KEMENTERIAN PENDIDIKAN DAN KEBUDAYAAN
UNIVERSITAS NEGERI SURABAYA
FAKULTAS MATEMATIKA DAN ILMU PENGETAHUAN ALAM
Kampus Ketintang, Jalan Ketintang, Surabaya 60231
Telepon : +6231- 8296427, Faksimil : +6231- 8296427
Laman : http://fmipa.unesa.ac.id, email : fmipa@unesa.ac.id

SURAT KETERANGAN

Nomor: B/47380/UN38.3/KP.05/2020

Yang bertanda tangan di bawah ini, Dekan FMIPA Universitas Negeri Surabaya menerangkan dengan sesungguhnya bahwa:

Nama	: Dr. Nita Kusumawati, M.Sc
NIP	: 198207042005012002
NIDN	: 0004078201
Pangkat/Golongan	: Pembina (IV/a)
Unit Kerja	: Jurusan Kimia FMIPA Universitas Negeri Surabaya

Bahwa dosen tersebut di atas, **berdasarkan pemeriksaan dokumen pada tahap I oleh Tim PAK Pusat (8-12 Juli 2020), yang menyatakan**:

1. Ditemukan 36 karil yang diupload.
2. Karil No. 1-8 dapat digunakan sebagai syarat khusus.
3. Karil No. 10-15 diterbitkan pada jurnal yang dikelola kurang baik: coverage (2010-2010) dan karil No. 16 dan 22 diminta untuk diklarifikasi karena ada kecenderungan plagiasi, similarity index dan primary source relative tinggi.
4. Jumlah kum yang diperoleh adalah 190.8

Selanjutnya hasil pemeriksaan dokumen pada tahap II oleh Tim PAK Pusat (22-30 September 2020), yang menyatakan:

1. Ditemukan surat klarifikasi sebagaimana permintaan PAK pusat pada pemeriksaan terdahulu (8-12 Juli 2020). Instansi terkait tidak melihat catatan per karil sehingga menyatakan coverage 2010-2010.
2. Institusi terkait memberikan klarifikasi tentang adanya kemungkinan plagiarisme.

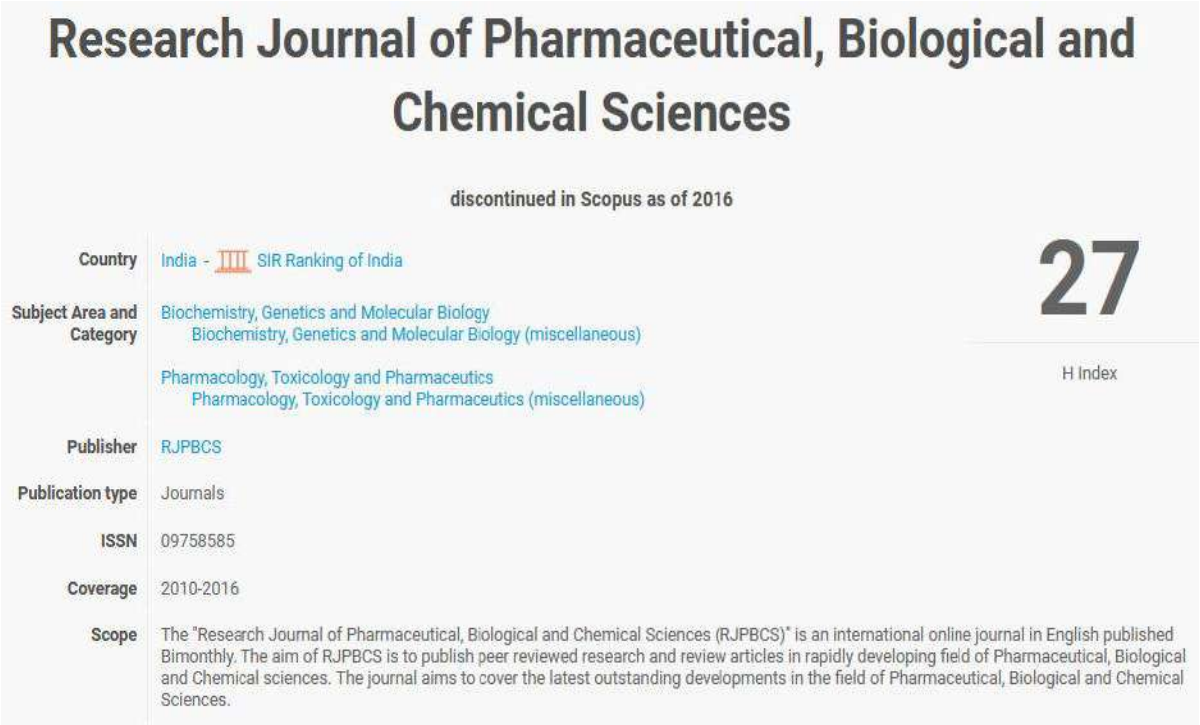
Dan solusi yang diberikan oleh Tim PAK Pusat berdasarkan hasil pemeriksaan tersebut di atas, yang meliputi:

1. Diminta klarifikasi terhadap Karil No 10 - 15 dan No.16 dan 22 sesuai temuan pada kesimpulan;
2. Diminta hasil Similarity checking terbaru dilengkapi dengan full textnya sehingga dapat cepat diperiksa.
3. Diminta semua menyurat sebagaimana dinyatakan pada surat Klarifikasi.
4. Klarifikasi diminta dari institusi pengusul.

Yang bersangkutan telah memberikan respon dan klarifikasi tertulis dengan penjelasan sebagai berikut:

1. Terkait dengan karil no. 10, yang dinyatakan "diterbitkan oleh jurnal yang dikelola kurang baik", dapat disampaikan klarifikasi dan bukti korespondensi sebagai berikut:

   Karil berjudul: The Influence of Casting Solution Composition and Stirring Conditions Against Mechanical Strenght and Performace of Polyvinylindene Fluoride (PVDF) Polysulfone (PSF) Composite Membran on Textile Industrial Wastewater Treatment diterbitkan pada tahun 2015 oleh *Research Journal of Pharmaceutical, Biological and Chemical Science* (RJPBCS), yang menurut hasil penelusuran yang pada website "Scimago Journal and Country Rank" pada saat itu masih terindeks scopus dengan bukti sebagai berikut.



Research Journal of Pharmaceutical, Biological and Chemical Sciences

discontinued in Scopus as of 2016

Country	India - SIR Ranking of India
Subject Area and Category	Biochemistry, Genetics and Molecular Biology Biochemistry, Genetics and Molecular Biology (miscellaneous) Pharmacology, Toxicology and Pharmaceutics Pharmacology, Toxicology and Pharmaceutics (miscellaneous)
Publisher	RJPBCS
Publication type	Journals
ISSN	09758585
Coverage	2010-2016
Scope	The "Research Journal of Pharmaceutical, Biological and Chemical Sciences (RJPBCS)" is an international online journal in English published Bimonthly. The aim of RJPBCS is to publish peer reviewed research and review articles in rapidly developing field of Pharmaceutical, Biological and Chemical sciences. The journal aims to cover the latest outstanding developments in the field of Pharmaceutical, Biological and Chemical Sciences.

27

H Index

   Sebagai pelengkap, data berikut merupakan bukti korespondensi dengan pihak jurnal selama proses review berlangsung:

Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI, Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.
Indexed Copernicus Value: 4.62

On Thu, Oct 30, 2014 at 9:38 PM, Nita Kusumawati <nitakusumawati82@yahoo.com> wrote:

> Dear editor, I really hope to be able to publish my scientific articles concerning the preparation process of membrane from synthetic polymer materials in your journal.
> Thank you.
>
> Best regard,
>
> Nita Kusumawati

--

Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI,
Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.
Indexed Copernicus Value for the year 2010: 4.62

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Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI,
Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.
Indexed Copernicus Value for the year 2010: 4.62

--

Re: Acceptance: RJPBCS / 2010-4307.

Dari: RJPBCS RJPBCS (editor.rjpbcs@gmail.com)

Kepada: nitakusumawati82@yahoo.com

Tanggal: Selasa, 18 November 2014 21.13 GMT+7

Dear Author,

Received payment, your manuscript will be publishes in 6(1) issue.

Thanks for your support towards RJPBCS.

With regards

Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI, Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.

On Mon, Nov 17, 2014 at 4:54 AM, Nita Kusumawati <nitakusumawati82@yahoo.com> wrote:

Dear editor, along with this email, I've posted the scan of payment proof via Mandiri Bank - Indonesia

Pada Selasa, 11 November 2014 12:51, RJPBCS RJPBCS <editor.rjpbcs@gmail.com> menulis:

Research Journal of Pharmaceutical, Biological and Chemical Sciences

(ISSN: 0975-8585)

CODEN: RJPBBP

Impact factor 0.35

Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.

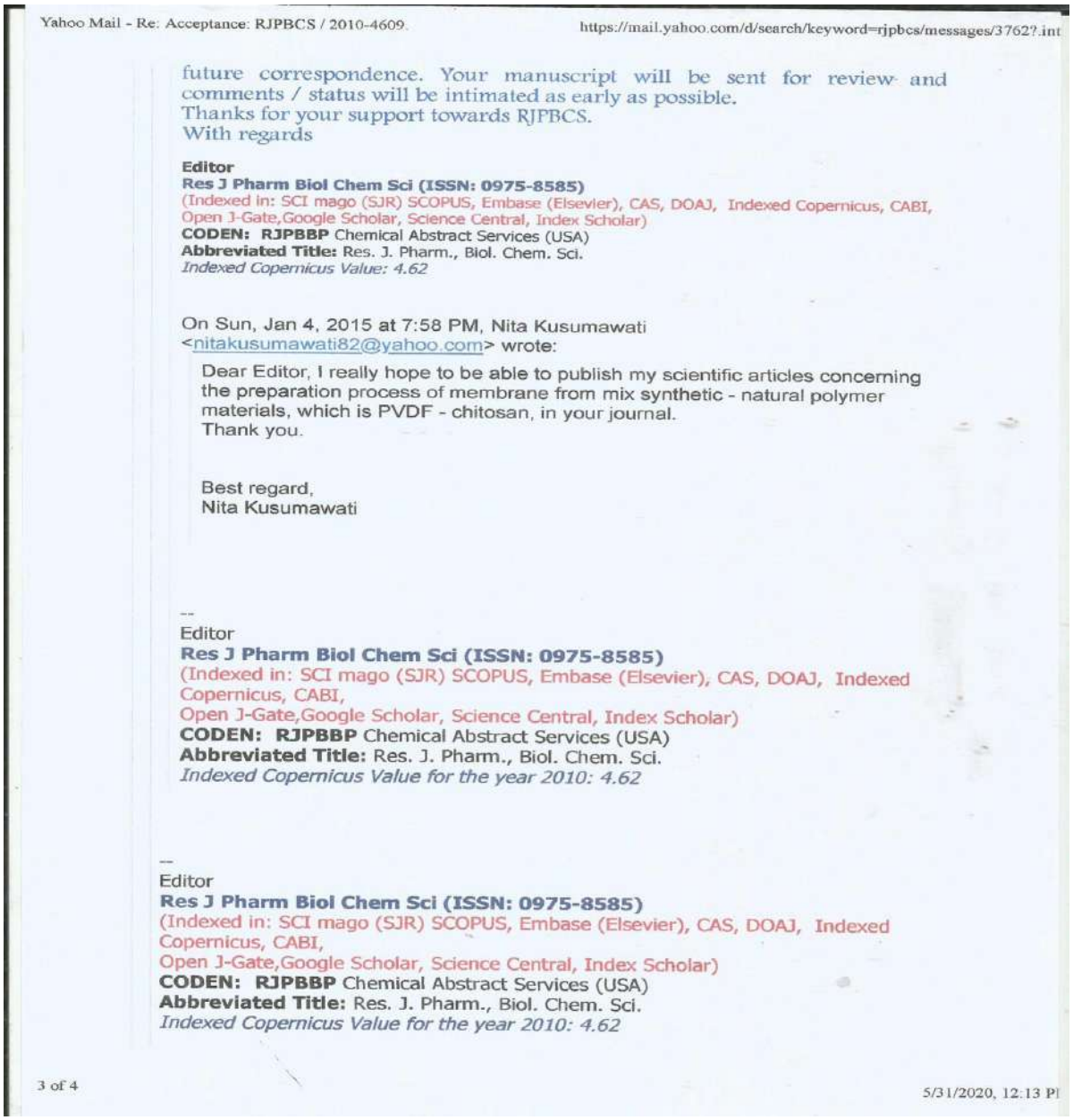
Indexed and Abstracted in

SCI mag (SJR), Scopus, Embase (Elsevier), CAS, DOAJ, AYUSH Research Portal (Govt. Of India), Indexed Copernicus (ICV: 4.62), CABI, Open J-Gate, Google Scholar, Science Central, Index Scholar etc.

Dear **Nita Kusumawati, Toeti Koestiari, and Maria Monica.**

2. Terkait dengan karil no. 11, yang dinyatakan "diterbitkan oleh jurnal yang dikelola kurang baik", dapat disampaikan klarifikasi dan bukti korespondensi sebagai berikut:

   Karil berjudul: Manufacture of PVDF-Kitosan Composite Membrane and its Utilization in Batik Industrial Wastewater Treatment diterbitkan pada tahun 2015 oleh *Research Journal of Pharmaceutical, Biological and Chemical Science* (RJPBCS), yang menurut hasil penelusuran pada website "Scimago Journal and Country Rank" pada saat itu masih terindeks scopus. Sebagai pelengkap, data berikut merupakan bukti korespondensi dengan pihak jurnal proses review berlangsung:



Yahoo Mail - Re: Acceptance: RJPBCS / 2010-4609. https://mail.yahoo.com/d/search/keyword=rjpbcs/messages/3762?int

future correspondence. Your manuscript will be sent for review and comments / status will be intimated as early as possible.
Thanks for your support towards RJPBCS.
With regards

Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI, Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.
Indexed Copernicus Value: 4.62

On Sun, Jan 4, 2015 at 7:58 PM, Nita Kusumawati <nitakusumawati82@yahoo.com> wrote:

Dear Editor, I really hope to be able to publish my scientific articles concerning the preparation process of membrane from mix synthetic - natural polymer materials, which is PVDF - chitosan, in your journal.
Thank you.

Best regard,
Nita Kusumawati

--
Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI,
Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.
Indexed Copernicus Value for the year 2010: 4.62

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Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI,
Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.
Indexed Copernicus Value for the year 2010: 4.62

3 of 4 5/31/2020, 12:13 PM

Science Central,
Index Scholar etc.

Dear **Nita Kusumawati, Asri Wijiastuti, and Agus Budi Santoso.**
Ref.No: RJPBCS/2010-4609.
I am pleased to inform you that your manuscript entitled **"Manufacture of PVDF-Kitosan Composite Membrane and its Utilization in Batik Industrial Wastewater Treatment"** is accepted for publication in RJPBCS Volume 6 Issue 2. Kindly send the scanned copy of copyrights transfer form along with Pay in slip of manuscript handling charges for 01 article of $100.00 (One hundred USD only) to the following Account. Copyrights transfer form you get it from www.rjpbcs.com website. If you have any queries feel free to mail me to editor.rjpbcs@gmail.com.
Important Note: Please make payment before 24/01/2015 otherwise it may delay the publication and mention manuscript reference number on transaction scan copy.
For Bank Transaction

Payment to be Made	$100.00
Account Holder (Beneficiary account name)	Research and Reviews
Account Number	0694102000003285 (Current Account)
Beneficiary Account Holder Address	#29, SHIG-02D, 5th Phase, Yelahanka New Town, Bangalore 560064, Karnataka, India
Name of Bank	IDBI Bank Ltd
Branch Code	000694
IFSC Code	IBKL0000694
Swift Code	IBKLINBB008
Bank Address	IDBI Bank Ltd, Dinakaran Complex, No. 9, HIG, Sector-A, Yelahanka New Town, Bangalore-560064, Karnataka, India

For other than Bank Transaction

Western Union/ Money gram / Express money	**Full Name: Hemalatha M** **Address:** #29, Madanamma Temple, Chikkabanavara, Bangalore North (Tq), Bangalore District – 560090, Karnataka, India.

With Regards,
Managing Editor

On Sun, Jan 4, 2015 at 11:18 PM, RJPBCS RJPBCS <editor.rjpbcs@gmail.com> wrote:

Research Journal of Pharmaceutical, Biological and Chemical Sciences
(ISSN: 0975-8585)

Dear Author,

Thank you for submission of manuscript to RJPBCS. Your Manuscript number is **RJPBCS / 2010-4609**. Please quote manuscript number for

Re: Acceptance: RJPBCS / 2010-4609.

Dari: RJPBCS RJPBCS (editor.rjpbcs@gmail.com)

Kepada: nitakusumawati82@yahoo.com

Tanggal: Selasa, 10 Februari 2015 21.56 GMT+7

Dear Author,

Received payment, your manuscript will be publishes in 6(2) issue.

Thanks for your support towards RJPBCS.

With regards

Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI, Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.

On Thu, Feb 5, 2015 at 4:21 AM, Nita Kusumawati <nitakusumawati82@yahoo.com> wrote:

Dear publisher, by this email, I send to you a payment proof of manuscript: RJPBCS/2010-4609, undertaken on 26 January 2015.

