UNIVERSITAS NEGERI YOGYAKARTA

- eli_rohaeti <eli_rohaeti@uny.ac.id>

Acknowledge letter

1 pesan

CMUJS <cmjs@cmu.ac.th> 30 Januari 2018 10.3
Kepada: "eli_rohaeti@uny.ac.id" <eli_rohaeti@uny.ac.id>

Dear Authors,

Thank you very much for your submission of the manuscript entitled "**Application of silver nanoparticles synthesized by using Ipomoea batatas L. waste to improve antibacterial properties and hydrophobicity of polyester cloths**" for publication in Chiang Mai Journal of Science. This is a formal acknowledgement of the receipt of your manuscript which has been assigned the following manuscript number: **CMJS.26.01.18-8873**. Your manuscript will be subjected to evaluation by experts in the field. I will let you know the outcome of referees' assessments as soon as we have received their reports.

Your sincerely,
Asst. Prof. Dr. Wasu Pathom-aree
Editor

Form Acknowledge_CMJS.26.01.18-8873.pdf
76K

Chiang Mai Journal of Science
Journal of the Science Faculty of Chiang Mai University

Chiang Mai Journal of Science
Published by Faculty of Science
Chiang Mai University
Chiang Mai 50200, Thailand

30 January 2018

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Yours sincerely,

(Asst. Prof. Dr. Wasu Pathom-aree)
Editor-in-Chief

UNIVERSITAS NEGERI YOGYAKARTA

- eli_rohaeti <eli_rohaeti@uny.ac.id

Please suggest some reviewers

10 pesan

CMJS journal <cmjs.chem@gmail.com> 26 Februari 2018 11.
Kepada: eli_rohaeti@uny.ac.id

Ms.No.: CMJS.26.01.18-8873 (C712) "Application of silver nanoparticles synthesized by using Ipomoea batatas L. waste to improve antibacterial properties and hydrophobicity of polyester cloths"

Dear Dr. Eli Rohaeti, eli_rohaeti@uny.ac.id

Your manuscript is in the process of evaluation. There is no reviewer accepted to review this work although we have invited many reviewers (including your previous suggested reviewres). To expedite the evaluation process of this MS, could you please suggest several appropriate reviewers? Please be reminded that the reviewer should not be your colleague or collaborator.

Best regards,

Jaroon Jakmunee
Associate Editor
Chiang Mai Journal of Science

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Faculty of Science
Chiang Mai University Fax + 66 53 941909
Chiang Mai
THAILAND 50200 E-mail cmjs.chem@gmail.com
http://it.science.cmu.ac.th/ejournal/index.php

- eli_rohaeti <eli_rohaeti@uny.ac.id> 27 Februari 2018 15.
Kepada: CMJS journal <cmjs.chem@gmail.com>

Dear Jaroon Jakmunee
Associate Editor
Chiang Mai Journal of Science

We are sorry to submit the names of reviewers as follows:
1. Prof. Riyanto, Ph.D. ; Chemistry Department, FMIPA, Universitas Islam Indonesia; email: riyanto@fmipa.uii.ac.id
2. Dr. Indriana Kartini; Chemistry Department, FMIPA Universitas Gadjah Mada; email: indriana@ugm.ac.id
3. Dr. Hendrawan, M.Si.; Chemistry Department, FPMIPA UPI Bandung; email: hendrawan@upi.edu
Further information is greatly awaited.

Best reagards,
Eli Rohaeti
Dept. of Chem. Edu. FMIPA
Yogyakarta State of Univ.

[Kutipan teks disembunyikan]

- eli_rohaeti <eli_rohaeti@uny.ac.id> 27 Februari 2018 15.
Kepada: CMJS journal <cmjs.chem@gmail.com>

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Further information is greatly awaited.

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Eli Rohaeti No.: CMJS.26.01.18-8873 (C712) "Application of silver nanoparticles synthesized by using Ipomoea batatas L. waste to improve antibacterial properties and hydrophobicity of polyester cloths"
Dept. of Chem. Edu. FMIPA
Yogyakarta State of Univ.
[Kutipan teks disembunyikan]

CMJS journal <cmjs.chem@gmail.com> 1 Maret 2018 06.
Kepada: - eli_rohaeti <eli_rohaeti@uny.ac.id>

Thanks

[Kutipan teks disembunyikan]
[Kutipan teks disembunyikan]
[Kutipan teks disembunyikan]

–
Jaroon Jakmunee
Associate Editor
Chiang Mai Journal of Science

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http://it.science.cmu.ac.th/ejournal/index.php

CMJS journal <cmjs.chem@gmail.com> 9 Maret 2018 05.
Kepada: - eli_rohaeti <eli_rohaeti@uny.ac.id>

Dear Dr. Eli Rohaeti,

None of your suggested reviewers accepted to review. Please suggested other persons.

Best regards,
Jaroon

[Kutipan teks disembunyikan]
[Kutipan teks disembunyikan]
[Kutipan teks disembunyikan]

–
Jaroon Jakmunee
Associate Editor
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- eli_rohaeti <eli_rohaeti@uny.ac.id> 9 Maret 2018 10.2
Kepada: CMJS journal <cmjs.chem@gmail.com>

Dear Jaroon Jakmunee
Associate Editor
Chiang Mai Journal of Science

We submit the names of reviewers as follows:
1. Prof. I Made Arcana, Ph.D. ; Chemistry Department, FMIPA, Bandung Institute of Technology Indonesia; email: arcana@chem.itb.ac.id
2. Dr. Agus Haryono, M.Sc. Chemical Research Center of Indonesian Institute of Sciences, Jakarta, Indonesia; email: agus.haryono@lipi.go.id/ agus063@lipi.go.id/ haryonolipi@yahoo.com
3. Dr. Diah Mardiana; Chemistry Department, FMIPA Universitas Brawijaya Malang, Indonesia; email: mdiah@ub.ac.id
Further information is greatly awaited.

Best reagards,
Eli Rohaeti
Dept. of Chem. Edu. FMIPA
Yogyakarta State of Univ.
[Kutipan teks disembunyikan]

CMJS journal <cmjs.chem@gmail.com> 9 April 2018 07.1
Kepada: - eli_rohaeti <eli_rohaeti@uny.ac.id>

None of your suggested reviewers accepted to review. Please suggested other persons.

Best regards,
Jaroon

[Kutipan teks disembunyikan]

CMJS journal <cmjs.chem@gmail.com> 9 April 2018 07.
Kepada: - eli_rohaeti <eli_rohaeti@uny.ac.id>

None of your suggested reviewers accepted to review. Please suggested other persons.

Best regards,
Jaroon

[Kutipan teks disembunyikan]

- eli_rohaeti <eli_rohaeti@uny.ac.id> 9 April 2018 09.
Kepada: CMJS journal <cmjs.chem@gmail.com>

Dear Dr. Jaroon Jakmunee
Associate Editor
Chiang Mai Journal of Science

I suggest the reveiwers of our article, i.e.
1. Sunu Brams Dwandaru, Ph. D. ; Department of Physics Education, Faculty of Mathematics and Natural Sciences, Universitas Negeri Yogyakarta, Indonesia; email: wipsarian@uny.ac.id
2. Prof. Dr. Endang Widjajanti LFX, ; Department of Chemistry Education, Faculty of Mathematics and Natural Sciences, Universitas Negeri Yogyakarta, Indonesia; email: endang_widjajanti@uny.ac.id
3. Prof. Dr. Zainal Abidin Tholib; Department of Physics, Universiti Putra Malaysia, Malaysia; email: zainalat@upm.edu.my
4. Prof. Dr. Phill. Hari Sutrisno; Department of Chemistry Education, Faculty of Mathematics and Natural Sciences, Universitas Negeri Yogyakarta, Indonesia, email: sutrisnohari@uny.ac.id

Best regards,
Eli Rohaeti
No.: CMJS.26.01.18-8873 (C712) "Application of silver nanoparticles synthesized by using Ipomoea batatas L. waste to improve antibacterial properties and hydrophobicity of polyester cloths"
eli_rohaeti@uny.ac.id
[Kutipan teks disembunyikan]

CMJS journal <cmjs.chem@gmail.com> 10 April 2018 18.
Kepada: - eli_rohaeti <eli_rohaeti@uny.ac.id>

thanks
[Kutipan teks disembunyikan]

CMJS journal <cmjs.chem@gmail.com> 9 April 2018 07.
Kepada: - eli_rohaeti <eli_rohaeti@uny.ac.id>

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Best regards,
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[Kutipan teks disembunyikan]

- eli_rohaeti <eli_rohaeti@uny.ac.id> 9 April 2018 09.
Kepada: CMJS journal <cmjs.chem@gmail.com>

Dear Dr. Jaroon Jakmunee
Associate Editor
Chiang Mai Journal of Science

I suggest the reveiwers of our article, i.e.
1. Sunu Brams Dwandaru, Ph. D. ; Department of Physics Education, Faculty of Mathematics and Natural Sciences, Universitas Negeri Yogyakarta, Indonesia; email: wipsarian@uny.ac.id
2. Prof. Dr. Endang Widjajanti LFX, ; Department of Chemistry Education, Faculty of Mathematics and Natural Sciences, Universitas Negeri Yogyakarta, Indonesia; email: endang_widjajanti@uny.ac.id
3. Prof. Dr. Zainal Abidin Tholib; Department of Physics, Universiti Putra Malaysia, Malaysia; email: zainalat@upm.edu.my
4. Prof. Dr. Phill. Hari Sutrisno; Department of Chemistry Education, Faculty of Mathematics and Natural Sciences, Universitas Negeri Yogyakarta, Indonesia, email: sutrisnohari@uny.ac.id

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CMJS journal <cmjs.chem@gmail.com> 10 April 2018 18.
Kepada: - eli_rohaeti <eli_rohaeti@uny.ac.id>

thanks
[Kutipan teks disembunyikan]

UNIVERSITAS NEGERI YOGYAKARTA

- eli_rohaeti <eli_rohaeti@uny.ac.id

Review results of manuscript CMJS.26.01.18-8873 (C712)

3 pesan

CMJS journal <cmjs.chem@gmail.com> 26 April 2018 06.1
Kepada: - eli_rohaeti <eli_rohaeti@uny.ac.id>

MS No.: CMJS.26.01.18-8873 (C712) "Application of silver nanoparticles synthesized by using Ipomoea batatas L. waste to improve antibacterial properties and hydrophobicity of polyester cloths"

Dear Dr. Eli Rohaeti,

I am now able to inform you that your above manuscript has now passed through our refereeing procedure and has been recommended for revision as details in the attached files. Please consider comments/suggestions of the referees for improving of your article.

