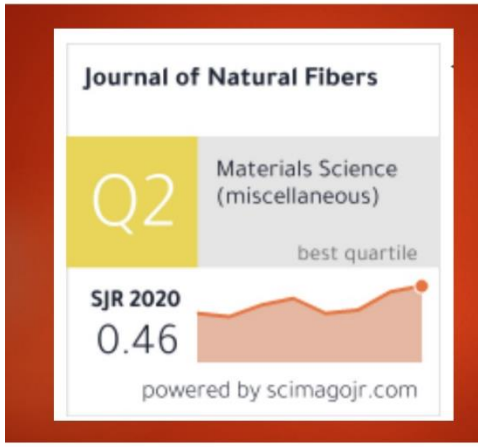
Bukti Korespondensi & Proses Review

Paper 2 Syarat Khusus Ajuan Guru Besar 2023

Isolation and Characterization of New Cellulose Microfibers Pandan Duri (Pandanus tectorius) for Sustainable Environment"

Pengusul
Dr. Edi Syafri, ST, M.Si



Citation metrics

- **3.507 (2021)** Impact Factor
- **Q1** Impact Factor Best Quartile
- **3.760 (2021)** 5 year IF
- **5.6 (2021)** CiteScore (Scopus)
- **Q1 (2021)** CiteScore Best Quartile
- **1.270 (2021)** SNIP
- **0.480 (2021)** SIR

1. Proses Submission

Journal of Natural Fibers - Manuscript ID WJNF-2021-1223 has been submitted online Kotak Masuk ×

Journal of Natural Fibers <onbehalfof@manuscriptcentral.com> Kam, 23 Des 2021, 11.38

kepada saya

Inggris > Indonesia Terjemahkan pesan Nonaktifkan untuk: Inggris ×

22-Dec-2021

Dear Dr Syafri:

Your manuscript entitled "Isolation and Characterization of New Cellulose Microfibers Pandan Duri (Pandanus tectorius) for Sustainable Environment" has been successfully submitted online and is presently being given full consideration for publication in Journal of Natural Fibers.

Your manuscript ID is WJNF-2021-1223.

Please mention the above manuscript ID in all future correspondence or when calling the office for questions. If there are any changes in your street address or e-mail address, please log in to ScholarOne Manuscripts at https://mc.manuscriptcentral.com/jnf and edit your user information as appropriate.

You can also view the status of your manuscript at any time by checking your Author Center after logging in to https://mc.manuscriptcentral.com/jnf.

Thank you for submitting your manuscript to Journal of Natural Fibers.

Sincerely,
Journal of Natural Fibers Editorial Office

Isolation and Characterization of New Cellulose Microfibers Pandan Duri (*Pandanus tectorius*) for Sustainable Environment

Abstract

Cellulose is the main component of natural fibers whose content varies greatly depending on the type of plant and its treatment. Therefore, it is necessary to examine the effect of chemical treatment on natural fiber properties to determine the optimal chemical treatment of its cellulose content. This study successfully extracted the content from Pandan duri (*Pandanus tectorius*) fiber through alkalization, bleaching, and acid hydrolysis. The effect of these chemical treatments on the characteristics of fiber surface, shape, chemical composition, crystallinity, and thermal properties was examined and analyzed. Subsequently, the cellulose components in the structure and the removal of lignin groups were characterized by fourier transform infrared (FTIR) spectroscopy spectrum analysis. The surface morphology analysis was done by scanning electron microscopy (SEM) that showed the raw fiber surface was still in the structure of bundles. However, it was decomposed into cellulose fibrils after chemical treatment with a diameter of about 2µm – 20µm. The chemical composition for cellulose content increased by 90.5%, while hemicellulose decreased by 89.6%

2. Revisi1 Submission

Journal of Natural Fibers - WJNF-2021-1223 - changes required to your submission Kotak Masuk ×

Journal of Natural Fibers <onbehalfof@manuscriptcentral.com> Sel, 28 Des 2021, 21.18

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28-Dec-2021

Dear Dr Edi Syafri:

1. Please delete acknowledgements from the main document and insert them in the title page.
2. Correct your references according to the Chicago style (see attachment).
3. Figures should be supplied in one of Journal preferred file formats: JPEG, TIFF, EPS, PS. Microsoft Word (DOC or DOCX) files are acceptable only for figures that have been drawn in Word.

Your above referenced manuscript, entitled "Isolation and Characterization of New Cellulose Microfibers Pandan Duri (Pandanus tectorius) for Sustainable Environment" requires some further changes before it is ready for reviewing in Journal of Natural Fibers. Your submission has been returned to you and is located in your Author Center as a draft, so that you can make the required changes to it and submit it again.

Your submission along with all files you submitted is now in your Author Center, at https://mc.manuscriptcentral.com/jnf Please read the Quick Guide to Continuing your Submission, which shows how you can access your manuscript, and submit it back to the site. The Guide is located at http://mc.manuscriptcentral.com/societyimages/tandf_qs0/Continuning%20a%20Submission_screenshot.pdf

Your manuscript must be set out in our journal's style before we will review it. Please revise it in line with our style, and submit an improved manuscript. See the Instructions for Authors for details, which can be reached via the "Instructions & Forms" link at the top right of this page.

You may contact the Editorial Office if you have further questions.

Sincerely,

Journal of Natural Fibers - Manuscript ID WJNF-2021-1223 has been submitted online

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Sab, 1 Jan 2022, 22.56

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01-Jan-2022

Dear Dr Syafri:

Your manuscript entitled "Isolation and Characterization of New Cellulose Microfibers Pandan Duri (Pandanus tectorius) for Sustainable Environment" has been successfully submitted online and is presently being given full consideration for publication in Journal of Natural Fibers.

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Please mention the above manuscript ID in all future correspondence or when calling the office for questions. If there are any changes in your street address or e-mail address, please log in to ScholarOne Manuscripts at https://mc.manuscriptcentral.com/jnf and edit your user information as appropriate.