Thank You

Best Regard,
Nita Kusumawati

Pada Sabtu, 17 Januari 2015 13:42, RJPBCS RJPBCS <editor.rjpbcs@gmail.com> menulis:

Research Journal of Pharmaceutical, Biological and Chemical Sciences

(ISSN: 0975-8585)

CODEN: RJPBBP

Impact factor 0.35

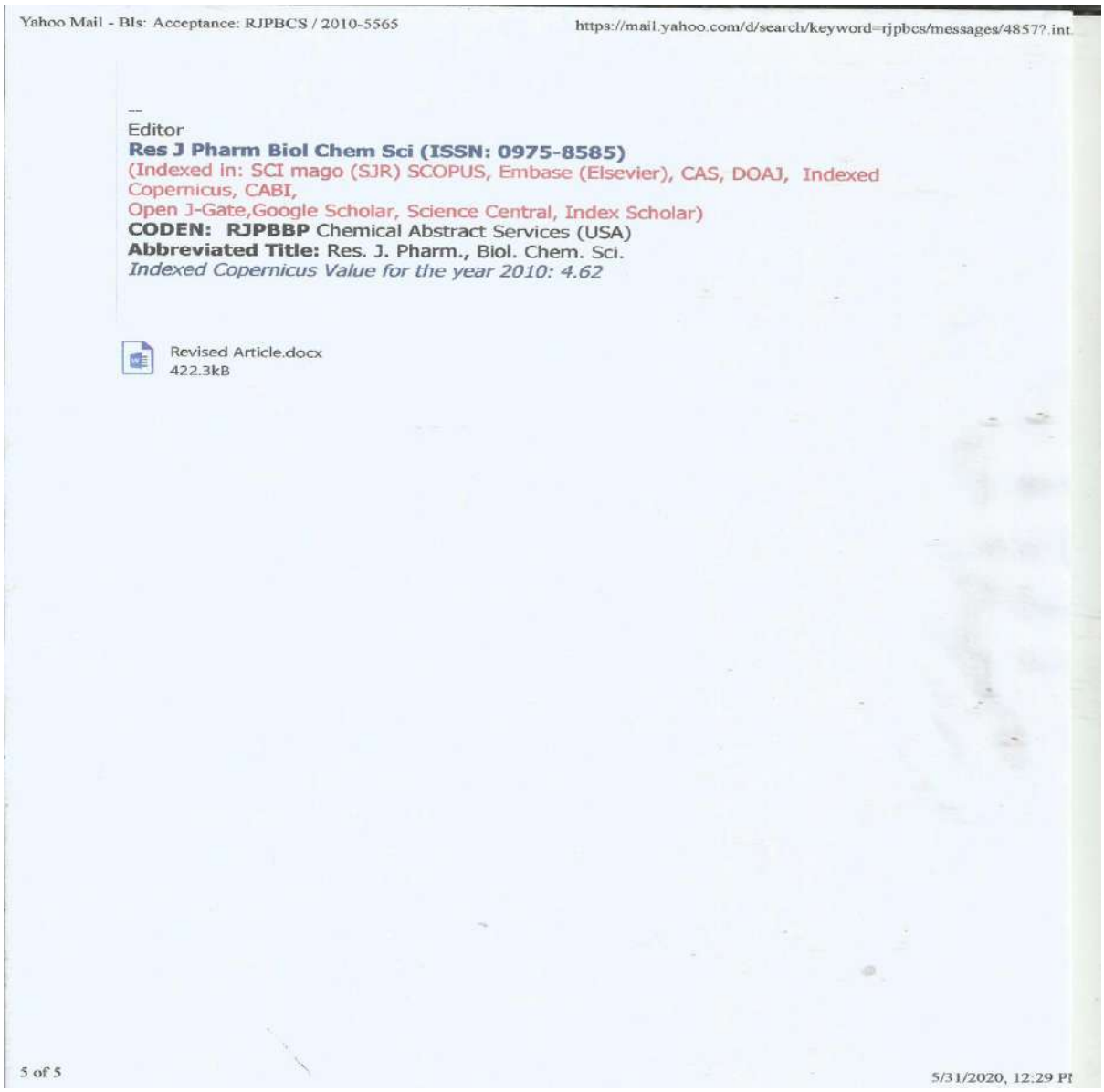
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.

Indexed and Abstracted in

SCI mag (SJR), Scopus, Embase (Elsevier), CAS, DOAJ, AYUSH Research Portal (Govt. Of India), Indexed Copernicus (ICV: 4.62), CABI, Open J-Gate, Google Scholar,

3. Terkait dengan karil no. 12, yang dinyatakan "diterbitkan oleh jurnal yang dikelola kurang baik", dapat disampaikan klarifikasi dan bukti korespondensi sebagai berikut:

   Karil berjudul: The Influence of Washing Process Using TRO on Indigosol Dyeing Quality, Leaching Percetage, and Mechanical Strength of Mori Fabric diterbitkan pada tahun 2015 oleh *Research Journal of Pharmaceutical, Biological and Chemical Science* (RJPBCS), yang menurut hasil penelusuran pada website "Scimago Journal and Country Rank" pada saat itu masih terindeks scopus. Sebagai pelengkap, data berikut merupakan bukti korespondensi dengan pihak jurnal proses review berlangsung:



Yahoo Mail - Bls: Acceptance: RJPBCS / 2010-5565 https://mail.yahoo.com/d/search/keyword=rjpbcs/messages/4857?.int

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Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI,
Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.
Indexed Copernicus Value for the year 2010: 4.62

Revised Article.docx
422.3kB

5 of 5 5/31/2020, 12:29 PI

publish it on RJPBCS. Great expectations of me so that the article can be published to the public. Thank You.

Best Regard,
Nita Kusumawati

--
Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI,
Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.
Indexed Copernicus Value for the year 2010: 4.62

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Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI,
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CODEN: RJPBBP Chemical Abstract Services (USA)
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CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.
Indexed Copernicus Value for the year 2010: 4.62

For Bank Transaction

Payment to be Made	$150.00
Account Holder (Beneficiary account name)	Research and Reviews
Account Number	0694102000003285 (Current Account)
Beneficiary Account Holder Address	#29, SHIG-02D, 5th Phase, Yelahanka New Town, Bangalore 560064, Karnataka, India
Name of Bank	IDBI Bank Ltd
Branch Code	000694
IFSC Code	IBKL0000694
Swift Code	IBKLINBB008
Bank Address	IDBI Bank Ltd, Dinakaran Complex, No. 9, HIG, Sector-A, Yelahanka New Town, Bangalore-560064, Karnataka, India

For other than Bank Transaction

Western Union/ Money gram / Express money	Fist Name: Vrushabendra Last Name: Swamy City: Bangalore Country: India.

With Regards,
Managing Editor

On Wed, Jul 1, 2015 at 9:27 AM, RJPBCS RJPBCS <editor.rjpbcs@gmail.com> wrote:

Research Journal of Pharmaceutical, Biological and Chemical Sciences

(ISSN: 0975-8585)

Dear Author,

Thank you for submission of manuscript to RJPBCS. Your Manuscript number is **RJPBCS / 2010-5565**. Please quote manuscript number for future correspondence. Your manuscript will be sent for review and comments / status will be intimated as early as possible.
Thanks for your support towards RJPBCS.
With regards

Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI,
Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.
Indexed Copernicus Value: 4.62

On Tue, Jun 30, 2015 at 5:39 PM, Nita Kusumawati <nitakusumawati82@yahoo.com> wrote:

Dear editor. Along with this email, I attach a scientific article that I want to

Received payment, your manuscript will be publishes in 6(5) issue.
Thanks for your support towards RJPBCS.
With regards
Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI, Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.

On Fri, Aug 7, 2015 at 3:12 PM, Nita Kusumawati <nitakusumawati82@yahoo.com> wrote:

Dear editor, by this email i attach the proof
of article payment Ref.No:RJPBCS/2010-5565. Thank you.

Best regard,
Nita Kusumawati

Pada Minggu, 12 Juli 2015 14:24, RJPBCS RJPBCS <editor.rjpbcs@gmail.com> menulis:

Research Journal of Pharmaceutical, Biological and Chemical Sciences

(ISSN: 0975-8585)

CODEN: RJPBBP

Impact factor 0.35

Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.

Indexed and Abstracted in

SCI mag (SJR), Scopus, Embase (Elsevier), CAS, DOAJ, AYUSH Research Portal (Govt. Of India), Indexed Copernicus (ICV: 4.62), CABI, Open J-Gate, Google Scholar, Science Central, Index Scholar etc.

Dear **Nita Kusumawati, Toeti Kostiari, Agus Budi Santoso.**
Ref.No: RJPBCS/2010-5565.
I am pleased to inform you that your manuscript entitled **"The Influence of Washing Process Using TRO on Indigosol Dyeing Quality, Leaching Percentage, and Mechanical Strenght of Mori Fabric**
." is accepted for publication in RJPBCS volume 6 issue 5. Kindly send the scanned copy of copyrights transfer form along with pay in slip of manuscript handling charges for 01 article of $150.00 (one hundred and fifty USD only) to the following account. Copyrights transfer form you get it from www.rjpbcs.com website. If you have any queries feel free to mail me to editor.rjpbcs@gmail.com.
Important Note: Important Note: Please make payment before 20/07/2015 otherwise it may delay the publication and mention manuscript reference number on transaction scan copy. For Fast and rapid action make payment through Western Union / Moneygram

Bls: Acceptance: RJPBCS / 2010-5565

Dari: Nita Kusumawati (nitakusumawati82@yahoo.com)

Kepada: editor.rjpbcs@gmail.com

Tanggal: Rabu, 16 September 2015 07.19 GMT+7

Dear editor, I honestly do not understand why such a thing could happen in the management of major journals like you. Moreover, when it happens, there is no any information I received and it information just presented to me when I questioned. I hope in the future, there is no longer such an event, considering I was one of your loyal customers. Thank's before for your attention

Best regard,
Nita Kusumawati

Pada Senin, 14 September 2015 12:45, RJPBCS RJPBCS <editor.rjpbcs@gmail.com> menulis:

Dear Dr,

We are sorry for not publishing in 6(5), actually we sent for editing, but it has been missed by our technical editor, find attached file, if any corrections required edit yourself and mail revised version.

With regards

Managing Editor

On Mon, Sep 14, 2015 at 10:38 AM, Nita Kusumawati <nitakusumawati82@yahoo.com> wrote:

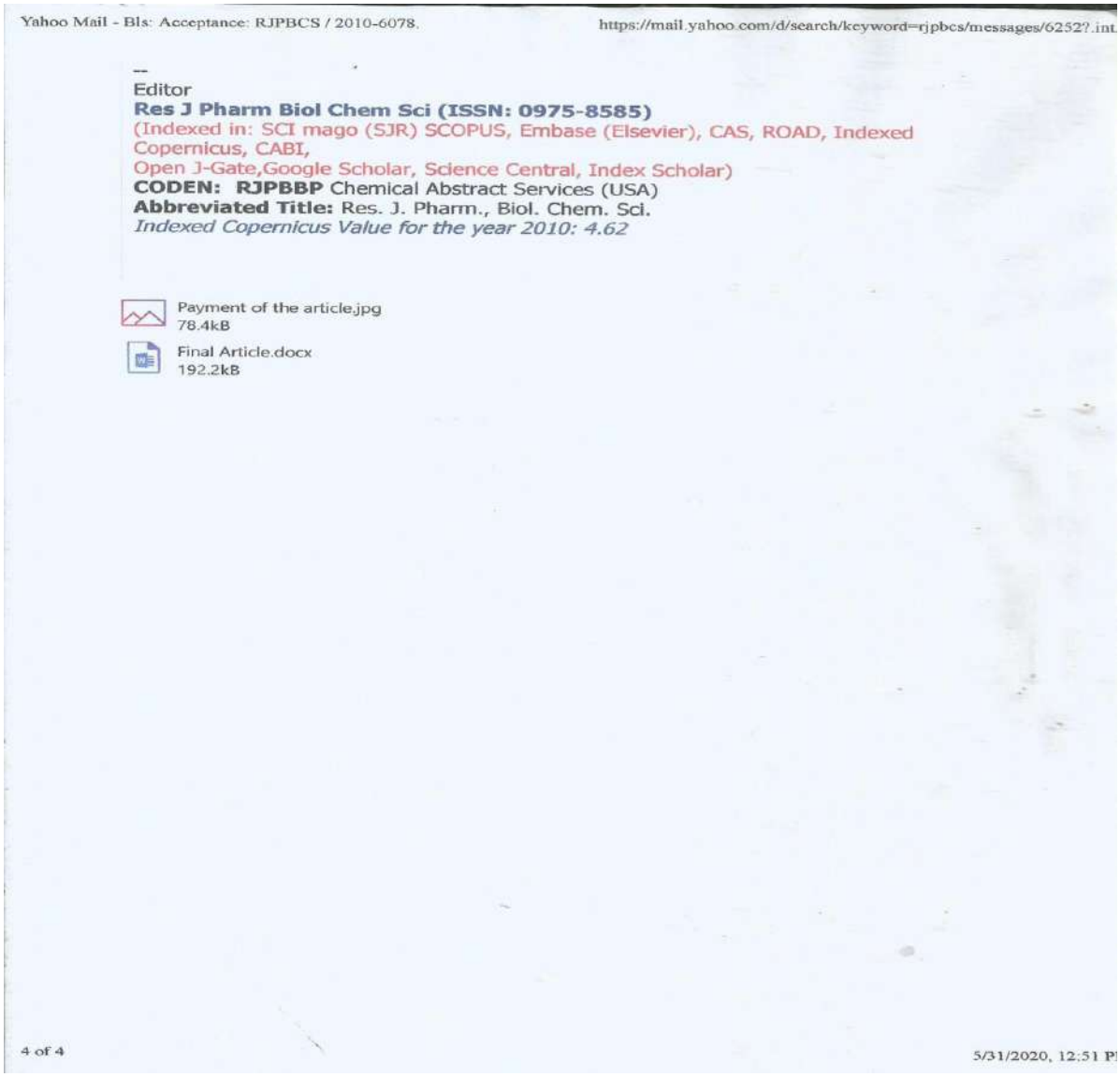
> Dear editor,
> I need your confirmation about the publication of my article, which according to information in the last email from you, will be published in 6 (5) issue. But it turns out, I can not find my article on 6 (5) issue.
>
> Best regard,
> Nita Kusumawati

Pada Sabtu, 8 Agustus 2015 10:57, RJPBCS RJPBCS <editor.rjpbcs@gmail.com> menulis:

Dear Author,

4. Terkait dengan karil no. 13, yang dinyatakan “diterbitkan oleh jurnal yang dikelola kurang baik”, dapat disampaikan klarifikasi dan bukti korespondensi sebagai berikut:

   Karil berjudul: Optimization of Batik Wax Waste Reutilization by Blending Method Using Gum Rosin, Paraffin, and Lard on Handmade Batik Fabric Production Towards Zero Waste Management diterbitkan pada tahun 2016 oleh *Research Journal of Pharmaceutical, Biological and Chemical Science* (RJPBCS), yang menurut hasil penelusuran pada website “Scimago Journal and Country Rank” pada saat itu masih terindeks scopus. Sebagai pelengkap, data berikut merupakan bukti korespondensi dengan pihak jurnal proses review berlangsung:



Yahoo Mail - Bls: Acceptance: RJPBCS / 2010-6078. https://mail.yahoo.com/d/search/keyword=rjpbcs/messages/6252?.int

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Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, ROAD, Indexed Copernicus, CABI,
Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.
Indexed Copernicus Value for the year 2010: 4.62

Payment of the article.jpg
78.4kB

Final Article.docx
192.2kB

4 of 4 5/31/2020, 12:51 P

Thanks for your support towards RJPBCS.
With regards

Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI, Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.
Indexed Copernicus Value: 4.62

On Sun, Oct 4, 2015 at 7:51 AM, Nita Kusumawati <nitakusumawati82@yahoo.com> wrote:

> Dear Editor, by this email, I inform to you that there are some errors in the scientific article that I sent through the 2 previous email. Therefore, by this email, I have also attached the final revision of my scientific article. Thank You.
>
> Best Regard,
> Nita Kusumawati

--
Editor
Res J Pharm Biol Chem Sci (ISSN: 0975-8585)
(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI,
Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.
Indexed Copernicus Value for the year 2010: 4.62

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Editor
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(Indexed in: SCI mago (SJR) SCOPUS, Embase (Elsevier), CAS, DOAJ, Indexed Copernicus, CABI,
Open J-Gate,Google Scholar, Science Central, Index Scholar)
CODEN: RJPBBP Chemical Abstract Services (USA)
Abbreviated Title: Res. J. Pharm., Biol. Chem. Sci.
Indexed Copernicus Value for the year 2010: 4.62

AYUSH Research Portal (Govt. Of India),
Indexed Copernicus (ICV: 4.62), CABI,
Open J-Gate, Google Scholar,
Science Central,
Index Scholar etc.

Dear **Nita Kusumawati, Anang Kistyanto, Deny Arifiana.**
Ref.No: RJPBCS/2010-6078.

I am pleased to inform you that your manuscript entitled **"Optimization of Batik Wax Waste Reutilization by Blending Method using Gum rosin, Paraffin, and Lard on Handmade Batik Fabric Production Towards Zero Waste Management"** is accepted for publication in RJPBCS Volume 6 Issue 6. Kindly send the scanned copy of copyrights transfer form along with Pay in slip of manuscript handling charges for 01 article of $125.00 (One hundred Fifty USD only) to the following Account. Copyrights transfer form you get it from www.rjpbcs.com website. If you have any queries feel free to mail me to editor.rjpbcs@gmail.com.

