The three-dimensional flower-like Bi$_2$WO$_6$ assisted by ethanolamine through a microwave method for efficient photocatalytic activity

Xingchen Liu, SuZhen Wang, Song Wang, Han Shi, Xiaolong Zhang and Zhicheng Zhong

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Review timeline
Original submission: 7 September 2018
1st revised submission: 10 January 2019
2nd revised submission: 16 January 2019
Final acceptance: 21 January 2019

Note: Reports are unedited and appear as submitted by the referee. The review history appears in chronological order.

Review History
RSOS-181422.R0 (Original submission)

Review form: Reviewer 1 (Girish Kumar)

Is the manuscript scientifically sound in its present form?
Yes

Are the interpretations and conclusions justified by the results?
Yes

Is the language acceptable?
No

Is it clear how to access all supporting data?
Yes

Do you have any ethical concerns with this paper?
No
Have you any concerns about statistical analyses in this paper?
I do not feel qualified to assess the statistics

Recommendation?
Accept with minor revision (please list in comments)

Comments to the Author(s)

Dear authors,

The specific insights into the exceptional role of ethanolamine on the morphological evolution of Bi$_2$WO$_6$ sheets and further assembling into flower-like structure are explained in detail. The further discussions related to the shifting of band edge positions, photoluminescence spectroscopy and Mott-Schottky plots that substantiates the influence of ethanolamine are the other interesting aspects. However, few clarifications and thorough language editing are still essential for improved understanding.

Major comments
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Minor comments
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(2) The role of ethanolamine in altering the pressure of the reaction and on the crystallization of Bi$_2$WO$_6$ must be highlighted.
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(1) The significant results obtained highlighting the role of ethanolamine may be presented.

Reviewed by: S. Girish Kumar, Asst. Professor
Dept. of Chemistry, CMR UNIVERSITY
KARNATAKA, INDIA.
https://publons.com/author/1300703/s-girish-kumar#profile

Review form: Reviewer 2

Is the manuscript scientifically sound in its present form?
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Is the language acceptable?
Yes

Is it clear how to access all supporting data?
Not Applicable

Do you have any ethical concerns with this paper?
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Have you any concerns about statistical analyses in this paper?
No
Recommendation?
Major revision is needed (please make suggestions in comments)

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Decision letter (RSOS-181422.R0)

13-Nov-2018

Dear Miss Liu:

Title: The 3-D flower-like Bi2WO6 assisted by ethanolamine through microwave method for efficiency photocatalytic activity
Manuscript ID: RSOS-181422

Thank you for your submission to Royal Society Open Science. The chemistry content of Royal Society Open Science is published in collaboration with the Royal Society of Chemistry.

The editor assigned to your manuscript has now received comments from reviewers. We would like you to revise your paper in accordance with the referee and Subject Editor suggestions which can be found below (not including confidential reports to the Editor). Please note this decision does not guarantee eventual acceptance.

Please submit your revised paper before 06-Dec-2018. Please note that the revision deadline will expire at 00.00am on this date. If we do not hear from you within this time then it will be assumed that the paper has been withdrawn. In exceptional circumstances, extensions may be possible if agreed with the Editorial Office in advance. We do not allow multiple rounds of revision so we urge you to make every effort to fully address all of the comments at this stage. If deemed necessary by the Editors, your manuscript will be sent back to one or more of the original reviewers for assessment. If the original reviewers are not available we may invite new reviewers.
To revise your manuscript, log into http://mc.manuscriptcentral.com/rsos and enter your Author Centre, where you will find your manuscript title listed under "Manuscripts with Decisions." Under "Actions," click on "Create a Revision." Your manuscript number has been appended to denote a revision. Revise your manuscript and upload a new version through your Author Centre.

When submitting your revised manuscript, you must respond to the comments made by the referees and upload a file "Response to Referees" in "Section 6 - File Upload". Please use this to document how you have responded to the comments, and the adjustments you have made. In order to expedite the processing of the revised manuscript, please be as specific as possible in your response.

Please also include the following statements alongside the other end statements. As we cannot publish your manuscript without these end statements included, if you feel that a given heading is not relevant to your paper, please nevertheless include the heading and explicitly state that it is not relevant to your work.