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Thank you for submitting your manuscript to Journal of Natural Fibers.

Sincerely,
Journal of Natural Fibers Editorial Office

3. Decision Revision1 (R1)

Journal of Natural Fibers - Decision on Manuscript ID WJNF-2021-1223 Kotak Masuk ×

Journal of Natural Fibers <onbehalfof@manuscriptcentral.com> Sen, 7 Feb 2022, 16.34
kepada saya

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07-Feb-2022

Dear Dr Syafri:

Your manuscript entitled "Isolation and Characterization of New Cellulose Microfibers Pandan Duri (Pandanus tectorius) for Sustainable Environment", which you submitted to Journal of Na Fibers, has been reviewed. The reviewer comments are included at the bottom of this letter.

The reviewer(s) would like to see some revisions made to your manuscript before publication. Therefore, I invite you to respond to the reviewer(s)' comments and revise your manuscript.

When you revise your manuscript please highlight the changes you make in the manuscript by using the track changes mode in MS Word or by using bold or colored text.

To start the revision, please click on the link below:

*** PLEASE NOTE: This is a two-step process. After clicking on the link, you will be directed to a webpage to confirm. ***

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This will direct you to the first page of your revised manuscript. Please enter your responses to the comments made by the reviewer(s) in the space provided. You can use this space to doc any changes you made to the original manuscript. Please be as specific as possible in your response to the reviewer(s).

This link will remain active until you have submitted your revised manuscript. If you begin a revision and intend to finish it at a later time, please note that your draft will appear in the "Revise Manuscripts in Draft" queue in your Author Center.

Reminder: Your Revision for Journal of Natural Fibers is pending

Kotak Masuk ×

Journal of Natural Fibers <onbehalfof@manuscriptcentral.com>
kepada saya

Sel, 8 Feb 2022, 13.12

Inggris > Indonesia Terjemahkan pesan

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08-Feb-2022

Dear Dr Edi Syafri:

Recently, you received a decision on Manuscript ID WJNF-2021-1223, entitled "Isolation and Characterization of New Cellulose Microfibers Pandan Duri (Pandanus tectorius) for Sustainable Environment." This email is simply a reminder that your revision is due on 28-Feb-2022.

To start the revision, please click on the link below:

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https://mc.manuscriptcentral.com/jnf?URL_MASK=3c5984c446c4417382cb3f17670c28fc

This will direct you to the first page of your revised manuscript in your Author Center. The manuscript and decision letter are located here also.

This link will remain active until you have submitted your revised manuscript. If you have already begun a revision, you can click on the link to continue your revision. Please note that your draft will appear in the “Revised Manuscripts in Draft” queue in your Author Center.

If it is not possible for you to submit your revision by 28-Feb-2022, we will consider your paper as a new submission.

Please contact the Editorial Office if you are unable to submit within this time.

Journal of Natural Fibers - Manuscript ID WJNF-2021-1223.R1 has been submitted online

Journal of Natural Fibers <onbehalfof@manuscriptcentral.com>
kepada saya

Rab, 9 Feb 2022, 19.00

Inggris > Indonesia Terjemahkan pesan

Nonaktifkan untuk: Inggris

09-Feb-2022

Dear Dr Syafri:

Your manuscript entitled "Isolation and Characterization of New Cellulose Microfibers from Pandan Duri (Pandanus tectorius) for Sustainable Environment" has been successfully submitted online and is presently being given full consideration for publication in Journal of Natural Fibers.

Your manuscript ID is WJNF-2021-1223.R1.

Please mention the above manuscript ID in all future correspondence or when calling the office for questions. If there are any changes in your street address or e-mail address, please log in to ScholarOne Manuscripts at https://mc.manuscriptcentral.com/jnf and edit your user information as appropriate.

You can also view the status of your manuscript at any time by checking your Author Center after logging in to https://mc.manuscriptcentral.com/jnf.

Thank you for submitting your manuscript to Journal of Natural Fibers.

Sincerely,
Journal of Natural Fibers Editorial Office

Journal of Natural Fibers - WJNF-2021-1223.R1 - changes required to your submission

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Kam, 24 Feb 2022, 22.04

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24-Feb-2022

Dear Dr Edi Syafri:

Please correct your references accoridng to the Chicago style (see attachment).

Your above referenced manuscript, entitled "Isolation and Characterization of New Cellulose Microfibers from Pandan Duri (Pandanus tectorius) for Sustainable Environment" requires some further changes before it is ready for reviewing in Journal of Natural Fibers. Your submission has been returned to you and is located in your Author Center as a draft, so that you can make the required changes to it and submit it again.

Your submission along with all files you submitted is now in your Author Center, at https://mc.manuscriptcentral.com/jnf Please read the Quick Guide to Continuing your Submission, which shows how you can access your manuscript, and submit it back to the site. The Guide is located at http://mc.manuscriptcentral.com/societyimages/tandf_qs0/Continuning%20a%20Submission_screenshot.pdf

Your manuscript must be set out in our journal's style before we will review it. Please revise it in line with our style, and submit an improved manuscript. See the Instructions for Authors for details, which can be reached via the "Instructions & Forms" link at the top right of this page.

You may contact the Editorial Office if you have further questions.

Sincerely,

Journal of Natural Fibers Editorial Office

4. Decision Revision2 : Minor Revision

Journal of Natural Fibers - Decision on Manuscript ID WJNF-2021-1223.R1 Kotak Masuk ×

Journal of Natural Fibers <onbehalfof@manuscriptcentral.com> Jum, 6 Mei 2022, 15.15

kepada saya

Inggris > Indonesia Terjemahkan pesan Nonaktifkan untuk: Inggris ×

06-May-2022

Dear Dr Edi Syafri:

Your manuscript entitled "Isolation and Characterization of New Cellulose Microfibers from Pandan Duri (Pandanus tectorius) for Sustainable Environment", which you submitted to Journal of Natural Fibers, has been reviewed. The reviewer comments are included at the bottom of this letter.

