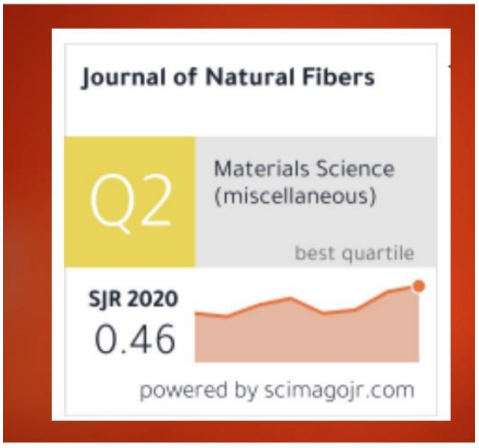
Bukti Korespondensi

Paper17

Effect of ultrafine grinding and ultrasonication duration on theperformance of polyvinyl alcohol (PVA) agave gigantea cellulosemicro fiber (CMF) bio-composite film

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

Submission & Revision

Dr. Edi Syafri

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

January 1, 2022

Dear Prof. Ryszard Kozlowski (Editor-in-Chief, Journal of Natural fiber)

I wish to submit an original research article entitled "**Isolation and Characterization of New Cellulose Microfibers Pandan Duri (*Pandanus tectorius*) for sustainable environment**" for consideration by Journal of Natural fiber.

I confirm that this work is original and has not been published elsewhere, nor is it currently under consideration for publication elsewhere.

Highlights of this Investigation:

- This study successfully extracted the content from Pandan duri (*Pandanus tectorius*) fiber through alkalization, bleaching, and acid hydrolysis
- The chemical composition for cellulose content increased by 90.5%, while hemicellulose decreased by 89.6% after acid hydrolysis treatment.
- Thermal gravimetric analysis (TGA) showed higher degradation temperature of micro cellulose offered better thermal stability compared to raw fibers.

We have no conflicts of interest to disclose. Please address all correspondence concerning this manuscript to me at edisyafri11@gmail.com.

Thank you for your consideration of this manuscript.

Sincerely,

Dr. Edi Syafri

237582259 (Journal of Natural Fibers) A revise decision has been made on your submission

Kotak Masuk ×

Journal of Natural Fibers <onbehalfof@manuscriptcentral.com> Kam, 19 Jan, 16.53

kepada saya

Inggris › Indonesia Terjemahkan pesan Nonaktifkan untuk: Inggris ×

19-Jan-2023

Dear Dr Edi Syafri:

Your manuscript entitled "Effect of Ultrafine Grinding and Ultrasonication Duration on the Performance of Polyvinyl Alcohol (PVA) Agave gigantea Cellulose Micro Fiber (CMF) Bio-Composite Film", 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 receivi 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 submit a revision, go to https://rp.tandfonline.com/submission/flow?submissionId=237582259&step=1. If you decide to revise the work, please submit a list of changes or a rebuttal against each point which is being raised when you submit the revised manuscript.

If you have any questions or technical issues, please contact the journal's editorial office at WJNF-peerreview@journals.tandf.co.uk.

IMPORTANT: Your original files are available to you when you upload your revised manuscript. Please delete any redundant files before completing the submission.

237582259.R1 (Journal of Natural Fibers) A revise decision has been made on your submission

Kotak Masuk ×

Journal of Natural Fibers <onbehalfof@manuscriptcentral.com> Sen, 20 Feb, 18.05

kepada saya

Inggris > Indonesia Terjemahkan pesan Nonaktifkan untuk: Inggris ×

20-Feb-2023

Dear Dr Edi Syafri:

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Because we are trying to facilitate timely publication of manuscripts submitted to Journal of Natural Fibers, your revised manuscript should be uploaded by 13-Mar-2023. If it is not possible for you to submit your revision by this date, we may have to consider your paper as a new submission.

	ADM: Muthukumar, Aswathi • Accept (06-Mar-2023) view decision letter Contact Journal	WJNF-2023-0018.R2 (237582259.R2)	Effect of Ultrafine Grinding and Ultrasonication Duration on the Performance of Polyvinyl Alcohol (PVA) Agave gigantea Cellulose Micro Fiber (CMF) Bio-Composite Film View Submission	03-Mar-2023	06-Mar-2023
a revision has been submitted (WJNF-2023-0018.R2)	ADM: Muthukumar, Aswathi • Minor Revision (20-Feb-2023) • a revision has been submitted view decision letter Contact Journal	WJNF-2023-0018.R1 (237582259.R1)	Effect of Ultrafine Grinding and Ultrasonication Duration on the Performance of Polyvinyl Alcohol (PVA) Agave gigantea Cellulose Micro Fiber (CMF) Bio-Composite Film View Submission	14-Feb-2023	20-Feb-2023
a revision has been submitted (WJNF-2023-0018.R1)	ADM: Muthukumar, Aswathi • Minor Revision (19-Jan-2023) • a revision has been submitted	WJNF-2023-0018 (237582259)	Effect of Ultrafine Grinding and Ultrasonication Duration on the Performance of Polyvinyl Alcohol (PVA) Agave gigantea Cellulose Micro Fiber (CMF) Bio-Composite Film View Submission	12-Jan-2023	19-Jan-2023

Decision Letter (WJNF-2023-0018)

From: ryszard.kozlowski@escorena.net

To: edisyafri11@gmail.com

CC:

Subject: 237582259 (Journal of Natural Fibers) A revise decision has been made on your submission

Body: 19-Jan-2023

Dear Dr Edi Syafri:

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Because we are trying to facilitate timely publication of manuscripts submitted to Journal of Natural Fibers, your revised manuscript should be uploaded by 09-Feb-2023. If it is not possible for you to submit your revision by this date, we may have to consider your paper as a new submission.