• Ethics statement
Please clarify whether you received ethical approval from a local ethics committee to carry out your study. If so please include details of this, including the name of the committee that gave consent in a Research Ethics section after your main text. Please also clarify whether you received informed consent for the participants to participate in the study and state this in your Research Ethics section.
*OR*
Please clarify whether you obtained the necessary licences and approvals from your institutional animal ethics committee before conducting your research. Please provide details of these licences and approvals in an Animal Ethics section after your main text.
*OR*
Please clarify whether you obtained the appropriate permissions and licences to conduct the fieldwork detailed in your study. Please provide details of these in your methods section.

Once again, thank you for submitting your manuscript to Royal Society Open Science and I look forward to receiving your revision. If you have any questions at all, please do not hesitate to get in touch.

Yours sincerely,
Dr Laura Smith
Publishing Editor, Journals
Royal Society of Chemistry
Thomas Graham House
Science Park, Milton Road
Cambridge, CB4 0WF
Royal Society Open Science - Chemistry Editorial Office

On behalf of the Subject Editor Professor Anthony Stace and the Associate Editor Professor Eva Hevia.

******************************************************************************

RSC Associate Editor:
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Reviewer: 2

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Author’s Response to Decision Letter for (RSOS-181422.R0)

See Appendix A.

RSOS-181422.R1 (Revision)

Review form: Reviewer 1 (Girish Kumar)

Is the manuscript scientifically sound in its present form?  
Yes

Are the interpretations and conclusions justified by the results?  
Yes

Is the language acceptable?  
No

Is it clear how to access all supporting data?  
Not Applicable

Do you have any ethical concerns with this paper?  
No

Have you any concerns about statistical analyses in this paper?  
I do not feel qualified to assess the statistics

Recommendation?  
Accept with minor revision (please list in comments)

Comments to the Author(s)  
Dear authors,

The point-to-point response for all the comments deserves appreciation and revised manuscript provides more insight into the work. The listed comments may be considered for better readability.
Minor comments
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Reviewed by: S. Girish Kumar, Asst. Professor
Dept. of Chemistry, CMR UNIVERSITY
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Review form: Reviewer 2

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Have you any concerns about statistical analyses in this paper?
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Recommendation?
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The authors have either revised this submission according to the comments and suggestions from the reviewers satisfactorily, or responded to the concerns from the reviewers well. I suggest this
manuscript be accepted for publication in Royal Society Open Science after the authors correct
the following problems.
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were presented with whole names (such as References 1 and 2), and some with abbreviated ones
(such as References 3 and 4).
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not (such as References 5 and 6).
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numbers of (Chem Eng J, 2018, 353: 533-541) were not provided.
The authors should correct these minor mistakes to meet the standard of publication.

Decision letter (RSOS-181422.R1)

14-Jan-2019

Dear Miss Liu:

Title: The 3-D flower-like Bi2WO6 assisted by ethanolamine through microwave method for
efficiency photocatalytic activity
Manuscript ID: RSOS-181422.R1

Thank you for submitting the above manuscript to Royal Society Open Science. On behalf of the
Editors and the Royal Society of Chemistry, I am pleased to inform you that your manuscript will
be accepted for publication in Royal Society Open Science subject to minor revision in accordance
with the referee suggestions. Please find the reviewers' comments at the end of this email.

The reviewers and handling editors have recommended publication, but also suggest some minor
revisions to your manuscript. Therefore, I invite you to respond to the comments and revise your
manuscript.

Because the schedule for publication is very tight, it is a condition of publication that you submit
the revised version of your manuscript before 23-Jan-2019. Please note that the revision deadline
will expire at 00.00am on this date. If you do not think you will be able to meet this date please let
me know immediately.

To revise your manuscript, log into https://mc.manuscriptcentral.com/rsos and enter your
Author Centre, where you will find your manuscript title listed under "Manuscripts with
Decisions". Under "Actions," click on "Create a Revision." You will be unable to make your
revisions on the originally submitted version of the manuscript. Instead, revise your manuscript
and upload a new version through your Author Centre.