The reviews are in general favourable and suggest that, subject to minor revisions, your paper could be suitable for publication. Please consider these suggestions, and I look forward to receiving your revision.

When you revise your manuscript please highlight the changes you make in the manuscript by using the track changes mode in MS Word or by using bold or colored text.

To start the revision, please click on the link below:

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This link will remain active until you have submitted your revised manuscript. If you begin a revision and intend to finish it at a later time, please note that your draft will appear in the "Revised

5. Submission Revision2 (R2)

Revison 2 : Manuscript ID WJNF-2021-1223.R1

Edi Syafri <edisyafri11@gmail.com>
kepada ryszard.kozlowski, rkscience.biuro

Sab, 14 Mei 2022, 10.40

May 13, 2022

Dear Editor Journal of Natural Fibers

We cordially apologize for your any inconvenience.

Sir/Madam, we are very pleased to be able to communicate you. We have revised our manuscript according your suggestion. We would be happy to provide any additional information you may need regarding our manuscript.

We would like to thank the editors and reviewers who gave their valuable recommendations to make our work more appealing and informative. Authors have revised the manuscript and incorporate all the suggestions. The point wise response to the comments given by the reviewers is provided in the following section.

Thank you again for your valuable time and kind consideration.

We will look forward your amiable reply

Journal of Natural Fibers - Decision on Manuscript ID WJNF-2021-1223.R2

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Journal of Natural Fibers <onbehalfof@manuscriptcentral.com>
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Sab, 14 Mei 2022, 15.41

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14-May-2022

Dear Dr Edi Syafri:

Ref: Isolation and Characterization of New Cellulose Microfibers from Pandan Duri (Pandanus tectorius) for Sustainable Environment

Our reviewers have now considered your paper and have recommended publication in Journal of Natural Fibers. We are pleased to accept your paper in its current form which will now be forwarded to the publisher for copy editing and typesetting. The reviewer comments are included at the bottom of this letter, along with those of the editor who coordinated the review of your paper.

You will receive proofs for checking in due course.

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The publisher also requests that proofs are checked through the publisher's tracking system and returned within 48 hours of receipt. Your paper will be exported to Taylor&Francis by us and the editorial system in due course (there is a queue). It will take some more weeks until you will be contacted by Taylor & Francis for Proof and Agreement. Check your e-mails.

Thank you for your contribution to Journal of Natural Fibers and we look forward to receiving further submissions from you.

Sincerely,

Proses Review Manuskrip

To:	edisyafri11@gmail.com
CC:	
Subject:	Journal of Natural Fibers - Decision on Manuscript ID WJNF-2021-1223

Body:

Dear Dr Syafri:

Your manuscript entitled "Isolation and Characterization of New Cellulose Microfibers Pandan D
you submitted to Journal of Natural Fibers, has been reviewed. The reviewer comments are inc

The reviewer(s) would like to see some revisions made to your manuscript before publication.
comments and revise your manuscript.

When you revise your manuscript please highlight the changes you make in the manuscript by
colored text.

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your draft will appear in the "Revised Manuscripts in Draft" queue in your Author Center.

IMPORTANT: Your original files are available to you when you upload your revised manuscript
submission.

Because we are trying to facilitate timely publication of manuscripts submitted to Journal of Na
28-Feb-2022. If it is not possible for you to submit your revision by this date, we may have to

Once again, thank you for submitting your manuscript to Journal of Natural Fibers and I look fo

Sincerely,
Professor Kozlowski
Editor in Chief, Journal of Natural Fibers
ryszard.kozlowski@escorena.net, rkscience.biuro@gmail.com

Reviewer(s)' Comments to Author:

Reviewer: 1

Comments to the Author
The investigation points out the differences from related research also using acidic hydrolysis.
promising alternatives for designing eco-friendly treatments for obtaining cellulose microfibres
The paper has a valuable contribution in the area of the circular economy. The relevance of the
products obtained as reinforces of different composites. The vegetable wastes could be conside
Some prepositions may be missing from the title.
The introduction correctly highlights the current concerns in the area of bio-economy. The met
connections with others' work are declared.
Improving English readability and writing style is recommended, starting from the abstract sec
necessary to examine the effect of chemical treatment on natural fiber properties to determine
Meanwhile, the preparation which was previously coated with carbon was subsequently coated
Please explain this statement:" This is due to its several irregular amorphous areas.-pg. 6, ln.
Based on the specific characteristics determined after the investigation made and making conn
literature, please give examples for products' in which the cellulose microfibres obtained may a

	The paper could be accepted for publication after minor changes. The article has to be revised specified in the reviewer's comments. Reviewer: 2 Comments to the Author In this paper the authors studied the Pandanus tectorius fibers before and after some chemica composition, surface morphology by SEM, Fourier transform infrared - FTIR, x-ray diffraction - Comments: Page 4, line 30 the authors states: Lignin and hemicellulose were extracted by alkaline treatm The temperature is very high for cellulose fibers. I believe that under these conditions, the deg °C or 80 °C? Page 4, line 41 the number 2 is subscribed for sodium chlorite chemical. Page 4, line 48 the authors states that acid hydrolysis treatment was carried out at a hotplate hour with a ratio of fiber weight to a solution of 1:20. This conditions are very aggressive for cellulose fibers.The temperature is very high. The temp Page 5, line 56 the authors states that I002 = Intensity for 2θ=22.3°, which indicated the crys 2θ=22.3° is relative to the crystalline phase plus amorphous phase. Page 7, line 56 to page 8, line 3 the authors states: The presence of cellulose was also indicate According to these results, the intensity of the band was reduced after chemical treatment con Explain the results, once the non-cellulose components was removed. Page 8, line 7 the authors states that significant increase in the value (71.31%) after acid hydr amorphous structure. What the author think about the acid hydrolysis of the cellulose amorphous phase? Is possible In the chemical composition results, the lignin content is relative to the total lignin (insoluble li Overall, the paper provides informations from fiber characterization, there are no results about	
	07-Feb-2022	

RESPONSE TO REFEREES

General comment: We would like to thank the editors and reviewers who gave their valuable recommendations to make our work more appealing and informative. Authors have revised the manuscript and incorporate all the suggestions. The point wise response to the comments given by the reviewers is provided in the following section.