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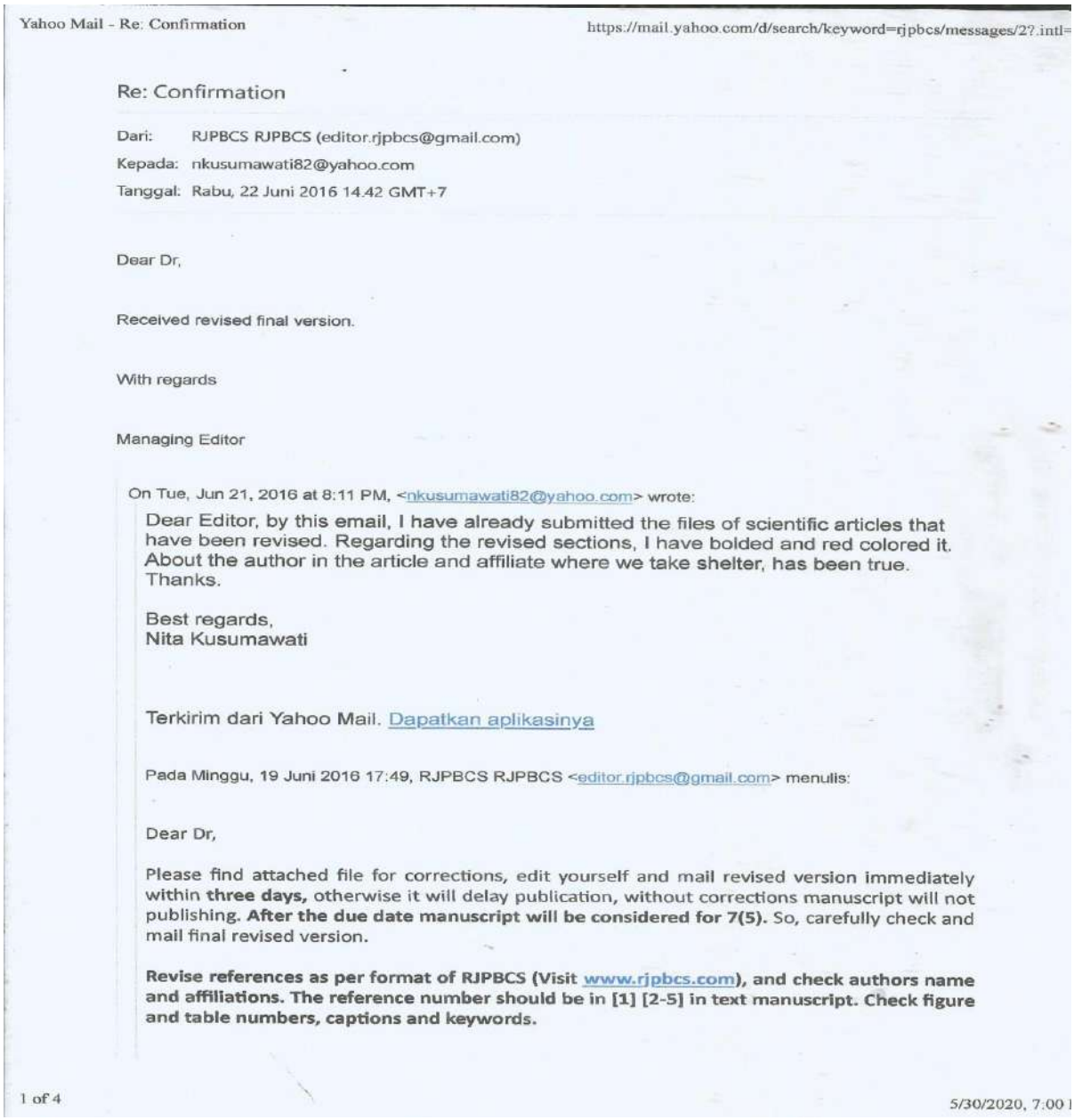

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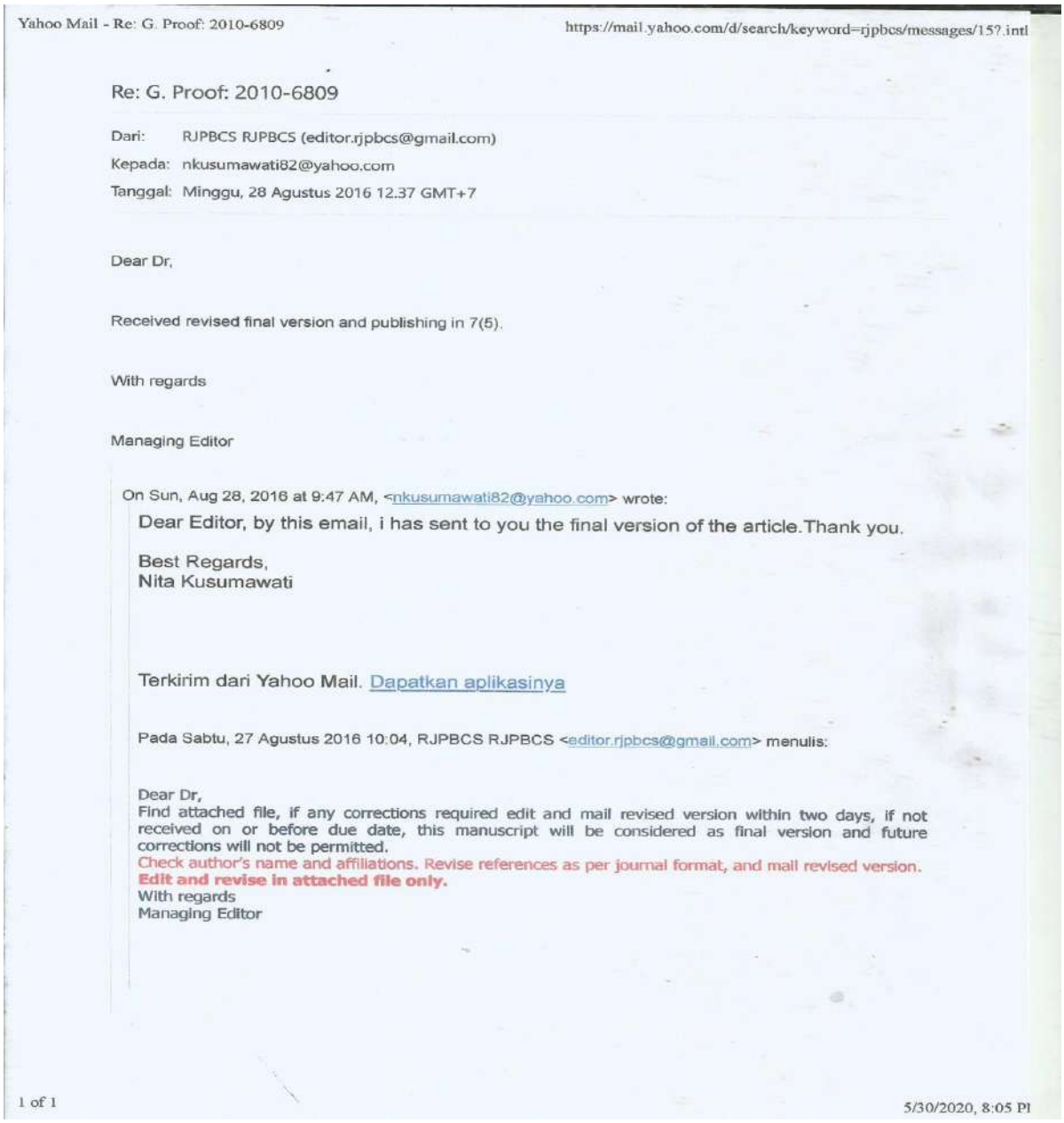

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Dari: Nita Kusumawati (nitakusumawati82@yahoo.com)

Kepada: editor.rjpbcs@gmail.com

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RJPBCS vol 7 no. 2 (2016)

by Nita Kusumawati

ISSN: 0975-8585

Research Journal of Pharmaceutical, Biological and Chemical Sciences

The Effect of Casting Solution and Non Solvent Composition on the Performance of Polysulfone Membranes against Chromium (VI) .

Keshinanta Dyah Ayu Maharani[1], and Nita Kusumawati[2]*.

[1]Departement of chemistry, Universitas Negeri Surabaya, Surabaya, Indonesia.
[2]Department of Chemistry, Universitas Negeri Surabaya, Surabaya, Indonesia.

ABSTRACT

In general, this research aims to study membrane preparation process which are capable to form asymmetry structures on the polysulfone membrane, resulting in increased permeability and selectivity. More specific, this study aims to determine the effect of casting solution and non-solvent composition on the formation of asymmetry structure, mechanical strength and membrane performance in the separation of chromium (VI). In this research, H_2O and CH_3OH are chosen as non-solvent solution which is used to induce phase inversion. To determine the effect of casting solution and non-solvent composition on membrane performance, casting solution composition was varied in the range of PSF/NMP/PEG400 (% w/w) 12/84 /4-16/84/0, and the non-solvent composition was varied in the range H_2O/CH_3OH (%v/v) 100/0, 75/25, 50/50. Permeability (flux) measurement result of PSf membrane using Dead end membrane reactor showed that the range of flux was 8060.39 $L/m^2.h$ - 468.78 $L/m^2.h$. Membrane with the smallest PSf composition on the casting solution and the biggest H_2O composition on the non-solvent has the largest flux and smallest selectivity (rejection percentage). From the results of selectivity (rejection percentage) measurement against chromium (VI), which performed by UV-Vis Spectrophotometer, shows the range of 12.61% - 49.19%. Meanwhile, mechanical strength measurement results for the membrane with the smallest composition of PSf and biggest H_2O is 2458.89 N/m^2, and for the biggest composition of the PSf with the smallest composition of H_2O is 3427.06 N/m^2. Generally, in this research, polysulfone membrane having obtained the high mechanical strength and performance.

Keyword: Membranes, Polysulfone, Polyethylene glicol, Chromium (VI)

Corresponding author

ISSN: 0975-8585

INTRODUCTION

In present, the textile industries are some manufacturing industries which have important effect in Indonesia [1]. However, the textile industry also gives serious problems to the environment [2]. The main serious problem caused by the textile industry is the problem of waste, where the levels of total chromium is one of the parameters that can be used as an indicator of polluted water, and considered dangerous for the environment [3]. In the textile industry, chromium mainly used in dyeing process both as a dye and a mordant (binding color) [4]. Generally, the chromium ion is found as trivalent chromium [Cr (III)] and ionic hexavalent chromium [Cr (VI)], which hexavalent chromium ion is more toxic and carcinogenic [5], [6]. One methode that can be used for the treatment of heavy metal chromium (VI) is the membrane technology.

During this time, preparation of PSf membranes has been successfully done by two kind methods, namely sintering and track-etching [7]. But on further development, those methods began to be abandoned because of the membrane produced by those methods still has a high mass transfer resistance, cause of symmetry structure [8]. The high mass transfer will produce membrane with distant ratio differences of flux and rejection, because the membrane is dominated by the sponge pore structure. The condition in which the membrane is dominated by sponge structure thus, can increase the likelihood of fouling. Therefore, to obtain the PSf membrane with an equal ratio of flux and rejection, in research will be pursued formation of asymmetry structure. The asymmetry structure of the membrane can be produced through a phase inversion method with immersion precipitation technique [9], [10].

In order to produce PSf membrane with equal ratio of flux and rejection, PSf membrane that will be used for treatment of chromium hexavalent, made with varied composition of casting solution and non-solvent composition using phase inversion method which is induced by immersion precipitation techniques [11]. The casting solution composition this research was PSf/NMP/PEG (%w/w) 12/84/4; 12.5/84/3.5%; 13/84/3; 13.5/84/2.5; 16/84/0), while the non-solvent composition was H_2O/CH_3OH (%v/v) 100/0, 75/25, 50/50. The results of this research analyzed using several instruments: "Dead-end" membrane reactor for measuring permeability, spectrophotometer (UV-Vis) to measure the selectivity (rejection), autograph to measure the mechanical strength, and scanning electron microscopy (SEM) instrument for imaging the surface and cross section morphology of the membrane [12].

EXPERIMENTAL

Material

Materials research include: Polysulfone (PSf) (Sigma-Aldrich, d= 1.24 g/mL at 25 ^{0}C), N-methyl pyrrolidynone (NMP) (Sigma -Aldrich, MW: 99.13 g/mole), technical methanol (PT. BRATACO, batch: K-02801-3), PEG (PT. BRATACO, mw : 400, batch : X14648), distilled water, sulfuric acid (H_2SO_4) (Ajax Finichem Pty Ltd, >98%), 1.5-diphenylcarbazid (Merck, GR for analysis and redox indicator), and gauze for supporting layer of the membrane (Kasa Husada Indonesia, 254 Mesh).

Equipment

Research equipment includes: Ohaus analytical balance, NESCO LAB MS-H280-Pro magnetic stirrer with temperature control for mixing the membrane material, Seiko stopwatch, coagulation bath (self-made), and OP-211 pH meter. While the research instruments include: Shimadzu AG-10TE autograph, dead-end membrane reactors (self-made) which is connected with compressor as a pressure source, Shimadzu UV-1800 UV-Visible spectrophotometer, and Zeiss EVO MA10 Scanning Electron Microscopy (SEM).

Preparation of Polysulfone Membrane

The PSf membrane was made by phase inversion methods. Polysulfone was dissolved in NMP then added PEG [13-16]. The ratio of casting solution composition: (PSF/NMP/PEG) (%w/w): 12/84/4; 12.5/84/3.5; 13/84/3; 13.5/84/2.5; 16/84/0. The solution was stirred with a magnetic stirrer at 60 ^{0}C for two hours [8]. A homogeneous polymer solution then casted using casting knife on a glass plate that coated with polyester [17-18], with 0.8 mm thickness, and then soaked in a coagulation bath containing a mixture of distilled water and methanol with ratio H_2O/CH_3OH (%v/v): 100/0; 75/25; 50/50. A solid PSf membrane formed then washed

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by using distilled water to remove the remnants of the solvent. After that, the solid membrane dried on the open air.

Polysulfone Membrane Performance Test

The performance of PSf membrane was determinated by measuring flux and rejection of the membrane against chromium (VI). For PSf membrane permeability test using Dead end membrane reactor, dried membrane was cut ± 6 cm. On the equipment was applied pressure 1 bar as a driving force of the 5 ppm Cr (VI) feed solution. The feed solution flowed through the membrane, and the time when all of the feed solution filtered out is recorded. Furthermore, according to the recorded time, the volume of feed solution, and the surface area of the membrane, the flux was calculated.

Furthermore, to determine rejection coefficient of the PSf membrane, feed solution with maximum wavelength of 542 nm, measured concentrations before and after passing through the PSf membrane using UV-Vis spectrophotometer, where in every measurement process, feed solution was controlled at pH 2 using H_2SO_4 1M and 1,5-diphenylcarbazide $1{,}44x10^{-4}$ M was added too into the solution as complex agent Cr (VI) [19].

Polysulfone Membranes Characterization

The characterization of PSf membrane on this research includes physical and mechanical characterization. The physical characterization of the PSf membranes is done by imaging the surface and cross section morphology using SEM. While the mechanical characterization done by measure the mechanical strength of the PSf membrane using Shimadzu AG-10TE autograph. Mechanical strength test of PSf membrane gave the magnitude of stress and strain which experienced by PSf membrane over the force of 100 kN [20], then they were compared to calculate the young's modulus .

RESULTS AND DISCUSSION

Polysulfone Membrane Preparation

In present, phase inversion is commonly used as preparation method for polysulfone membrane because of its simple and easy to produce membrane, so it's cost less in production process. Preparation of PSf membrane was used casting solution composition range (PSF/NMP/PEG) (%w/w) 12/84/4 – 16/84/0 and non-solvent composition range (H_2O/CH_3OH) (%v/v) 100/0 – 50/50. Casting solution was stirred in specific temperature 60 ^{0}C for two hours. The temperature was chosen because that the higher stirring temperature used, the greater pore size formed in the membrane. Meanwhile the selection of NMP as PSf solvent in this study is due to smaller solubility parameter differences between PSf and NMP than PSf with the other solvent was probably used on the PSf membrane preparation. The solubility parameter differences between PSf, NMP, and DMAc shown in table 1.

Table 1. Hansen solubility parameters between PSf and the solvents (MPa^{½}) [13]

Material	δ_D	δ_P	δ_H	δ_t	$[\delta_{tPSf} - \delta_t Solvent]$
PSf	19,7	8,3	8,3	22,93	
NMP	18	12,3	7,2	22,96	0.03
DMAc	16,8	11,5	10,2	22,77	0.16

On the preparation process of PSf membrane, Polyethylene Glycol (PEG) added as additive substance, in order to increase porosity which automatically will be increase the permeability of PSf membrane. However, uses of additive with high molecular weight, such as PEG, can increase the formation of macrovoid which can decrease the mechanical strength of the membrane. Because of that, uses of non solvent from the combination of H_2O and CH_3OH, expected will be decrease the solubility parameter differences between solvent and non-solvent, so it can reduce macrovoid. A homogeneous polymer solution cast used a casting knife on a glass plate (thickness of 0.8 mm) that coated with polyester. The illustration of casting knife methode can be observed in figure 1. In the casting process of PSf membrane, was used polyester as supporting layer. Polyester was used to increase the mechanical strength of PSf membrane.

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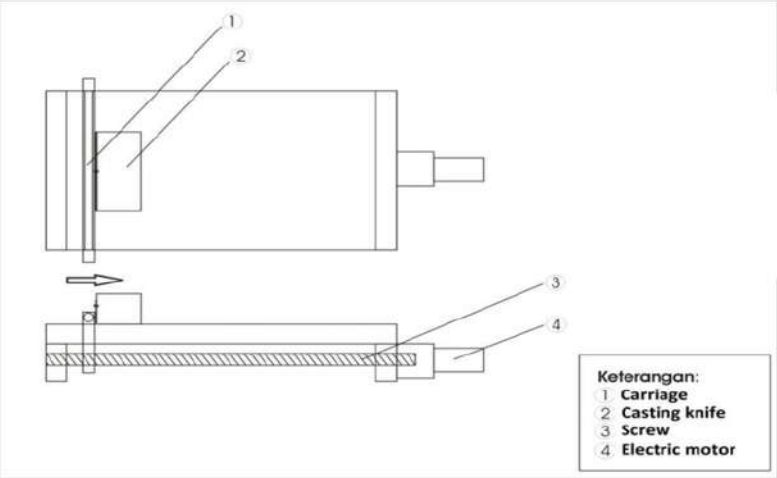

Figure 1. Illustration of polysulfone membrane casting with casting knife methode

On this research, the preparation of PSf membrane has been done using phase inversion method with immersion precipitation technique, where the liquid phase changes into the solid phase induced by particle change processes between solvent (NMP) with non-solvent (H_2O and CH_3OH). The differences between the solvent and non-solvent solubility parameter that used in the membrane preparation process will be increase the mixing temperature (ΔH) of the casting solution, that will be followed by ΔH and ΔG (Gibb's free energy) increases. The increased of Gibb's free energy will trigger the occurrence of membrane casting solution coagulation, so it can generated the solid PSf membrane [21]. The solubility parameter differences between solvent and non-solvent that are used on the PSf membrane preparation shown in table 2.

Table 2. Hansen solubility parameters between solvent and non-solvent [21]

Non Pelarut	δ_d (MPa$^{1/2}$)	δ_p (MPa$^{1/2}$)	δ_H (MPa$^{1/2}$)	δ_t (MPa$^{1/2}$)	$\Delta\delta$
NMP	18	12.3	7.2	22.96	
Metanol	11,6	13,0	24,0	29,7	6,74
Air	12,2	22,8	40,4	48,0	25,04

The interactions between components of PSf membrane affect in differences of the phase inversion timing for each membrane composition, it can be observed in figure 3. Interactions that may occur between the components of PSf membrane can be observed in figure 2.

Characterization of PSf Membrane Performance

The performance of PSf membrane (permeability and selectivity) characterized with dead-end reactor and a spectrophotometer (UV-Vis).

Permeability of PSf Membrane

Flux value that was gotten, showed the amount of permeate volume which passed by the membrane at the specific wide of membrane and specific flow rate time. Permeability (flux) test aims to inform characteristic of the membrane's pore, include: porosity, pore size, and pore structure.