When submitting your revised manuscript, you will be able to respond to the comments made by
the referees and upload a file "Response to Referees" in "Section 6 - File Upload". You can use this
to document any changes you make to the original manuscript. In order to expedite the
processing of the revised manuscript, please be as specific as possible in your response to the
referees.

When uploading your revised files please make sure that you have:

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and figure captions. Do not upload a PDF as your "Main Document".
2) A separate electronic file of each figure (EPS or print-quality PDF preferred (either format should be produced directly from original creation package), or original software format)
3) Included a 100 word media summary of your paper when requested at submission. Please ensure you have entered correct contact details (email, institution and telephone) in your user account
4) Included the raw data to support the claims made in your paper. You can either include your data as electronic supplementary material or upload to a repository and include the relevant doi within your manuscript
5) All supplementary materials accompanying an accepted article will be treated as in their final form. Note that the Royal Society will neither edit nor typeset supplementary material and it will be hosted as provided. Please ensure that the supplementary material includes the paper details where possible (authors, article title, journal name).

Supplementary files will be published alongside the paper on the journal website and posted on the online figshare repository (https://figshare.com). The heading and legend provided for each supplementary file during the submission process will be used to create the figshare page, so please ensure these are accurate and informative so that your files can be found in searches. Files on figshare will be made available approximately one week before the accompanying article so that the supplementary material can be attributed a unique DOI.

Once again, thank you for submitting your manuscript to Royal Society Open Science. The chemistry content of Royal Society Open Science is published in collaboration with the Royal Society of Chemistry. I look forward to receiving your revision. If you have any questions at all, please do not hesitate to get in touch.

Best wishes,

Dr Laura Smith
Publishing Editor, Journals

Royal Society of Chemistry
Thomas Graham House
Science Park, Milton Road
Cambridge, CB4 0WF
Royal Society Open Science - Chemistry Editorial Office

On behalf of the Subject Editor Professor Anthony Stace and the Associate Editor Professor Eva Hevia.

**********************************************************************

RSC Associate Editor:
Comments to the Author:
(There are no comments.)

RSC Subject Editor:
Comments to the Author:
(There are no comments.)

**********************************************************************
Reviewer comments to Author:
Reviewer: 1

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See Appendix B.

Decision letter (RSOS-181422.R2)

21-Jan-2019

Dear Miss Liu:

Title: The 3-D flower-like Bi2WO6 assisted by ethanolamine through microwave method for efficiency photocatalytic activity
Manuscript ID: RSOS-181422.R2

It is a pleasure to accept your manuscript in its current form for publication in Royal Society Open Science. The chemistry content of Royal Society Open Science is published in collaboration with the Royal Society of Chemistry.

The comments of the reviewer(s) who reviewed your manuscript are included at the end of this email.

Thank you for your fine contribution. On behalf of the Editors of Royal Society Open Science and the Royal Society of Chemistry, I look forward to your continued contributions to the Journal.

Yours sincerely,
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On behalf of the Subject Editor Professor Anthony Stace and the Associate Editor Professor Eva Hevia.

********

RSC Associate Editor
Comments to the Author:
(There are no comments.)

********

Reviewer(s)' Comments to Author:
Appendix A

Dear Editor and Reviewers:

Thank you for your letter and for the reviewers’ comments concerning our manuscript entitled “The 3-D flower-like Bi$_2$WO$_6$ assisted by ethanolamine through microwave method for efficiency photocatalytic activity”. Those comments are all valuable and very helpful for revising and improving our paper, as well as the important guiding significance to our researches. We have studied comments carefully and have made correction which we hope meet with approval. Revised portion are marked in red in the paper. The main corrections in the paper and the responds to the reviewer’s comments are as flowing:

Responds to the reviewer’s comments:
Reviewer #1:
1. Response to comment: Abstract(1) Indicate the reaction temperature and time within the parenthesis after the sentence (microwave method) and specify concentration of ethanolamine for clarity. (2) The role of ethanolamine in altering the pressure of the reaction and on the crystallization of Bi$_2$WO$_6$ must be highlighted. (3) The results derived from materials characterization should be briefly emphasized.
Response: We have made corrections according to the Reviewer’s comments. All the corrections were written in red.