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
Overall objective and scope of this manuscript named "Effect of Ultrafine Grinding and Ultrasonication Duration on the Performance of Polyvinyl Alcohol (PVA) Agave gigantea Cellulose Micro Fiber (CMF) Bio-Composite Film" seems to be appropriate for "Journal of Natural Fibers". The authors produced cellulose micro fiber/PVA composites. The authors determined mechanical, XRD, SEM, and TGA analysis of the composite materials. However, the authors should enhance quality of the manuscript before publication.

More literature search should be done on micro/nano cellulose-based materials/composites and summarize in the Introduction section of this manuscript.
It is highly recommended that the authors should read and summarize the below articles into the manuscript.

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⎩ Tozluoglu, A. et al. 2023. Nanocellulose in Paper and Board Coating. In: Taghiyari, H.R., Morrell, J.J., Husen, A. (eds) Emerging Nanomaterials. Springer, Cham. https://doi.org/10.1007/978-3-031-17378-3_8.
⎩ Poyraz, B., Tozluoglu, A., Candan, Z., Demir, A., Yavuz, M., Buyuksari, U., Unal, H.I., Fidan, H., Saka, R.C. 2018. TEMPO-treated CNF composites: pulp and matrix effect. Fibers and Polymers 19(1): 195 – 204.
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Design of experiments (composite groups) should be given in a table.

What about homogeneity of the composites?
The authors should add more photographs of the raw materials and experimental set-up.
More details about TGA analysis should be given.
DSC analysis of the composites should be performed.
Thickness and density values of the composites?
Some conclusions and suggestions regarding with industrial perspective should be added into the Conclusions section of the manuscript.

Reviewer: 2

Comments to the Author
The paper is approaching a current topic. It is proposed the valorization of agro-products to reduce nonrenewable raw materials. The results show the feasibility of the treatment procedures for obtaining new products with a high degree of biodegradation.
The authors could consider the following recommendations:
• Review the entire manuscript in respect of the English style.
• Edit the text according to the journal specifications.
• Underline the advantages/disadvantages, suggestions for utilization domains, sustainability from the economic point of view, etc. Include a comparison of the biocomposite developed and others present in the market based on valuable references.

The paper could be accepted for publication after minor changes. It 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 submit your revised manuscript via new Submission Portal.

Associate Editor: 1
Comments to the Author:
(There are no comments.)

Date Sent: 19-Jan-2023

ecision Letter (WJNF-2023-0018.R1)

From: ryszard.kozlowski@escorena.net

To: edisyafri11@gmail.com

CC:

Subject: 237582259.R1 (Journal of Natural Fibers) A revise decision has been made on your submission

Body: 20-Feb-2023

Dear Dr Edi Syafri:

Your manuscript entitled "Effect of Ultrafine Grinding and Ultrasonication Duration on the Performance of Polyvinyl Alcohol (PVA) Agave gigantea Cellulose Micro Fiber (CMF) Bio-Composite Film", which you submitted to Journal of Natural Fibers, has been reviewed. The reviewer comments are included at the bottom of this letter.

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If you have any questions or technical issues, please contact the journal's editorial office at WJNF-peerreview@journals.tandf.co.uk.

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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
It is recommended to review the form of the phrases. The same word is repeated in some sentences, e.g.: ….", excellent flexibility, and

excellent physical properties…."
To the last suggestion, the authors responded partially. They did not underline the novelty presented by their product compared to the others already present on the market based on valuable references.

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

Reviewer: 2

Comments to the Author
It seems that the authors revised the manuscript.

Editor's Comments to Author:
Please submit your revised manuscript via new Submission Portal.

Associate Editor: 1
Comments to the Author:
(There are no comments.)

Date Sent: 20-Feb-2023

Decision Letter (WJNF-2023-0018.R2)

From: ryszard.kozlowski@escorena.net

To: edisyafri11@gmail.com

CC:

Subject: Journal of Natural Fibers - Decision on Manuscript ID WJNF-2023-0018.R2

Body: 06-Mar-2023

Dear Dr Edi Syafri:

Ref: Effect of Ultrafine Grinding and Ultrasonication Duration on the Performance of Polyvinyl Alcohol (PVA) Agave gigantea Cellulose Micro Fiber (CMF) Bio-Composite Film

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.

Once the Taylor & Francis production department receives and performs an initial check on your article, they will send you a link to complete your online article publishing agreement. This is an essential step. Your completed agreement must be accepted by the publisher before we can publish any version of your paper.

The publisher also requests that proofs are checked through the publisher's tracking system and returned within 48 hours of receipt.

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

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 have considered the recommendations previously made.

Editor's Comments to Author:

There is one more review Accept

/.

Date Sent: 06-Mar-2023

RESPONSE TO REFEREES

Dear Editor.

Thank very much for major correction of our paper. The manuscript has been already revised based on reviewer's comments and journal format. The revised words are marked by RED color in the manuscript. Following are my answers (italicized words) of the reviewer comments.