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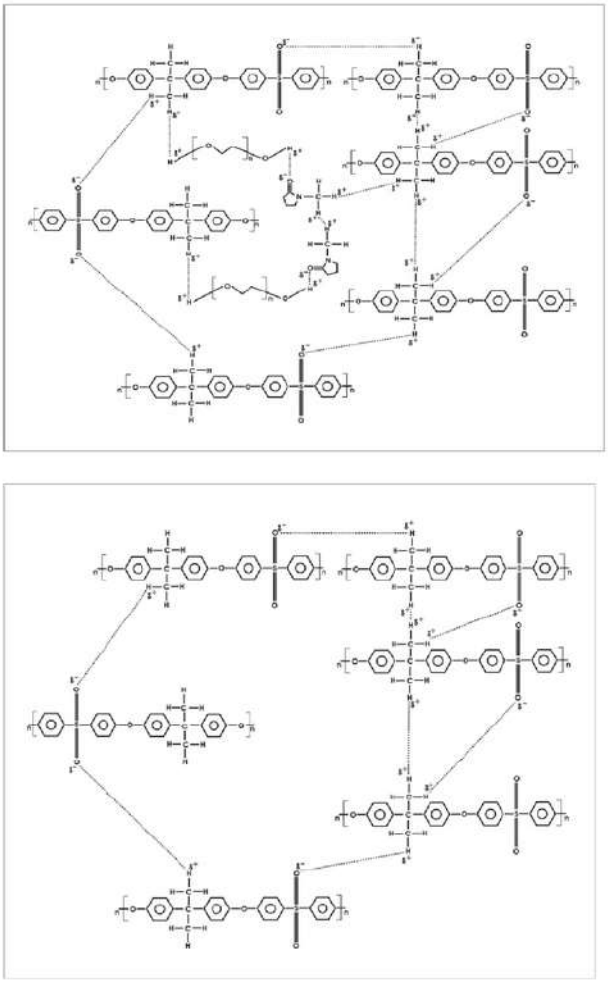

Figure 2. Interaction between the components of PSf membrane: on casting solution (a); solid membrane (b) (hyphothetic)

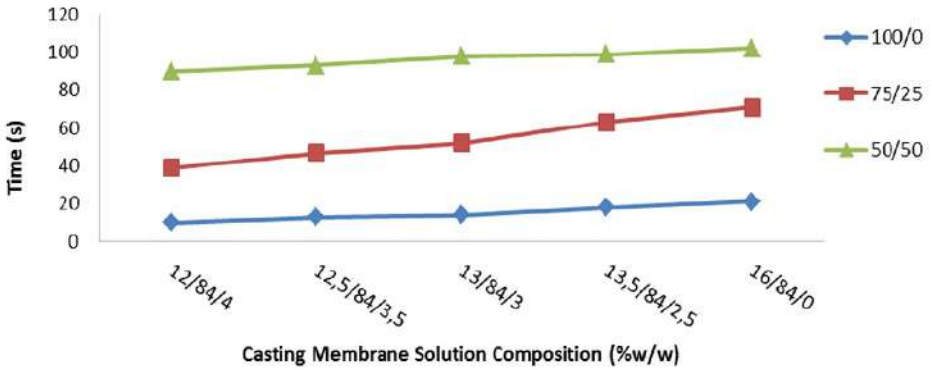

Figure 3. Phase inversion timing

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In this research, permeability test done by using $K_2Cr_2O_7$ as a feed solution with 5 ppm concentration, and 250 mL volume. The pressure was set at 1 bar. The interaction between PSf membrane and Cr (VI) showed in figure 4, and the illustration of dead-end reactor membrane showed in figure 5.

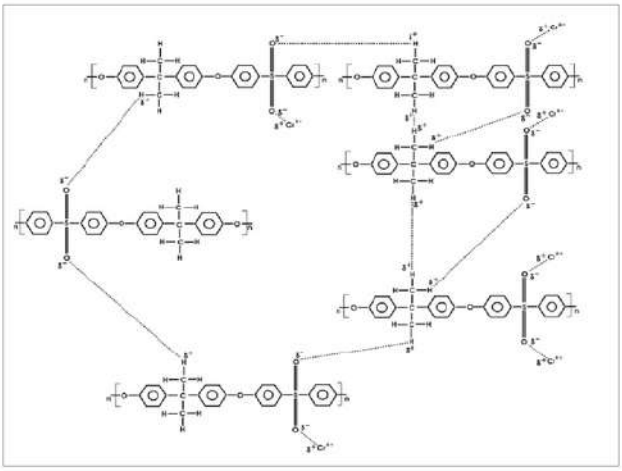

Figure 4. Interaction between solid membrane and Cr (VI) (hypothetic)

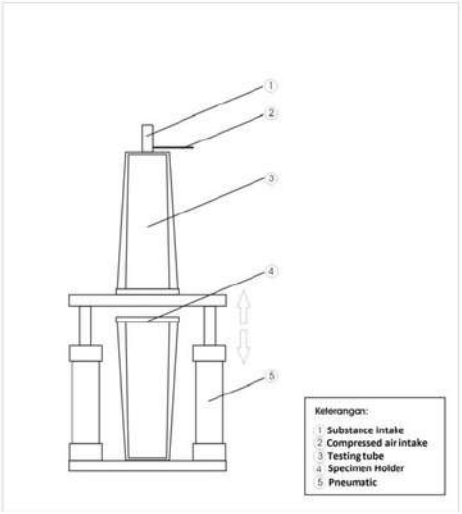

Figure 5. Illustration of dead-end reactor membrane

The PSf polymer composition in casting solution and the H_2O composition on the non solvent greatly affects the permeability of the membrane. Polysulfone membrane with the smallest amount of PSf polymer and biggest volume amount of H_2O has the highest flux value, which amounted at 8578.07 $Lm^{-2}h^{-1}$. Whereas, polysulfone membrane with the biggest amount of PSf polymer and the smallest volume amount of H_2O has the lowest flux value, which amounted at 468.74$Lm^{-2}h^{-1}$. In addition, the effect of the casting solution and non solvent composition on the membrane permeability can be explained using the equation [11]:

$$\Delta G = \Delta H - T\,\Delta S \quad (1)$$

$$\Delta H = \varphi_1\varphi_2 V(\delta_1 - \delta_2)^2 \quad (2)$$

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When the difference in solubility parameter between the solvent and non solvent becomes higher, ΔH reaction will also increase. The increasing of ΔH, will be followed by an increase in ΔG reaction, that will cause an unspontaneous in the casting solution system. These conditions made phase inversion process of the casting solution becomes faster. Faster phase inversion process has resulted in less availability of time that sufficient to form more complex interaction between the membrane molecules. Then, this led to form larger pores size on the membrane. The pore size determines the time it takes the feed solution ($K_2Cr_2O_7$) to flow through the PSf membrane will be shorter, resulting a greater flux value, and vice versa. Flux data of the PSf membrane can be observed in figure 6.

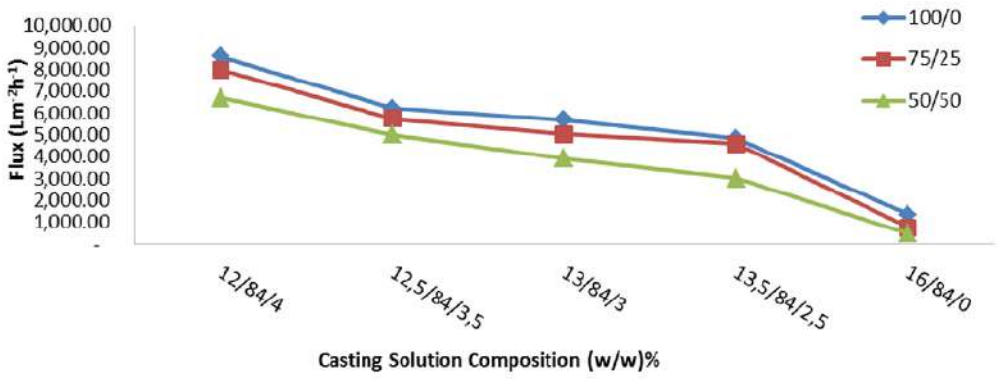

Figure 6. Flux of the PSf membrane against $K_2Cr_2O_7$

PSf membrane selectivity

Selectivity is one of the parameters that can determine the ability of the membrane to withstand a particular species and oth species that passed in this case a very influential to it is the large size of the particular species and the pore size of the membrane. The parameter used to measure the selectivity of the membrane, called the rejection coefficient. In this research, $K_2Cr_2O_7$ feed solution with a maximum wavelength of 542 nm, measured concentrations before and after passing through the PSf membrane using UV-Vis spectrophotometer. The rejection coefficient of the PSf membrane shown in figure 7.

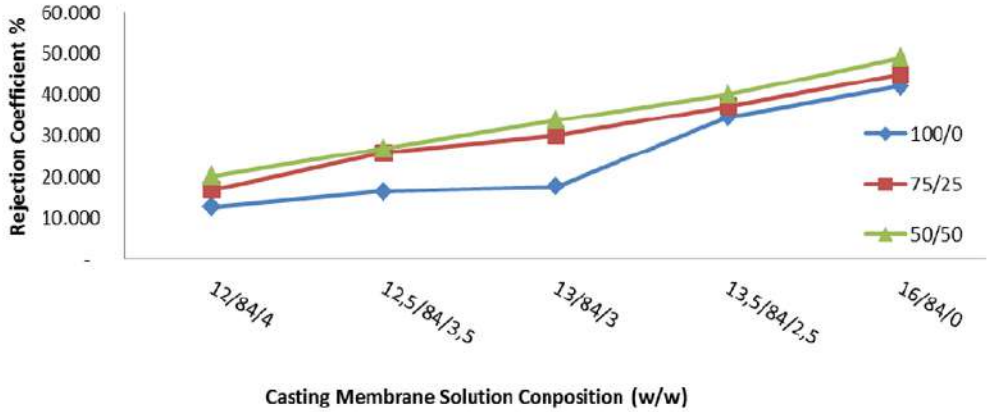

Figure 7. The rejection coefficient of the PSf membrane against $K_2Cr_2O_7$

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Based on data from the rejection coefficient, the membrane with the smallest amount of PSf polymer and the biggest amount of H_2O volume has a minimum rejection coefficient, which amounted 12.67%. While the membrane with the biggest amount of PSf polymer and the smallest amount of H_2O volume has a maximum rejection value amounting to 49.19%. In this research, the highest value of selectivity PSf membrane, only reached 49.19%, those values are still far from the maximum value of selectivity, i.e. by 100%. It indicates that filtration role doesn't really exist to filter Cr (VI) ions on membrane filtration, PSf. That is because the ionic formed of Cr (VI) are relatively smaller than the molecule of water. So it is confirmed that the ions Cr (VI) which only filtered less than 50%, and 50% other passed along with the water through the membrane of the PSf. The highest value of this selectivity has been obtained 49.19% of this research, because the selectivity of PSf membrane more caused by adsorption phenomena than filtration phenomena. The adsorption phenomena on this case worked through physical interactions between the membrane and chromium (VI). The physical interactions occur by electrostatic force between positive charge of the chromium (VI) and the opposite charge on the membrane in which case are oxygen atoms. Statements regarding the existence of the membrane filtration with adsorption function is also reinforced in the research on PSf ultrafiltration membrane in addition to get filtration function also has adsorption role. Adsorption role is caused due to a physical interaction between the surface membrane with the feed solution, which in that research is protein. Besides that, larger pore size would cause $K_2Cr_2O_7$ component that successful rejected lower, and vice versa, smaller pore sizes would cause the membrane selectivity against the KCr_2O_7 feed solution becomes higher.

Mechanical strength of PSf membrane

The mechanical strength gave the magnitude of stress and strain on PSf membrane. The magnitude of mechanical strength that belongs to PSf membrane measured by young's modulus, that is the ratio of the stress to the strain. The mechanical strength data of the PSf membrane, can be observed in table 3.

Table 3. Young's Modulus of PSf membrane

Casting Solution Composition (%b/b)	Non Solvent Composition (H_2O/CH_3OH) (%v/v)	Young's Modulus (N/m^2)
12/84/4	100/0	2458.89
16/84/0	50/50	3427.05

The data showed that the casting solution composition affect the mechanical strength of the PSf membrane, where the membrane with the largest quantity of PSf, will tend to form a tighter space between the particles. This caused a membrane having a smaller pore size and a higher mechanical strength. Likewise, the membrane with the smallest quantity of PSf, will tend to form the larger space between the membrane particles and led to the lower mechanical strength of the membrane.

The surface and cross section morphology of the PSf membrane

Based on the results of SEM analysis in figure 8, PSf membrane with casting solution composition of PSf/NMP/PEG (%w/w) 12/84/4 and non-solvent composition of H_2O/CH_3OH (%v/v) 100/0, has a larger pore size, compared with membrane with casting solution composition of PSf/NMP/PEG (%w/w) 16/84/0 and non-solvent composition of H_2O/CH_3OH (%v/v) 50/50, has a smaller pore size. But unfortunately, despite having obtained the surface morphology characteristic of the membrane, but not significantly observed the formation of the asymmetry structure in the polysulfone membrane that formed.

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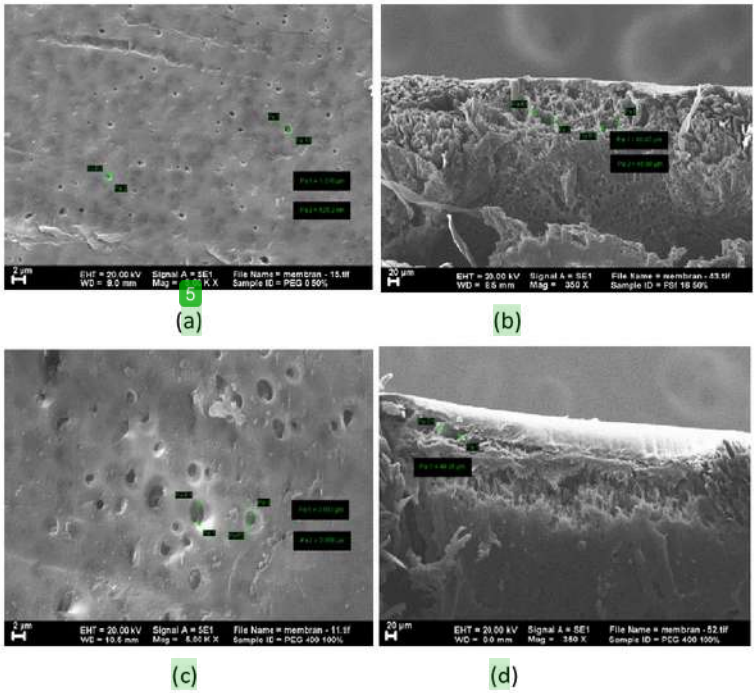

Figure 8. The surface morphology of the PSf membrane from 12%PSf/84%NMP/4%PEG (a) and 16%PSf/84%NMP (c); the cross section morphology of the PSf membrane from 100%H_2O (b) and 50%H_2O/50%CH_3OH (d)

CONCLUSION

Based on the results of the data that has been obtained, the conclusion that can be drawn are: (1) casting solution and non solvent composition affect the permeability of PSf membrane, where the membrane flux obtained were in the range 8603.39 $L.m^{-2}.h^{-1}$ – 468,78 $L.m^{-2}.h^{-1}$, (2) casting solution and non solvent composition affect the selectivity of PSf membrane, where the membrane rejection obtained were in the range 12.61% - 49.19; (3) the results of the mechanical strength test (Young's modulus) show that membrane with largest permeability (flux) has the smallest Young's modulus, which is 2458.89 N/m^2 and membrane with largest selectivity has the largest Young's modulus, which is 3427.05 N/m^2; (4) the imaging result of the surface morphology shows that the PSf membrane resulted from preparation using casting solution composition 12%PSf/84%NMP/4%PEG and non solvent composition 100%H_2O has larger pores than PSf membrane resulted from preparation using casting solution composition 16%PSf/ 84%NMP and non solvent composition 50%H_2O/50% CH_3OH, at the same magnification. But unfortunately, despite having obtained the surface morphology characteristic of the membrane, but not significantly observed the formation of the asymmetry structure in the polysulfone membrane that formed.

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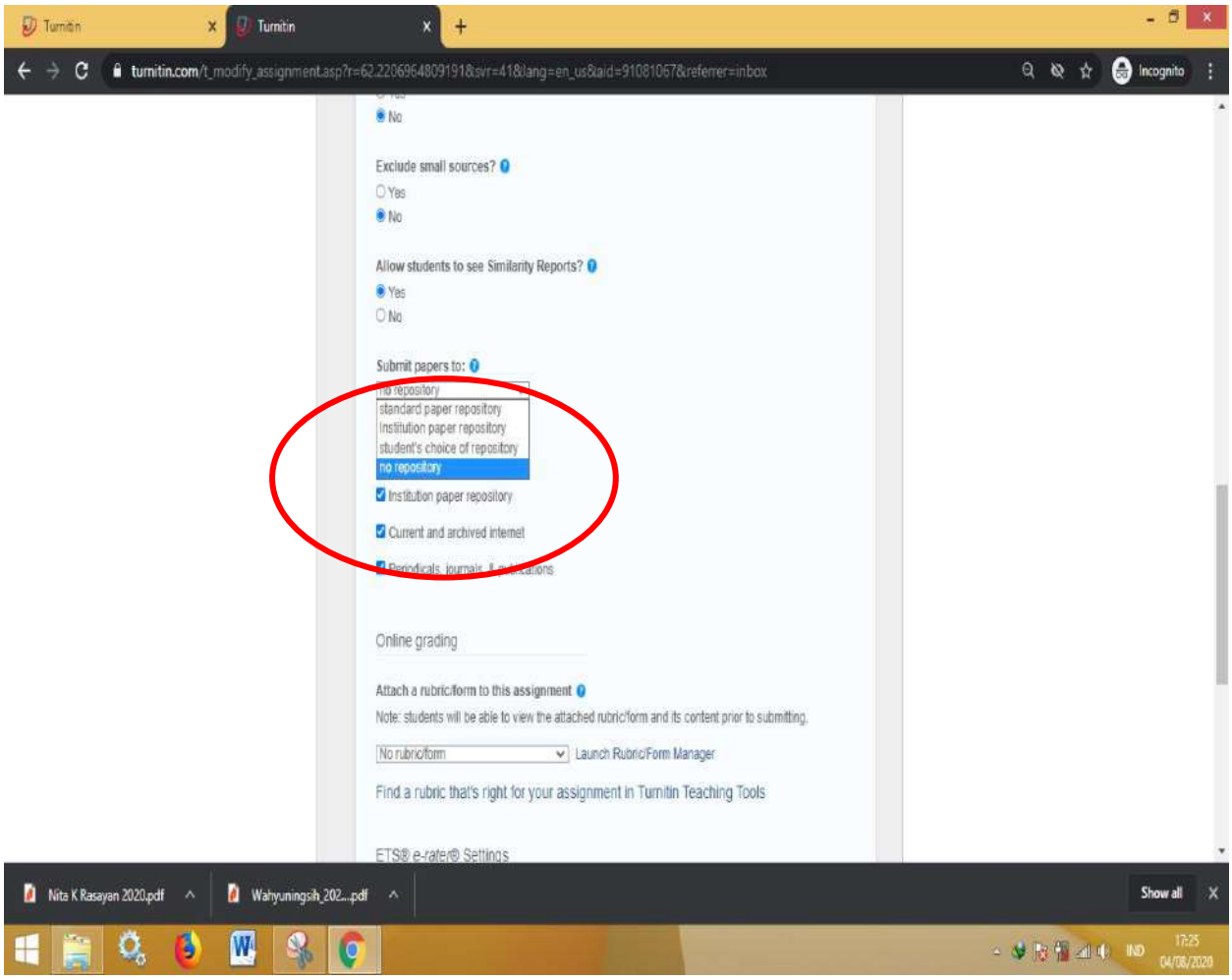