2. Response to comment: Section 1.0(1) Modify the starting phrase ‘In recently, …. pollution'! (avoid using pollution twice in the same phrase). (1) Add few review articles related to Bi$_2$WO$_6$ along with ref. [2,3]: Applied Surface Science 355 (2015) 939-958; Catalysis Science and Technology 2 (2012) 694-706; Chemical Society Reviews 43 (2014) 5276-5287.(2) Remove ‘more than 10 hours’ and generalize as ‘longer time’, while discussing hydrothermal method.
Response: We have made corrections according to the Reviewer’s comments and mentioned references have been added. All the corrections were written in red.
3. Response to comment: Section 2.1(1) The preparative method should be detailed out completely to ensure reproducibility: (i) volume of Bi precursors and tungstate precursors taken should be indicated; (ii) concentration of ethanolamine must be specified. (2) The apparatus in which these solutions were mixed must be indicated. (3) The samples may be abbreviated as ‘EA:X’ instead of ‘EA X’.
Response: Thank you for your comment. (1) (i) The amount of Bi precursors and tungstate precursors has been shown in the paper, which is 2 mmol and 1 mmol, respectively. (ii) the concentration of used ethanolamine is about 99%, and which has been indicated in the article. (2) The whole experiment was going on microwave reactor (Biotage Sweden). No additional mixing apparatus were used. (3) We have re-written this part according to the Reviewer’s suggestion. All the corrections were written in red.

4. Response to comment: Section 2.3(1) The details of excitation source including its intensity, wavelength maxima and distance between light source and solution surface must be provided. (2) Specify the solution pH at which degradation reaction were carried out.
Response: The details of excitation source and the solution pH (pH=7) have been added in the article, which were written in red.

5. Section 3.2(1) The XRD studies requires few clarifications: (i) is it possible to observe crystallized Bi2O3 if only Bi precursor are used in the similar experimental condition?; (ii) reflections of Bi2O3 and Bi2WO6 in the XRD patterns may be distinguished using symbols (like, asterisk ‘*’ for Bi2O3); (iii) can pure Bi2WO6 formation is feasible if reaction time is prolonged for EA 1-1?.
(2) Typo: ration!
Response: (1) Thank you for your comment. (i) We have supplemented this data. The crystallized Bi2O3 was obtained if only Bi precursor is used in the similar experimental condition (Fig.S1). (ii) We have re-written this part according to the Reviewer’s suggestion. All the corrections were written in red. (iii) We have supplemented this data. When the reaction time is prolonged for EA
1-1, the diffraction peaks characteristic of Bi2O3 gradually decreased, and that of Bi2WO6 enhanced gradually (Fig.S2).

(2) We have re-written this part according to the Reviewer’s suggestion. All the corrections were written in red.

6. Section 3.6 (1) devolved!
Response: We have re-written this part according to the Reviewer’s suggestion. All the corrections were written in red.

7. Section 3.8 (1) The phrase ‘excessive –NH2 makes Bi3+ reduced to Bi0’ may be supported with suitable literature. (2) It should be ‘adsorbed –OH groups’ instead of ‘absorbed’!
Response:
(1) Thank you for your comment. Since –NH2 is reducible, excessive –NH2 makes Bi3+ reduced to Bi0. Our experiments prove this point (Fig. 11). In addition, we also added relevant references. (2) We have re-written this part according to the Reviewer’s suggestion. All the corrections were written in red.

8. Conclusion (1) The significant results obtained highlighting the role of ethanolamine may be presented.
Response: The role of ethanolamine has been added in the article, which was written in red.

Reviewer #2:
1. on Abstract: the authors should provide the detailed results and conclusions, and describe briefly if and how the structure and catalytic performance of Bi2WO6 change with the assist of ethanolamine.
Response: We have re-written this part according to the Reviewer’s suggestion. All the corrections were written in red.