I would be most grateful if you could send a list of answers to all the referees comments and return your revised paper with yellow background highlight on the change/modify being made (or by other means to help follow the change). Please send them to this e-mail within 2 months, the revised MS sending after 2 months will be considered as a new submission. Please also pay attention on format of the article, the example paper for your guideline for the current format of CMJS journal is also attached herewith (No need to arrange the MS as two columns).

I thank you for your interesting in Chiang Mai Journal of Science.

Yours respectfully,

Jaroon Jakmunee
Associate Editor
Chiang Mai Journal of Science

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- eli_rohaeti <eli_rohaeti@uny.ac.id> 10 Mei 2018 09.4
Kepada: CMJS journal <cmjs.chem@gmail.com>

CHIANG MAI JOURNAL OF SCIENCE

Referee's Evaluation Form

Ms. No.: CMJS.26.01.18-8873(C712)

Please consider the following points in the table below and evaluate each one in turn (tick ✓ the appropriate column).

POINTS TO CONSIDER	EVALUATION			
	Inadequate	Adequate	Good	Excellent
Originality of the work			✓	
Clarity of expression			✓	
Standard of English		✓		
Use of figures and tables			✓	
Validity of arguments		✓		
Adequacy of references			✓	
Overall impression		✓		

Based on the above evaluation, you recommend that this paper should be (please tick ✓)

☐ accepted as it is without revision
✓ accepted subject to minor revision
☐ accepted subject to major revision
☐ rejected
☒

In the event that you consider that the paper requires revision, or would be more appropriate as a ***Short Communication***, or should simply be rejected, please give your reasons overleaf so that we, in turn, can explain them clearly to the authors in our own combined Referees' Report.

REFEREE'S COMMENTS

Please give details below of your suggestions for revision of the paper or explain your reasons for recommending that the paper be accepted as a ***Short Communication*** or be rejected outright.

Summary of this manuscript:

Strength:

This article, more or less discussed green chemistry which was then followed by the application in preparing a healty fabric or cotton. The procedure was simple but on the right track in preparing a series of compounds to confirm the antibacterial activity.

Weakness:

In this work, the number of samples might be multiplied to have a stronger conclusion. The English expression is less impressive.

Specific Comments/suggestions:

Consider the following corrections:

1. Please insert the word "for" in the following sentence p. 2 (Introduction)

   Plant extracts effectively reduce metal ions more significantly for ions that have high positive electrochemical potentials such as Ag^+ [4].

2. The extracellular synthesis of silver nanoparticles could be conducted economic and safe by using leaf extract of *Catharanthus roseus Linn. G.Don* [25].

   Change in to ..... could be conducted economically and safely by .....

3. The antioxidant properties of phenolic compounds through their tendency to metal [3].

   Seme words missing. It can be as the following:

   The antioxidant properties of phenolic compounds might occur through their tendency to chelate metal [3].

4. Some references are without DOI and /or address to download, [1-2], [4], [14-16], [27].

5. Tables are NOT correctly presented as in guide line. NO need lines for columns.

6. In Figure 6 the word "wavenumber" should be indicated in the absis.

Thank you very much indeed for your time and effort in completing this evaluation

Review Comment:

This paper is dealing with the synthesis silver nanoparticle by using peel extract of Ipomoea batatas L. to improve antibacterial properties and hydrophobicity of polyester cloths. Polyester cloths were modified by coating silver nanoparticles and hexadecyltrimethoxysilane (HDTMS), and the modified polyester was characterized by ATR-FTIR, SEM, contact angle, and antibacterial activity test. This paper contains some interesting information, but the analysis of the data is not clear and complete, so that it needs some clarification according to the following comments.

Other comments:

1. Experimental part is lacking details of quality of all chemicals used. In the test of hydrophobicity by measuring contact angle was used a liquid that was dropped on surface sample, but in the manuscripts the liquid used is not clear.

2. In the synthesis of silver nanoparticles was used 12 mL of 1% PVA as a stabilizer agent. What is the PVA molecular weight used in the synthesis of nanoparticles, and in polyester modification was carried out by coating on the surface of the polyester with a colloidal nanoparticle silver, please explain how the PVA is present in the modified polyester. In the manuscript, there is no explanation of the existence of the PVA in the modified polyester.

3. In the test of antibacterial activity, was carried out in an agar medium (solid) and a liquid nutrient broth medium. However, in the results and discussion, there is no explanation about the influence of the both mediums on the antibacterial activity in modified polyesters, and how many samples were used in each experiment of antibacterial activity test for the same conditions?

4. In the synthesis of silver nanoparticles, how to know resulted colloidal silver has a particle size of nanometers? And in the explanation, it is stated that in the process of forming silver nanoparticles using skin extracts of Ipomoea batatas L. occurs reaction between -OH phenolic of anthocyanin compounds with silver ions to produce silver nanoparticles and quinone compounds that cause the color of the solution to reddish brown. Is there any evidence to support this explanation e.g. phenolic conversion to be quinone like analysis by FTIR or NMR?

5. Figure 5 is not clear. In the manuscript is stated that the modified polyester shows the presence of silver nanoparticles deposited on the surface of polyester (Fig. 5b and 5c). Please show the evidence the presence of silver nanoparticles contaminants in modified polyester in Fig. 5b and 5c, whereas from SEM images show that Fig. 5a and 5b are almost identical (P0 and P3), and they are different

with Fig. 5c (P4), so that the surface between pure polyester (P0) and the modified polyester (P3) are practically no different.

6. In Figure 6, why modification polyesters by silver nanoparticles and HDTMS compound are not produce new functional groups in FTIR? However, In the manuscript is stated that the contact angles between polyester modified by silver nanoparticle and followed by HDTMS with modified polyester by HDTMS and followed by silver nanoparticle are different, so why the modification method influence on the contact angle of polyesters. In the manuscript, there is no explanation about this result.

7. In the antibacterial activity of modified polyester is stated that the interaction between the ester groups of polyester and the -OH groups if HDTMS produces a hydrophobic functional group, so that the hydrophobic surface may cause the bacteria to inhibit its growth when interacting with the modified polyester. It should be explained by the molecular structure, how the interaction takes place between group ester from polyester with -OH group from HDTMS and silver ions, so that can inhibit the bacteria growth?

8. It should be explained why the presence of hydrophilic and hydrophobic compounds from antibacterial agents can damage the cytoplasmic membrane and kill bacterial cells? The explanation in the manuscript is not clear.

9. In Figure 8 and 9, It should be explained clearer in the manuscript why at incubation time less than 55-60 hours, the diameter of clear zone increases, on the other hand after incubation time more than 55-60 the diameter of clear zone decreases, and the maximum diameter of clear zone was observed at incubation time between 55-60 hours? And why in Figure 8 the maximum diameter of clear zone was observed in sample of P-HDTMS, whereas in Figure 9 it was observed in sample of P-HDTMS-Ag? The explanation in the manuscript is not clear.

10. The explanation of Table 1-3 is not clear in the manuscript.

11. The writing in English needs to be improved

CHIANG MAI JOURNAL OF SCIENCE

Referee's Evaluation Form

Ms. No.:

Please consider the following points in the table below and evaluate each one in turn (tick ✓ the appropriate column).

POINTS TO CONSIDER	EVALUATION			
	Inadequate	Adequate	Good	Excellent
Originality of the work		✓		
Clarity of expression				
Standard of English	✓			
Use of figures and tables		✓		
Validity of arguments	✓			
Adequacy of references			✓	
Overall impression		✓		

Based on the above evaluation, you recommend that this paper should be (please tick ✓)

- ☐ accepted as it is without revision
- ☐ accepted subject to minor revision
- ☐ accepted subject to major revision
- ☒ rejected

☒

In the event that you consider that the paper requires revision, or would be more appropriate as a ***Short Communication***, or should simply be rejected, please give your reasons overleaf so that we, in turn, can explain them clearly to the authors in our own combined Referees' Report.

CHIANG MAI JOURNAL OF SCIENCE

Referee's Evaluation Form

Ms. No.: ***CMJS.26.01.18-8873 (C712)***

Please consider the following points in the table below and evaluate each one in turn (tick ✓ the appropriate column).

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	Inadequate	Adequate	Good	Excellent
Originality of the work				√
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Standard of English			√	
Use of figures and tables			√	
Validity of arguments			√	
Adequacy of references			√	
Overall impression			√	

Based on the above evaluation, you recommend that this paper should be (please tick ✓)

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REFEREE'S COMMENTS

Please give details below of your suggestions for revision of the paper or explain your reasons for recommending that the paper be accepted as a ***Short Communication*** or be rejected outright.

Summary of this manuscript:

Strength:

This article explains the utilization of natural materials to produce functional materials. The novelty of this research is found in the use of sweet purple potatoes extract.

Weakness:

This article does not specify the application of the product (polyester cloth)

Specific Comments/suggestions:

It is necessary to explain the type of method of nanoparticles preparation and include with the reaction

Thank you very much indeed for your time and effort in completing this evaluation

Dear Jaroon Jakmunee,
Associate Editor
Chiang Mai Journal of Science

I send a list of answers to all the referees comments and our revised paper with yellow background highlight on the change/modify being made.

Sincerely yours.
Eli Rohaeti (No.: CMJS.26.01.18-8873 (C712) "Application of silver nanoparticles synthesized by using Ipomoea batatas L. waste to improve antibacterial properties and hydrophobicity of polyester cloths")
[Kutipan teks disembunyikan]

2 lampiran

C712 REF(Eli).doc
197K

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1428K

CMJS journal <cmjs.chem@gmail.com> 10 Mei 2018 23.5
Kepada: - eli_rohaeti <eli_rohaeti@uny.ac.id>

Dear Dr. Eli Rohaeti,

Your revised manuscript was received for reconsideration for publication in Chiang Mai Journal of Science. You will be informed the final result within 15 days, please remind us if you don't receive the final result after 15 days.

Yours respectfully,
Jaroon Jakmunee
Associate Editor (Chemistry Section)
Chiang Mai Journal of Science

[Kutipan teks disembunyikan]
[Kutipan teks disembunyikan]

–
Jaroon Jakmunee
Associate Editor
Chiang Mai Journal of Science

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Fax + 66 53 941909
E-mail cmjs.chem@gmail.com

REVISION TO REFEREE'S COMMENTS

Ref. 1

1. *Weakness:* In this work, the number of samples might be multiplied to have a stronger conclusion.