Best regards,
Edi Syafri

Referee(s)' Comments to Author:

Comments from the editors and reviewers:

Reviewer: 1

Comments to the Author

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More literature search should be done on micro/nano cellulose-based materials/composites and summarize in the Introduction section of this manuscript.

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11. Yildirim, M., Candan, Z., Gonultas, O. 2021. Chemical performance analysis of nanocellulose/boron compounds reinforced hybrid UF resin. Green Materials, 1-8.
12. Candan, Z., Gardner, D. J., Shaler, S. M. 2016. Dynamic mechanical thermal analysis (DMTA) of cellulose nanofibril/nanoclay/pMDI nanocomposites. Composites Part B: Engineering 90: 126 – 132.

Response: *Thank you very much for the suggestion. We have revised our manuscript and added the reference.*

Design of experiments (composite groups) should be given in a table.

What about homogeneity of the composites?

Response: *The solution within petri dish might be still contained trapped bubble and the solution was not evenly distributed in the petri dish. The vibration from ultrasonic probe was needed to make the solution more homogeneous.*

The authors should add more photographs of the raw materials and experimental set-up.

More details about TGA analysis should be given.

DSC analysis of the composites should be performed.

Thickness and density values of the composites?

Response: *We have added DSC characterization, thickness and density values of the composites to the manuscript.*

Some conclusions and suggestions regarding with industrial perspective should be added into the Conclusions section of the manuscript.

Response: *Thank you very much for the suggestion. We have added the industrial perspective as shown in manuscript.*

Reviewer: 2

Comments to the Author

The paper is approaching a current topic. It is proposed the valorization of agro-products to reduce nonrenewable raw materials. The results show the feasibility of the treatment procedures for obtaining new products with a high degree of biodegradation.

The authors could consider the following recommendations:

- Review the entire manuscript in respect of the English style.
- Edit the text according to the journal specifications.
- Underline the advantages/disadvantages, suggestions for utilization domains, sustainability from the economic point of view, etc. Include a comparison of the biocomposite developed and others present in the market based on valuable references.

Response: *We are grateful for these valuable comments. We have revised the language in manuscript.*

Revisions

Effect of Ultrafine Grinding and Ultrasonication Duration on the Performance of Polyvinyl Alcohol (PVA) Agave gigantea Cellulose Micro Fibre (CMF) Bio-Composite Film

Abstract
This paper aims to assess the effect of ultrafine grinding and ultrasonication treatment on the bio-composite characteristics of polyvinyl alcohol (PVA) Agave gigantea (AG) Cellulose Micro Fiber (CMF) bio-composite film performance. This study included five different types of samples. FESEM investigation of PVA/CMF revealed a fiber diameter of 10-15 μm. According to X-ray diffraction, the CMF bio-composite has the highest crystallinity index (87%). The bio-composite film was as transparent as the pure PVA film, demonstrating that the CMF was uniformly dispersed throughout the film. Tensile testing revealed that the ultrafine grinding and ultrasonication treatment for 2 hours (PVA/U2) increased tensile strength by 43% compared to the untreated PVA/CMF sample. This finding is confirmed by thermogravimetry analysis (TGA) and derivative (DTG) analyses, which show that the PVA/U2 sample has the most significant degree of thermal stability when compared to other samples. An ANOVA output supports the results of this experiment with an R2 value of 0.94600232 at a 95% confidence level. The p-value of 0.000079 and F-value of 10.80004 for ultrafine grinding and ultrasonication duration on AG leaf-based PVA bio-composite revealed a statistically significant influence on the studied parameters.
Keywords**:** *Agave gigantea*, cellulose microfiber, ANOVA, superior thermal stability, and high transparency

Introduction

Polyethylene (PE) (Mohammadi and Babaei 2022; Moradi et al. 2020), polypropylene (PP) (Akoueson et al. 2023; da Silva et al. 2022), polystyrene (PS) (Abolghasemi-Fakhri et al. 2019; Pilevar et al. 2019), polyvinyl chloride (PVC) (Chen et al. 2022; H. Wang et al. 2022), and polyethylene terephthalate (PET) (Yuan et al. 2023; Zhai et al. 2022) are now the most common raw materials employed in the manufacture of traditional plastics for packaging applications. However, because they are non-biodegradable, these plastics generate severe worldwide environmental issues (Haider et al. 2019; Taghavi et al. 2021). Therefore, biodegradable polymers are used to replace the plastics material due to its environmentally friendly, abundant availability, and sustainable. Among biodegradable polymers, Polyvinyl Alcohol (PVA) has superior properties: biocompatible, semi-crystalline, non-toxic, water-soluble, chemical resistant, transparency, excellent flexibility, and excellent physical properties (Allafchian et al. 2020; Cano et al. 2015; Dara et al. 2021; Poyraz, Tozluoğlu, Candan, Demir, et al. 2017). PVA can form hydrogen bonds with the surface of hydrophilic materials and serves as a matrix for bio-composite material (Lisdayana et al. 2020; Sarwar et al. 2018).