2. On 1st paragraph: Better background information on microwave applications and organic pollutant removal by other methods including advanced oxidation and biological processes should be presented to the prospective audiences, and these following recent articles in this field could serve this purpose in some aspects: Scientific Reports, 2017, 7(1): 1668 (p1-8); Chemical
3. On 2nd paragraph: The use of photocatalysis for organic pollutants has received much interests. More recent articles on this topic should be reviewed, for example: Microstructure and performance of Z-scheme photocatalyst of silver phosphate modified by MWCNTs and Cr-doped SrTiO3 for malachite green degradation. Applied Catalysis B Environmental, 2018, 227: 557-570.
Response: We have made corrections according to the Reviewer’s comments and mentioned references have been added. All the corrections were written in red.

4. On 3rd paragraph: Major properties of the model pollutant “methyl orange” should be mentioned. Why MO was selected as the model pollutant?
Response: Thank you for your comment. Methyl orange (MO) is stable and does not decompose under the action of light. Moreover, the degradation rate of MO can be calculated by absorbance and concentration standard curves. Therefore, MO is used as a model for photocatalytic experiments. We have explained this point in the article. All the corrections were written in red.

5. Line 48-53 in Page 3: The authors should consider to list some typical long chain compounds, compare them with ethanolamine, and describe the advantages and disadvantages.
Response: We have re-written this part according to the Reviewer’s suggestion. All the corrections were written in red.

6. In section 2.1, the symbol for temperature should be presented in an accepted style.
Response: We have made corrections according to the Reviewer’s comments. All the corrections were written in red.
7. Line 30-41 in Page 6: Viscosity of EA is much higher than that of water, which could affect the mass transfer rate for ions in the system. This claim needs more reliable explanations and experimental evidences or data.
Response: Thank you for your comment. We have added the relevant references have been added (25). All the corrections were written in red.

8. Line 25-27 in Page 7: More explanations and evidences including literature citation for “the high dispersion of EA makes the precursor sodium tungstate and bismuth nitrate to be fully in contact, thus contributing to the formation of Bi2WO6” are needed. The following literature could be referred for the dispersion: Influences of anion concentration and valence on dispersion and aggregation of titanium dioxide nanoparticles in aqueous solutions. Journal of Environmental Sciences, 2017, 54: 135-141.
Response: We have made corrections according to the Reviewer’s comments and mentioned references have been added. All the corrections were written in red.

9. Fig.2 shows that the EA 2-1 sample has the highest purity and crystallinity, but the authors only compared EA 7-1 to EA 5-1, indicated that the EA 7-1 sample has a higher purity and crystallinity. That comparison and explanation is incomplete and cannot support the following conclusion. In addition, the XRD spectra of ethanolamine should be provided to eliminate its effects for the crystal phase and structure of Bi2WO6.
Response: Thank you for your comment. In this paper, we focus on the 3D flower-like structure. Seen from Fig.3, since only EA 5-1 and EA 7-1 samples formed the 3D flower-like structure, we only compared EA 7-1 to EA 5-1. We have omitted the expression in this part in the article. In addition, since EA is liquid at room temperature, the XRD spectrum of which has no peak, there is no interference with the crystal phase and structure of Bi2WO6. So we think it is not necessary to supplement its XRD spectrum.

10. Line 47-56 in Page 7: The authors should edit this manuscript more carefully. Such as Line 52-53 in Page 7, the clerical error in the sentence “This result proves that EA 2-1 is a mixture of two
substances, which is consistent with the XRD result”. It should be EA 1-1, not EA 2-1.

Response: Thank you for your comment. We have made corrections according to the Reviewer’s comments and mentioned references have been added. All the corrections were written in red.

11. Line 55 in Page 8: The sentence “The surface area of all the samples is similar, shown in Table 1, which can indicate that the difference in adsorption capacity is not caused by the BET difference” should be placed in section 3.7 to explain the difference in adsorption capacity.

Response: Thank you for your comment. We have made corrections according to the Reviewer’s comments and mentioned references have been added. All the corrections were written in red.

12. Line 3-19 in Page 11: the amount of EA could promote the increased atomic ratio of OS in Bi2WO6, the authors should explain it.

Response: Thank you for your comment. The O 1s core level at 532.25 eV (OS) can be ascribed to the oxygen in –OH. EA molecules contain active hydroxyl and secondary amino group. With the proportion of EA increasing through hydrogen bonding, the association of –OH and Bi2WO6 molecular becomes greater. So we think the amount of EA could promote the increased atomic ratio of OS in Bi2WO6. We have explained this reason in article 3.8.

