[Sustainability] Manuscript ID: sustainability-1602796 - Submission Received External Indox ×

Editorial Office <sustainability@mdpi.com> to me, Apip, Asep, Novi, Edy, Navid, Surachai, Yukihiko 💌

Dear Dr. Farobie,

Thank you very much for uploading the following manuscript to the MDPI submission system. One of our editors will be in touch with you soon.

Journal name: Sustainability Manuscript ID: sustainability-1602796 Type of manuscript: Article Title: Slow-pyrolysis of Ulva lactuca (Chlorophyta) for sustainable production of bio-oil and biochar Authors: Apip Amrullah, Obie Farobie *, Asep Bayu, Novi Syaftika, Edy Hartulistiyoso, Navid R Moheimani, Surachai Karnjanakom, Yukihiko Matsumura Received: 2 February 2022 E-mails: apip.amrullah@ulm.ac.id, obiefarobie@apps.ipb.ac.id, edyhartulistiyoso@apps.ipb.ac.id, asepbayu@yahoo.co.id, novi017@brin.go.id, N.Moheimani@murdoch.edu.au, surachai ka@rsu.ac.th, mat@hiroshima-u.ac.jp

You can follow progress of your manuscript at the following link (login required):

Gambar 1. Bukti submission (2 February 2022).

[Sustainability] Manuscript ID: sustainability-1602796 - Minor Revisions (External) Inbox ×

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Sustainability Editorial Office

to me, Apip, Asep, Novi, Edy, Navid, Surachai, Yukihiko, Sustainability 💌

Dear Dr. Farobie,

Thank you again for your manuscript submission:

Manuscript ID: sustainability-1602796 Type of manuscript: Article Title: Slow-pyrolysis of Ulva lactuca (Chlorophyta) for sustainable production of bio-oil and biochar Authors: Apip Amrullah, Obie Farobie *, Asep Bayu, Novi Syaftika, Edy Hartulistiyoso, Navid R Moheimani, Surachai Karnjanakom, Yukihiko Matsumura Received: 2 February 2022 E-mails: apip.amrullah, ac.id, obiefarobie@apps.ipb.ac.id, edyhartulistiyoso@apps.ipb.ac.id, asepbayu@yahoo.co.id, novi017@brin.go.id, n.moheimani@murdoch.edu.au, surachai.ka@rsu.ac.th, mat@hiroshima-u.ac.jp

Your manuscript has been reviewed by experts in the field. Please find your manuscript with the referee reports at this link:

Gambar 2. Bukti korespondensi minor revision (revisi tahap 1).

Mon, Feb 28, 10:24 AM

Dear Prof. Dr. Marc A. Rosen/ Dr. Zoe Zhou,

We would like to thank you for your kind consideration in allowing us to revise our manuscript (ID: sustainability-1602796) entitled "Slow-pyrolysis of *Ulva lactuca* (Chlorophyta) for sustainable production of biooil and biochar." The manuscript has been revised for better readability according to the suggestions of the Editor and Reviewers by including the response to the reviewers' comments and revised manuscript.

The novelty of this study is highlighted as follows: (1) performing a comprehensive study for co-production of bio-oil and bio-char from *U. lactuca*, including their characteristics which have not been reported elsewhere and (2) providing the detailed reaction pathway for the conversion of *U. lactuca* into high-value compounds identified in bio-oil. The key findings of this study also have been mentioned in the manuscript. We believe that this manuscript will attract the readers of the journal of *Sustainability* and other sustainable energy-related journals since the significance and novelty of this work have already been insisted on.

We again appreciate the kindness of the Editor and Reviewers in helping improve the manuscript. All authors have reviewed and agreed to the submission of the revised manuscript. We are attaching the response to the reviewers' comments below. We also sent the response to each reviewer following the guideline of *Sustainability*. We hope that the manuscript is now acceptable for publication. Please do not hesitate to contact me if there are any questions.

Dr. Eng. Obie Farobie Department of Mechanical and Biosystem Engineering, IPB University. E-mail: obiefarobie@apps.ipb.ac.id Tel: +62812-898-11381

Response to Reviewer 1 Comments

Point 1: What is the main reason for selecting this feedstock for this study? Is it available throughout the world? **Response 1:** *Ulva lactuca* is one of the green algae with 125 species from the genus Ulva that can be found worldwide [1]. *Ulva sp* commercialization among green algae is one of the largest, although not as massive as other commercial species such as *Laminaria japonica* (brown algae), *Euchema* sp., or *Gracillaria* sp. (red algae) [2]. The global production of *Ulva* sp. accounted for 2,356 tons in 2019, still far below the aforementioned seaweeds. On the other hand, *Ulva* sp. is highly nutrient level tolerant in its habitat. It is often found as the most predominant species (52%) in algal bloom, causing environmental problems [3,4]. Therefore, it is significant to explore the valorization of *Ulva* sp. to various products, in this case, are bio-oil and bio-char. This study improves the understanding of Ulva sp. behavior as feedstock and the characteristic of its product. With the utilization of *Ulva* sp for bio-oil and bio-char, the value-added of this organism can be improved. In addition, the diverse utilization will potentially reduce the *Ulva* sp. eutrophication so that the impact on the environment can be minimized.