However, PVA films have poor mechanical and thermal properties, which could limit their utility in packaging applications (Cazón, Vázquez, and Velazquez 2018b, 2018a; Huang et al. 2019). There are several investigations on cellulose-reinforced PVA matrix to increase the tensile strength and thermal stability of PVA composite (Hu and Wang 2016; S. Singh, Gaikwad, and Lee 2018; Wu et al. 2022). Cellulose fibres are widely used as polymer reinforcement because they are renewable, sustainable, abundant, and low-cost. Notably, cellulose has outstanding mechanical characteristics such as high tensile strength and elastic moduli owing to its semi-crystalline extended polymer chain (Candan et al. 2022; Poyraz et al. 2018; Tozluoglu et al. 2022). Cellulose provides a larger reaction surface with the matrix

because of the presence of hydrogen bonds, resulting in a better and promising strengthening effect in bio-composites production (Kalambettu et al. 2015). The cellulose structure can be deconstructed through chemical and mechanical treatments to reduce cellulose fibrefibres' size from raw and cellulose microfibers (CMF). CMF can be extracted from numerous sources, including leaves or stiff fibres, wood, cereal straws, seeds, fruits, bamboo fibre, sugar palm fibre, ginger, agave, and other plant fibres. Among them, Agave gigantea showed a cellulosic fibre content of around 55–70%, higher than wood, with values ranging from 40 to 50% (Azammi et al. 2020; S. K. Singh et al. 2021). In this work, various sizes of CMF from agave fibres through chemical treatment (alkalization and lightening), ultrafine crushing, and ultrasonication have been developed.

Furthermore, the CMF was used as reinforcement in PVA-based bio-composites, and its mechanical, thermal, transparency and functional groups were examined. FESEM was used to examine the fracture surface of composites and the shape and diameter of agave fibres from various treatments. This paper elaborates on how ultrafine grinding and ultrasonic treatment affect the mechanical and thermal properties of PVA films Agave gigantea bio-composite.

Materials and methods

Materials

The fibre in this work was extracted from the fresh leaves of the Agave gigantea (AG) plant. The leaves were sourced in the plantation area in Harau District, West Sumatera Indonesia. Polyvinyl alcohol with 99% hydrolyzed was supplied from Sigma-Aldrich, United States of America. The chemicals adopted in this experiment were sodium hydroxide (NaOH), sodium chlorite ($NaClO_2$), and glacial acetic acid (CH_3COOH).

Sample preparation

Fresh AG leaves were cleansed and sliced into 100-120 mm lengths before being immersed in boiling water at 100 °C for 3 h to enhance fibre release from other extractive elements. The outer skin of the fibre was then removed with a stainless cutter. The AG fibre was sun-dried for 3-4 days with a moisture level of 9 to 11%. The AG fibre was sliced into 7-12 mm lengths and mashed with a blender. On a hotplate, the fibrefibre was alkalized with 5% (w/v) NaOH for two hours at 80 °C. The bleaching was continued using NaClO2 with the condition 1.7% (wt% NaClO2), and then the sample after chemical treatment was labelled CMF. CMF pulp with a suspension concentration of 1 wt% and a dry weight of 20 grams of cellulose was suspended in 2000 mL of distilled water with a modified previous study (Syafri, Sari, et al. 2021; Syafri, Melly, et al. 2021). The solution was fed into the Masuko Sangyo ultrafine grinder MKCA6-3; Masuko Sangyo Co., Ltd., Japan, with various gaps of -10, -30, -50, -70, -90, and -110 m to obtain CMF, with five passes each in each gap. The resulting CMF suspension showed the characteristics of a gel labelled UFG AG. Furthermore, this cellulose suspension was ultrasonicated using SONIC RUPTOR 400 Omni International for 1 hour and 2 hours at 600 W with the labels U1 and U2 AG. The CMF formulation process is shown in the schematic diagram in Fig. 1.

Figure 1 (Here)

There are five samples in this study, namely PVA film (100 mL of distilled water and 10 g of PVA), PVA/CMF AG film (100 mL of distilled water, 10 g of PVA, and 5 wt% of CMF), PVA/UFG AG film (100 mL of distilled water, 10 g of PVA, and 5 wt% of UFG), PVA/U1 AG film (100 mL of distilled water, 10 g of PVA, and 5 wt% of pure UI) subjected with ultrasonication for 1 h, and PVA/U1 AG film (100 mL of distilled water, 10 g of PVA,

and 5 wt% of pure UI) subjected with ultrasonication for two h. A magnetic stirrer was used to mix the components to form a fibre suspension. Meanwhile, ultrasonication treatment was done to the samples to improve the sample components' dispersion in the suspension (Mahardika et al. 2019; Abral et al. 2019).

100 mL of Distilled water and 10 g of PVA were mixed to prepare PVA film. The suspension was heated at 70 °C and 500 rpm for two h until gelatinized using the magnetic stirrer Scilogex MS-H280-Pro. The resulting gel was sonicated by SONIC RUPTOR 400 Omni International ultrasonic for 5 min. The treated gel cast in a beaker was dried for 20 h in a 50 °C vacuum drying oven at 0.6 MPa.

5 wt% of CMF and 10 g of PVA were dispersed into 100 mL of distilled water PVA/CMF AG film. The suspension was heated using a magnetic stirrer at 70 °C and 500 rpm for two h until gelatinization. The resulting gel was treated with 600 W for 5 min using the ultrasonic SONIC RUPTOR 400 Omni International. The treated gel cast in a beaker was dried for 20 h in a 50 °C vacuum drying oven at 0.6 MPa.