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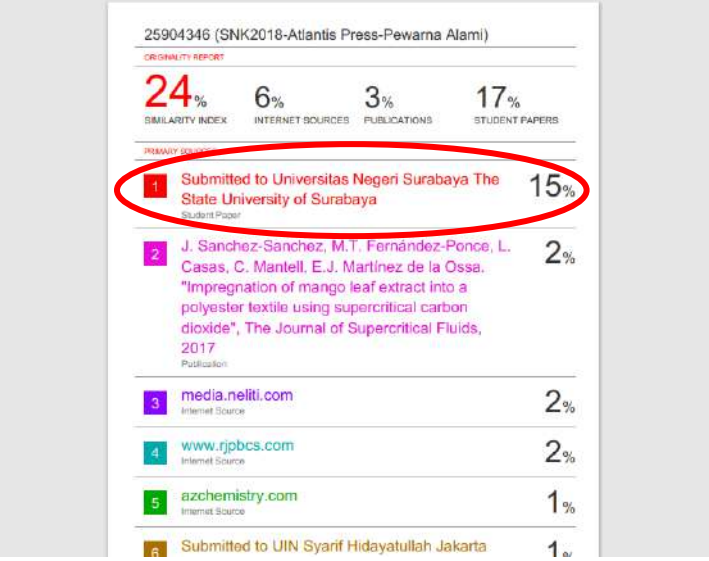

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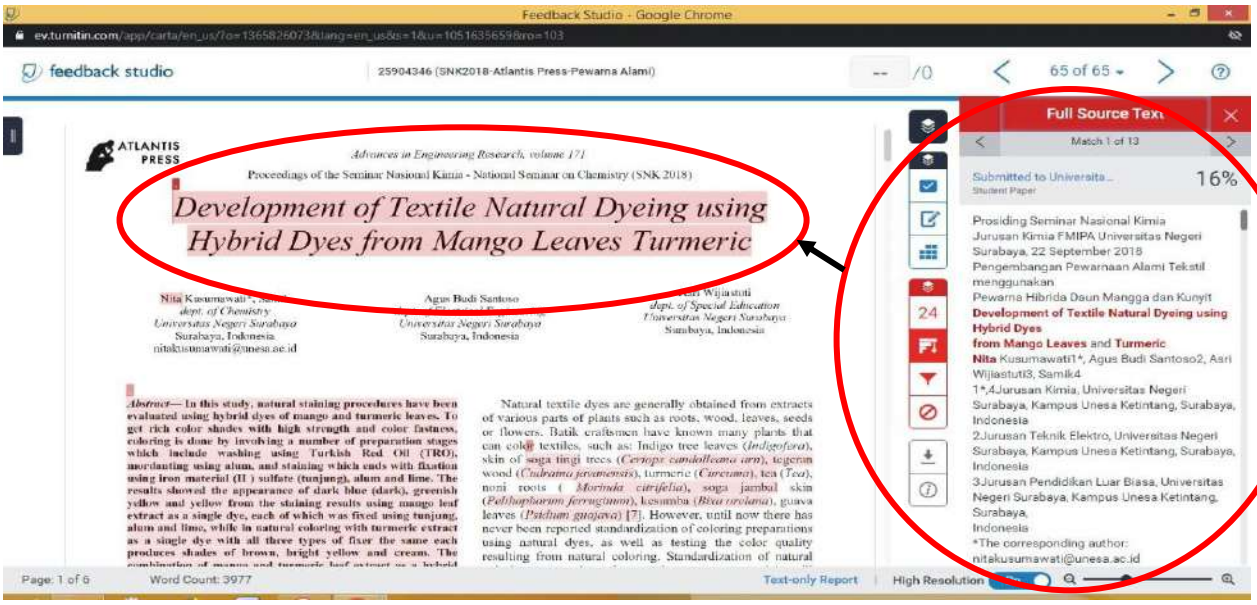

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Development of Textile Natural Dyeing using Hybrid Dyes from Mango Leaves Turmeric

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***Abstract*— In this study, natural staining procedures have been evaluated using hybrid dyes of mango and turmeric leaves. To get rich color shades with high strength and color fastness, coloring is done by involving a number of preparation stages which include washing using Turkish Red Oil (TRO), mordanting using alum, and staining which ends with fixation using iron material (II) sulfate (tunjung), alum and lime. The results showed the appearance of dark blue (dark), greenish yellow and yellow from the staining results using mango leaf extract as a single dye, each of which was fixed using tunjung, alum and lime, while in natural coloring with turmeric extract as a single dye with all three types of fixer the same each produces shades of brown, bright yellow and cream. The combination of mango and turmeric leaf extract as a hybrid coloring material has enriched the color shades produced with color strength 78.64% -90.16% for 25% DM / 75% K; 77.33% -90.70% to 50% DM / 50% K; and 75.87% -87.36% for 75% DM / 25% K. Furthermore, the use of hybrid dyes from a combination of mango and turmeric leaf extract has resulted in high color fastness. This can be seen from the staining scale of each dye, ie 2-3 (less) - 4 (good) for 25% DM / 75% K; 3-4 (good enough) - 4 (good) for 50% DM / 50% K; and 3 (enough) - 3-4 (good enough) for 75% DM / 25% K.**

Keywords—Textile, Dyes, Natural, Mango leaves, Turmeric

I. INTRODUCTION

Small industries have a very large role in development Indonesian economy. In 2003, a small industrial sector that was able to absorb 99.4% of the Indonesian workforce. One of the small industries that absorbs the most labor is the batik industry. The development of the batik industry from the beginning was recognized by UNESCO as a non-Indonesian original cultural heritage [1].

Unfortunately, the rapid development of the batik industry is not accompanied by an increase in awareness of batik craftsmen and entrepreneurs to attend and to manage their production waste [2]. During this time, a large amount of liquid waste produced every day by batik UMKM in almost all parts of Indonesia, was immediately thrown into the body of water [3]. This condition is worrying, considering that 15-85% of the total textile dyes are used, they do not bind to fabric fibers and are wasted as waste effluent [4] - [5]. The percentage of dyes that pass as an effluent increases the potential for environmental damage that can be caused [6]. One of the solutions to this problem is to minimize the use of synthetic dyes and re-promote the use of environmentally friendly natural dyes.

Natural textile dyes are generally obtained from extracts of various parts of plants such as roots, wood, leaves, seeds or flowers. Batik craftsmen have known many plants that can color textiles, such as Indigo tree leaves (*Indigofera*), skin of soga tingi trees (*Ceriops candolleana arn*), tegeran wood (*Cudraina javanensis*), turmeric (*Curcuma*), tea (*Tea*), noni roots (*Morinda citrifelia*), soga jambal skin (*Pelthophorum ferruginum*), kesumba (*Bixa orelana*), guava leaves (*Psidium guajava*) [7]. However, until now there has never been reported standardization of coloring preparations using natural dyes, as well as testing the color quality resulting from natural coloring. Standardization of natural coloring preparations from various source materials will greatly determine the homogeneity of coloring. The creation of the Standard Operating Procedure (SOP) for the preparation of natural dyes through this research, will make the quality of the coloring of batik cloth homogeneous and not dependent on the skills of the Human Resources (HR) who are the executors of coloring.

Mango (*Mangifera indica L.*) is a medicinal plant that belongs to the *Anacardiaceae* family. Mango is a plant native to Southeast Asia that is widely distributed to most tropical regions of the world. Mango is the third most popular tropical fruit worldwide and its production produces considerable agro-industrial wastes, such as skin, kernel seeds, leaves and bark derived from pruning and fruit processing industries. Many studies have shown that mango leaves are one of the main sources of polyphenols with powerful antioxidant functions and other pharmacological properties [8] - [9]. *Mangiferin* is one of the dominant polyphenols in mango leaves that has a function as a natural dye and has several pharmacological properties such as anti-oxidants, anti-bacterial, and anti-fungi [8] - [12]. Thus, the use of material in natural textile coloring is expected to provide 2 (two) benefits at once, namely coloring and providing protection from attacks by free radicals and microorganisms.

Other than *mangiferin, curcuminoids* are one of the producers of natural textile dyes, yellow, resulting from the extraction of turmeric root (*Curcuma longa L*), with the main constituent known as curcumin, along with small amounts of *demethoxycurcumin* and *bisdemethoxycurcumin* [13]. As well *mangiferin*, a large number of previous studies have also been successful in proving the functionality of *curcumin*, which is not only able to color silk fibers in brilliant yellow but is also capable of providing other

functional properties, such as antibacterial activity [14] and antioxidants [15]. Thus, the coloring of textiles using turmeric material will not only produce the appearance of brilliant yellow shades on the fiber, but also be able to make the related textile material odorless due to the minimal interaction between sweat users of textile products with some types of microorganisms that cause body odor.

II. MATERIAL AND METHOD

A. *Material*

The raw material used as a color producer in this study is mango and turmeric leaves, does not have special specifications. Turkish Red Oil (TRO) material used in this research was obtained from UD. Main Chemical Surabaya-Indonesia. Alum (Al2 (SO4) 3) which in this study played a dual role, as a mordanting agent and fixer and ash soda (Na2CO3) purchased from PT. Brataco Surabaya-Indonesia. Tunjung (FeSO4.7H2O) and lime (CaCO3), both of which were used as fixers in this study were purchased from UD. Jaya Murni Surabaya-Indonesia.

B. *Mordanting*

Mordanting is generally carried out to increase the interaction of coloring compounds with textile materials. This stage can produce more even and sharp coloring results. Mordanting is carried out in the following stages. Three pieces of 25x25cm textile material that will be colored, soaked in TRO 2 g / L for 6 hours. The textile material is then rinsed by soaking it in 2 liters of water while still stirring using a magnetic stirrer and then aerated. The textile material is further processed with mordanting using alum and soda ash.

The mordant solution was prepared by dissolving 16.7 grams of alum and 4.2 grams of soda ash in 2 liters of water. The solution is stirred with the magnetic stirrer until it dissolves completely (± 5 minutes). The solution is heated to 100 °C, then a fabric is inserted into it while continuing to heat for up to 1 hour. The fabric is left in a submerged condition for 12 hours. Flushing the fabric is done by soaking in 2 liters of water which is stirred continuously using a magnetic stirrer for 10 minutes. The cloth is then dried and ironed. After that, the fabric is ready to be colored.

C. *Fixer Preparation*

To find high fastness in natural coloring, the natural coloring of textiles usually ends with fixation or color locking. In general, the preparation of the fixer solution is carried out by dissolving 50 grams of tunjung in 1 liter of air. The solution is formed and then left to settle, the clear side of the solution is used for the fixation process.

D. *Natural Dyeing*

Extraction of natural coloring compounds from mango and turmeric leaf material was carried out by the following procedure. Five hundred grams of raw material is cut into small pieces and mashed to form a puree. The puree is put into a beaker and is added with water in a ratio of 1:10. The raw material is then heated until the volume of the solution becomes half of the first. To get a more concentrated dye solution, heating can continue until the volume of the solution becomes one-third of the original. At the final stage of the preparation of natural dyes, filtering of the dye solution is used using filter gauze in order to separate the dye solution with residue. The filtered dye extract is then cooled and ready for use.

E. *Dyeing Procedure*

Fabric dyeing using natural dyes from the material of mango and turmeric leaves follows the procedure as follows. A total of 210 mL extracts of natural coloring were put into the dying place and then inserted into a 25x25 cm (7 gram) cloth that had passed the mordanting stage. Coloring is done for 50 minutes (10x immersion x @ 5 minutes). At the next stage, the fabric sample is ready to be fixed.

F. *Fixation Procedure*

In the fixation process, the fabric goes down to 210 mL of fixer solution: (a) Iron (II) sulfate ($FeSO_4.7H_2O$) (tunjung), (b) aluminum sulfate (alum) and (c) and calcium oxide (CaO). Fabric samples are soaked in a fixer solution for 10 minutes. Fabric samples are then rinsed, washed and dried.

G. *Analysis*

To find out the color quality resulting from the natural dyeing procedure using mango and turmeric leaf material, a number of analyzes were carried out which included: (a) Shimadzu Diffuse Reflection Ultra Violet (DRUV) UV-2401-PC Spectrophotometer using color intensity instrument to determine the strength the color of the hybrid produced; and (b) staining scale analysis carried out to determine hybrid color fastness resulting from natural coloring using mango and turmeric leaf material.

III. RESULT AND DISCUSSION

A. *Natural Hybrid Dyeing with mango and tumeric leaf material*

Hybrid dyeing is a coloring method by utilizing a combination of dyes or pigments to obtain a variety of color shades and high strength and colorfastness. Hybrid dyeing can also be one solution to the lack of abundance of one of the natural coloring material sources, given the combination of dyes that can produce the same type of chromophore and auxochrome will produce the same color. Textile dyeing results using more than one coloring material are expected to be able to be an alternative natural dyeing source material that is able to replace the role of seasonal material sources, through the appearance of the same color with strength and colorfastness that is not less strong. Thus, one of the weaknesses that have been triggering the assumption that the use of natural dyes in textile production is less practical can be overcome.

Before staining using natural dyes, fabric fibers will be prepared in advance through a number of stages, such as washing using TRO and mordanting using alum and soda ash. Fabric fiber preparation using TRO compounds aims to remove most or all of the contaminants found in the fabric fibers that will be colored. This treatment has an important

role in removing various compounds, both compounds with the same type of charge as fiber and contaminant compounds that have different charges with fiber. Considering that fabric fibers are dominated by negatively charged hydroxide functional compounds, the presence of contaminant compounds with a positive charge will trigger interactions between the two, thus potentially blocking interactions that may occur between coloring compounds, especially those with a positively charged functional group. In line with this, although in the compound structure is dominated by the same functional group (negative) with fabric fibers, the deposition of a large number of these compounds also has the potential to prevent possible interactions between negative functional groups of fabric fibers with positive groups of natural coloring compounds. In Fig. 1 there is an illustration of interactions that can occur between functional groups of fabric fibers with specific contaminants and natural dyes.

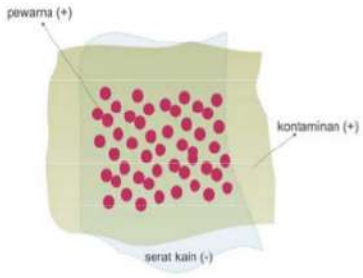

Fig. 1. Illustration of interactions that can occur between fabric fibers, contaminants and specific dyes

After ensuring the minimum contaminants contained in the fabric fibers, to optimize the interactions that occur between the functional groups of fabric fibers and coloring compounds, especially those that are negatively charged, the preparation of fabric fibers is followed by mordanting, which is the procedure of preparing fabric fibers through soaking fabric fibers in solution specific mordant. Not all chemical compounds can act as mordant compounds. Most of the mordanting functions are presented by complex metal compounds with more than one valence, such as Al2 (SO4) 3 or better known as alum. The mordant compound acts as an intermediate compound that allows interaction between negatively charged fabric fibers and natural coloring compounds which are also dominated by the same charge. One of the positive charges of the mordant compound will interact with the negative charge of the fabric fiber and the remaining positive charge will interact with the negative group of the coloring compound. The greater the positive valence possessed by the mordant compound, the higher the quantity of dye bound, resulting in higher strength and color fastness. In Figure 2 there is an illustration of the interaction that occurs between the fabric fibers and the mordanting agent.

In figure 3, it appears the dye molecular structure of *mangiferin* is a green colorant contained in mango leaf extract. As seen in the molecular structure, *mangiferin* compounds are dominated by negatively charged hydroxide functional groups. Coloring compounds with negative charges such as *mangiferin* will not produce color when directly interacted with fabric fibers which are not through the previous mordanting process. However, in some cases, staining has been reported on the appearance of colors on fabric fibers that do not go through the mordanting stage. In such cases, it is predicted that a large number of coloring compounds have interacted with contaminants that are positively charged. This kind of contaminant compound is capable of carrying out the role as is the mordanting agent. However, it should be noted that in this case staining generally has a lower color strength with low fastness. This condition is triggered by the minimum interaction that occurs between fabric fibers and dyes due to the absence of high valence in the positive charge of the contaminant compound compared to most mordant compounds.

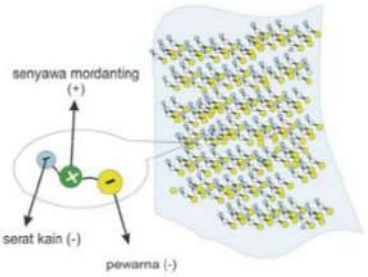

Fig.2. Illustration of interactions that can occur between fabric fibers with mordant compounds

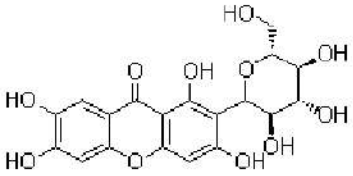

Fig.3. The molecular structure of *magiferin*

In line with the molecular structure of *mangiferin* in mango leaf extract, turmeric extract also contains a natural coloring compound, namely *curcumin*, a bright yellow dye producer dominated by negatively charged functional groups, one of which is a hydroxide group. However, slightly different from *mangiferin,* in the turmeric extract there is also a complementary compound which also strengthens the appearance of typical yellow shades of turmeric, namely *demethoxycurcumin* (Fig. 5) and *bisdemethoxycurcumin* (Fig. 6).

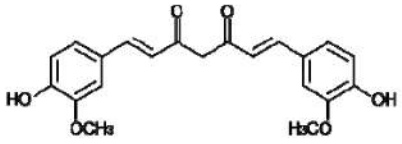

Fig. 4. The molecular structure of *curcumin*

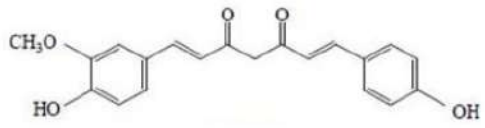

Fig. 5 .The molecular structure of *demethoxycurcumin*

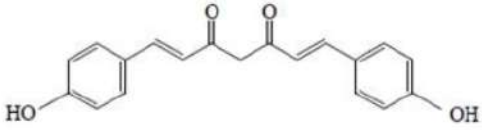

Fig. 6. The molecular structure of *bisdemethoxycurcumin*

In addition to the washing stages using TRO and mordanting using alum, the fixation stage uses three types of fixer compounds, is tunjung, alum and lime which are an integral part of the natural dyeing process. The fixation stage is done as a final stage, where the fixer compound will act as a color lock. The color locking mechanism that is carried out at the fixation stage is activated by minimizing the reactivity of active functional groups in coloring compounds through interactions that occur with various types of fixer compounds. However, in its development, almost most textile businesses do not understand the procedure for selecting fixer compounds, especially if they do not want the changes in color shades that appear. Tunjung, tawas and limestone are three types of fixer compounds that are widely used by textile businesses with natural dyes throughout Indonesia. To get color shades that are similar to the original extract, the fixer compound used must be ensured that it does not contain different chromophore and auxochrome groups with coloring extracts. In the coloring of fabric fibers using *mangiferin* dye which ends with a fixation process using tunjung, alum and lime each have resulted in the emergence of different shades of color with the dye extract, is blackish green, greenish yellow and yellow. While coloring with *curcumin*, each has produced the appearance of yellowish brown, yellow and cream shades from fixation using tunjung, tawas and lime. In table 1, there are a number of color shades resulting from staining using *mangiferin, curcumin*, and a hybrid of both.