In this work, we synthesized silver nanoparticles which applied to polyester fabric resulting in 5 variations of polyester products. Modification of Polyester performed as many as 5 times repetition. Similarly, the antibacterial and hydrophobicity test were performed 3 replications.

2. Specific Comments/suggestions:

1. Please insert the word "for" in the following sentence p. 2 (Introduction)

   Plant extracts effectively reduce metal ions more significantly for ions that have high positive electrochemical potentials such as Ag^{+} [4].

2. The extracellular synthesis of silver nanoparticles could be conducted economic and safe by using leaf extract of *Catharanthus roseus Linn. G.Don* [25].

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6. In Figure 6 the word "wavenumber" should be indicated in the absis.

We've done all the improvement suggestions.
All the articles ([1-2], [4], [14-16], [27]) do not have DOI.

Ref. 2

1. Experimental part is lacking details of quality of all chemicals used. In the test of hydrophobicity by measuring contact angle was used a liquid that was dropped on surface sample, but in the manuscripts the liquid used is not clear.

   We have completed the material specifications.
   In the test of hydrophobicity, the liquid is replaced with water.

2. In the synthesis of silver nanoparticles was used 12 mL of 1% PVA as a stabilizer agent. What is the PVA molecular weight used in the synthesis of nanoparticles, and in polyester modification was carried out by coating on the surface of the polyester with a colloidal nanoparticle silver, please explain how the PVA is present in the modified polyester. In the manuscript, there is no explanation of the existence of the PVA in the modified polyester.

   We have completed a molar mass of PVA of 29,365.96 g/mol. PVA acts only as a plasticizer during the preparation of silver nanoparticles, so it does not play a direct role in polyester modification. Silver nanoparticles which produced in this work only 1 type, that is with the addition of PVA.

3. In the test of antibacterial activity, was carried out in an agar medium (solid) and a liquid nutrient broth medium. However, in the results and discussion, there is no explanation about the influence of the both mediums on the antibacterial activity in modified polyesters, and how many samples were used in each experiment of antibacterial activity test for the same conditions?

   In this study, liquid media was used for bacterial growth while solid media for antibacterial test.
   Thus in this study only use 1 type of media (solid media) on antibacterial analysis of the sample.
   We did not compare the antibacterial differences of the sample by using liquid media and solid media.
   We have added the number of samples in this test

4. In the synthesis of silver nanoparticles, how to know resulted colloidal silver has a particle size of nanometers? And in the explanation, it is stated that in the process of forming silver nanoparticles using skin extracts of Ipomoea batatas L. occurs reaction between -OH phenolic of anthocyanin compounds with silver ions to produce silver nanoparticles and quinone compounds that cause the color of the solution to reddish brown. Is there any evidence to support this explanation e.g. phenolic conversion to be quinone like analysis by FTIR or NMR?

   Particle size has been performed with PSA tools. Detailed characteristics of the nanoparticles we have reported in other articles (reviewed process).
   In this paper we focus on the use of silver nanoparticles as a material to increase the hydrophobicity and antibacterial polyester.
   However, in this paper we have revealed the size of the nanoparticles. The explanation that peel extract acts as a reducing agent which converts

the silver nitrate solution into a silver atom, which is indicated by the colloidal color change occurring. We explained that because the anthocyanin in the peel extract had turned into quinone, we took it from the reference.
In this study, we focused on ATR-FTIR of polyester fabric before and after modification with nanoparticle.

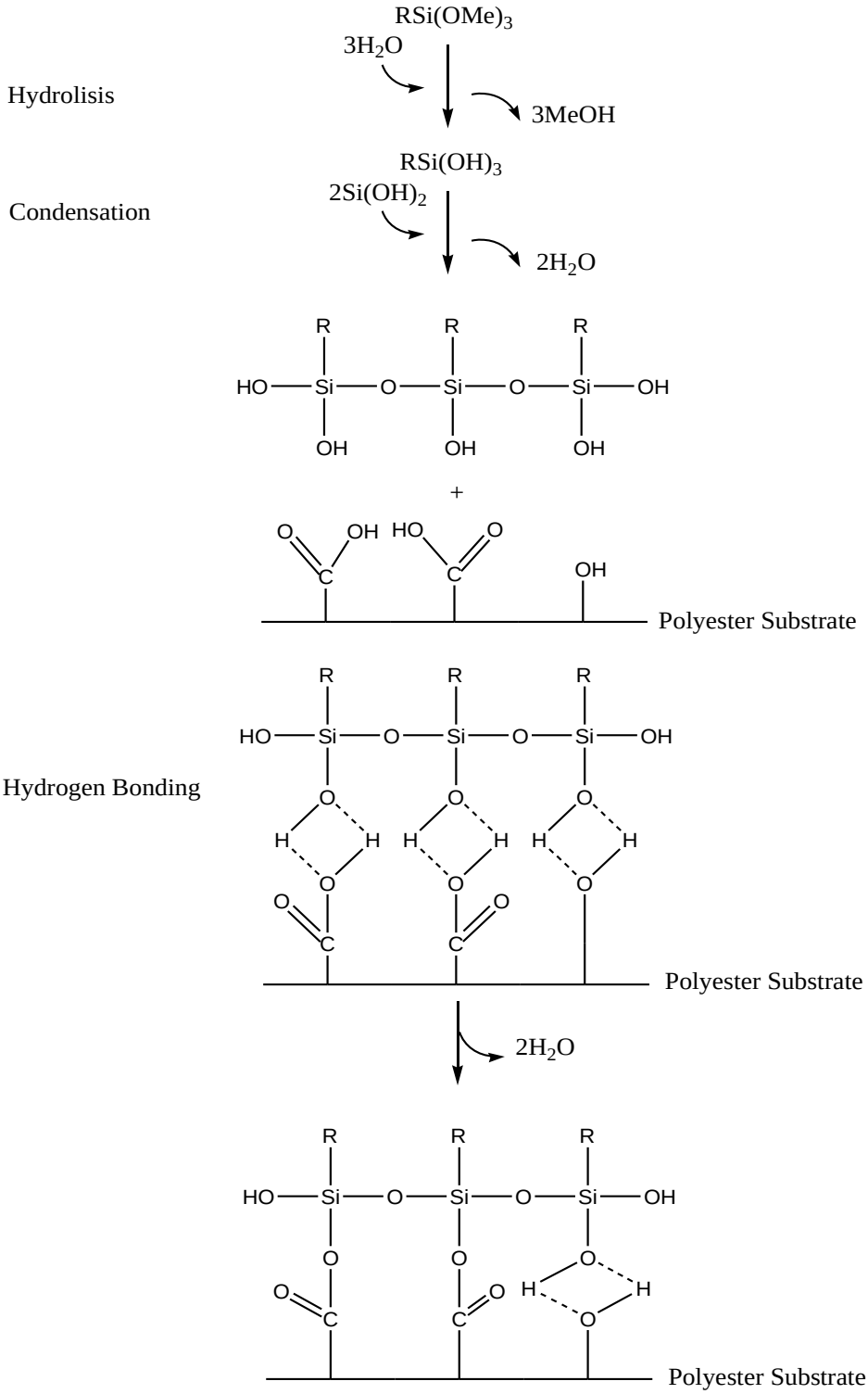
5. Figure 5 is not clear. In the manuscript is stated that the modified polyester shows the presence of silver nanoparticles deposited on the surface of polyester (Fig. 5b and 5c). Please show the evidence the presence of silver nanoparticles contaminants in modified polyester in Fig. 5b and 5c, whereas from SEM images show that Fig. 5a and 5b are almost identical (P0 and P3), and they are different with Fig. 5c (P4), so that the surface between pure polyester (P0) and the modified polyester (P3) are practically no different.

The surface between pure polyester (P0) and the modified polyester (P3) are practically no different.
P3 is a modified polyester with a silver coating and followed the HDTMS while P4 is a modified polyester with a HDTMS coating and followed the silver nanoparticles.
P0 and P3 practically do not differ significantly surface. This could be caused by the HDTMS layer covered the silver nanoparticle so that the nanoparticle of silver can't be detected on surface images of P3, whereas in P4 the layer of HDTMS was covered by the silver nanoparticles so that the surface images are different, the silver nanoparticles are very much on the surface of the cloth P4.

6. In Figure 6, why modification polyesters by silver nanoparticles and HDTMS compound are not produce new functional groups in FTIR? However, In the manuscript is stated that the contact angles between polyester modified by silver nanoparticle and followed by HDTMS with modified polyester by HDTMS and followed by silver nanoparticle are different, so why the modification method influence on the contact angle of polyesters. In the manuscript, there is no explanation about this result.

The contact angle is related to the surface free energy. The free energy of the surface is inversely proportional to the contact angle.
The addition of nanosilver particles to hydrophobic materials, such as polyester composite (P4), caused a decrease in surface free energy and an increase of the contact angle of P4.
The presence of a hydrophobic functional group increases the angle contact of water.
The method of modification of the polyester fabric, qualitatively does not change the type of functional group present (proved by ATR-FTIR), but that affects the number of hydrophobic groups. Thus, the addition of nanosilver particles to hydrophobic materials, such as polyester composite, caused an increase of the hydrophobic groups and a decrease in surface free energy, so that an increase of the contact angle of P4.

7. In the antibacterial activity of modified polyester is stated that the interaction between the ester groups of polyester and the -OH groups if HDTMS produces a hydrophobic functional group, so that the hydrophobic surface may cause the bacteria to inhibit its growth when interacting with the modified polyester. It should be explained by the molecular structure, how the interaction takes place between group ester from polyester with -OH group from HDTMS and silver ions, so that can inhibit the bacteria growth?



the interaction among the ester, the hydroxyl groups of polyester and the -OH groups of HDTMS produces a hydrophobic functional group, so that the

hydrophobic surface may cause the bacteria to inhibit its growth when interacting with the modified polyester.
And also interaction between silver in modified polyester and microbes can be ionic or covalent interaction.

8. It should be explained why the presence of hydrophilic and hydrophobic compounds from antibacterial agents can damage the cytoplasmic membrane and kill bacterial cells? The explanation in the manuscript is not clear.