5% of pure UFG AG suspension, 10 g of PVA, and 10 mL of distilled water was heated on the magnetic stirrer at 70 °C and 500 rpm for two h until gelatinization to form PVA/UFG AG film. The resulting gel was treated with 600 W ultrasonic for 5 min. The treated gel cast in a beaker was dried for 20 h in a 50 °C vacuum drying oven at 0.6 MPa.

The suspension (10 g PVA, 5% pure UI, and 100 mL distilled water) was heated by the magnetic stirrer at 70 °C and 500 rpm for two h until gelatinization to form PVA/U1 AG film. The resulting gel was sonicated at 600 W for 5 min. The sonicated gel was cast in a beaker and dried for 20 h in a 50 °C vacuum drying oven at 0.6 MPa as suggested in the previous study (Syafri, Sari, et al. 2021).

PVA/U2 AG film was prepared by mixing 10 g of PVA, 10 g PVA, 5% pure UI, and 100 mL of distilled water pure UI in the suspension (10 g PVA, 5% pure UI, and 100 mL distilled water) was heated by the magnetic stirrer at 70 °C and 500 rpm for two h until gelatinization. The resulting gel was sonicated at 600 W for 5 min. The sonicated gel was cast in a beaker and dried for 20 h in a 50 °C vacuum drying oven at 0.6 MPa. All films were stored in a closed desiccator device at 50% relative humidity and 25 C before sample characterization (Yun et al. 2022).

Field Emission Scanning Electron Microscopy Characterization

A field emission scanning electron microscope (FESEM) Quattro S, Thermo Fisher Scientific, Waltham, MA, USA, was employed to assess the morphological fracture surfaces of the films. As suggested by previous researchers, the sample was placed in the carbon tube due to the non-conductive sample type (A Nugroho et al. 2021; Taspika et al. 2020). The FESEM images of the film were recorded at 500 and 2000 magnifications under a high vacuum and working distance of 10±0.5 mm with 3.0 kV accelerating voltage as suggested by previous researchers (Doustdar, Olad, and Ghorbani 2022)(Kashyap et al. 2022).

Film Transparency Characterization

A Shimadzu UV 1800 spectrophotometer was employed to measure the transparency of films according to ASTM D 1003-00 (ASTM D1003 2006). An equal-weight rectangular sample (10 mm × 25 mm) was positioned in the spectrophotometer by a transmittance spectrum of 200 to 800 nm. The transparency of the film is based on the area under the transmittance curve as recommended by previous researchers (da Silva et al. 2022).

Tensile Stress Assessment

Tensile strength was adopted according to the ASTM D638-type V standard (ASTM D638-V 2012). The width and thickness of the samples were measured with 0.01 µm accuracy with digital callipers Mitutuyo Universal Testing Machine AGS-X series 5 kN, Shimadzu, Japan, was employed to govern tensile strength (TS), elastic modulus (ME), and strain at break (SB) of the samples at room temperature. A 30 mm/min tensile test speed was used. Previous researchers (Ding, Tang, and Zhu 2022; Y. Han, Jiang, and Hu 2020) suggested that the tensile tests were repeated five times for each sample to ensure the consistency and reliability of the data (Joshi and Patel 2022; Poyraz, Tozluoğlu, Candan, and Demir 2017).

X-Ray Diffraction Assessment

Shimadzu XRD-700 Maxima X series from Shimadzu Corp., Kyoto, Japan, was employed to conduct X-ray diffraction testing at 24 ºC, 40 kV, and 30 mA using Cu Kα radiation (λ = 0.15406 nm) (C. Wang et al. 2020). All samples were scanned from 2θ = 10º to 50º every 2º/min. The Gaussian function was used for the crystallinity calculation (Xc) of the area of the crystalline region and the area of the amorphous region, respectively. Eq. 1 was adopted to calculate the degree of crystallinity:

$$X_c\ (\%) = (F_c/F_a + F_c) \times 100 \quad (1)$$

F_c is the crystalline area (2θ = 20-23º), and F_a is the non-crystalline / amorphous region (2θ = 15-16 º).

Thermogravimetry analysis (TGA) and derivative (DTG) Assessment

The samples in this work were analyzed using a TGA 4000 thermal analysis instrument from Perkin Elmer, Hopkinton, MA, United States of America. 10 mg of the film was placed on a microbalance inside the furnace. The instrument had a nitrogen flow rate of 20 mL/min. The test was carried out from 30 °C up to 600 °C. Pyris software (Version 11, Pyris, Washington, MA, USA) assessed weight loss, weight loss rate, and residue percentages (Candan, Gardner, and Shaler 2016).

Differential Scanning Calorimetry (DSC)

DSC analysis of the samples was performed using differential scanning calorimetry (DSC) type PerkinElmer 4000, United States. The samples were encapsulated in an aluminum pan then scanned from 30 ºC to 400 ºC with a heating rate of 10 ºC/min under nitrogen gas (Sultana et al. 2020).

Statistical analysis

The variance analysis (ANOVA) was employed to evaluate the tensile property data of films. The difference between samples was determined to be significant at alpha 0.05 with a 95% confidence level.