Reviewer: 1

Comments to the Author

The investigation points out the differences from related research also using acidic hydrolysis. It introduces the idea that might stimulate others to develop promising alternatives for designing eco-friendly treatments for obtaining cellulose microfibres from Pandanus tectorius. The paper has a valuable contribution in the area of the circular economy. The relevance of the information presented opens new perspectives to view the products obtained as reinforces of different composites. The vegetable wastes could be considered practical alternatives for raw materials.

Thank you for your positive comments and feedback on our paper.

Some prepositions may be missing from the title.

We have modified our paper title as per your suggestion.

The introduction correctly highlights the current concerns in the area of bio-economy. The methods used are clearly explained and presented. Relevant connections with others' work are declared.

We are very much grateful for the insightful comments and for providing valuable suggestions to improve the quality of our paper. Changes have been highlighted.

Improving English readability and writing style is recommended, starting from the abstract section. The same word is repeated in the phrase:" Therefore, it is necessary to examine the

effect of chemical treatment on natural fiber properties to determine the optimal chemical treatment of its cellulose content….. Meanwhile, the preparation which was previously coated with carbon was subsequently coated with gold to reduce the electron charge."

Thank you very much for your valuable comments. The typographical error and tenses are corrected with the help of a language expert and updated in the revised manuscript.

Please explain this statement:" This is due to its several irregular amorphous areas.-pg. 6, ln. 47", including if it is possible references.

Thank you very much for your valuable suggestion to improve the quality of the manuscript. The changes have been amended in the revised manuscript according to your valuable comments.

Based on the specific characteristics determined after the investigation made and making connections with the information found within the particular literature, please give examples for products' in which the cellulose microfibres obtained may add value.

Thank you very much for your valuable suggestion to improve the quality of the manuscript. The changes have been amended in the revised manuscript according to your valuable comments.

The paper could be accepted for publication after minor changes. The article has to be revised by the author(s) and resubmitted with suggested modifications specified in the reviewer's comments.

We are thankful for the valuable comments and acceptance provided by your side.

We have incorporated all mistakes in the revised manuscript.

Reviewer: 2

Comments to the Author

In this paper the authors studied the Pandanus tectorius fibers before and after some chemical treatments. The fibers were characterized by chemical composition, surface morphology by SEM, Fourier transform infrared - FTIR, x-ray diffraction - XRD and thermogravimetric analysis - TGA.

We appreciate the time and effort that you and the reviewers dedicated to providing feedback on our manuscript. We are very much grateful for the insightful comments and for providing valuable suggestions to improve the quality of our paper.

Comments:

Page 4, line 30 the authors states: Lignin and hemicellulose were extracted by alkaline treatment of 5% (w/v) NaOH for 2 hours at 800 °C on a hotplate. The temperature is very high for cellulose fibers. I believe that under these conditions, the degradation of cellulose will occur. The temperatura used was 800 °C or 80 °C?

It is 80 °C. The typographical error is corrected and updated in the revised manuscript.

Page 4, line 41 the number 2 is subscribed for sodium chlorite chemical. Page 4, line 48 the authors states that acid hydrolysis treatment was carried out at a hotplate temperature of 500°C with 200 rpm with 5 M HCl solution for 1 hour with a ratio of fiber weight to a solution of 1:20. This conditions are very aggressive for cellulose fibers.The temperature is very high. The temperatura used was 500 °C or 50 °C?

It is 50 °C. The typographical error is corrected and updated in the revised manuscript.

Page 5, line 56 the authors states that I002 = Intensity for 2θ=22.3°, which indicated the crystal region. But, according the Segal method the intensity in 2θ=22.3° is relative to the crystalline phase plus amorphous phase.

The changes have been amended in the revised manuscript according to your valuable comments.

Page 7, line 56 to page 8, line 3 the authors states: The presence of cellulose was also indicated by the bands of 2903 cm-1 which is the C–H strain. According to these results, the intensity of the band was reduced after chemical treatment compared to raw fiber.

Explain the results, once the non-cellulose components was removed.

The following peak, identified in the region 1400 cm^{-1} to 1425 cm^{-1} for both raw and chemically treated fibers, confirms the vibrational stretching of CH_2 denotes the presence of aromatic rings in hemicellulose (Vijay et al. 2019). The vibration of C-O attributed to the acetyl groups in lignin is shown by a small intensity peak in the region 1200 cm^{-1} to 1225 cm^{-1} (Vinod et al. 2021). The last intensity peak, which can be found in the range of 1025 cm^{-1} to 1035 cm^{-1}, reflects the stretching and vibration of C-H in the presence of cellulose (Shravanabelagola Nagaraja Setty et al. 2020).

Page 8, line 7 the authors states that significant increase in the value (71.31%) after acid hydrolysis treatment is due to the reduced non-cellulose amorphous structure. What the author think about the acid hydrolysis of the cellulose amorphous phase? Is possible in this case?

We are not sure about this. So, we will consider it as a scope for future work.

In the chemical composition results, the lignin content is relative to the total lignin (insoluble lignin + soluble lignin)?

It denotes total lignin content in the fiber.

Overall, the paper provides informations from fiber characterization, there are no results about potential application. It's well written in my point of view.

We are thankful for the valuable comments and acceptance provided by your side.

We have incorporated all mistakes in the revised manuscript.