The -OH functional groups from the polyester cloth and the HDTMS (hydrophilic) and hydrophobic **bound to the silver will interact to -S-H on the cytoplasmic membrane to form a bond -S-S- and produce S-Ag clusters that cause inhibition of cell respiration and kill bacterial cells.**

9. In Figure 8 and 9, It should be explained clearer in the manuscript why at incubation time less than 55-60 hours, the diameter of clear zone increases, on the other hand after incubation time more than 55-60 the diameter of clear zone decreases, and the maximum diameter of clear zone was observed at incubation time between 55-60 hours? And why in Figure 8 the maximum diameter of clear zone was observed in sample of P-HDTMS, whereas in Figure 9 it was observed in sample of P-HDTMS-Ag? The explanation in the manuscript is not clear.

The explanation of Fig. 8.
The interaction between the ester groups in the polyester and the -OH group in HDTMS produces a hydrophobic functional group so that the hydrophobic surface may cause the bacteria to inhibit its growth when interacting with the polyester fabric. Compounds containing hydrophilic and hydrophobic groups (P-HDTMS) are antibacterial agents that can damage cytoplasmic membranes and kill the bacterial cells.

The explanation of Fig. 9.
This is due to the interaction between ester groups with –OH groups of HDTMS resulting in hydrophobic groups which are not favored by bacteria followed with modification by deposition of Ag nanoparticles, which is an antibacterial agent as well, thus increasing activity of antibacterial from polyester fabric (P-HDTMS-Ag). Thus, the polyester modification method using a combination of HDTMS and nanoparticles increases the antibacterial activity of the fabric.

P-HDTMS has the ability to inhibit the highest growth of E. coli compared to other polyester samples. Thus, gram-negative bacteria are more easily inhibited by modified polyesters by silane compounds. The hydrophobic functional groups are more able to inhibit gram-negative bacteria.
P-HDTMS-Ag has the ability to inhibit the highest growth of S. aureus compared to other polyester samples. Thus, gram-positive bacteria are more easily inhibited by modified polyesters by silane compounds and followed by coating of silver nanoparticles. The presence of hydrophobic function groups and silver nanoparticles may inhibit gram-positive bacteria.

In other words, gram-positive bacteria can be inhibited to the maximum by using polyester composites, this is because the gram-positive bacteria

consist of a thick layer of peptidoglycan and contains a layer of teapixic acid is polar and tend to be negatively charged, so that antibacterial material is required that can inhibit its growth in the form of composites polyester with HDTMS and silver nanoparticles

10. The explanation of Table 1-3 is not clear in the manuscript.

Explanation of Table 1.
The type of polyester sample and the incubation time affect the antibacterial activity. The five types of polyester samples have different antibacterial activity significantly. Similarly with incubation time, different incubation times showed different antibacterial activity significantly as well.

Explanation of Table 2.
All of the modified polyester shows significantly higher antibacterial activity than the unmodified polyester. Thus, modification can increase antibacterial activity of polyester cloths.
However, within the modified polyesters (P1-P4) no significant difference in theirs antibacterial activity was observed.
Modification of polyester cloths by using silver nanoparticle, HDTMS, silver nanoparticle and HDTMS, and also HDTMS and silver nanoparticle has the similar effect toward antibacterial activity of polyester.
Thus, the type of addition material and methods did not effect antibacterial activity of polyester cloths against *S.aureus*.

The results of LSD test in Table 2 shows that there are 7 variations of samples which have significant influence between two samples on antibacterial activity against *Escherichia coli* ATCC 35218.
Therefore, it can be concluded that modification can affect antibacterial activity of polyester cloths. The addition of silver nanoparticles, HDTMS, and combinations of the two might enhance the antibacterial activity of unmodified polyester fabrics to inhibit the growth of *E.coli*.

Explanation of Table 3.
Each sample of the polyester fabric shows no difference in antibacterial activity to inhibit the growth of *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923.
Thus, the ability each sample to inhibit the *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923 is the same.

11. The writing in English needs to be improved

This article has been reviewed by Prof in our Institution which usually write articles of international standard.

Ref. 3

1. *Weakness:*

   This article does not specify the application of the product (polyester cloth)

   It has been added to the conclusion section, ie

   "Based on the antibacterial properties of the fabric then the modified polyester product with nanoparticles and HDTMS can be used as antibacterial materials for biomedical applications."

2. Specific Comments/suggestions:

   It is necessary to explain the type of method of nanoparticles preparation and include with the reaction

   It has been described the type of method of nanoparticles preparation, that is biosynthesis by using bioreductor of peel extract (Methods)

   Reaction between peel extract of *Ipomoea batatas L* and silver nitrate solution forming nanoparticle of silver has been explained in Figure 4 and in Results and Discussion.

Application of silver nanoparticles synthesized by using *Ipomoea batatas L.* waste to improve antibacterial properties and hydrophobicity of polyester cloths

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ABSTRACT

Synthesis of colloidal silver nanoparticle by using peel extracts of *Ipomoea batatas L*., its deposition on polyester cloths, and the modification with hexadecyltrimethoxysilane (HDTMS) have been conducted in this work. The silver nanoparticles were characterized by using Uv-Vis spectrophotometer, functional groups of unmodified polyester and those of modified polyester were characterized by ATR-FTIR spectrophotometer, surface images of polyester were observed by Scanning Electron Microscopy tool, antibacterial activity of unmodified polyester cloth and modified polyester against *Staphylococcus aureus* and *Escherichia coli* were determined with a diffusion method, and hydrophobicity of polyester was measured by using asessile drop method. Silver nanoparticles were successfully produced using peel extracts of *Ipomoea batatas L* as indicated by the absorption peaks at 436 nm. SEM images confirmthat the silver nanoparticles coat onto polyester cloths. Modification with nanoparticle and HDTMS do not affect the functional groups of polyester. The polyester fabric with the addition of HDTMS compound and silver nanoparticles showedthe largest contact angle and the antibacterial activity to inhibit *Staphylococcus aureus*. Samples ofpolyester – HDTMS – silver nanoparticle show the highest antibacterial activity against*S. aureus*with a strength 1.4 times greater than unmodified polyester. There is a difference in the antibacterial activity of the polyester among the unmodified polyester and the modified polyester fabric at different incubation times inhibiting the growth of *Escherichia coli* and *Staphylococcus aureus*. Each sample ofthe unmodified polyester and the modified polyester fabric shows the same ability to inhibit the growth of *Staphylococcus aureus* and *Escherichia coli*.

Keywords: Antibacterial Activity, Hydrophobicity, *Ipomoea batatas L.*, Polyester, Silver Nanoparticles.

1. INTRODUCTION

Peelof purple sweet potato is an untapped waste, however, the anthocyanin content in a peelof the purple sweet potato is actually still high,being 110.51 mg/100 g. Pigments of anthocyanins in *Ipomoea batatas L.* are more stable than anthocyanins from other sources, such as red cabbage, elderberries, blueberries and red corn [1]. However, anthocyanins contain phenolic hydroxyl groups which are easily oxidized into quinones, and thus skin extract of *Ipomoea batas L.* can be used as a reducing agent to convert silver ions (Ag^{+}) into silver nanoparticles (Ag^{0}). The reduction of particle size to nano-size of silver can be caused by the H radicals formed in phenolic compounds that are antioxidants [2]. Phenolics have hydroxyl and carbonyl groups that can bind metals. The antioxidant properties of phenolic compounds might occur through their tendency to chelate metal [3]. The efficiency of the synthesis of metal nanoparticles depends on the electrochemical potential of the reduced ions. Plant extracts effectively reduce metal ions more significantly for ions that have high positive electrochemical potentials such as Ag^{+} [4].

Nanoparticles can be synthesized using various methods, for examples using microwave irradiation method [5], reduction method [6], photochemical or photosynthesis method [7, 8], ball milling method [9], electrochemical method [10], biosynthesis by using actinomycete or actinobacteria [11, 12], and biosynthesis by using dried fruit extract method [13]. In this study, silver nanoparticles were prepared by applying green chemistry using skin extract of sweet purple potato (*Ipomoea batatas L.*). Utilization of plants to synthesize nanoparticles can be done based on the ability of these plants to absorb metal ions from the environment. The ions will be reduced through complex metabolic processes and accumulated in certain organs. Plants are known to have organic compounds that serve as reducing agents that can be used to replace or complement inorganic reducers. The use of

plant organic compounds for the synthesis of nanoparticles is known as biosynthesis and is an eco-friendly method, as well as simpler and more efficient than chemical procedures [14].

The process of applying nanoparticles to textile fibers is done by composting nanometer-scale particles into textile fibers. The three-dimensional nanostructured surface particles and gel-producing additives produce hydrophobic fabric products that do not reduce the breathability and comfort of the fabric when worn. However, the use of silane compounds causes the impurities attached to the fabric to be easily released when watered, however, the fabric remains dry.

Textile materials developed into self-cleaning textile and antibacterial products in this workare polyester fabrics. Naturally, polyester fibers have hydrophobic properties as well as apparel materials, polyester fibers are also commonly used as materials for sportwear, underwear and sheets [15]. The antibacterial polyester can be developed through modification with silver nanoparticles. Antibacterial textiles have been developed by modifying textile materials with nanoparticles and vegetable oil, such as nylon [16], cotton [17], silk cloths [6], polyurethane [18], leather [19], and wool fiber [20].

Polyester with self-cleaning properties can be developed through modification with silane compounds. Several types of silane compounds used to modify materials are hexamethyldisilazane [21], octyltriethoxysilane [22], and hexadecyltrimethoxysilane [16, 17, 23]. The addition of silane-based molecules of HDTMS compounds to textiles can increase water contact angle or hydrophobicity of the fabric [16]. The objectives of our research were to synthesize silver nanoparticle by using peel extract of *Ipomoea batatas L.*, to modifypolyester cloths by coating silver nanoparticles and HDTMS, and to study hydrophobicity property of polyester, and also to determine antibacterial activity of unmodified polyester and modified polyester.

2. MATERIALS AND METHODES

2.1. Materials

Silver nitrate (E-Merck), polyvinyl alcohol (PVA) with molar mass 29,365.96 g/mol (E-Merck), ethanol, acetone, and hexadecyltrimethoxysilane (Aldrich) were purchased as commercial products and used without any further purification. Polyester cloth was purchased from a store in Yogyakarta, Indonesia. Nutrient Agar (NA) and Nutrient Broth (NB) were purchased from Oxoid. *Staphylococcus aureus* ATCC 25923 and *Escherichia coli* 32518 were obtained from a collection of Faculty of Medicine, Universitas Gadjah Mada (UGM) Yogyakarta, Indonesia.