Results and discussion

Field Emission Scanning Electron Microscopy Analysis

Fig. 2. shows the fracture surface of the PVA/CMF AG bio-composite and the AG fiber's surface morphology after alkalization, bleaching, and ultrasonication for 1 and 2 h. Fig. 2(f) shows the surface morphology of cellulose with a magnification of 8000× shows fibre measurements with an average fibre diameter of 8 µm. The presence of non-cellulose material

causes a rough surface. Fig. 2(g) shows the surface morphology of the bundled microfibrils. Because of the applied chemical treatment, the fibre diameter was lower (10-15 µm) than the raw AG fibre. Figure 2d depicts the smooth surface structure of the fibres in various sizes. Mechanical ultrafine grinding treatment promoted the cellulose to shrink and form micro-dimensional fibres, also known as cellulose micro-fibres.

Figure 2 (Here)

Fig. 2(a) shows a smooth fracture surface of pure PVA due to crack propagation in the presence of resistance. The addition of MCF presence of the surface rougher, as shown in Fig. 2(b), because the fibres inhibited crack propagation (Jain, Singh, and Chauhan 2017)(Solikhin et al. 2018). Fig. 2(c) shows that the fibre surface is still micro-sized in the PVA matrix. A beach mark due to cellulose micro-fibre interfering with crack growth is shown in Fig. 2(d). The fracture surface of the bio-composite film shows uniformly distributed beach marks over the entire surface, indicating good CMF dispersion after ultrasonication, as shown in Fig. 2(e). Besides, the porosities are not visible in Fig. 2(e). This event can be explained by the fact that all extracted CMF are well dispersed in the suspension.

Film Transparency Assessment Analysis

The transmittance of PVA and PVA/AG fibres' optical properties were determined in the range of 200-800 nm as shown in various sizes as shown in Fig. 3. Film transparency, % transmittance at 280 and 660 nm, and UV block for PVA films with different sizes of AG fibre were analyzed (Table 1). % T film predominantly depends on the dispersion of the AG fibre in the PVA matrix. The PVA film became relatively opaque with the AG fibre content after UFG, decreasing from 76% T for pure PVA to 65% T. The UFG cellulose suspension reduced the light transmittance of the PVA bio-composite films concerning the higher cellulose content revealing better dispersion in the matrix. PVA films with a mixture of CMF, U1, and U2 AG fibres exhibited high transparency of about 90%, 87%, and 89%, respectively. Another advantage of this film is its high UV absorption capacity, blocking 61%, 52%, and 47% of UV-B, respectively. UFG causes a decrease in the transparency of the film. It significantly lowers the UV absorption capacity. For the chemical composition of cellulose, the smaller the size of the cellulose leads to a more significant increase in UV absorption. These results are supported by previous research (Fu et al. 2015)(Q. Wang et al. 2018).

Figure 3 (Here)

Tensile Stress Properties Analysis

Fig. 4. depicts the mechanical properties of the tensile strength (TS), elastic modulus (ME), and strain at break (SB) of PVA films and PVA bio-composites with the addition of cellulose AG fibres of different sizes. The cellulose microfiber AG in the PVA matrix observed a significant decrease in tensile strength. A significant increase was observed using UFG, U1, and U2 AG fibres (33 MPa, 37 MPa, and 39 MPa, respectively). A similar trend has resulted when analyzing the elastic modulus of the films.

After ultrafine grinding and ultrasonication, the addition of cellulose AG fiber improved the mechanical properties of PVA films. This improvement was attributed to three factors: (i) inherent chain stiffness; (ii) homogeneous distribution of cellulose AG fibre in the PVA matrix; and (iii) high compatibility between cellulose AG fibre and PVA, resulting in a strong interaction through hydrogen bonding. Although introducing various cellulose AG fibre diameters provided a comparable result, not all of them strengthened the PVA matrix similarly.

The PVA film's tensile strength was calculated to be 27.40 MPa. It did not increase significantly with the addition of UFG to 33.55 MPa. However, after adding U2 AG, the tensile strength increased by 17% (39.20 MPa).

Figure 4 (Here)

In contrast, the elastic modulus increased with the addition of UFG, U1, and U2 by increasing the values of 708.64 MPa, 921.08 MPa, and 898.83 MPa, respectively, compared to pure PVA (538.21 MPa). Variations in the cellulose AG fibrefibre size can explain the difference in the strengthening effect of cellulose macro-fibre. The ultrafine grinding and ultrasonication process promoted the micro-cellulose's surface area enhancement. This event escalated the pure PVA film's cellulose microfibers' tensile properties (Abdulkhani, Echresh, and Allahdadi 2020; Li et al. 2022). The cellulose AG fibrefibre's size can affect the PVA matrix's strengthening effect. Previous studies (Anwer et al. 2015; Kumar, Kumar, and Bhowmik 2018) reported that cellulose micro-fibre produces a higher specific surface area which causes cross-linking, contributing to an increase in tensile properties. In addition, during the mechanical treatment, the strain at break increased due to the cellulose micro-fibre content. This phenomenon can be attributed to the between and within the molecular hydrogen bonding of cellulose micro-fibre with PVA increasing its elasticity and tensile properties. These results are supported by previous research (Nurazzi et al. 2021).

X-Ray Diffraction Assessment Analysis

The XRD patterns of all the formulated samples are compared in Fig. 5 with different sizes of cellulose, namely PVA/CMF AG, PVA/UFG AG, PVA/U1 AG, and PVA/U2 AG. The PVA film's crystallinity and bio-composite were computed using the Segal method. PVA and bio-composite PVA peaks at a value of 2θ from 20º.