TABLE 1. THE COLORATION OF THE DYEING RESULTS USING *MANGIFERIN* AND *CURCUMIN*

Color Composition	Fixer type		
	Tunjung	Alum	Lime
100% DM			
75%DM-25%K			
50%DM-50%K			
25%DM-75%K			
100%K			

B. *Analysis*

In table 2, the color intensity produced by staining using mangiferin, curcumin, and hybrids of the two coloring compounds appear. The results of the color intensity analysis of each color composition showed that the coloring using *mangiferin* produced the best color intensity, amounting to 97.06%, which resulted from fixation using a lime fixer, while in staining using *curcumin*, the highest intensity resulted from fixation using tunjung fixer (89 , 73%). Meanwhile, overall, hybrid coloring results 75% DM / 25% K; 50% DM / 50% K; and 25% DM / 75% K which ends with fixation using alum fixer produces the highest color intensity, each of which is 97.88%; 76.53%; and 87.63%. These results indicate that hybrid staining with a comparable composition of *mangiferin* and *curcumin* is unable to produce color strength comparable to other compositions. This condition is predicted to be influenced by the high level of competition among *mangiferin* and *curcumin* compounds which actually reduce the strength of the color produced.

TABLE 2. COLOR INTENSITY RESULTING FROM STAINING USING *MANGIFERIN* AND *CURCUMIN*

No.	Material	Composition (%v/v)	Color Intensity
1	DM1	100	88,96
2	DM2	100	79,89
3	DM3	100	97,06
4	K/DM1	25/75	74,56
5	K/DM2	25/75	97,88
6	K/DM3	25/75	87,79
7	K/DM1	50/50	57,36
8	K/DM2	50/50	76,53

9	K/DM3	50/50	72,38
10	K/DM1	75/25	35,60
11	K/DM2	75/25	87,63
12	K/DM3	75/25	37,89
13	K1	100	89,73
14	K2	100	77,61
15	K3	100	81,55
Note : 1. Tunjung, 2. Alum, 3. Lime			

TABLE 3. COLORFASTNESS RESULTING FROM STAINING USING *MANGIFERIN* AND *CURCUMIN* AGAINST WET WASHING

No.	Material	Komposisi (%v/v)	*Staining Scale*
1	DM1	100	3 (enough)
2	DM2	100	4 (good)
3	DM3	100	3-4 (worth)
4	K/DM1	25/75	3-4 (worth)
5	K/DM2	25/75	2 (less)
6	K/DM3	25/75	2-3 (less)
7	K/DM1	50/50	4 (good)
8	K/DM2	50/50	3 (less)
9	K/DM3	50/50	2 (less)
10	K/DM1	75/25	3-4 (worth)
11	K/DM2	75/25	2-3 (less)
12	K/DM3	75/25	2-3 (less)
13	K1	100	3-4 (worth)
14	K2	100	3-4 (worth)
15	K3	100	2-3 (less)
Note : 1. Tunjung 2. Alum 3. Lime			

Table 3 shows the color fastness scale of each dye composition and fixer type. The results of staining scale analysis showed the effectiveness and efficiency of tunjung as a fixer compound for staining using *mangiferin* and *curcumin*. This condition is evident from the higher staining scale resulting from fixation using tunjung compared to the other two types of fixers, namely alum and lime. However, it is important to note that the high complexity and quantity of chromophore and auxochrome groups found in fixer tunjung compounds have caused significant color changes produced compared to the original extract dyes. Therefore, to get a color feel that more closely resembles the original extract color, alum fixer is the best alternative that can be used.

IV. CONCLUSION

In this study natural dyeing procedures have been developed using hybrid dyes that combine *mangiferin* and *curcumin* which ends with fixation using tunjung fixers, alum and lime. In general, the results of the analysis showed the appearance of color shades which were yellow, brown and green gradations with color intensity of 35.60% - 97.88% and colorfastness of wet washing in the range of staining scale 2 (less) - 4 (good). In addition, the results of the analysis show the tendency for the darkest (blackish) color to emerge from the use of a raised fixer, and the color shades closest to the color of the extract from the use of alum fixers.

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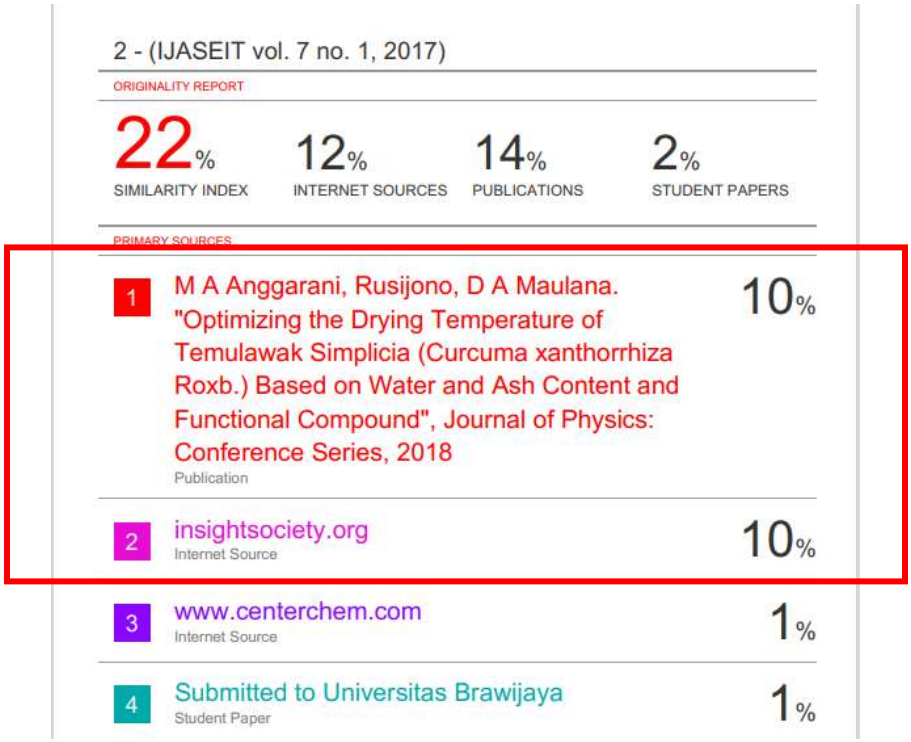

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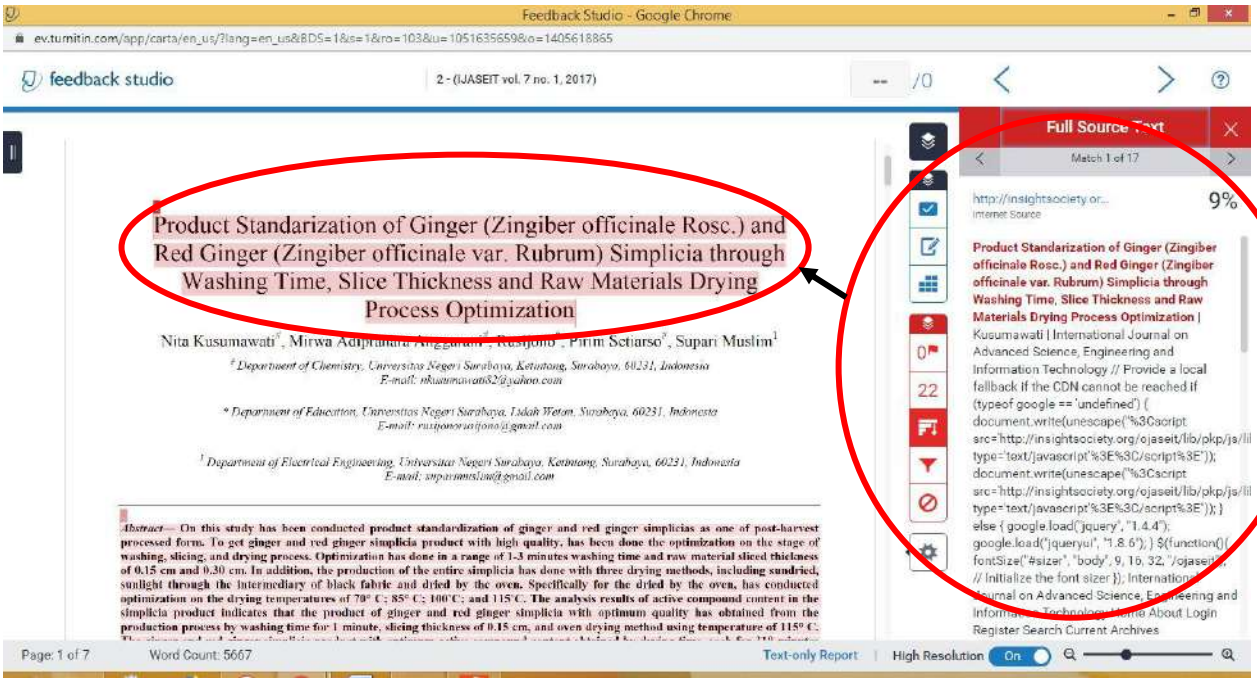

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Product Standarization of Ginger (Zingiber officinale Rosc.) and Red Ginger (Zingiber officinale var. Rubrum) Simplicia through Washing Time, Slice Thickness and Raw Materials Drying Process Optimization

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Abstract*— On this study has been conducted product standardization of ginger and red ginger simplicias as one of post-harvest processed form. To get ginger and red ginger simplicia product with high quality, has been done the optimization on the stage of washing, slicing, and drying process. Optimization has done in a range of 1-3 minutes washing time and raw material sliced thickness of 0.15 cm and 0.30 cm. In addition, the production of the entire simplicia has done with three drying methods, including sundried, sunlight through the intermediary of black fabric and dried by the oven. Specifically for the dried by the oven, has conducted optimization on the drying temperatures of 70° C; 85° C; 100°C; and 115°C. The analysis results of active compound content in the simplicia product indicates that the product of ginger and red ginger simplicia with optimum quality has obtained from the production process by washing time for 1 minute, slicing thickness of 0.15 cm, and oven drying method using temperature of 115° C. The ginger and red ginger simplicia product with optimum active compound content obtained by drying time, each for 310 minutes and 280 minutes. The ginger simplicia products contain essential oils, oleoresins, zingiberene in essential oils, gingerol and shogaol in the oleoresins, each by 1.7%; 0.86%; 56.90%; 42.50%; and 21.54%. Meanwhile, the red ginger simplicia products contain essential oils, oleoresins, zingiberene in essential oils, gingerol and shogaol in the oleoresins, each by 2.11%; 0.88%; 78.60%; 80.06%; and 8.02%. Thus, it can be said that, overall, red ginger simplicia product contains an active compound higher than the ginger simplicia product.

***Keywords*— simplicia; ginger; red ginger; washing; slicing; drying**

I. INTRODUCTION

Material handling after harvesting time needs to be considered because it affects the quality of the processed products. The quality and safety of biofarm product, which one of which in the form of simplicia, determined by the quality of raw materials, post-harvest handling, and processing techniques. The post-harvest handling techniques consist of the stages of sorting, washing, drying/draining [1]-[3], sorting/grading, packaging, labeling, and storage, either before sold in fresh form or after further processing. However, until now there is no researcher who reported in detail about the standard operating procedures (SOP) for the manufacture of processed products after harvest, particularly in the form of simplicia, which are able to maintain active compound content in the raw materials.

Ginger (Zingiberofficinale (L.) Rosc) has a variety of benefits, both as herbs, essential oils, flavor concentrates, as well as the drug [4]. Traditionally, ginger is used to treat rheumatic diseases, asthma, stroke, tooth pain, diabetes, muscle pain, throat, cramps, hypertension, nausea, fever and infections [5]-[8]. Based on the shape, color, and size of the rhizome, there are three kinds of ginger known, namely the large white ginger/*gajah* ginger (Z. officinale var. Roscoe), small white ginger or *emprit* ginger (Z. officinale var. Amarum) and red ginger or *sunti* ginger (Z. officinale var.

Rubrum) [9]. In general, the three types of the ginger rhizome contained active compound content of which is divided into two group of compounds, namely: (1) the volatile compounds, which largely consist of sesquiterpene derivatives (> 50%) and monoterpenes. Two of these components provide the scent of ginger, with a relatively constant level, ie 1-3%. The sesquiterpene derivatives contained in the ginger rhizome include zingiberene (20-30%), ar-curcumene (6-19%), β-sesquiphelandrene (7-12%) and β-bisabolane (2-12%). Meanwhile, derivatives of monoterpenes contained α-pinene, bornyl acetate, borneol, camphene, ρ-cymene, cineol, citral, cumene, β-elemene, farnesene, β-phelandrene, geraniol, limonene, linalol, myrcene, β-pinene and sabinene; (2) non-volatile compounds, namely oleoresin (4.0 to 7.5%), with the content of gingerol, shogaol, gingediol, gingediasetat, gingerdion, and gingerenon. Those component has given a spicy flavor on the ginger [10].

Reference [8] shows that in general, the active compound in red ginger has a similarity with the active compounds contained in ginger. However, based on the reference [11], red ginger has essential oil content of (3.9%) and alcohol-soluble extract (9.93%) higher than *emprit* ginger which has a content of essential oil (3.5%) and alcohol soluble extracts of (7.29%), and *gajah* ginger containing essential oil (2.5%) and alcohol-soluble extract (5.81%).

Each element in ginger have their benefits and characteristics, such as: (1) anti-oxidants [13], including zingiberene essential oils, as well as gingerol and shogaol in oleoresin [14]; (2) inhibiting the growth of pathogenic bacteria by the essential oil [9], [15]-[16]; (3) antihepatotoxic against CCl_4 by gingerol and shogaol in oleoresin [17]; (4) reduced cardiotonic activity by gingerol in oleoresin [18]; (5) suppress intestinal contractions and antitussive properties by gingerol and shogaol in oleoresin [19]; and (6) the phitopharm deposit.

Based on its usefulness, on this research, the content of active compound in ginger include (1) essential oils; (2) oleoresin; (3) zingiberene essential oils; (4) gingerol, and (5) shogaol in oleoresin, become the nutritional content parameters of simplicia ginger products. Given the benefit magnitude of each active compounds contained in ginger, it is necessary to do a research about the effect of ginger simplicia producing stage process, which includes the washing process, slicing, and drying, to the content of active compounds in ginger simplicia.

II. Materials and Methods

A. Material

The main material used in this research is the *gajah* ginger (Z. officinalevar. Roscoe) and red ginger (Z. officinale var. Rubrum) rhizome. Meanwhile, to get the optimum quality of ginger and red ginger simplicia product, utilized a number of tools, including: washer (homemade) used in optimization stages of ginger raw material washing time by NESCO Lab MS-H280 Pro Magnetic Stirrer as a motor producing whirlpool which is used in order to remove contaminants attached to the raw materials that collected on the filter, as shown in Fig. 1; Bosch MUZ4DS3 slicer machine (Fig. 2) with slicing capacity of 60 g/min and the slicing thickness of 0.15 cm and 0.30 cm; and J-LabTech LDO-030E (Daihan LabTech Ltd. Co) drying machine which used at the optimization stage of the sliced thickness of ginger and red ginger with a range temperature of 50 °C - 250 °C and a drying time up to 60 minutes.

Fig. 1 Washing machine of ginger and red ginger

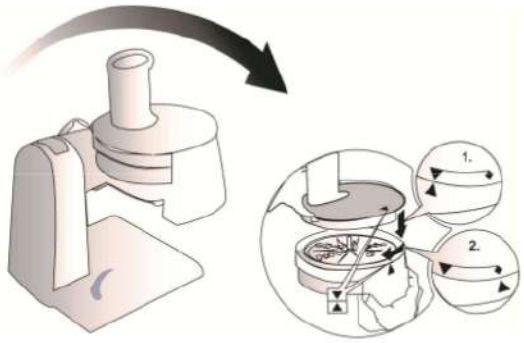

Fig. 2 Slicer machine of ginger and red ginger raw material

B. Manufacturing of Ginger and Red Ginger Simplicia

Generally, manufacturing process of ginger and red ginger simplicia at this stage used the same steps, covering the steps of washing, slicing, and drying of the material. In early stages of simplicia product manufacture, done the washing process of fresh raw materials for 1 minute. After being washed, raw materials dried in the sunlight for 24 hours. Raw materials that have been dried, then going through stages of slicing. The slicing process of raw materials is done with a thickness of 0.15 mm and 0.3 mm. Raw materials that have been sliced has ready for the drying process.

The drying process at this stage includes three methods, which was direct sunlight drying, sunlight drying with the utilize of black fabric as sample cover, and the drying process using an oven. Specifically for drying method with oven, the temperature used was 70 ° C; 85 ° C; 100 °C; and 115 °C. In addition, during the drying process has not performed samples reversing, and drying process is stopped as soon as constant mass is obtained.

C. *Water Content Testing of Ginger and Red Ginger Simplicia Product*

The testing procedure performed using the gravimetric method.

D. *Active Compound Content Testing of Ginger and Red Ginger Simplicia Product:*

The testing procedure of active compound content of simplicia products that performed on the stages includes essential oil content testing procedure using stahl distillation methods. Meanwhile, the testing procedure of oleoresins content, zingiberene content in essential oils, gingerol, and shogaol content in oleoresin, all four of them performed using Pharmaspec UV-1700 UV Visible Spectrophotometer.

III. Result and Discussion

A. *The Effect of Washing Time to Simplicia's Quality and Nutritional Content*

Appropriate washing time will make the ginger and red ginger simplicia has unsignificantly different on nutritional quality compared to the fresh ginger products. Thereby, the production of ginger simplicia will not only have longer shelf life but also has high nutritional content. At this stage, the active compound which acts as nutritional quality parameters of ginger and red ginger simplicia was (1) essential oils; (2) oleoresin; (3) zingiberene in essential oils; also (4) gingerol, and (5) shogaol in oleoresin. Fig. 1 shows the molecular structure of zingiberene, gingerol, and shogaol compounds contained in ginger.