Revisi 2

From: ryszard.kozlowski@escorena.net

To: edisyafri11@gmail.com

CC:

Subject: Journal of Natural Fibers - Decision on Manuscript ID WJNF-2021-1223.R1

Body: 06-May-2022

Dear Dr Edi Syafri:

Your manuscript entitled "Isolation and Characterization of New Cellulose Microfibers from Pandan Duri (Pandanus tectorius) for Sustainable Environment", which you submitted to Journal of Natural Fibers, has been reviewed. The reviewer comments are included at the bottom of this letter.

The reviews are in general favourable and suggest that, subject to minor revisions, your paper could be suitable for publication. Please consider these suggestions, and I look forward to receiving your revision.

When you revise your manuscript please highlight the changes you make in the manuscript by using the track changes mode in MS Word or by using bold or colored text.

To start the revision, please click on the link below:

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Once again, thank you for submitting your manuscript to Journal of Natural Fibers and I look forward to receiving your revision.

Sincerely,
Professor Kozlowski
Editor in Chief, Journal of Natural Fibers

ryszard.kozlowski@escorena.net, rkscience.biuro@gmail.com

Reviewer(s)' Comments to Author:

Reviewer: 1

Comments to the Author
The authors responded properly to most of the suggestions.
The recommendation:" Based on the specific characteristics determined after the investigation made and making connections with the information found within the particular literature, please give examples for products' in which the cellulose microfibres obtained may add value.", should be solved gradually in the" Results and Discussion" section or" Conclusions".

The paper could be accepted for publication after minor changes. The article has to be revised by the author(s) and resubmitted with suggested modifications specified in the reviewer's comments.

Editor's Comments to Author:
Please, make this revision quickly, submit and inform me about it by E-mail (to speed up). we deal with 1200 papers/year.

Date Sent: 06-May-2022

RESPONSE TO REFEREES

General comment: We would like to thank the editors and reviewers who gave their valuable recommendations to make our work more appealing and informative. Authors have revised the manuscript and incorporate all the suggestions. The point wise response to the comments given by the reviewers is provided in the following section.

Reviewer: 1

Comments to the Author

The authors responded properly to most of the suggestions.

Thank you for your positive comments and feedback on our paper.

The recommendation:" Based on the specific characteristics determined after the investigation made and making connections with the information found within the particular literature,

please give examples for products' in which the cellulose microfibres obtained may add value.", should be solved gradually in the" Results and Discussion" section or" Conclusions".

Thank you for your positive comments and we have answered this at conclusion part.

The paper could be accepted for publication after minor changes. The article has to be revised by the author(s) and resubmitted with suggested modifications specified in the reviewer's comments.

We are thankful for the valuable comments and acceptance provided by your side.

We have incorporated all mistakes in the revised manuscript.

Revisi 2

Isolation and Characterization of New Cellulosic Microfibers from Pandan Duri (*Pandanus tectorius*) for Sustainable Environment

Abstract

Cellulose is the main component of natural fibers whose content varies greatly depending on the type of plant and its treatment. Therefore, it is necessary to examine the effect of chemical treatment on natural fiber properties. This study successfully extracted the content from Pandan duri (*Pandanus tectorius*) fiber through alkalization, bleaching, and acid hydrolysis. The effect of these chemical treatments on the characteristics of fiber surface, shape, chemical composition, crystallinity, and thermal properties was examined and analyzed. Subsequently, the cellulose components in the structure and the removal of lignin groups were characterized by Fourier transform infrared (FTIR) spectroscopy spectrum analysis. The surface morphology analysis was

done by scanning electron microscopy (SEM) that showed the raw fiber surface was still in the structure of bundles. However, it was decomposed into cellulose fibrils after chemical treatment with a diameter of about 2μm – 20μm. The chemical composition for cellulose content increased by 90.5%, while hemicellulose decreased by 89.6% after acid hydrolysis treatment. Also, X-ray (XRD) analysis showed crystallinity increased from 39.5% for raw fibers to 67.7% after the hydrolysis. Thermal gravimetric analysis (TGA) showed higher degradation temperature of micro cellulose offered better thermal stability compared to raw fibers. In conclusion, the cellulose from *Pandanus tectorius* fiber can be used to reinforce biocomposites as an alternative to synthetic fibers for sustainability of environment.

Keywords: *Pandanus tectorius*, Chemical Treatment, Cellulose Fiber, Crystallinity, Thermal stability, Chemical Composition

Introduction

Residues from agricultural waste are not optimally used due to lack of waste utilization and management to improve their use-value, therefore, they are usually buried or burnt (Sanjay et al. 2018; Vinod et al. 2020). The primary components of plant residues in natural fibers are natural biopolymers, such as cellulose, hemicellulose, and lignin (Manimaran et al. 2018; Zhang et al. 2015; Yang et al. 2007). Cellulose can be used as reinforcement in specific applications like food packaging (Mahardika et al. 2019; Syafri et al. 2019; Asrofi, Abral, Kurnia, et al. 2018; Rangappa et al. 2021; Syafri et al. 2017; 2018), biomedical field, and electro-conductive (Ul-Islam et al. 2015). The superior characteristics of its fibers include high mechanical strength or stiffness, biodegradability, good thermal resistance properties, recyclability, abundant availability in nature, low density, and cost (Mahmud et al. 2021;

Masmoudi et al. 2016; Nagarajan et al. 2021). With these advantages, it can be used as a reinforcement and filler in biocomposite polymers, structural applications, insulation materials, and automotive applications, such as car panels as well as other interior applications (Adesina et al. 2019; Sari et al. 2021; Muthu Chozha Rajan et al. 2020; Rangappa et al. 2022; Madhu et al. 2020).