The research was conducted in the following stages: extraction of *Ipomoea batatas L*. peel, synthesis of Ag nanoparticle by using bioreductor, addition of colloidal silver nanoparticle on polyester cloths, modification of polyester cloths by adding HDTMS and colloidal silver nanoparticle, and characterization of unmodified polyester and modified polyester.

2.2. Apparatus

Ultra Violet – Visible (Uv-Vis) spectrophotometer (Shimadzu UV2450, Japan) wasused to study thesuccessful formation of colloid of silver nanoparticle by observing absorption peaks of silver nitrate solution and colloidal silver nanoparticle. Attenuated Total Reflection - Fourier Transfrom Infra-red (FTIR) spectra were recorded on a FTIR – ATR spectrophotometer (Shimadzu prestige 21, Japan). Morphological images of unmodified polyester and modified polyester were observed by using a scanning electron microscope tool (SEM Jeol T300, USA).

2.3. Preparation of Peel Extract

A total of 20 grams of *Ipomoea batatas L.* peel was freshly cleaned with aquadest and inserted it into a 500 mL beaker glass. About 100 mL of distilled water was added into it and then boiled for 15 minutes. Allowing to stand the mixture until room temperature after boiling, the extract of *Ipomoea batatas L.* skin was then filtered using Whatmann no. 42.

2.4. Biosynthesis of Silver Nanoparticle

About 1 mL of peel extract of *Ipomoea batatas L.*in an erlenmeyer was added into 40 mL of 1 x 10^{-3} M silver nitrate solution. The mixture was allowed to stand for 2 hours to react. To the mixture, 12 mL of 1% PVA solution was added and then it was allowed to stand for 1 hour. Finally, the solution was stirred for 3 days to form colloidal silver nanoparticles. The colloid was then characterized using a UV-Vis spectrophotometer.

2.5. Deposition of Nanoparticle on Polyester Cloths

The sample of polyester fiber (P0) with size 5 cm x 5cmwas soaked in colloidal silver nanoparticles in erlenmeyer 50 mL and shaked using a shaker at 155 rpm for 24 hours. It was then dried at room temperature for one day. Thus, a polyester- nanoparticle sample (P1) was produced.

2.6. Modification of Polyester Cloth with HDTMS

The polyester (P0) and polyester – nanoparticle (P1) were immersed in the solution of HDTMS in 4% ethanol which was stirred for 6 hours prior to use. The immersion of samples in HDTMS solution was performed for 60 minutes at room temperature using a shaker at 155 rpm. Samples of P0 and P1 that have been reacted with HDTMS were dried with a blow dryer and then continued by curing at 110^{o}C for 60 minutesto obtain the sample of polyester - HDTMS (P2),and of polyester - nanoparticle - HDTMS (P3). Then the sample of P2 was re-immersed in the silver nanoparticle to form polyester - HDTMS - nanoparticle (P4). Each polyester sample was prepared for 5 replications.

2.7. Characterization of Silver Nanoparticle with UV-Vis Spectrophotometer

The spectrum of colloidal silver nanoparticles was analyzed at a wavelength range of 200-600 nm.

2.8. Characterization of Polyester

Characteristic of unmodified polyester and modified polyester was determined by analysis of functional groups, observation of surface images, hydrophobic property, and antibacterial activity. Analysis of functional group of unmodified polyester and modified polyester was performed by using Attenuated Total Reflection (ATR) - FourierTransformInfrared (FTIR) spectrophotometer and observation of surface photo by Scanning Electron Microscopy (SEM).

2.8.1. Test of Hydrophobicity

Characterization of hydrophobic properties was perfomed with sessile drop method as has been previously conducted [16]. The water was dropped from a height of 1 cm from a sample with a volume of 0.01 mL. After the water was dripped then a water droplet image on the surface of the cloth was photographed using a camera. The contact angle difference of the five polyester fiber samples was done by using Corel Draw. Each polyester sample was characterized for 3 replications.

2.8.2. Test of Antibacterial Activity of Polyester Cloths

The antibacterial activity of P0, P1, P2, P3, and P4 were performed against *Staphylococcus aureus* ATCC 25923 as gram-positive bacteria and against *Escherichia coli* ATCC 35218 as gram-negative bacteria as described in the previous work [16, 17]. *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923 were rejuvenated on an agar medium of nutrient agar (NA). Inoculated *Escherichia coli* ATCC 35218 and *Staphylococcus aureus*ATCC 25923 were rejuvenated into a liquid nutrient broth (NB) medium. Samples of polyester, polyester - nanoparticle, polyester - HDTMS, polyester

- nanoparticle - HDTMS, and polyester - HDTMS - nanoparticle were cut off using paper piercing with diameter of 6 mm,and placed on microbial cultures in the petridish. The diameter of the clear zone of samples were measured using a sliding range every 6 hours for 72 hours. Antibacterial activity of each polyester sample was measured for 3 replications. Activity of unmodified polyester and modified polyester in inhibiting the bacterial growth is indicated by the diameter of inhibition zone around the sample pieces.

2.9. Test of Statistic

These results were analyzed using a quantitative descriptive method with ANOVA, Least Significant Different (LSD), and t-independent. The ANOVA test is conducted to determine the significant differences in antibacterial activity of the five polyester samples, to study the effect of treatment or modification and incubation time toward the antibacterial activity. This was continued with LSD. The LSD test was performed to find out the differences in antibacterial activity of different samples. Tests of t-independent were performed to determine the differences in antibacterial activity of the five samples against *Escherichia coli* and *Staphylococcus aureus* bacteria by using software IBM SPSS.

3. RESULTS AND DISCUSSION

3.1. Characteristic of Silver Nanoparticle

Bioreductor in this study are peel extract of *Ipomoea batatas L*. (Figure 1) which reduced $AgNO_3$ to nanoparticles and polyvinyl alcohol (PVA) as stabilizer agent. The colloids of silver nanoparticle isreddish brown color as in Figure 2. The UV-Vis spectrum (Figure 3 (a)) shows the absorbance of a 10^{-3} M of silver nitrate solution at a wavelength of 218.50 nm. Figure 3 (b) is UV-Vis spectrum of the colloidal silver nanoparticles. A peak appears at a wavelength of 436 nm and diameter of silver particle of 61.8 nm. This absorption peak indicates that the Ag^+ has been reduced to Ag^o. The silver nanoparticles have a characteristic of surface plasmon resonance (SPR) peak at 433 nm with a reddish brown

colour [24]. Colloidal silver nanoparticles provide a peak at wavelengths of around 400-500 nm which is a typical peak uptake of silver nanoparticles.The UV-Vis spectra of silver nanoparticles showed absorption peak in the range of 400 - 450 nm [6].

Thus, the synthesis of nanoparticles can be done successfully by utilizing plant extracts, especially skin extract of *Ipomoea batatas L*. The process of reduction of Ag^{+} ions into nanoparticles occurs outside the cell.The extracellular synthesis of silver nanoparticles could be conducted economically and safely by using leaf extract of *Catharanthus roseus Linn. G. Don* [25]. The extracellular synthesis was done by reacting metal ions with water or plant extracts. The use of plants in the synthesis of silver nanoparticles is relatively simpler and cheaper compared to other microorganisms. The types of plants that have been used for nanoparticle synthesis have been widely practiced by researchers. The biomolecule components contained in the plant are thought to act as a reducing agent, solvents and stabilizers in the formation of silver nanoparticles such as flavonoids, terpenoids, polysaccharides, alkaloids, and other secondary metabolites [25]. Three varieties of *Ipomoea batatas L.* have a major anthocyanin of cyanidin type [26]. However, anthocyanins can be oxidized to quinone. Thus it can be disclosed that in the process of forming silver nanoparticles using peel extracts of *Ipomoea batatas L* occurs reactions between hydroxyl groups (-OH phenolic) in anthocyanin compounds with silver ions to produce silver nanoparticles and quinone that cause the color of the solution to be reddish brown.

Each plant produces a variety of secondary metabolites. One of the most important groups of metabolites is a phenolic compound. Phenolics have at least one aromatic ring (C6) carrying a hydroxyl group. The antioxidant properties of phenolic compounds through their tendency to metal [3]. Based on the standard potential value, reaction (1) can be written as reactions (2) and (3). The sum of reaction (2) and (3) yields a cell potential of -0.445 volts. The negative cell potential value indicates that this redox reaction can’t occur spontaneously.

$$Ag^{+}_{(aq)} + \text{-OH phenolic}_{(aq)} + H_2O_{(l)} \longrightarrow Ag^{0}_{(s)} + \text{quinone}_{(aq)} + H^{+}_{(aq)} + O_{2(g)} \quad (1)$$

$$Ag^{+} + e \longrightarrow Ag^{0} \qquad E^{o} = 0.799 \text{ volt} \quad (2)$$

$$2\,H_2O \longrightarrow 4H^{+} + O_2 + 4e \qquad E^{o} = -1.224 \text{ volt} \quad (3)$$

Although the cell potential of netreaction is negative, the Ag^{+}can be reduced to Ag^{0} in the presence of –OH phenolic from plant extracts (a) (Figure 4). A complex formed (c2) (Figure 4) by the reaction between the -OH group of phenolic and the silver ion has an important role in the reduction and formation of silver nanoparticles. Many researchers concluded that complexes formed from citrate ion and silver ion will catalyze the reduction of Ag^{+} to Ag^{O} even slowly in chemical procedures. The probability of reaction mechanism in extracellular synthesis of silver nanoparticle by supporting peel extract of *Ipomoea batatas L*. is shown in Figure 4. Anthocyanin has a structure as shown in 4(a), and simple structure is shown in 4(b) and 4(c1). When silver ion is reduced to form silver, -OH anthocyanin is oxidized to C=O quinon (c3).