Figure 5 (Here)

However, the prominent peak of PVA was observed at 19.5, representing the (11O) plane of the partially semi-crystalline region of PVA (Jahan, Niazi, and Gregersen 2018; Niazi et al. 2017). In PVA films, the crystallinity was slightly lower than that of PVA/CMF AG. The addition of cellulose microfiber AG into the PVA bio-composite film increased the intensity (11O), increasing crystallinity. PVA/CMF AG had the highest crystallinity value compared to all films, 87%. However, in all PVA bio-composites, the addition of AG fibre showed increased crystallinity compared to pure PVA films. The lowest crystallinity was observed for PVA/UFG AG films (83%) and the highest for PVA/CMF AG (87%).

Table 1 (Here)

The PVA/U2 AG peak at a value of 2θ 19.5º (2 0 0 field) also started to appear at higher concentrations of AG fibre. In PVA/U1 AG and PVA/U2 AG, the trend was not significant to increase crystallinity. have the same crystallinity. This event can be described to the insignificant amount of cellulose micro-fibre in the PVA/U AG (wt./wt.) films. After that, crystallinity showed a slight increase with a 5% concentration in PVA/CMF AG films.

Thermogravimetry analysis (TGA) and derivative (DTG) Assessment Analysis

Fig. 5. shows the thermal stability of PVA and PVA/CMF AG bio-composites through the TGA and DTG curves. The curves have the same patterns in the three weight loss regions in the temperature range of 75-410 °C. The first area shows the evaporation of water vapour in the temperature range (80-165°C) with a weight loss range of 10-15 wt%. The second transition

region (310-410 °C) shows the decomposition of the structure of the PVA/MCF AG bio-composite films with a total weight loss of about 65-80 wt%.

Figure 6 (Here)

The highest thermal stability of all PVA and PVA/CMF AG bio-composite films samples was PVA/U2 AG samples. This phenomenon caused the addition of CMF after ultrasonication, causing the crystal structure to increase. This finding was validated by the measurement of the crystallinity index, as indicated in Table 1, and previous research. Because of the strong hydrogen connections formed between the PVA, cellulose, and matrix hydroxyl groups, this behaviour suggests that adding CMF AG fibre can boost the heat stability of pure PVA (Sun et al. 2019)(Khalili et al. 2022). The third region above 410 °C shows residual char formed from samples of PVA bio-composite films with a total weight loss above 96 wt% at 550 °C. Another result that supports this discovery is that the PVA/U2 AG solution is extremely well dispersed throughout the samples, promoting a strong connection between the cellulose molecules and the PVA matrix. This finding is in line with the recent findings of previous researchers (X. Han et al. 2023; S. S. Singh et al. 2022; Tiwari and Sarangi 2022; Tozluoğlu et al. 2017). The physical product of each sample is shown in Fig. 7. According to the figure, the PVA U2 sample has a superior look and more uniformly stable surface stability than the other samples.

Figure 7 (Here)

Differential Scanning Calorimetry (DSC), Density, and Thickness Analysis

The thickness and density of each sample are explained in the Figure 8. The thickness of the PVA sample is around 0.13 mm, smaller than the other samples. Meanwhile, the thickness of PVA/CMF AG was 0.22 mm, higher than the PVA/UFG, PVA/U1 AG, and PVA/U2 AG samples, with a thickness of around 0.19 mm. Meanwhile, the density of PVA is around 1.077 g/cm3, while the density of PVA/CMF AG is around 1.163 g/cm3, which is higher than the density because it is partially filled with cellulose. The density of the PVA/UFG, PVA/U1 AG, and PVA/U2 AG samples was higher, considering that the presence of cellulose microfiber in a tiny size does not cause large pores.

Figure 8 (Here)

DSC thermograms of all samples are presented in Figure 9. Figure 9 shows the heating process of the PVA composite from 25 to 300 °C. For the PVA homopolymer, the DSC displays a glass transition (t_g) peak around 94.22 °C with an enthalpy change $\Delta H \approx 204.20$ J/g. Another sharp endotherm melting transition (t_m) at 186.06 °C with $\Delta H \approx 29.44$ J/g. The heat required to melt 100% PVA crystals is 138.6 J/g (Mathers et al. 2022). The presence of micro cellulose increased the value (t_g) of PVA to 113.57 °C and the melting temperature to 187.74 °C. The t_g temperature slowly shifts to a lower temperature, where the t_g of PVA/U2 AG is 106.83 °C with a t_m of around 185.67 °C. The enhancement of the glass transition can be attributed to the semicrystalline nature of the material. The t_g and t_m values are more consistent with previous research (Okahisa et al. 2020). The enthalpy change from tg PVA/U2 AG is 225.43 J/g, and the enthalpy change from tm is 18.37 J/g.s.

Figure 9 (Here)

ANOVA Output

Table 2 displays the ANOVA output from the tensile strength test for each sample. Each sample was represented by five specimens: PVA, PVA/CMF, PVA/UFG, PVA/U1, and PVA/U2.