Point 2: What are the main applications of the bio-oil obtained from the pyrolysis of U.Lactuca plant? Please add brief information about the importance of these products in the manuscript?

Response 2: Bio-oil after being upgraded, can be used as a substitute for fossil resources for various applications such as fuel to generate heat and power and for chemical production. Improved quality bio-oil can be utilized as a fuel in the boiler, furnace, turbine, diesel engines, power generation, and industrial processes [5,6]. As for chemical production, bio-oil can be further processed to obtain phenol for resins and wood adhesives, molded plastic and foam insulations, and calcium and magnesium acetate for biodegradable deicers, fertilizers, levoglucosan, hydroxy-acetaldehyde, and various food seasonings and essences.

The brief information about the importance of bio-oil has been added in the manuscript (introduction section, Lines 111-113).

Point 3: The pyrolysis of U.Lactuca feedstock studied at 400c to 600c. Did you try the pyrolysis at below 400c? **Response 3:** We did not investigate the pyrolysis below 400 °C since the bio-oil yield is too low. The pyrolysis temperatures of 400 °C to 600 °C were selected following the previous studies on biomass pyrolysis [7–12].

Point 4: Please conclude the suitable temperature and time for the pyrolysis U.Lactuca feedstock to obtained the highest yield of bio-oil and biochar?

Response 4: The maximum bio-oil yield (24.05%) was achieved at 500 °C within 50 min. Meanwhile, the highest biochar yield (88.47%) was obtained at 400 °C within 10 min.

The information about the highest yield of bio-oil and biochar has been added in the conclusion following the reviewer's suggestion.

Point 5: Please add brief information about previous reported work and results using this feedstock with different methods to compare the results of your study?

Response 5: The authors would like to thank you for your excellent recommendation. Brief information about previous reported work and results using this feedstock with different methods have been added in the manuscript (Lines 191-195).

Point 6: Please add the GCMS chromatograms with high resolution and main products peak? (Remove the noise from the chromatogram).

Response 6: The great suggestion from the reviewer is appreciated. The authors understand that GCMS chromatograms with high resolution should be presented in the manuscript. However, the GCMS instrument did not support the excel file for the time being. Alternatively, a printed version of GCMS chromatograms is presented in the supplementary material.

The author would like to ask the reviewer's understanding.

Point 7: Please see the attached file for the additional comments.

Point 7.1: How did you decide the time and temperature for the drying? Add reference or statement of justification.

Response 7.1: The pretreatment process of macroalgae was adapted from the previous work of Wu et al. [13]. They used the high temperature of 70 °C and 2 h for the drying. To prevent the degradation of chemical constituent, the temperature of 50 °C for 3 h was used in this study.

Point 7.2: How did you decide the sample size? Is reactor need to completely fill with the sample? Do we need to moniotor any pressure inside the reactor? If pressure is build up in the reactor, is it effect on yield? Pelase address these questions in the manuscripts.

Response 7.2: The sample size used in this study was based on our previous study [14]. No, the reactor is not entirely filled with the sample. We don't need to monitor the pressure inside the reactor since the pyrolysis is conducted at atmospheric pressure. Hence, we did not investigate the effect of pressure following the typical pyrolysis process. This information has been added in the manuscript (Lines 151-153).

Point 7.3: Is this purging throughout the reactions? If it is throughout the reaciton, how did you maintain the pressure inside the reactor?.

Response 7.3: Thank you for the question. No, it is not purging throughout the reactions. The purging with N2 was conducted only at the beginning to remove the air inside the reactor.

Point 7.4: It is better to add the GC condition in the manuscript.

Response 7.4: The GC condition has been added in the manuscript following the reviewer's suggestion (Lines 164-166).

