining the full range of data may provide more detailed information about the reaction kinetics. However, to the best of our knowledge, the Tafel slope is usually calculated for the potential just after the onset potential until the current increases horizontally. This specific curve is the center of interest for Tafel analysis, as it provides information about reaction kinetics. While it is indeed important to show the Tafel slope for at least 1 mA, we are pleased to confirm that our Tafel analysis fulfills this requirement. And this approach is also supported by the

articles referred by the reviewer (Rare Metals, 41 (2022): 3069-3077; J. Colloid. Interf. Sci., 630 (2023) 888 doi.org/10.1016/j.jcis.2022.10.152; Rare Metals, 41 (2022): 2237-2242).

Comment 5: The related Bode plot should be given.

Response: Thank you for your valuable suggestion, the manuscript has been modified according to reviewer suggestion.

Change in manuscript.

The related bode plot is shown in Fig. S2 [13].

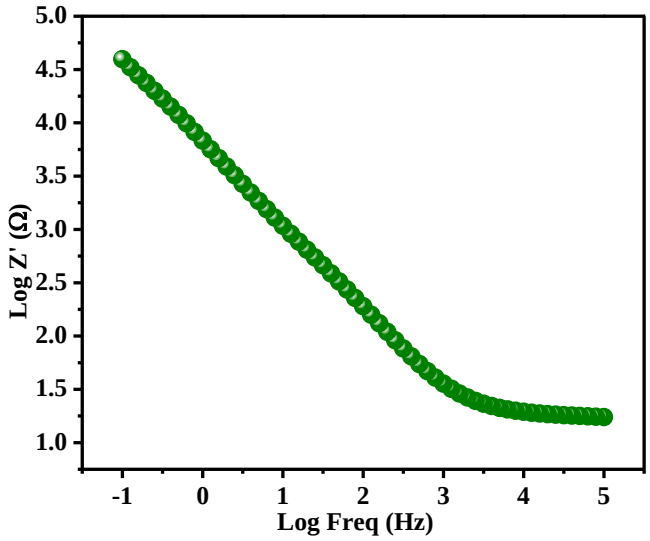

Fig. S2. Bode plot related to EIS