Several previous studies that have utilized this component in the manufacture of biocomposites include cellulose nanocrystals with chitosan (Adel et al. 2019), polyester with bacterial cellulose reinforcement (Panaitescu, Frone, and Chiulan 2016), PLA with cellulose nanocrystal reinforcement (Yin et al. 2017), and others. Recently, various new sources of cellulose have been identified and characterized, namely cellulose in bark *Thespesia populnea* fiber (Kathirselvam et al. 2019), *roselle* fiber (Kian et al. 2017), *oil palm mesocarp* (Chieng et al. 2017), *Acacia nilotica* L. (Kumar et al. 2020), *Citrullus lanatus* (Khan, Vijay, et al. 2020), *Phaseolus vulgaris* (Babu et al. 2020), *Vachellia farnesiana* (Vijay et al. 2020), *Momordica Charantia* (Khan, Raghunathan, et al. 2020), *Eucalyptus* spp. (Carrillo-Varela, Pereira, and Mendonça 2018) and other fibers. Pandan duri *(Pandanus tectorius)* grows a lot in tropical areas like Indonesia and its fiber potentially serves as auspicious source of cellulose, considering its abundant availability in the country.

Several chemical treatments that have been successfully used for cellulose extraction include alkalization, bleaching, and acid hydrolysis (Abral, Ariksa, et al., 2019; Afolabi et al., 2019; Ilyas et al., 2018; Mahardika et al., 2018; Sanjay et al. 2019; Owolabi & Megat-Yusoff, 2018). Ilyas et al. 2018 reported that alkalizing treatment succeeded in increasing the cellulose content of sugar palm fibers by 46.7% compared to those without treatment (Ilyas, Sapuan, and Ishak 2018). Previous studies on the extraction of cellulose from *Pandanus tectorius* (Screwpine) only used alkali and combined alkali-bleach treatment. (Owolabi et al., 2019). The

results obtained a maximum cellulose content of 78% at the concentration of 2 wt% NaOH for 120 minutes and 2 wt% $NaClO_2$ (Owolabi et al., 2019).

Furthermore, the process of extracting the component using this study's method has not been examined by previous research teams. This study aims to determine the characteristics of the fiber before and after chemical treatment. The fiber was extracted by different chemical treatments, such as alkalization, bleaching, and acid hydrolysis. Furthermore, it was analyzed for XRD, FTIR, thermal properties, and its surface morphology was observed using SEM. This study will help compare Pandanus tectorius fiber's characteristics with other natural fibers and determine the optimal cellulose content with various chemical treatments. After being processed, the material properties of the species showed that the development of the business was minimal.

Materials and Methods

Materials

The primary material used in this study was Pandan duri (PD) (*Pandanus tectorius*) leaves as the primary source of cellulose fiber. The leaves were obtained from a plantation area in Harau, Kabupaten Lima Puluh Kota, West Sumatera. The thorns on its edges were cleaned and cut 5-10 cm long, it was subsequently dried in the sun for four days with a moisture content of approximately 9 to 10%. Furthermore, the leaf's fibers were decomposed using a blender.

The Pure Analysis chemicals used include sodium hydroxide (NaOH 98% Sigma-Aldrich brand, HCl (KOH brand Millipore), sodium chlorite ($NaClO_2$ brand Pubchem), and glacial acetic acid (CH_3COOH).

Fiber Extract

The chemical methods used for fiber extraction include alkalization, bleaching, and acid hydrolysis treatment. Lignin and hemicellulose were extracted by alkaline treatment of 5% (w/v) NaOH for 2 hr at 80°C on a hotplate. The brown-colored fibers were washed until they were alkali-free with a pH of 7.

Alkalized PD fibers were bleached using a solution consisting of equal parts (v:v) acetic buffer (27 g NaOH and 75 mL glacial acetic acid, diluted to 1 L distilled water), and dilute sodium chlorite (1.7 wt% NaClO2). This treatment was repeated twice for 1 hr at 80°C, resulting in white PD fibers which were subsequently hydrolyzed with 5 M HCl solution for 1 hr with a ratio of fiber weight to a solution of 1:20. This process was carried out at a hotplate temperature of 50°C with 200 rpm. The hydraulic fibers in particles are known as Cellulose Micro Fibers (CMF) Pandan Berduri (PD). The extraction process is shown in Figure 1.

Insert Fig. 1 here

Analysis of Chemical Composition

Chemical composition analysis was based on the test method developed by Van Soest. Natural fiber consists of fiber soluble in neutral detergent (*Neutral Detergent Fiber*/NDF), soluble in acid detergent (*Acid Detergent Fiber*/ADF), hemicellulose, cellulose, and lignin. The Van Soest method can determine the content of cellulose, hemicellulose, and lignin in the PD fiber.

Scanning Electron Microscopy (SEM)

SEM observations were used to determine the shape of the fiber surface before and after the chemical treatment. Meanwhile, the surface morphology of the cellulose was observed using Scanning Electron Microscopy (SEM), Model: S-3400N, Hitachi, Ltd., Japan, with a voltage of 20 kV and a current of 8 mA probe. The test sample was placed on the SEM sample stub. Meanwhile, the preparation which was previously coated with gold to reduce the electron charge and obtain image clarity.

X-ray Diffraction (XRD)

The crystallinity index of the fibers before and after chemical treatment was measured using the X-ray diffraction (XRD) technique through X'pert PROPANalytical (Model: PW3040/60) with Cu Kα radiation (λ = 0.1542 nm). The spectrum was recorded between 5° and 50° at 40 kV and 30 mA. The crystallinity index (I_{cr}) was calculated using this formula:

$$CI = [(I_{002} - I_{am})/I_{002}] \times 100 \quad (1)$$

Where I_{002} = Intensity for 2θ=22.3°, which indicated the crystal region. I_{am} is an amorphous region at Intensity 2θ=18° (Segal et al. 1958).

Fourier Transform Infrared (FTIR)

FTIR characterization using PerkinElmer FTIR spectrometer (Frontier instrument, USA). This test was used to identify free functional groups from the fibers before and after chemical treatment. Spectrum scans were recorded with 4 cm^{-1} over a wavenumber range of 4000-600 cm^{-1} (Abral, Satria, et al. 2019).