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- 1. Baweja, P.; Kumar, S.; Sahoo, D.; Levine, I. *Biology of Seaweeds*; Elsevier Inc., 2016; ISBN 9780128027936.
- 2. Lakshmi, D.S.; Sankaranarayanan, S.; Gajaria, T.K.; Li, G.; Kujawski, W.; Kujawa, J.; Navia, R. A short review on the valorization of green seaweeds and ulvan: Feedstock for chemicals and biomaterials. *Biomolecules* **2020**, *10*, 1–20, doi:10.3390/biom10070991.
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- Aboulkas, A.; Hammani, H.; El Achaby, M.; Bilal, E.; Barakat, A.; El harfi, K. Valorization of algal waste via pyrolysis in a fixed-bed reactor: Production and characterization of bio-oil and bio-char. *Bioresour. Technol.* 2017, 243, 400–408, doi:10.1016/j.biortech.2017.06.098.

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- 13. Wu, P.; Zhang, X.; Wang, J.; Yang, J.; Xuanwei peng; Feng, L.; Zu, B.; Xie, Y.; Li, M. Pyrolysis of aquatic fern and macroalgae biomass into bio-oil: Comparison and optimization of operational parameters using response surface methodology. *J. Energy Inst.* **2021**, *97*, 194–202, doi:10.1016/j.joei.2021.04.010.
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Response to Reviewer 2 Comments

Point 1: I am returning my review of the paper title "Slow-pyrolysis of Ulva lactuca (Chlorophyta) for sustainable production of bio-oil and biochar". This is a great piece of research on valorization of marine macroalga Ulva Lactuca for bio-oil and bio-char production through a thermochemical process. I am very enthusiastic about the results. It is clear the lab work was executed very competently. However, minor corrections listed below are necessary to improve manuscript quality.

Response 1: The authors would like to thank you for the helpful comments from the reviewer. The manuscript has been revised for better readability, following the reviewer's suggestions.

Point 2: The introduction should mention the problematic of feedstock management (high moisture, inorganics, calorific value for energy applications for example) and the need of pre-treatment processes such as slow pyrolysis to produce value-added by-products.

Response 2: The great suggestion from the reviewer is appreciated. The feedstock problem has been mentioned in the introduction part, following the reviewer's suggestion (Lines 100-104).

Point 3: Compare the results to other feedstocks typically used for high quality bio-oil and bio-char production **Response 3:** The authors would like to thank you for the excellent recommendation from the reviewer. Comparison with other feedstocks has been provided in the discussion section (Lines 233-236, Lines 249-254).

Point 4: I suggest to add statistical analysis to compare the obtained results. Significance difference analysis would help to visualize if the results were improved significantly compared to the raw material.

Response 4: Thank you very much for the great suggestion. We agree that the statistical analysis is essential to visualize whether the results were improved significantly compared to the raw material. However, following some published papers in reputable international journals [1–10], the error bars and standard deviations are enough to present the statistical analysis in the field of pyrolysis of biomass. The authors would like to ask the reviewer's understanding.

Point 5: In figure 5 remove the background lines to keep same figure style along the paper **Response 5:** The authors would like to thank you for your excellent recommendation. The background lines in figure 5 have been removed following the reviewer's recommendation.

Point 6: In Equation 4 state that the initial feedstock was dry as in equation 3 **Response 6:** The outstanding recommendations from the reviewer are appreciated. Equation 4 has been revised following the reviewer's suggestion.

References

- 1. Gautam, R.; Shyam, S.; Reddy, B.R.; Govindaraju, K.; Vinu, R. Microwave-assisted pyrolysis and analytical fast pyrolysis of macroalgae: Product analysis and effect of heating mechanism. *Sustain. Energy Fuels* **2019**, *3*, 3009–3020, doi:10.1039/c9se00162j.
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- 5. Zhou, S.; Liang, H.; Han, L.; Huang, G.; Yang, Z. The influence of manure feedstock, slow pyrolysis, and hydrothermal temperature on manure thermochemical and combustion properties. *Waste Manag.* **2019**, *88*, 85–95, doi:10.1016/j.wasman.2019.03.025.
- 6. Zhang, L.; Yang, Z.; Li, S.; Wang, X.; Lin, R. Comparative study on the two-step pyrolysis of different lignocellulosic biomass: Effects of components. *J. Anal. Appl. Pyrolysis* **2020**, *152*, 104966, doi:10.1016/j.jaap.2020.104966.
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- 9. Hao, J.; Qi, B.; Li, D.; Zeng, F. Catalytic co-pyrolysis of rice straw and ulva prolifera macroalgae: Effects of process parameter on bio-oil up-gradation. *Renew. Energy* **2021**, *164*, 460–471, doi:10.1016/j.renene.2020.09.056.
- 10. Iaccarino, A.; Gautam, R.; Sarathy, S.M. Bio-oil and biochar production from halophyte biomass: effects of pre-treatment and temperature on Salicornia bigelovii pyrolysis. *Sustain. Energy Fuels* **2021**, *5*, 2234–2248, doi:10.1039/d0se01664k.