Table 2 (Here)

Table 3 (Here)

Table 2 summarizes the ANOVA analysis's number, average, and variance on the results of the tensile strength evaluation on the five types of samples. Based on the statistics, the pure PVA sample has a more excellent value than the PVA/CMF combination. According to the table, increasing the ultrasonication period increases the average tensile strength value. This summary of ANOVA statistics agrees well with the tensile strength result shown in Fig. 4. (a). The ANOVA output is displayed in table 3, where the square sum of inter-group ANOVA is 418.862412, and the sum of squares within groups is 193.917127. These two parameters represent the correlation of variation in the tensile strength population, with degrees of freedom of 4 and 20, respectively. Based on the results of table 3, it is determined that there is a high correlation between the investigated factors and the response. The coefficient of determination value of 0.94600232 supports this event (Amroune et al. 2022). Although this investigation employed organic material, the coefficient of determination yielded a reasonably high value. The sample preparation and data retrieval of experimental findings performed five times enhances the data's confidence and reliability. This discovery is in line with previous researchers' coefficient of determination results (Choudhary, Sachdeva, and Kumar 2020; Agus Nugroho et al. 2022).

The ANOVA output in this investigation fits well with the hypothesis that ultrafine grinding and ultrasonication time can enhance tensile strength with an F value of 10.80004. The p-value is lower than alpha 0.05, which is 0.000079. As a result, the hypothesis rejected Ho in this study's statistical analysis. This parameter is supported by the critical F value of 2.866081, much lower than the F value of 10.80004. This statistical evaluation is valid with a confidence level of 95% and a standard error of 0.95434569. Thus, the error of this statistical analysis is within 10%.

Conclusions

The production of PVA and PVA/CMF AG bio-composite films was successfully developed. CMF AG fibre has a diameter of 10-15 micrometres obtained from FESEM macroscopic and microscopic form of cellulose after chemical treatment (alkalization and bleaching) and ultrasonication method for 1 and 2 h. PVA/CMF bio-composite films were proven by analysis of mechanical properties, transparency, and thermal characterization. The effect of CMF in the PVA matrix has been investigated compressively. The tensile strength test on each sample revealed that the PVA/U2 bio-composite sample treated with ultrafine grinding and ultrasonication for two hours had the most excellent tensile strength value compared to the other samples. The TGA and DTG analyses yielded similar results. This event demonstrates that the PVA/U2 sample has the most significant level of thermal stability. As a result, the PVA/U2 sample is an appropriate AG leaf-based microfiber sample that could be suggested for sustainable packaging applications. ANOVA output with an R2 value of 0.94600232 at a 95% confidence level supports the outcomes of this experiment. The p-value of 0.000079 and the F-value of 10.80004 for ultrafine grinding and ultrasonication time on AG leaf-based PVA bio-composite demonstrated strong significance, above the F-critical value of 2.866081. As a result, the F value fulfilled the statistical criteria for supporting the hypothesis (H1) and rejecting the null hypothesis (H0) within a 10% standard error.

Microfibers have been suggested as a potential material for packaging from an industrial perspective due to their unique properties. One of the main advantages of using microfibers in packaging is their high strength and durability. Microfibers are able to withstand high levels of

stress and tension, making them suitable for packaging heavy or fragile items. This can help to prevent damage to the packaged items during transport and storage.

Another advantage of microfibers in packaging is their high barrier properties. They are able to prevent the penetration of gases, liquids, and bacteria, which can help to extend the shelf life of packaged products. Microfibers can also be used to create packaging that is resistant to punctures, tears, and other types of damage. Microfibers are also lightweight, which can help to reduce the overall weight and cost of packaging. This is important for shipping and transportation, as it can help to reduce fuel costs and lower the carbon footprint. Overall, microfibers are a versatile material for packaging, which can be tailored to the specific requirements of the packaged product, and their properties can be optimized for a specific application. Research in this area is ongoing to further develop their potential.

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Dear Dr Edi Syafri:

Your manuscript entitled "Effect of Ultrafine Grinding and Ultrasonication Duration on the Performance of Polyvinyl Alcohol (PVA) Agave gigantea Cellulose Micro Fiber (CMF) Bio-Composite Film", which you submitted to Journal of Natural Fibers, has been reviewed. The reviewer comments are included at the bottom of this letter.

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Effect of ultrafine grinding and ultrasonication duration on the performance of polyvinyl alcohol (PVA) agave gigantea cellulose micro fiber (CMF) bio-composite film

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ABSTRACT

This paper aims to assess the effect of ultrafine grinding and ultrasonication treatment on the bio-composite characteristics of polyvinyl alcohol (PVA) Agave gigantea (AG) Cellulose Micro Fiber (CMF) bio-composite film performance. This study included five different types of samples. FESEM investigation of PVA/CMF revealed a fiber diameter of 10-15 µm. According to X-ray diffraction, the CMF bio-composite has the highest crystallinity index (87%). The bio-composite film was as transparent as the pure PVA film, demonstrating that the CMF was uniformly dispersed throughout the film. Tensile testing revealed that the ultrafine grinding and ultrasonication treatment for 2 h (PVA/U2) increased tensile strength by 43% compared to the untreated PVA/CMF sample. This finding is confirmed by thermogravimetry analysis (TGA) and derivative (DTG) analyses, which show that the PVA/U2 sample has the most significant degree of thermal stability when compared to other samples. An ANOVA output supports the results of this experiment with an R2 value of 0.94600232 at a 95% confidence level. The p-value of 0.000079 and F-value of 10.80004 for ultrafine grinding and ultrasonication

KEYWORDS

Agave gigantea; cellulose microfiber; ANOVA, superior thermal stability and high transparency

关键词

龙舌兰; 纤维素微纤维; 优异的热稳定性和高透明度