3.2. SEM Image, Functional Groups, and Water Contact Angle of Polyester

SEM images of unmodified and modified polyesters (Figure 5) show that fabrics are composed of fibril. Figure 5(a) shows no contaminants on the surface of unmodified polyester fabrics. The modified polyester shows the presence of silver nanoparticles deposited on the surface of the polyester fabric (Figure 5(b) and 5(c)). Some of the microparticles cover the surface of the fabric, since the nanoparticles undergo agglomeration to form larger particles [22]. In this study, silver nanoparticles successfully cover the surface of polyester fabric. The addition of silver nanoparticles results in a rougher cloth surface than a cloth without the addition of nanoparticles. However the surface between pure polyester (P0) and the modified polyester (P3) are practically no different. This could be caused by the HDTMS layer covered the silver nanoparticle so that the nanoparticle of silver can't be detected on surface images of P3, whereas in P4 the layer of HDTMS was covered by the

silver nanoparticles so that the surface images are different, the silver nanoparticles are very much on the surface of the cloth P4.

ATR-FTIR spectra of polyester (Figure 6) shows that polyester contains functional groups of -OH, -C-H, -COO esther, C=O, and C-O. Modification with silver nanoparticle and HDTMS compound does not show a new functional group. Thus, it does not effect the functional groups of polyester. This result is in accordance with the previous studies on the modification of cotton cloth with silver nanoparticles and HDTMS compounds [16]. The addition of the HDTMS compound can decrease absorption band in the spectrum of FTIR of modified Nylon [16].

The data of the contact angle in Figure 7 shows that the addition of one of the silver nanoparticles and HDTMS might increase the contact angle of the polyester fabric. However, the addition of HDTMS and the silver nanoparticles produces the highest contact angle on the resulting polyester. This is in contrast to the previous research [16, 17], that the addition of two types of compounds produced the highest contact angles. Note that the method of modification is different, in the previous research modifications were done with the addition of silver nanoparticle first and then followed with the addition of HDTMS. While in this study, the addition of HDTMS was firstly performed and then the addition of silver nanoparticle. Thus the modification method seems to affect the properties of the fabric produced. The polyester after modification with HDTMS compound and silver nanoparticles has the largest contact angle value. The presence of a hydrophobic functional group increases the angle contact of water. The method of modification of the polyester fabric, qualitatively does not change the type of functional group present, but that affects the number of hydrophobic groups. Thus, the addition of nanosilver particles to hydrophobic materials, such as polyester composite, caused an increase of the hydrophobic groups and a decrease in surface free energy, so that an increase of the contact angle of P4.

3.3 Antibacterial Activity of Polyester

The lowest inhibition zone of polyester in inhibiting the growth of *E. coli* bacteria is the P0 sample and the highest activity is the modified polyester (polyester – HDTMS) as shown in Fig.8. The highest inhibitory zone diameter of sample P-HDTMS occurs at the 60th hour. Incubation for 60 minutes is the maximum time for modified polyesters to inhibit bacterial growth of *E. coli*. Then the antibacterial activity decreases to the measurement at 72th hour. After 60 min, the ability of polyester in inhibiting bacterial growth decreased.

The P-HDTMS has the highest ability to inhibit the growth of *E. coli* compared to other polyester samples. Thus, gram-negative bacteria are more easily inhibited by modified polyesters by silane compounds. The hydrophobic functional groups are more able to inhibit gram-negative bacteria. Modification of polyester by adding HDTMS compound can increase antibacterial activity of polyester cloths. The interaction between the ester groups and / or the –OH groups in the polyester and the -OH group in HDTMS produces a hydrophobic functional group so that the hydrophobic surface may cause the bacteria to inhibit its growth when interacting with the polyester fabric [27]. Compounds containing hydrophilic and hydrophobic groups are antibacterial agents that can damage cytoplasmic membranes and kill the bacterial cells [16]. The -OH functional groups (hydrophilic) from the polyester cloth and the HDTMS and hydrophobic groups of modified polyester bound to the silver will interact to -S-H on the cytoplasmic membrane by ionic or covalent bonds to form a bond -S-S- and produce S-Ag clusters that cause inhibition of cell respiration.

The lowest inhibition zone of polyesterin inhibiting the growth of *S. aureus* is shown by unmodified polyester (P) and the highest activity is shown by the P-HDTMS-Ag (Fig.9). In general, the graph shows the antibacterial activity of the polyester sample which tends to increase with the time of incubation, which then decreases after 60 hours of incubation. The highest inhibition zone diameter of sample of P-HDTMS-Ag occurs at the

60th hour. Thus, incubation for 60 minutes is the maximum time for modified polyesters to inhibit bacterial growth of *S. aureus*. P-HDTMS-Ag shows the highest antibacterial activity. This is due to the interaction between ester and or –OH groups with –OH groups of HDTMS (Fig. 10) resulting in hydrophobic groups which are not favored by bacteria followed with modification by deposition of Ag nanoparticles, which is an antibacterial agent as well, thus increasing activity of antibacterial from polyester fabric. Thus, the polyester modification method using a combination of HDTMS and nanoparticles increases the antibacterial activity of the fabric. The P-HDTMS-Ag has the highest ability to inhibit the growth of *S. aureus* compared to other polyester samples. Thus, gram-positive bacteria are more easily inhibited by modified polyesters by silane compounds and followed by coating of silver nanoparticles. The presence of hydrophobic function groups and silver nanoparticles may inhibit gram-positive bacteria. In other words, gram-positive bacteria can be inhibited by polyester composites, this is because the gram-positive bacteria consist of a thick layer of peptidoglycan and a layer of teicoic acid and also tend to be negatively charged and polar, so that antibacterial material is required that can inhibit its growth in the form of composites polyester with HDTMS and silver nanoparticles.

Based on results of Anova test in Table 1, there are three tests, namely interaction test between incubation time and type of sample, effect of incubation time, and effect of sample type on antibacterial activity of polyester cloths. The significant value of the interaction test between the incubation time and the sample type was 0.000 ($p<0.05$) for both against *E.coli* and *S.aureus*. Interpretation of $p<0.05$ indicates the effect of interaction between incubation time and sample type on antibacterial activity. The significant value of effect test of the incubation time on antibacterial activity was 0.000 ($p <0.05$). The interpretation of $p <0.05$ was the effect of incubation time on antibacterial activity. The significant value of the sample effect test on antibacterial activity was 0.000 ($p< 0.05$). Interpretation of $p<0.05$ indicates the

influence of sample type on antibacterial activity. Thus, type of the polyester sample and the incubation time affect the antibacterial activity. The five types of polyester samples have different antibacterial activity significantly. Similarly with incubation time, different incubation times showed different antibacterial activity significantly as well.

The results of LSD tests in Table 2 show that the sample was a significant influence amongs the four modified samples with respect to P0 on the antibacterial activity (clear zone) for *Staphylococcus aureus* ATCC 25923, P0-P1, P0-P2, P0-P3 and P0-P4. All of the modified polyester shows significantly higher antibacterial activity than the unmodified polyester. Thus, modification can increase antibacterial activity of polyester cloths.

However, within the modified polyesters (P1-P4) no significant difference in theirs antibacterial activity was observed. Modification of polyester cloths by using silver nanoparticle, HDTMS, silver nanoparticle and HDTMS, and also HDTMS and silver nanoparticle has the similar effect toward antibacterial activity of polyester. Thus, the type of addition material and methods did not effect antibacterial activity of polyester cloths against *S.aureus*.

The results of LSD test in Table 2 shows that there are 7 variations of samples which have significant influence between two samples on antibacterial activity (clear zone) against *Escherichia coli* ATCC 35218, these are P0-P1, P0-P2 , P0-P3, P0-P4, P1-P3, P1-P4 and P2-P3. Therefore, it can be concluded that modification can affect antibacterial activity of polyester cloths. The addition of silver nanoparticles, HDTMS, and combinations of the two might enhance the antibacterial activity of unmodified polyester fabrics to inhibit the growth of *E.coli*.

Similarly, the addition method of nanoparticles and HDTMS or a combination of both might affect the antibacterial properties of polyester fabrics on inhibiting growth of *E.coli*. Considering that the differences in antibacterial activity between the following polyester

samples P1-P3, P1-P4 and P2-P3 are significantly different, it can be concluded that the modification of fabric with two chemicals in P3 and P4 shows significantly differences compared to modifications using a single material (only nanoparticles or HDTMS) in P1 and P2.

Based on the results of the t-independent test as shown in Table 3, each sample of the polyester fabric shows no difference in antibacterial activity to inhibit the growth of *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923. Thus,the ability each sample to inhibit the *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923 is the same. This can be due to the fact that each type of bacteria has almost the same of chemical content. The chemical composition in cell wall of gram positive bacteria and gram negative bacteria is almost the same, i.e the presence of peptidoglycan and lipopolysaccharide.

4. CONCLUSIONS

Based on the results of the research and discussion it could be concluded that the silver nanoparticles were successfully synthesized using a reducing agent of peel extract of *Ipomoea batatas L*. as confirmed at a wavelength of 436 nm with a reddish brown color. The unmodified polyester fabric has the lowest contact angle value while the polyester fabric with the modification of HDTMS compound and silver nanoparticles has the largest contact angle value. There is a difference in the antibacterial activity of the fabric between the unmodified polyester and the modified polyester fabric at different incubation times in inhibiting the growth of *Escherichia coli* and *Staphylococcus aureus*. Samples of polyester – HDTMS – silver nanoparticle show the highest antibacterial activity against *S. aureus* with a strength of about 1.4 times greater than unmodified polyester and it is the highest in hydrophobicity properties. Each polyester sample has the same ability in inhibiting the growth of *Staphylococcus aureus* and *Escherichia coli*. Based on the antibacterial properties

of the fabric then the modified polyester product of nanoparticles and HDTMS can be used as antibacterial materials for biomedical applications.