Response to Reviewer 3 Comments

Point 1: ABSTRACT: It should be about 150-250 words with concise text in a single paragraph.

Answer the questions: What problem did you study, and why is it important? What methods did you use? What were your main results? And what conclusions can you draw from your results? Please make your abstract with more specific and quantitative results.

Response 1: The authors would like to thank you for your excellent recommendation. Following the reviewer's suggestion, the abstract has been revised to be about 226 words. The abstract already included the problem statement, importance of the study, methods, significant findings, and conclusion. The abstract has been revised following the reviewer's suggestion.

Point 2: Introduction: author should include more details about the current research status; a deep discussion is required. The first, second, and third paragraphs of the introduction should be concise; there is no need to describe the background in detail. Introduction should be more scientific.

Response 2: The outstanding recommendations from the reviewer are appreciated. The first, second, and third paragraphs of the introduction have been revised to be more concise following the reviewer's suggestion.

Point 3: Author should include an error bar where it is necessary.

Response 3: The authors would like to thank you for the helpful comments from the reviewer. The error bar has been provided in Figure 3.

Point 4: Volatile matter is nearly 50 %. How author justify the product content in figure 1? **Response 4:** The great suggestion from the reviewer is appreciated. Please note that all experiments were carried out twice to ensure reproducibility. The yield of the products is calculated using the Eqs. (3)-(5).

Point 5: Author should increase the temperature to 800 °C and increase reaction time because it reflects the increasing gas and oil product.

Response 5: The authors would like to thank you for the excellent recommendation from the reviewer. We agree that the pyrolysis temperature could be higher (above 600 °C) and longer reaction time to increase the gas and oil products. However, it would be better to investigate the detailed higher temperature and longer reaction time in the future study. The authors would like to ask the reviewer's understanding.

[Sustainability] Manuscript ID: sustainability-1602796 - Manuscript Resubmitted External Index ×



Fri, Mar 4, 8:50 AM

Sustainability Editorial Office

to me, Apip, Asep, Novi, Edy, Navid, Surachai, Yukihiko 💌

Dear Dr. Farobie,

Thank you very much for resubmitting the modified version of the following manuscript:

Manuscript ID: sustainability-1602796 Type of manuscript: Article Title: Slow-pyrolysis of Ulva lactuca (Chlorophyta) for sustainable production of bio-oil and biochar Authors: Apip Amrullah, Obie Farobie *, Asep Bayu, Novi Syaftika, Edy Hartulistiyoso, Navid R Moheimani, Surachai Karnjanakom, Yukihiko Matsumura Received: 2 February 2022 E-mails: apip.amrullah@ulm.ac.id, obiefarobie@apps.ipb.ac.id, edyhartulistiyoso@apps.ipb.ac.id, asepbayu@yahoo.co.id, novi017@brin.go.id, n.moheimani@murdoch.edu.au, surachai.ka@rsu.ac.th, mat@hiroshima-u.ac.jp

Gambar 3. Bukti submission manuskrip yang telah direvisi (4 Maret 2022).

[Sustainability] Manuscript ID: sustainability-1602796 - Minor Revisions External Index ×

Sustainability Editorial Office

to me, Apip, Asep, Novi, Edy, Navid, Surachai, Yukihiko, Sustainability 💌 Dear Dr. Farobie,

Thank you again for your manuscript submission:

Manuscript ID: sustainability-1602796 Type of manuscript: Article Title: Slow-pyrolysis of Ulva lactuca (Chlorophyta) for sustainable production of bio-oil and biochar Authors: Apip Amrullah, Obie Farobie *, Asep Bayu, Novi Syaftika, Edy Hartulistiyoso, Navid R Moheimani, Surachai Karnjanakom, Yukihiko Matsumura Received: 2 February 2022 E-mails: apip.amrullah@ulm.ac.id, obiefarobie@apps.ipb.ac.id, edyhartulistiyoso@apps.ipb.ac.id, asepbayu@yahoo.co.id, novi017@brin.go.id, n.moheimani@murdoch.edu.au, surachai.ka@rsu.ac.th, mat@hiroshima-u.ac.jp

Your manuscript has been reviewed by experts in the field. Please find your manuscript with the referee reports at this link: https://susy.mdpi.com/user/manuscripts/resubmit/644fd314cbc64b436d7b8c51ef4f3d3b

Gambar 4. Bukti korespondensi bahwa draf manuskrip masih perlu direvisi (revisi tahap 2).