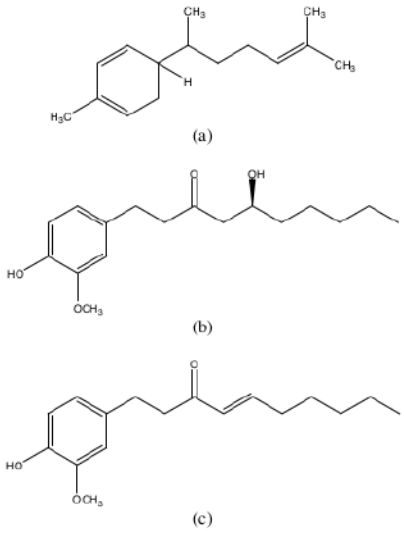

Fig. 1 A molecular structure of: (a) zingiberene; (b) gingerol; and (c) shogaol

Table 1 shows the data of drying time required and water content of dried simplicia generated from the washing stages application of raw materials for 1-3 minutes. Based on data in Table 1, note that duration of washing time applied in the production stage of ginger and red ginger simplisia greatly affect the drying time and water content on the simplicia products.

In the production process of ginger and red ginger material with washing time of 1 minute has produced simplicia with a drying time of 400 minutes and 380 minutes respectively. While washing time of ginger and red ginger material for 2 minutes and 3 minutes, each has produced ginger and red ginger simplicia with a drying time of 420 minutes and 560 minutes for the ginger simplicia product; and 390 minutes and 550 minutes for red ginger simplicia. Therefore, the ginger simplicia product has a water content higher than the red ginger simplicia product.

TABLE I
DRYING TIME OF GINGER AND RED GINGER SIMPLICIA RESULTED FROM WASHING TIME OPTIMIZATION

No	Simplicia's Type	Drying Time Resulted from Washing Time (minute)			Water Content (%) Resulted from Washing Time (minute)		
		1	2	3	1	2	3
1	Jahe	400	420	560	11.83	13.17	21.33
2	Jahe merah	380	390	550	12.83	21.17	23.00

Note :
1. Sliced thickness of 0,15 cm
2. Drying process using oven 100 °C
3. Drying chamber : 15cmx6.5cmx4cm
4. Simplicia's mass of 60 gram

Furthermore, in order to determine the optimum washing time in the production process of ginger and red ginger simplicia, has been analyzed the active compound content of simplicias products. Based on data in Table 2, was detected decreased levels of active compounds which include essential oils, oleoresins, zingiberene in essential oils, gingerol and shogaol in oleoresin, along with the duration of raw materials time washing.

TABLE II
ACTIVE COMPOUND CONTENT OF GINGER AND RED GINGER RED GINGER SIMPLICIAS RESULTED FROM WASHING TIME OPTIMIZATION

No	Simplicias Type & Washing Time (minute)	Active Compound Content (%)				
		Essential Oil	Oleoresins	Zingiberene	Gingerol	Shogaol
1	Jahe					
	1 minute	3.56	1.62	85.12	67.82	34.05
	2 minute	3.41	1.40	83.90	65.48	32.90
	3 minute	2.31	0.90	70.27	50.70	26.08
2	Jahe merah					
	1 minute	2.80	1.18	81.96	83.11	8.41
	2 minute	2.38	1.02	81.54	80.44	8.32
	3 minute	2.24	0.90	80.42	78.95	8.05

Zingiberene has the lowest decrease level of content in line with the duration of washing time compared to gingerol and shogaol. This is mainly due to the presence of carbon and hydrogen atoms in the molecular structure of zingiberene which has created hydrophobic characteristics so that it can inhibit the active compound's solubility in polar solvents like water. Meanwhile, on the molecular structure of gingerol and shogaol, there are some polar functional groups, such as -OH, which has been increasing the polarity of gingerol and shogaol compounds. The improved polarity of the compound has increased the solubility of these two types of active compounds in polar solvents like water. However, compared with shogaol, gingerol has a high solubility in the aqueous solvent, which is caused by the more presence of polar functional groups in their molecular structure.

The analysis results of active compounds in ginger simplicia showed significant decreased levels when ginger raw material has been washed for 3 minutes, which was observed the presence of (a) reduction in essential oil content, respectively of 4.21% and 35.11%; (b) decreased levels of oleoresin, respectively 13.58% and 44.44%; (c) decreased levels of zingiberene content in essential oil, respectively 1.43% and 17.45%; (d) decreased levels of gingerol content in oleoresin, respectively 3.45% and 25.24%; and (e) decreased levels of shogaol contenct in oleoresin, respectively 3.38% and 23.41% when the washing time is increased from 1 minute to 2 minutes and 3 minutes.

Meanwhile, the analysis results of the red ginger's simplicia active compounds showed a significant decline when the material has washed for 3 minutes, had observed the presence of (a) reduction in essential oil content, respectively 15% and 20%; (b) decreased levels of oleoresin content, respectively 13.56% and 23.73%; (c) decreased levels of zingiberene content in essential oil, respectively 0.51% and 1.88%; (d) decreased levels of gingerol content in oleoresin, respectively of 3.21% and 5.01%; and (e) decreased levels of shogaol content in oleoresin, respectively 1.07% and 4.28% when the material washing time has increased from 1 minute to 2 minutes and 3 minutes. Thereby, compared with ginger simplicia products, red ginger simplicia products experienced a lower decline when the washing process has carried out for 3 minutes.

B. The Effect of Slicing Thickness and Drying Method to Simplicia's Quality and Nutritional Content

The data in Table 3 indicate that the production process of ginger simplicia with a slice thickness of 0.3 cm, required drying time 25.30%; 8.21%; and 29.73% longer than the simplicias product manufactured with a slice thickness of 0.15 cm which each dried by direct sunlight, the sunlight with intermediaries of black fabric, and the oven on the temperature of 100 °C. Longer drying times have produced ginger simplicia with a water content of 32.35% - 59.15%. The water content of ginger simplicia with a thickness of 0.30 cm has equal to 56.34%; 59.15%; and 32.35% higher than the ginger simplicia product with a slice thickness of 0.15 cm with each drying method has direct sunlight, sunshine with intermediaries of black fabric and use an oven with temperature of 100 ° C.

Meanwhile, in the production process of red ginger simplicia with a thickness of 0.3 cm required drying time which 22.91%; 10.29%; and 5.41% longer than drying process of red ginger with a slice thickness of 0.15 cm and each has gone through the drying process using direct sunlight, sunshine with intermediaries black fabric, and the oven with temperature of 100 °C.

Longer drying times have produced red ginger simplicia with water content, respectively by 18.33%; 18.50%; and 18.00% for red ginger simplicia product which resulted from the drying method using direct sunlight, sunshine with intermediaries black fabric, and the oven with a temperature of 100 °C. The water content of red ginger simplicia with a thickness of 0.30 cm respectively by 23.60%; 14.43%; and 40.26% higher than the red ginger simplicia products with a slice thickness of 0.15 cm which each dried using direct sunlight, sunshine with intermediaries black fabric, and the oven with a temperature of 100 °C.

TABLE III
DRYING TIME OF GINGER AND RED GINGER SIMPLICIAS PRODUCT RESULTED FROM SLICES THICKNESS AND DRYING METHOD OPTIMIZATION

No	Simplisia's Type	Drying Time (minute)			Drying Time (minute)		
		Slice Thickness 0,15 cm			Slice Thickness 0,30 cm		
		Direct Sunlight	Sunlight, Black Fabric	Oven, T100 °C	Direct Sunlight	Sunlight, Black Fabric	Oven, T100 °C
1	Ginger	838	1,060	370	1,050	1,147	480
2	Red Ginger	838	1,040	370	1,030	1,147	390

TABLE IV
WATER CONTENT OF GINGER AND RED GINGER SIMPLICIA RESULTED FROM SLICES THICKNESS AND DRYING METHOD OPTIMIZATION

No	Simplisia's Type	Water Content (%)			Water Content (%)		
		Slice Thickness 0,15 cm			Slice Thickness 0,30 cm		
		Direct Sunlight	Sunlight, Black Fabric	Oven, T100 °C	Direct Sunlight	Sunlight, Black Fabric	Oven, T100 °C
1	Ginger	11.8	11.8	11.3	18.5	18.8	15.0
2	Red Ginger	14.8	16.2	12.8	18.3	18.5	18.0

The analysis results of active compound content in ginger and red ginger simplicia produced with variations in slice thickness and drying methods show that: (a) either at a slice thickness of 0.15 cm or 0.30 cm, a drying method using sunlight with a black fabric as intermediary has produced ginger and red ginger simplicia with the highest levels of active compound compared with those produced by drying using direct sunlight and oven with a temperature of 100 °C; (b) ginger and red ginger simplicia with a slice thickness of

0.30 cm had higher levels of active compounds compared to products with a slice thickness of 0.15 cm; and (c) the products of red ginger simplicia have higher levels of active compound compared with ginger simplicia products resulting from the production process with the same variety methods of drying and slicing thickness. Table 5 and 6 show the active compound content of simplicia products produced by the variation in the slice thickness and drying metho[6].

Water is a major component in foods that affect appearance, texture, and taste of ingredients. The water content in food ingredients also determines the acceptability of a food ingredient [20]. The sensitivity of a commodity against water losses due to evaporation depends on the deficit of atmospheric pressure in the vicinity as well as the structure of the surface layer of the commodity in question.

TABLE V
ACTIVE COMPOUND CONTENT OF GINGER SIMPLICIAS RESULTED FROM SLICES THICKNESS AND DRYING METHOD OPTIMIZATION

	Simplicias Type & Drying Method	Active Compound Content (%)				
		Essential Oil	Oleoresin	Zingiberene in Essential Oil	Gingerol in Oleoresin	Shogaol in Oleoresin
Direct Sunlight	0.15 cm	2.68	1.05	74.55	56.10	27.42
	0.30 cm	2.96	1.24	78.64	58.40	28.90
Sunlight, Black Fabric	0.15 cm	3.01	1.18	78.87	59.05	28.90
	0.30 cm	3.14	1.32	81.22	62.80	31.60
Oven, 100 °C	0.15 cm	1.80	0.98	58.52	42.70	21.82
	0.30 cm	1.88	1.03	58.66	43.10	22.35

TABLE VI
ACTIVE COMPOUND CONTENT OF RED GINGER SIMPLICIAS RESULTED FROM SLICES THICKNESS AND DRYING METHOD OPTIMIZATION

	Simplicias Type & Drying Method	Active Compound Content (%)				
		Essential Oil	Oleoresin	Zingiberene in Essential Oil	Gingerol in Oleoresin	Shogaol in Oleoresin
Direct Sunlight	0.15 cm	2.89	1.05	81.96	84.75	8.95
	0.30 cm	3.02	1.18	83.61	84.90	9.04
Sunlight, Black Fabric	0.15 cm	3.35	1.42	84.08	85.01	9.22
	0.30 cm	3.48	1.54	86.05	87.62	10.32
Oven, 100 °C	0.15 cm	2.26	0.91	78.98	80.26	8.06
	0.30 cm	2.62	0.96	83.43	80.52	8.86

Drying is a very important process in the manufacture of simplicia.The purpose of drying in the process of making simplicia is to lower the water content, so it is not easily covered with mold and bacteria, eliminates the activity of enzymes that can decipher the active compound content, facilitate further processing, so it can be more compact, durable and easily stored. The drying process, in addition, to extend the shelf life also determines the quality of the simplicia. Things to consider during the drying process is drying temperature, air humidity, air flow, the drying time and surface area materials. During the material drying process, these factors must be considered in order to obtain dried simplicia that is not easily damaged during storage.

Incorrect drying method can lead to a face hardening, which is a state where the outer material is dried while the inside is still wet. It can be caused by material sliced which too thick, the drying temperature which too high or by any other circumstances that led to the water evaporation of surface material are much faster than the diffusion of water from the inside to the surface, so the surface of the material becomes hard and inhibit to subsequent drying.

As shown by the active compound content of ginger and red ginger simplicia products in Table 5 and Table 6, it is known that the product of ginger and red ginger simplicia which dried using direct sunlight has lower active compound content than the one which dried using sunlight with intermediaries black fabric. This can happen because the drying process using a higher temperature on products that dried in direct sunlight. Drying method which has involved the use of a higher temperature in the manufacturing process of ginger and red ginger simplicia has resulted in the evaporation of volatile active compounds in the material.

Besides the drying method, slice thickness is also one of the variables that determine the quality of ginger and red ginger simplicias products. Based on data from the active compound content which shown in Table 7, it is known that the simplicia products with a slice thickness of 0.30 cm contain the higher active compound than the simplicia product manufactured with a slice thickness of 0.15 cm. This is caused by the lower slicing thickness (0.15 cm) which has resulted in higher damage to the matrix of the ginger material, making it easier to evaporate volatile active compounds in gingers simplicia along with the increasing of drying temperatures used. Likewise, if it thicker (0.30 cm), matrix damage that occurs in the raw material of gingers lower. The lower damage level matrix of ginger's raw materials will prevent the evaporation of volatile active compounds in the materials.

C. The Effect of Drying Temperature on Simplicia's Quality and Nutritional Content

Based on data in Table 7, it is known that the drying process by using an oven with a temperature of 70 °C has produced ginger and red ginger simplicia with a water content of respectively 20.83% and 16.33% which obtained through the stages of drying using the oven, each for 1,610 minutes and 1,820 minutes.

The drying process with a higher temperature generates products with lower water content and shorter drying time. The minimum drying process capable of saving drying time by up to 80.75% in the production of ginger simplicia, and

84.52% in the production of red ginger simplicia, may occur when the drying has done at a temperature of 115 °C, respectively for 310 minutes and 280 minutes. The product of ginger and red ginger simplicia manufactured using a drying temperature of 115 °C has a water content respectively by 11.17% and 12.50%. Water content data for each ginger and red ginger simplicia product produced using a temperature of 70 °C; 85 °C; 100 °C; and 115 °C appear in Table 8.

TABLE VII
DRYING TIME OF GINGER AND RED GINGER SIMPLICIA PRODUCT RESULTED FROM DRYING TEMPERATURE OPTIMIZATION

No	Simplicias Type	Drying Time (minute)			
		T 70 °C	T 85 °C	T 100 °C	T 115 °C
1	Ginger	1,610	1,540	370	310
2	Red Ginger	1,820	1,540	370	280

TABLE VIII
WATER CONTENT OF GINGER AND RED GINGER SIMPLICIA RESULTED FROM DRYING TEMPERATURE OPTIMIZATION

No	Simplicia Type	Drying Time (minute)			
		T 70 °C	T 85 °C	T 100 °C	T 115 °C
1	Ginger	20.83	12.83	11.33	11.17
2	Red Ginger	16.33	15.67	12.83	12.50

On the previous data has been shown that the lowest water content of ginger and red ginger simplicia can be obtained when dried at a temperature of 115 °C. However, to obtain an optimum drying temperature in the production process of ginger and red ginger simplicia, at this stage also conducted an analysis of active compounds content in simplicia products which dried using an oven on temperature 70 °C; 85 °C; 100 °C; and 115 °C. Based on data from the active compounds content of ginger and red ginger simplicia products which shown in Table 9, note that: (a) increasing the drying temperature from 85 °C to 100 °C caused a significant decrease in the active compounds content of ginger simplicia, which is 45.12% on the content of essential oils; 27.94% on the content of oleoresin; 28.99% on the content of zingiberene in essential oils; 33.33% on the content of gingerol in oleoresin; and 32.74% on shogaol content in oleoresin; (b) under the same conditions, the increase in drying temperature from 85 °C to 100 °C has not cause a significant reduction on the content of active compounds of red ginger simplicia, which is 35.43% on essential oil content; 18.75% on the content of oleoresin; 10.07% on the content of zingiberene in essential oils; 9.19% on the the content of gingerol in oleoresin; and 27.06% on the content of the oleoresin's shogaol. Thereby, it can be said that the red ginger material has higher stability towards drying temperature increased.

Furthermore, in order to know the manufacturing procedure of ginger and red ginger simplicia with an optimum quality, has been done an analysis using the ginger simplicia quality standard issued by Materia Med Indonesia (Table 10). Based on an optimization result which done in the production process of ginger and red ginger simplicia, includes the washing, slicing and drying stage, as well as the quality standard of gingers simplicia set by Materia Med Indonesia, it is known that the ginger simplicia products with optimum quality obtained from the production stage with (1) washing time for 1 minute; (2) slice thickness of 0.15 cm; (3) using the oven drying method; and (4) using a drying temperature of 115 °C. An optimum ginger simplicia product has a water content of 11.17% with a 1.7% essential oil content; 0.86% oleoresin; 56.90% zingiberene content in essential oils; 42.50% gingerol content in oleoresin; and 21.54% shogaol content in oleoresin. Meanwhile, red ginger simplicia products with an optimum quality have a water content of 12.50% and 2.11% essential oil content; 0.88% oleoresin; 78.60% zingiberene content in essential oils; 80.06% gingerol content in oleoresin; and 8.02% shogaol content in oleoresin.

TABLE IX
ACTIVE COMPOUND CONTENT OF GINGER AND RED GINGER SIMPLICIA RESULTED FROM DRYING TEMPERATURE OPTIMIZATION

No	Simplicias Type & Drying Temperature (°C)	Active Compund Content (%)				
		Essential Oil	Oleoresin	Zingiberene in Essential Oil	Gingerol in Oleoresin	Shogaol in Oleoresin
1	Ginger					
	70 °C	3.52	1.53	84.05	66.32	33.81
	85 °C	3.28	1.36	82.41	64.05	32.44
	100 °C	1.80	0.98	58.52	42.70	21.82
	115 °C	1.7	0.86	56.90	42.50	21.54
2	Red Ginger					
	70 °C	3.62	1.68	87.82	88.92	11.07
	85 °C	3.50	1.12	87.82	88.38	11.05
	100 °C	2.26	0.91	78.98	80.26	8.06
	115 °C	2.11	0.88	78.60	80.06	8.02

TABLE X
QUALITY STANDARD OF GINGER SIMPLICIA ISSUED BY MATERIA AND MED INDONESIA [21]

Characteristic	Value
Water content	Max. 12%
Essential oil content	Max. 1.5%
Ash content	Max. 8.0%
Patogenic content	None
Foreign material (impurities)	Max. 2.0%

IV. Conclusions

The results showed that the washing time, the slicing thickness, drying method and drying temperature affect the water content and active compounds of ginger and red ginger simplicia products. These effects include an increase in washing time material which increases the water content of simplicia products with lower levels of active compounds, while the increased of slice thickness increase the water content as well as an active compound of simplicia products. Meanwhile, the optimization result shows that drying method using sunlight with a black fabric as intermediary produced ginger and red ginger simplicia with the highest water content. Lower water levels produced from the drying process with direct sunlight and the lowest water content obtained from products which have dried in the oven. Furthermore, the increase in drying temperatures has produced simplicia with lower water content and reaches the maximum when used drying temperature of 115 ° C.