ACKNOWLEDGEMENT

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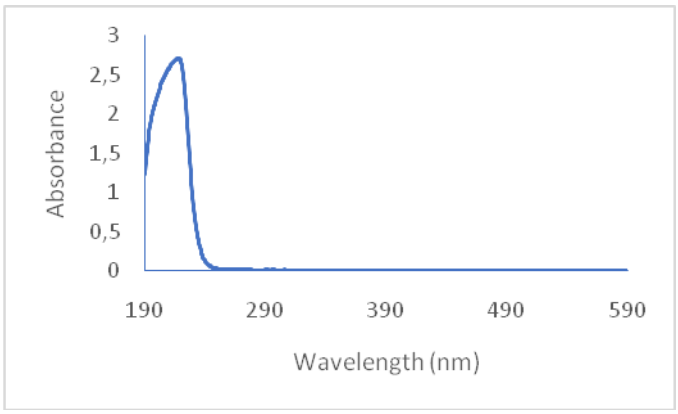
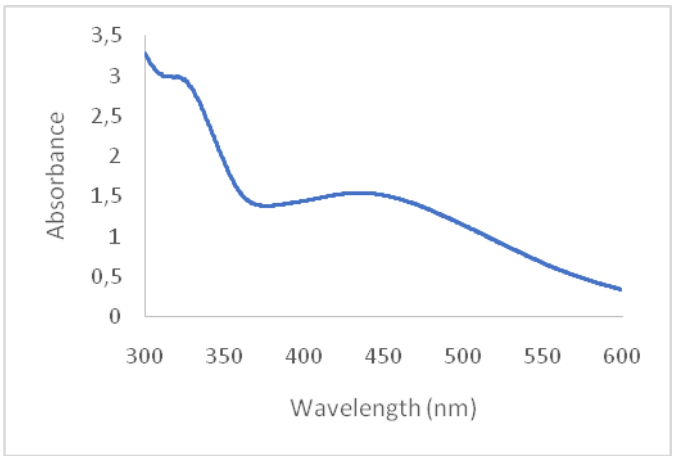
Figure 1.*Ipomoea batatas L.*

Figure 2. The colloidal of silver nanoparticle



(a)



(b)

Figure 3. UV-Vis spectrum of $AgNO_3$solution of $1x10^{-3}$ M (a) and silver nanoparticle (b)

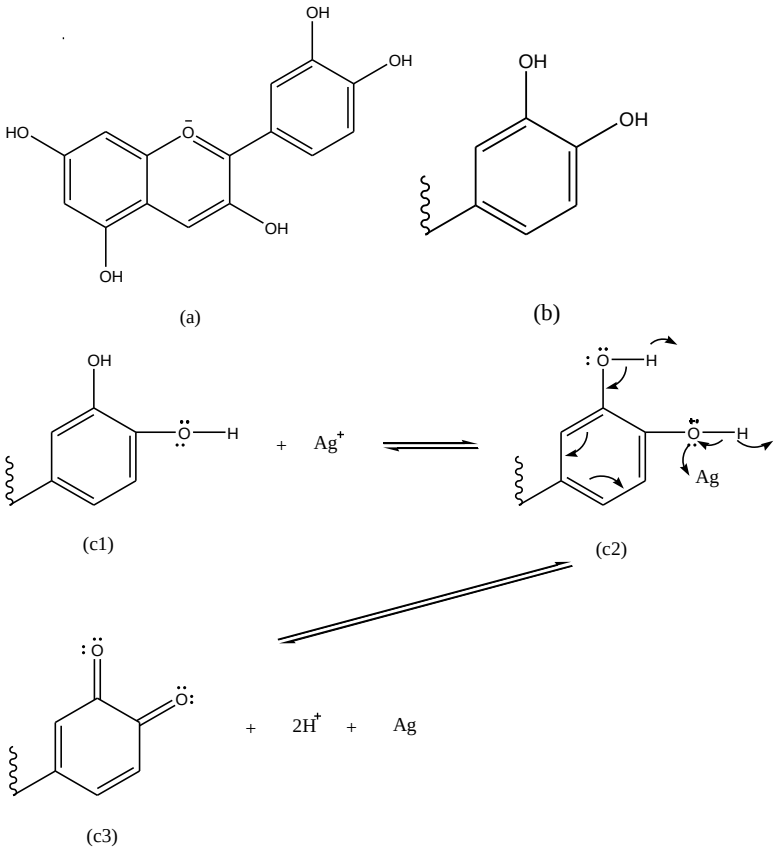
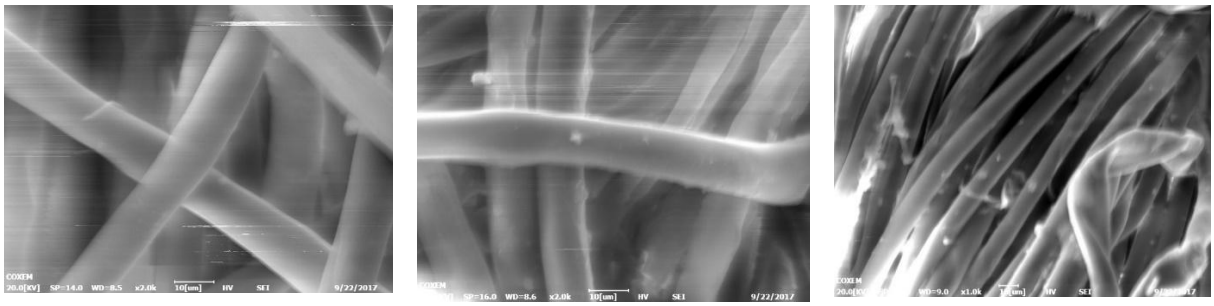
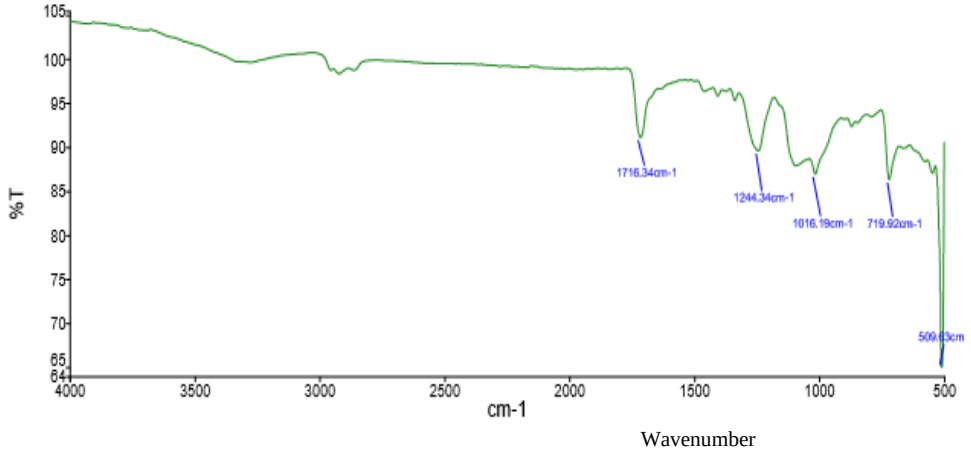


Figure 4. Reaction mechanism of formation the silver nanoparticles from anthocyanin (a) and silver ion

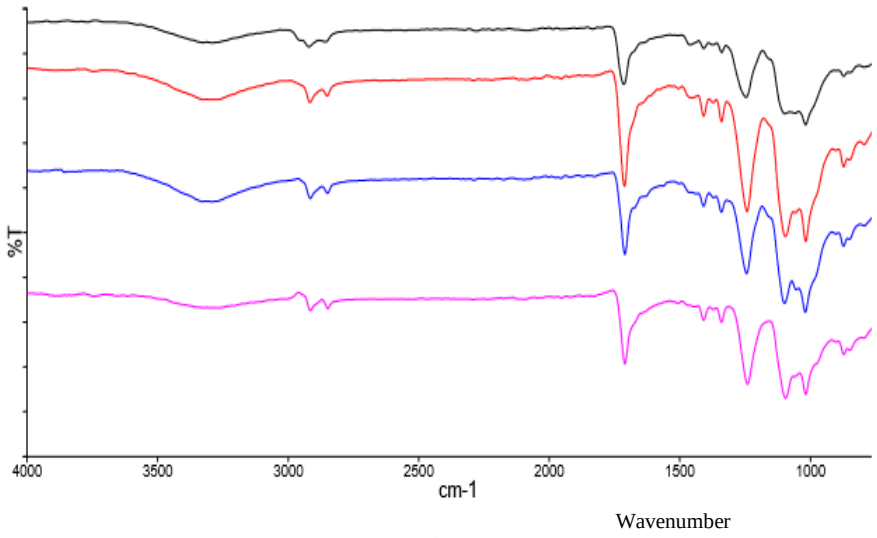


(a) (b) (c)

Figure 5. The SEM image of unmodified polyester (P0) (a), modified polyester (P3) (b), and P4 (c)

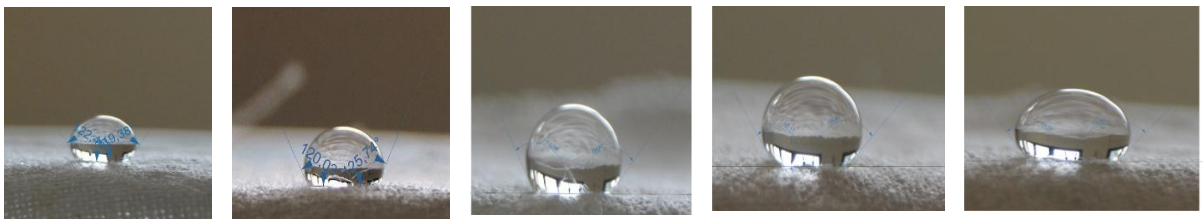


(a)



(b)

Figure 6. The ATR-FTIR spectra of unmodified polyester (P0)(a) and modified polyester (P1----, P2----, P3----, and P4----) (b)



θ = 120.92 θ = 122.89 θ = 128.63 θ = 130.08 θ = 133.29

(a) (b) (c) (d) (e)

Figure 7. The water contact angle for P0 (a), P1(b), P2 (c), P3(d), and P4(e)

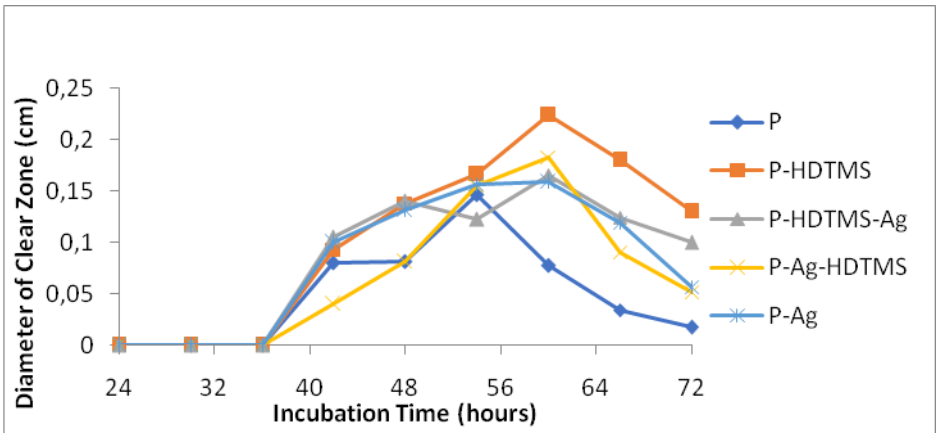


Figure 8. The antibacterial activity of polyester against *Escherichia coli* ATCC 35218