Mon, Mar 7, 9:40 AM

Dear Prof. Dr. Marc A. Rosen/ Dr. Zoe Zhou,

We would like to thank you again for your kind consideration in allowing us to revise our manuscript (ID: sustainability-1602796) entitled "Slow-pyrolysis of *Ulva lactuca* (Chlorophyta) for sustainable production of biooil and biochar." The manuscript has been revised for the second time for better readability according to the suggestions of the Editor and Reviewers by including the response to the reviewers' comments and revised manuscript.

The novelty of this study is highlighted as follows: (1) performing a comprehensive study for co-production of bio-oil and bio-char from *U. lactuca*, including their characteristics which have not been reported elsewhere and (2) providing the detailed reaction pathway for the conversion of *U. lactuca* into high-value compounds identified in bio-oil. The key findings of this study also have been mentioned in the manuscript. We believe that this manuscript will attract the readers of the journal of *Sustainability* and other sustainable energy-related journals since the significance and novelty of this work have already been insisted on.

We again appreciate the kindness of the Editor and Reviewers in helping improve the manuscript. All authors have reviewed and agreed to the submission of the revised manuscript. We are attaching the response to the reviewers' comments below. We also sent the response to each reviewer following the guideline of *Sustainability*. We hope that the manuscript is now acceptable for publication. Please do not hesitate to contact me if there are any questions.

Dr. Eng. Obie Farobie Department of Mechanical and Biosystem Engineering, IPB University. E-mail: obiefarobie@apps.ipb.ac.id Tel: +62812-898-11381

Response to Reviewer 3 Comments (Round 2)

Point 1: Paper can be accepted after minor revision. Author should include error bar in figure 2. **Response 1:** The great suggestion from the reviewer is appreciated. The error bar in figure 2 has been provided following the reviewer suggestion. The manuscript has been revised accordingly. Again, we would like to appreciate for the kindness of reviewer in helping improve the manuscript.

[Sustainability] Manuscript ID: sustainability-1602796 - Accepted for Publication (External) Inhox ×

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Sustainability Editorial Office

Mon, Mar 7, 3:20 PM

to me, Apip, Asep, Novi, Edy, Navid, Surachai, Yukihiko, Sustainability, Zoe 💌 Dear Dr. Farobie,

Congratulations on the acceptance of your manuscript, and thank you for your interest in submitting your work to Sustainability:

Manuscript ID: sustainability-1602796 Type of manuscript: Article Title: Slow-pyrolysis of Ulva lactuca (Chlorophyta) for sustainable production of bio-oil and biochar Authors: Apip Amrullah, Obie Farobie *, Asep Bayu, Novi Syaftika, Edy Hartulistiyoso, Navid R Moheimani, Surachai Karnjanakom, Yukihiko Matsumura Received: 2 February 2022 E-mails: apip.amrullah@ulm.ac.id, obiefarobie@apps.ipb.ac.id, edyhartulistiyoso@apps.ipb.ac.id, asepbayu@yahoo.co.id, novi017@brin.go.id, n.moheimani@murdoch.edu.au, surachai.ka@rsu.ac.th, mat@hiroshima-u.ac.jp

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Gambar 5. Bukti korespondensi bahwa draf manuskrip telah diterima

[Sustainability] Manuscript ID: sustainability-1602796; doi: 10.3390/su14063233. Paper has been published. External Index ×

Sustainability Editorial Office

to me, Sustainability, Zoe 💌

Dear Dr. Farobie,

We are pleased to inform you that "Slow Pyrolysis of Ulva lactuca (Chlorophyta) for Sustainable Production of Bio-Oil and Biochar" by Apip Amrullah, Obie Farobie *, Asep Bayu, Novi Syaftika, Edy Hartulistiyoso, Navid R. Moheimani, Surachai Karnjanakom, Yukihiko Matsumura has been published in Sustainability and is available online:

Abstract: <u>https://www.mdpi.com/2071-1050/14/6/3233</u> HTML Version: <u>https://www.mdpi.com/2071-1050/14/6/3233/htm</u> PDF Version: <u>https://www.mdpi.com/2071-1050/14/6/3233/pdf</u>

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https://www.mdpi.com/journal/sustainability/toc-alert/ to receive notifications.

Gambar 6. Bukti korespondensi bahwa draf manuskrip telah terbit.

Wed, Mar 9, 11:26 PM (1)