13. Line 55 in Page 9: in the paper, the values of band gaps of EA 0, EA 1-1, EA 2-1, EA 5-1 and EA 7-1 were calculated in the Fig.5, and the variation of energy band positions were observed from the XPS valence band spectra in the Fig.9. It’s better to write out the calculation. In order to further confirm the energy band positions and its variation, other means, such as Mott-Schottky calculation process should be also provided. The literature mentioned in Comment 3 could provide such information.

Response: Thank you for your comment. The XPS valence band spectra of EA 7-1, EA 5-1 and EA 2-1, the VB potentials are 1.77 eV, 1.81 eV and 1.97 eV, respectively. In addition, as shown in Fig. 10, the flat band potentials are estimated to be -0.67 V, -0.65 V, -0.59 V for
EA 2-1, EA 5-1 and EA 7-1 respectively. We have already explained this in article.

14. On Conclusions: The results show that the addition of EA can improve the photocatalytic activity of materials and the increase of the amount of EA, the photocatalytic activity gradually increases. The author only compared the performances of EA 0, EA 1-1, EA 2-1, EA 5-1 and EA 7-1, while did not conclude how the photocatalytic activity change when EA was increased further. Data on more higher proportion should be supplemented to find the optimal proportion, so as to make the conclusion and research more valuable.

Response: Thank you for your comment. We have supplemented this data, as shown in Fig.S4.
Appendix B

Dear Editor and Reviewers:

Thank you for your letter and for the reviewers’ comments concerning our manuscript entitled “The 3-D flower-like Bi$_2$WO$_6$ assisted by ethanolamine through microwave method for efficiency photocatalytic activity”. Those comments are all valuable and very helpful for revising and improving our paper, as well as the important guiding significance to our researches. We have studied comments carefully and have made correction which we hope meet with approval. Revised portion are marked in red in the paper. The main corrections in the paper and the responds to the reviewer’s comments are as flowing:

Responds to the reviewer’s comments:

Reviewer #1:
1. Response to comment: Title: Replace ‘efficiency’ by ‘efficient’!
Response: We have made corrections according to the Reviewer’s comments. All the corrections were written in red.

2. Response to comment: Abstract(1) Remove the phrase ‘The whole process took only ten minutes’!
Response: We have already removed the phrase in abstract.

3. Response to comment: Introduction (1) The ethanolamine should be abbreviated as ‘EA’ in its first appearance in the text.
Response: We have already made corrections according to the Reviewer’s comments. All the corrections were written in red.

4. Response to comment: Section 2.3 (1) Is it 460 or 464 nm! It may be verified.
Response: We have already changed the maximum absorption wavelength as 464 nm.

5. Response to comment: Section 3.0
(1) It should be ‘Figure’ instead of ‘figure’ throughout.
(2) The term ‘compound rates’ could not be followed. (near ref. 30 and at other places as well)
(3) The spin orbit coupling values must be made subscript to the orbital in the XPS studies.
(4) Figure 7-2?
(5) It should be ‘XRD patterns’ instead of ‘XRD images’!
Response: (1) We have already made corrections in 3.2. The correction was written in red.
(2) We have already changed “compound rates” to “recombination rate”. The correction was written in red.
(3) We have already labeled the spin orbital coupling value in Fig.6 and the corresponding explanations have been in 3.6.
(4) We have already changed “Figure 7-2” to “Figure 7 (b)”. The correction was written in red.
(5) We have made corrections according to the Reviewer’s comments. The correction was written in red.

Reviewer #2:
Response to comment:
1. There are many spelling or style problems in the reference list. For example, some journals were presented with whole names (such as References 1 and 2), and some with abbreviated ones (such as References 3 and 4).
2. Some of whole journal titles were listed in upper case (such as References 3 and 4), and some not (such as References 5 and 6).
3. Some bibliographic information are lost, such as Reference 12 whose volume and page numbers of (Chem Eng J, 2018, 353: 533-541) were not provided.
The authors should correct these minor mistakes to meet the standard of publication.
Response: We have made corrections according to the Reviewer’s comments in the reference list. The correction was written in red.