Thermogravimetric Analysis (TGA)

Measurement of the fiber's thermal stability without and after chemical treatment was carried out using the DTG-60 SHIMADZU (Kyoto, Japan). Furthermore, thermal analysis was conducted in a nitrogen atmosphere at a flow rate of 50 mL/min, a heating rate of ten °C/min, and a range temperature of 30-550°C.

Results and Discussion

Chemical Composition

The chemical composition of the PD fiber is presented in Table 1. According to the table, the cellulose content was 43.21% which is lower compared to other natural fiber sources, such as *Eucalyptus* (50.30%) (Carrillo-Varela, Pereira, and Mendonça 2018), banyan tree roots (67.32%) (Ganapathy et al. 2019), and sugar palm fibers (43.88) (Ilyas, Sapuan, and Ishak 2018). This is due to its several irregular amorphous areas (Karakoti et al. 2018; Saravanakumaar et al. 2018; Senthamaraikannan et al. 2018).

Insert Table 1 here

After the 5% NaOH alkalizing treatment, the cellulose content increased by 64.92%, while the lignin content decreased by 68.10% compared to raw fiber. The reduction in this content was due to the dissolution by alkalizing treatment. The bleaching process succeeded in dissolving hemicellulose with a reduction of 89.62% compared to raw fiber. Most of the lignin and hemicellulose were extracted from the pandan duri fiber, hence, increasing the degree of cellulose crystallinity as well as the strength and thermal properties of the fiber (Asrofi, Abral, Kasim, et al. 2018; Ilyas, Sapuan, and Ishak 2018).

Morphological Analysis

The fiber's surface looks rough, as shown in Figure 2a with a magnification of 750 times. According to the image, the diameter of its bundle before chemical treatment was ± 20µm. Figure 2a showed the presence of fibril-shaped fiber bundles that are still bound to lignin, pectin, and hemicellulose (Mahardika et al. 2018). Meanwhile, Figure 2b showed a smoother surface due to alkalization treatment. The alkalization process can remove impurities, such as wax and fatty substance on the surface (Kathirselvam et al. 2019). After the bleaching process, the bundle's length, as well as the lignin and hemicellulose levels were reduced, as shown in Figure 2c. These results were supported by testing the chemical composition of the fiber according to previous studies. Meanwhile, Figure 2d showed a very smooth fiber surface with

the shape of fibrils after acid hydrolysis treatment. The results indicated that the treatment increased the cellulose content by 90.5% compared to raw fiber.

Insert Fig. 2 here

Infrared spectroscopy analysis

The hydrophilic nature of cellulose with free hydroxyl groups regulates the water absorption of the fiber and its water absorption ability (Asrofi et al. 2017). The fiber's functional groups were identified through FTIR spectrum results, as shown in Figure 3. The wavelength in the range of 3331 cm^{-1} showed hydroxyl group (O–H) of cellulose compounds in the structure (Zhang et al. 2015). The presence of cellulose was also indicated by the bands of 2903 cm^{-1} which is the C–H strain (Khan, Raghunathan, et al. 2020). According to these results, the intensity of the band was reduced after chemical treatment compared to raw fiber. The strain of the carbonyl group (C–O) in lignin and hemicellulose was shown at wavenumbers 1732 cm^{-1} and 1647 cm^{-1} (Yang et al. 2007). Figure 2 showed the disappearance of the peak at a wavenumber of 1732 cm^{-1} which indicated that the treatment succeeded in extracting the lignin content. These results were also supported by chemical composition testing (Table 1) and previous studies (Mahardika et al. 2018). The following peak, identified in the region 1400 cm^{-1} to 1425 cm^{-1} for both raw and chemically treated fibers, confirms the vibrational stretching of CH_2 denotes the presence of aromatic rings in hemicellulose (Vijay et al. 2019). The vibration of C-O attributed to the acetyl groups in lignin is shown by a small intensity peak in the region 1200 cm^{-1} to 1225 cm^{-1} (Vinod et al. 2021). The last intensity peak, which can be found in the range of 1025 cm^{-1} to 1035 cm^{-1}, reflects the stretching and vibration of C-H in the presence of cellulose (Shravanabelagola Nagaraja Setty et al. 2020).

Insert Fig. 3 here

X-ray Diffraction (XRD) Analysis

The XRD curve of the sample is shown in Figure 4. According to this analysis, the crystallinity index of the fiber can be calculated before and after chemical treatment. The X-ray diffraction pattern was similar to the previous study's, which showed the intensity diffraction peaks around 16.2°, 22.3°, and 34.6° for Pandan Duri fiber as a typical polymorph of cellulose I (Supian et al. 2020; Mahardika et al. 2018). The crystallinity index was calculated using the Segal equation (Segal et al. 1958). The 22.3° indicated the crystalline plus amorphous regions of cellulose and and 18°peaks is relative to the amorphous phase. The crystallinity index value is shown in Table 1. According to the measurement results, the crystallinity index was 39.49%. Meanwhile, there was a significant increase in the value (71.31%) after acid hydrolysis treatment. This is due to the reduced non-cellulose amorphous structure, and the increased crystalline structure (Abral, Ariksa, et al. 2019). These results were supported by testing the chemical composition of the fibers (Table 1) and previous studies (Ilyas, Sapuan, and Ishak 2018).

Insert Fig. 4 here

Thermogravimetric Analysis (TGA)

The thermal stability was tested before and after chemical treatment by using thermogravimetric analysis. Figure 5a,b showed the thermogravimetric analysis (TGA) and derived thermogravimetric curves (DTG). There were three regions of temperature drop which occurred around 100°C, 250-400°C, and 400-550°C, respectively. The weight loss of about 100°C was probably due to the evaporation of water (Abral et al. 2021). This is indicated as a small trough on the left side of the DTG curve. All samples showed a large weight loss in the 250-400°C region as the cellulose was depolymerized and damaged (Khan, Raghunathan, et al. 2020). After the alkalization, bleaching, and hydrolyzation treatments, the degradation

temperatures of raw fiber were 309, 354, 357, and 346°C, respectively, as shown in the DTG curve (Figure 5b). Therefore, it had thermal stability than chemically treated fiber.