Ginger and red ginger simplicia product with an optimum quality obtained from the production stage with: (1) the washing time of 1 minute; (2) slice thickness of 0.15 cm; (3) a method of drying in an oven; and (4) using drying temperature of 115 ºC. An optimum ginger simplicia product has a water content of 11.17% with a 1.7% essential oil content; 0.86% oleoresin; 56.90% zingiberene content in essential oils; 42.50% gingerol content in oleoresin; and 21.54% shogaol content in oleoresin. Meanwhile, red ginger simplicia products with an optimum quality have a water content of 12.50% and 2.11% essential oil content; 0.88% oleoresin; 78.60% zingiberene content in essential oils; 80.06% gingerol content in oleoresin; and 8.02% shogaol content in oleoresin.

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Exploration of Natural Dyes by Using a Combination of Caesalpinia sappan and Leucaena leucocephala L. Leaves.

by Nita Kusumawati

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Exploration of Natural Dyes by Using a Combination of Caesalpinia sappan and Leucaena leucocephala L. Leaves.

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***Abstract*—In this research an exploration of the potential use of Caesalpinia sappan and Leucaena leucocephala L. has been carried out as an alternative to environmentally friendly dyeing. To optimize the quality of the shades produced, pre-treatment is done using Turkish Red Oil (TRO) (washing) as well as alum and soda ash (mordanting). Both are applied to minimize the presence of contaminants and create intermediates for reactions between dyes and fabric fibers, while fixation is applied to suppress the reactivity of natural dye compounds to the surrounding environment. Thus, the color fastness can be improved. Natural dyeing uses 75% KKS/25% DL; 50% KKS/50% DL; and 25% KKS/75% DL have produced shades of yellowish, brownish, reddish, purple and black with a color intensity in the range of 73.23%-81.08%; 70.27%-84.32%; and 79.73% -83.14% for fixation with iron(II)sulfate, alum and lime, respectively. Furthermore, the results of the staining scale analysis, dyeing with 75% KKS/25% DL; 50% KKS/50% DL; and 25% KKS/75% DL with fixation using iron(II)sulfate, alum and lime successively produce fastness on a scale of 3 (fair); 3-4 (good enough); and 3-4 (good enough).**

***Keywords*—Textile, natural dyes, *Caesalpiniasappan*, *Leucaena leucocephala*, brazillin, tannin**

I. INTRODUCTION

Three basic ingredients that cannot be released from human life are clothing, food, and shelter. Clothing is one of the basic needs that is experiencing rapid development in seconds every day. Clothing is experiencing rapid development in terms of models and colors used. The dyes used are mostly synthetic dyes. Synthetic dyes have their own charm in the world of textiles because they have several advantages. These advantages include a more affordable price, a stronger and more stable color, a more practical use, and an easily available presence. However, the use of these dyes has a large number of negative impacts on the environment and health [1-3].

Synthetic dyes contain a number of pollutants including heavy metals that are very dangerous for the environment and health. A number of heavy metals that accompany the presence of synthetic dyes in textile wastewater include Cu, Ni, Cr, Hg, and Co. These heavy metal pollutants generally come from the mordanting stage in dyeing. The intensive production of textiles will trigger the entry of large quantities of mutagenic dyes and heavy metals into the aquatic environment and pollute all life in them, including water sources for human life. Some countries such as the Netherlands and Germany have banned the use of this dye [4]. This condition is the background of the need for environmentally friendly alternative dyes, namely natural dyes [5-6]. Natural dyes can be obtained from various components of biological resources, such as root and wood bark, flowers, and leaves of some plants [7-11].

Utilization of a number of natural dyeing sources such as tarum (*Indigofera tinctoria*) leaf, areca nut (*Areca catechu*), turmeric (*Curcuma longa L.*), mangosteen rind (*Garcinia mangostana*), kesumba (*Bixa orellana*), mahogany root (*Morinda citrifolia*), brazilwood bark (*Caesalpinia sappan*), tinggi bark (*Ceriops candolleana Arn*), ketapang (*Terminalia catappa*) leaf, teak leaf (*Tectona grandis*), and lamtoro (*Leucaena leucocephala L.*) leaf[12-14] Color instability has become a problem for practitioners and researchers in textile technology, and standardization of procedures is needed to overcome this problem.

Brazilwood (*Caesalpinia sappan*) is a type of plant that has been known as the main ingredient of herbal medicine [15]. Brazilwood has yellow flowers with yellowish green fruit and oval-shaped. The substances contained in brazilwood include gallic acid, brazilin, delta-α-phellandrene, oscimene, resorcinin, essential oils, and tannins [16]. Meanwhile, brazilwood leaves contain essential oils up to 0.20%, colorless and odorless. The bark and root of the barzilwood produce a deep red color, which is produced by the brazilin compound. Besides having dyeing ability, this compound also has antioxidant and antimicrobial properties. The deep red color will appear when the bark and root of the brazilwood are dissolved in a suitable solvent. Solvents that can be used include water, ethanol and methanol. Apart from brazilwood, lamtoro also has body parts that can be used as natural dyeing agents.

Lamtoro (*Leucaena leucocephala L.*), also known as Chinese petai, is a tree that is generally used as a greening media and to prevent soil erosion. Lamtoro can grow to as high as 5-15 m with 4-8 pairs of double pinnate leaves. Lamtoro tree trunks are brown with flat fruit. Lamtoro contains several substances including alkaloids, saponins, flavonoids, mimosin, leukanin, and tannins. Lamtoro leaves produce a yellowish green color that comes from the tannin compounds in it [17]. This color will appear if the lamtoro leaves are dissolved in polar solvents such as water, ethanol, and methanol

II. MATERIAL AND METHODS

A. Material

A number of main materials used for the preparation and use of natural dyes include the brazilwood bark and lamtoro leaf material, each obtained from UD Sinar Agung and the environment around Universitas Negeri Surabaya. Other ingredients used are: (1) Turkish Red Oil (TRO, 70%) derived from CV. Dunia Kimia (Surabaya, Indonesia), (2) mordanting agent including alum ($Al_2(SO_4)_3$, ≥17%) and soda ash (Na_2CO_3, ≥48%), each obtained from PT. Brataco and CV. Water (Surabaya, Indonesia); (3) fixers, including iron (II) sulfate ($FeSO_4.7H_2O$, d 2.84 g/cm^3) and calcium oxide ($CaCO_3$, ≥90%), each obtained from PT. Nusa Indah Megah and Mitra Water (Surabaya, Indonesia). Especially for alum fixer, the specifications of the material used are the same as those used in the mordanting stage.

B. Mordanting

Mordanting is needed to improve the reaction of dyes with textile fibers, so that a stable color can be produced on the fiber. Mordanting is done through the preparation of 25cmx25cm fabric fibers. To optimize the mordant that can be attached to the fiber, immersing the fabric in 34.2 mL of 2 g/L TRO solution for 6 h. To ensure the fabric fibers are free of residues, the fabric fibers are rinsed three times, each using 34.2 mL of water. The fabric fibers are then dried in the open air and ready for use in order. Mordant solution was prepared by dissolving 34.2 grams of alum and 2.74 grams of soda ash in 0.68 mL of water. Mordant solution is stirred for 5 min using a magnetic stirrer, to ensure homogeneity of the solution. The mordant solution is then heated to 100 °C, and fabric fibers are inserted into it for 1 h while continuing to heat. Immersing in a mordant solution is continued for 24 h. To get a mordant residue-free fabric, rinse three times, each using 34.2 mL of water and then dried. The fiber fiber is then ironed to get a uniform fiber orientation and is ready to be dyed.

C. Natural Dyeing

The preparation of natural dyeing extracts from the brazilwood bark and lamtoro leaves is done through the preparation of 500 g of raw material that has been blended. Subtle ingredients are then extracted using a water solvent with a material/solvent ratio of 1:10, while being heated. The extraction process with heating is carried out until the volume of the solution is half from the beginning. The dye extract is filtered using two layers of fabric, to get a clear extract. The natural dyeing extract is cooled and ready to be used in dyeing. Fabric dyeing is done by immersing 7 g of fabric fiber measuring 25x25 cm using 36 mL extracts of natural dyes. The dyeing is carried out through 10 dips, each for 5 min, with pauses between immersions for 1 min. To reduce the reactivity of the dye with the surrounding environmental components, fixation is carried out.

D. Fixation

Color locking or fixation is done in order to increase color fastness. The fixer solution was prepared by dissolving 50 grams (iron(II)sulfate, alum, and lime) in 1 L of water. The solution is allowed to stand for 24 h to get the transparent part. For the fixation process, a fabric fiber is immersed in the fixer solution for 10 min, and dried.

E. Analysis

The quality of colors produced by natural dyes on cotton fibers was evaluated by a number of analyzes: (a) color intensity using Shimadzu Ultra Violet Diffuse Reflection (DRUV) UV-2401-PC Spectrophotometer; and (b) color fastness using the Gray Staining Scale method

III. RESULT AND DISCUSSIONS

A. Dyes Preparation

In this study, dyeing was carried out using a combination of two or more dyes, known as hybrid dyeing. The application of this method is done to enrich the color shades that can be produced by natural dyes. This relates to the presence of natural dyes which so far have been limited to the brownish and blackish shades characteristic of bark material, which tend to be boring. In addition, the hybrid dyeing method can be a solution for the absence of seasonal raw materials, through the provision of the same color shades. Thus, the sustainability of raw materials is no longer a barrier to natural dyeing. A hybrid dyeing method was applied in this study involving brazilwood bark and lamtoro leaf material.

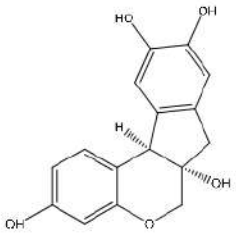

Figure 1Brazillin molecular structure

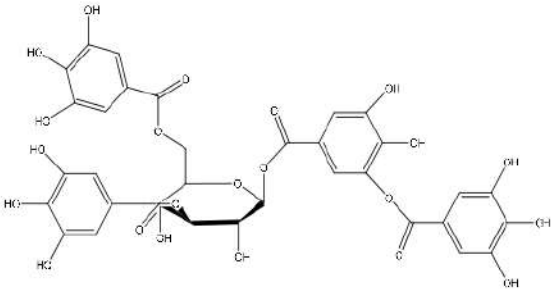

Figure 2 Tannin molecular structure

To optimize dyeing, the fabric fibers go through a pre-treatment stage. Preparations include washing using TRO and mordanting using alum and soda ash. Washing will eliminate contamination, both by polar and non-polar compounds, which inhibits the entry of mordanting agents. Like other surfactants, TRO has both polar and non-polar functional groups in its molecular structure. The existence of these polar functional groups will trigger the solubility of water-based contaminants, while non-polar functional groups will induce solubility of fat-based contaminants. The minimum contaminants in fabric fibers will open the broadest way for reactions with mordanting agents, which in this case act as intermediates for the reaction between fabric fibers and natural dyes which tend to have the same charge. Thus, the color intensity can be increased.

Mordanting is a part of fabric pre-treatment which also determines the success rate of dyeing. Mordanting determines the stability of the reaction between the dye and fabric fibers. Besides being closely related to color stability, the success of mordanting will also determine the distribution and sharpness of the resulting color. Metal mordant is one type of mordant that has been used extensively in textile natural dyeing. Alum and soda ash are two of the few types of mordant compounds. In dyeing alum and soda ash act as an intermediary for the reaction between fabric fibers and natural dyes that have the same charge tendency, which is negative. The same charge tendency causes the minimum reaction that occurs between fabric fibers and natural dyes. This condition triggers a low intensity of colors. As a solution to this problem, the existence of a positively charged mordant with a valence of more than one, as an intermediary reaction between the two is very necessary. The bonds formed between fibers, mordant, and natural dyes will play an important role in determining the change in color intensity in the fabric caused by washing, sunlight, heat from ironing and also sweat.

In dyeing, the type of chromophore and auxochrome that accompany will be the determinant of the color shades that appear. In Fig. 1 you can see brazillin compounds contained in the brazilwoodbark material, which will trigger the appearance of strong red shades on cotton fibers. Specifically, the brazilin compound specifically has the C=C chromophore group accompanied by auxochromes C-O and -OH which are responsible for the appearance of this color shades. Meanwhile, tannin compounds have more varied chromophore and auxochrome groups, through the presence of C=C and C=O as chromophores and C-O and -OH as auxochromes. Specifically for tannin dyeing, the resulting color shade will be determined by tannin classification, where the presence of hydrolyzed tannins and condensed tannins is responsible for the appearance of bright colors to dark red, brownish and even blackish.

The application of a hybrid dyeing method that combines brazillin in brazilwood bark and tannin in lamtoro leaves has produced a more varied color shade. Dyeing using lamtoro leaf extract gives a touch of yellowish green color derived from tannin compounds in it, while dyeing using lamtoro leaf provides a brighter and softer color effect compared to dyes from brazilwood bark.

After pre-treatment and dyeing, fixation becomes the final step that serves to improve the color fastness that has been produced. Fixation is done to lock the dye that has reacted with the fabric. Besides being able to lock colors, fixers are also able to induce color differences, before and after fixation.

TABLE I. HIBRID DYEING RESULTS WITH A COMBINATION OF BRAZIL WOOD BARK AND LAMTOROLEAF MATERIAL

No.	Material Composition (%v/v)	Fixer	Color Shade
1	KKS (100)	Iron (II) sulfate	
		Alum	
		Lime	
2	KKS/DL (75/25)	Iron (II) sulfate	
		Alum	
		Lime	
3	KKS/DL (50/50)	Iron (II) sulfate	
		Alum	
		Lime	
4	KKS/DL (25/75)	Iron (II) sulfate	
		Alum	
		Lime	
5	DL (100)	Iron (II) sulfate	
		Alum	
		Lime	

The color changes that occur depend on the chromophore and auxochrome groups in the molecular structure of each fixer. Utilization of iron (II) sulfate as a fixer tends to produce fiber colors that are darker than the extract color. Meanwhile, the

use of alum and lime, each producing a fabric color that is similar to the original color and the color is brighter than the original color. Table 1 shows the color shades produced after fixation using the three fixer compounds.

B. Analysis

The intensity of the colors produced by each of the dyeing operational conditions can be seen in Table 2. The resulting color intensities are derived from brazilin in brazilwood bark and tannin on lamtoro leaves, as well as a mixture of both. Brazillin dyeing with the highest intensity (89.36%) was produced from alum fixation, while the highest intensity (87.60%) in tannin dyeing was obtained from fixation using iron (II) sulfate. Hybrid dyeing that combines dyeing with brazilin and tannin produces the highest color intensity (89.08%) resulting from dyeing with a composition of 75% KKS/25% DL and fixation using iron(II)sulfate. In general, the presence of tannins in hybrid dyes tends to reduce the color intensity of the results of alum and lime fixation. Contrary to this result, in hybrid dyeing and fixation using iron(II)sulfate tends to increase the intensity of the resulting color. These results indicate a more effective reaction between a combination of brazillin and tannins with an iron(II)sulfate fixer. The large size of the alum molecule minimizes the reactions that occur with fiber due to the effect of space tightness. Meanwhile, although not as significant as the decrease in intensity resulting from alum fixation, positive charges with lower valence in the lime fixer have caused suboptimal reactions formed by fixers with natural dyes on the fiber.

TABLE II. COLOR INTENSITY WITH VARIOUS COMPOSITIONS OF HYBRID NATURAL DYES AND FIXERS

No.	Dyes Composition (%v/v)	Fixer	Color Intensity (%)
1	KKS (100)	Iron (II) sulfate	80.94
2	KKS (100)	Alum	89.36
3	KKS (100)	Lime	82.54
4	KKS/DL (75/25)	Iron (II) sulfate	89.08
5	KKS/DL (75/25)	Alum	73.23
6	KKS/DL (75/25)	Lime	74.27
7	KKS/DL (50/50)	Iron (II) sulfate	84.32
8	KKS/DL (50/50)	Alum	70.27
9	KKS/DL (50/50)	Lime	75.14
10	KKS/DL (25/75)	Iron (II) sulfate	80.58
11	KKS/DL (25/75)	Alum	79.73
12	KKS/DL (25/75)	Lime	83.14
13	DL (100)	Iron (II) sulfate	87.6
14	DL (100)	Alum	30.89
15	DL (100)	Lime	80.44

Table 3 shows the color fastness that results from hybrid dyeing of brazillin and tannin. The fastness scale indicates that hybrid dyeing with iron(II)sulfate fixation has higher fastness compared to alum and lime. The size of the iron (II) sulfate molecule that is between alum and lime is the advantage of this fixer. Molecular size not as large as alum has allowed more iron(II)sulfate molecules to enter and react with natural dyes on the fiber. The more intensive barrier created by this fixer will prevent the re-hydrolysis of the dye in the fiber and the high reactivity of the dye with the surrounding environmental components. Thus, the color intensity and fastness can be maintained or even increased. However, when viewed in terms of the resulting color shade, the use of alum fixers is able to produce the red color visualization closest to the color of brazilwood bark extract. Much darker colors are produced by iron (II) sulfate, while brighter colors are produced by alum. The appearance of this color shade is strongly influenced by the combination of chromophore and auxochrome groups which are also present in the molecular structure of each fixer.

TABLE III. COLORFASTNESS WITH VARIOUS COMPOSITIONS OF HYBRID NATURAL DYES AND FIXERS

No.	Material Composition (%v/v)	Fixer	Staining Scale (1-5)
1	KKS (100)	Iron (II) sulfate	3 (Fair)
2	KKS (100)	Alum	2 (Not good enough)
3	KKS (100)	Lime	2 (Not good enough)
4	KKS/DL (75/25)	Iron (II) sulfate	3 (Fair)
5	KKS/DL (75/25)	Alum	2 (Not good enough)
6	KKS/DL (75/25)	Lime	2 (Not good enough)
7	KKS/DL (50/50)	Iron (II) sulfate	3-4 (Good enough)
8	KKS/DL (50/50)	Alum	3 (Fair)
9	KKS/DL (50/50)	Lime	3 (Fair)
10	KKS/DL (25/75)	Iron (II) sulfate	2 (Not good enough)
11	KKS/DL (25/75)	Alum	2 (Not good enough)
12	KKS/DL (25/75)	Lime	3-4 (Good enough)
13	DL (100)	Iron (II) sulfate	3-4 (Good enough)
14	DL (100)	Alum	3-4 (Good enough)
15	DL (100)	Lime	2-3 (Not good enough)

IV. Conclusion

Exploration and standardization of natural dyeing as a substitute for synthetic dyes has been carried out. Hybrid dyeing is done by combining brazillin from brazilwood bark and tannins from lamtoro leaves extract. The dyeing results show the appearance of reddish, purple, brownish and blackish shades on the cotton fibers. The application of this hybrid dyeing has resulted in a color intensity in the range of 30.89%-89.36% with fastness reaching a scale of 3 (fair) to 3-4 (good enough).

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