Insert Fig. 5 here

Based on 400-550°C (region 3), all samples completely decomposed to ash (Vijay et al. 2020). However, raw fiber had more residue compared to the chemically treated counterpart due to the leftover non-cellulose content. A similar trend can be observed in previous studies (Mahardika et al. 2018; Babu et al. 2020). The most significant TGA result was the higher bleaching temperature of the fiber degradation (357°C) which showed superior thermal stability over the raw fiber (309°C).

Conclusion

This study aims to extract cellulose from pandan duri fiber through alkalization, bleaching, and acid hydrolysis chemical treatments. According to the SEM observation, after acid hydrolysis treatment, the fiber's surface was smooth and in the form of long fibrils compared to the raw fiber, which was in the form of bundles with a rough surface. The FTIR spectrum Intensity indicated the presence of cellulose-related functional groups in the structure. Furthermore, the XRD results showed that the highest crystallinity index (67.65%) was indicated by fiber treated with acid hydrolysis. Meanwhile, the bleached sample showed the most increased thermal stability with a maximum degradation temperature of 357°C. Therefore, the results showed the superior characteristics of pandan duri fiber in its cellulose content compared to others. From the results, it is noteworthy to mention that the cellulosic microfibers from Pandan Duri (Pandanus tectorius) can be used to produce products for different applications such as such as personal protective textiles, skin grafts, tissue engineering scaffolds, and wound dressings, etc., and also it can be a competitive nanofabrication filler.

Competing Interests

“The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.”

Author Contribution

“All the authors contributed equally for data curation, formal analysis, methodology, conceptualization, project administration, resources, investigation, supervision and writing.”

Data Availability

“All data generated or analyzed during this study are included in this published article.”

Animal Research (Ethics)

“This article does not contain any studies with human participants or animals performed by the authors.”

Consent to Participate (Ethics)

“The authors give the consent to participate this research study.”

Consent to Publish (Ethics)

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Decision Letter (WJNF-2021-1223.R2)

From: ryszard.kozlowski@escorena.net
To: edisyafri11@gmail.com
CC:
Subject: Journal of Natural Fibers - Decision on Manuscript ID WJNF-2021-1223.R2
Body: 14-May-2022

Dear Dr Edi Syafri:

Ref: Isolation and Characterization of New Cellulose Microfibers from Pandan Duri (Pandanus tectorius) for Sustainable Environment

Our reviewers have now considered your paper and have recommended publication in Journal of Natural Fibers. We are pleased to accept your paper in its current form which will now be forwarded to the publisher for copy editing and typesetting. The reviewer comments are included at the bottom of this letter, along with those of the editor who coordinated the review of your paper.

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Sincerely,
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Editor's Comments to Author:
The paper was checked by ass. editor; the corrections are done according to reviewer suggestions, authors' response is satisfactory, tables and figures are up-loaded separately, which is well done and appreciated. The paper seems to be ready for publication in the Journal of Natural Fibers.

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Date Sent: 14-May-2022

Dr. Edi Syafri
Assoc. Professor at Department of Agricultural Technology,
Politeknik Pertanian Negeri Payakumbuh,
West Sumatra 26271, Indonesia

May 14, 2022

Dear Editor Journal of Natural Fibers

We cordially apologize for your any inconvenience.

Sir/Madam, we are very pleased to be able to communicate you. We have revised our manuscript according your suggestion. We would be happy to provide any additional information you may need regarding our manuscript.

We would like to thank the editors and reviewers who gave their valuable recommendations to make our work more appealing and informative. Authors have revised the manuscript and

incorporate all the suggestions. The point wise response to the comments given by the reviewers is provided in the following section.

Thank you again for your valuable time and kind consideration.

We will look forward your amiable reply

Sincerely yours

Dr. Edi Syafri

Journal of Natural Fibers

Isolation and Characterization of New Cellulose Microfibers from Pandan Duri (Pandanus tectorius) for Sustainable Environment

Journal:	*Journal of Natural Fibers*
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Isolation and Characterization of New Cellulosic Microfibers from Pandan Duri (*Pandanus Tectorius*) for Sustainable Environment

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ABSTRACT

Cellulose is the main component of natural fibers whose content varies greatly depending on the type of plant and its treatment. Therefore, it is necessary to examine the effect of chemical treatment on natural fiber properties. This study successfully extracted the content from Pandan duri (*Pandanus tectorius*) fiber through alkalization, bleaching, and acid hydrolysis. The effect of these chemical treatments on the characteristics of fiber surface, shape, chemical composition, crystallinity, and thermal properties was examined and analyzed. Subsequently, the cellulose components in the structure and the removal of lignin groups were characterized by Fourier transform infrared (FTIR) spectroscopy spectrum analysis. The surface morphology analysis was done by scanning electron microscopy (SEM) that showed the raw fiber surface was still in the structure of bundles. However, it was decomposed into cellulose fibrils after chemical treatment with a diameter of about 2 µm – 20 µm. The chemical composition for cellulose content increased by 90.5%, while hemicellulose decreased by 89.6% after acid hydrolysis treatment. Also, X-ray (XRD) analysis showed crystallinity increased from 39.5% for raw fibers to 67.7% after the hydrolysis. Thermal gravimetric analysis (TGA) showed higher degradation temperature of micro cellulose offered better thermal stability compared to raw fibers. In conclusion, the cellulose from *Pandanus tectorius* fiber can be used to reinforce biocomposites as an alternative to synthetic fibers for sustainability of environment.

KEYWORDS

Pandanus tectorius; chemical treatment; cellulose fiber; crystallinity; thermal stability; chemical composition

关键词

盖盘菌; 化学处理; 纤维素纤维; 结晶度; 热稳定性; 化学成分