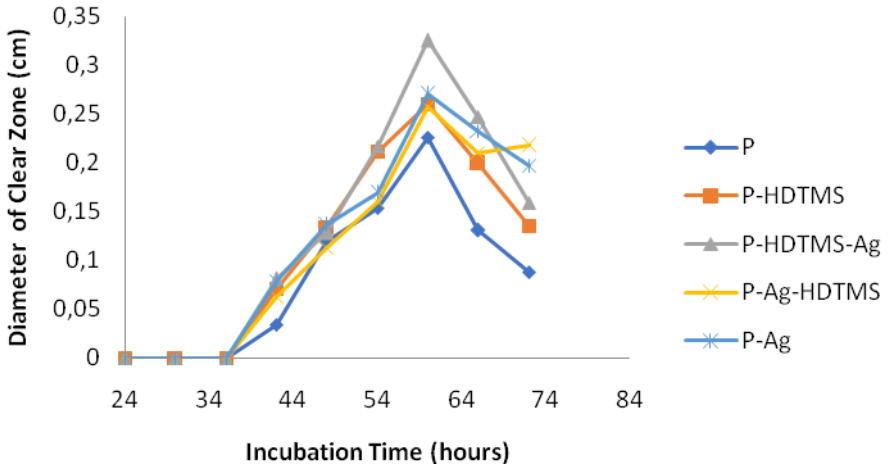


Figure 9. The antibacterial activity of polyester against *Staphylococcus aureus* ATCC 25923

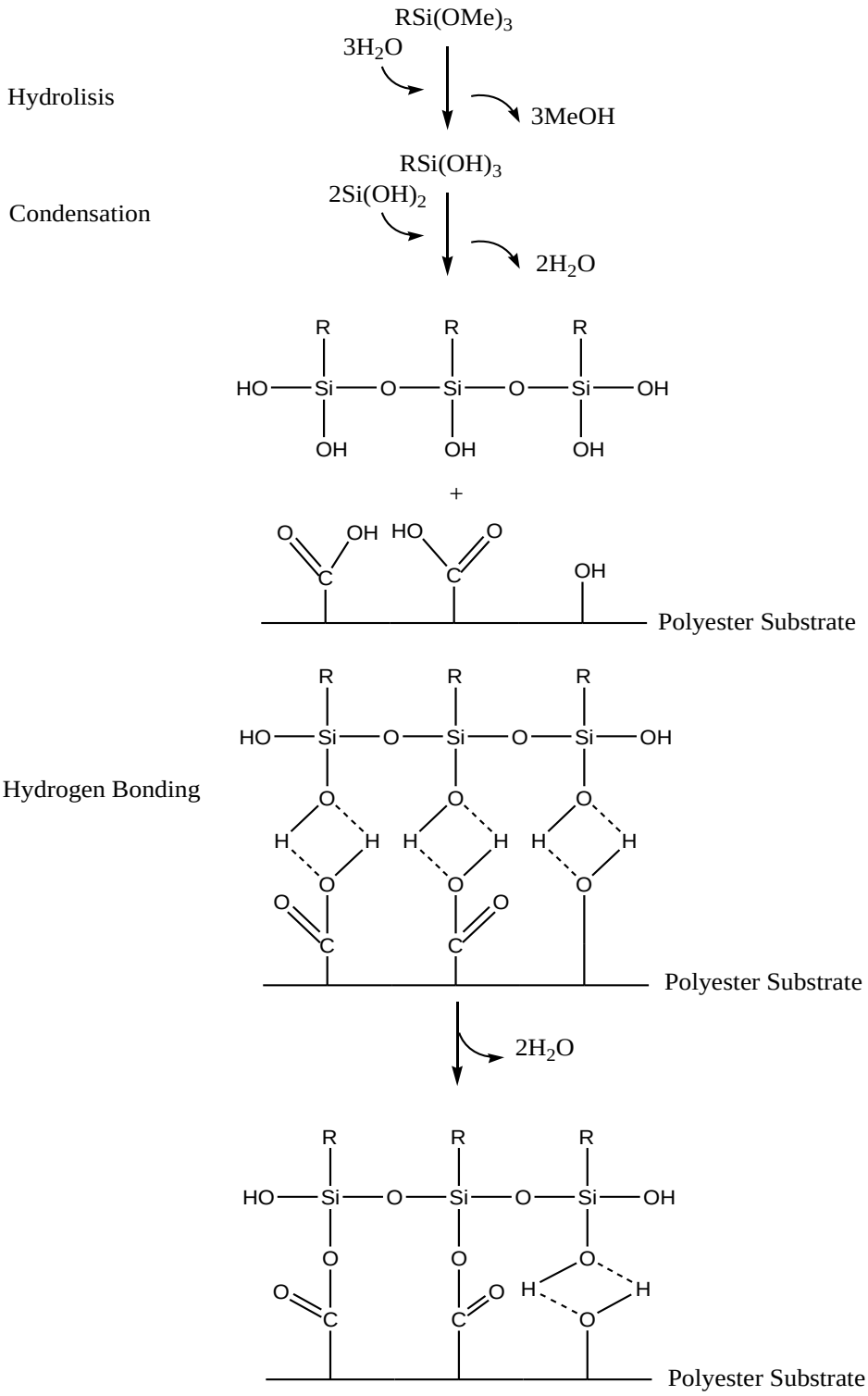


Figure 10. Interaction between the ester / the hydroxyl groups of polyester and the hydroxyl groups of HDTMS

Table 1. Significance of antibacterial activity of unmodified polyester and modified polyester against S*taphylococcus aureus* and *Escherichia coli*

Source of data	Sum	Df	Average	F	Sig.
Escherichia coli ATCC 35218					
Time	0.413	7	0.059	83.652	0.000
Sample	0.053	4	0.013	18.897	0.000
Time* Sample	0.057	28	0.002	2.880	0.000
Staphylococcus aureus ATCC 25923					
Time	0.990	7	0.141	138.091	0.000
Sample	0.035	4	0,.09	8.553	0.000
Time* Sample	0.052	28	0.002	1.821	0.000

Table 2. Antibacterial activity between 2 polyester samples against *Staphylococcus aureus* and *Escherichia coli*

Type of sample	Conclusion	
	Staphylococcus aureus	*Escherichia coli*
P0 – P1	Significant	Significant
P0 – P2	Significant	Significant
P0 – P3	Significant	Significant
P0 – P4	Significant	Significant
P1 – P2	Not significant	Not significant
P1 – P3	Not significant	Significant
P1 – P4	Not significant	Significant
P2 – P3	Not significant	Significant
P2 – P4	Not significant	Not significant
P3 – P4	Not significant	Not significant

Table 3. Interpretation of t-independent test for activity antibacterial of polyester against *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923

Polyester sample	t-independent
P0	No difference
P1	No difference
P2	No difference
P3	No difference
P4	No difference

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Contributed Paper

Application of Silver Nanoparticles Synthesized by Using *Ipomoea batatas L.* Waste to Improve Antibacterial Properties and Hydrophobicity of Polyester Cloths

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ABSTRACT

Synthesis of colloidal silver nanoparticle by using peel extracts of *Ipomoea batatas L.*, its deposition on polyester cloths, and the modification with hexadecyltrimethoxysilane (HDTMS) have been conducted in this work. The silver nanoparticles were characterized by using Uv-Vis spectrophotometer, functional groups of unmodified polyester and those of modified polyester were characterized by ATR-FTIR spectrophotometer, surface images of polyester were observed by Scanning Electron Microscopy tool, antibacterial activity of unmodified polyester cloth and modified polyester against *Staphylococcus aureus* and *Escherichia coli* were determined with a diffusion method, and hydrophobicity of polyester was measured by using asessile drop method. Silver nanoparticles were successfully produced using peel extracts of *Ipomoea batatas L* as indicated by the absorption peaks at 436 nm. SEM images confirmthat the silver nanoparticles coat onto polyester cloths. Modification with nanoparticle and HDTMS do not affect the functional groups of polyester. The polyester fabric with the addition of HDTMS compound and silver nanoparticles showedthe largest contact angle and the antibacterial activity to inhibit *Staphylococcus aureus*. Samples ofpolyester - HDTMS - silver nanoparticle show the highest antibacterial activity against*S. aureus*with a strength 1.4 times greater than unmodified polyester. There is a difference in the antibacterial activity of the polyester among the unmodified polyester and the modified polyester fabric at different incubation times inhibiting the growth of *Escherichia coli* and *Staphylococcus aureus*. Each sample ofthe unmodified polyester and the modified polyester fabric shows the same ability to inhibit the growth of *Staphylococcus aureus* and *Escherichia coli*.

Keywords: Antibacterial Activity, Hydrophobicity, *Ipomoea batatas L.*, Polyester, Silver Nanoparticles

1. INTRODUCTION

Peel of purple sweet potato is an untapped waste, however, the anthocyanin content in a peelof the purple sweet potato is actually still high,being 110.51 mg/100 g. Pigments of anthocyanins in *Ipomoea batatas L.* are more stable than anthocyanins from other sources, such as red cabbage, elderberries, blueberries and red corn [1]. However, anthocyanins contain phenolic hydroxyl groups which are easily oxidized into quinones, and thus skin extract of *Ipomoea batas L.* can be used as a reducing agent to convert silver ions (Ag^{+}) into silver nanoparticles (Ag^{0}). The reduction of particle size to nano-size of silver can be caused by the H radicals formed in phenolic compounds that are antioxidants [2]. Phenolics have hydroxyl and carbonyl groups that can bind metals. The antioxidant properties of phenolic compounds might occur through their tendency to chelate metal [3]. The efficiency of the synthesis of metal nanoparticles depends on the electrochemical potential of the reduced ions. Plant extracts effectively reduce metal ions more significantly for ions that have high positive electrochemical potentials such as Ag^{+} [4].

Nanoparticles can be synthesized using various methods, for examples using microwave irradiation method [5], reduction method [6], photochemical or photosynthesis method [7, 8], ball milling method [9], electrochemical method [10], biosynthesis by using actinomycete or actinobacteria [11, 12], and biosynthesis by using dried fruit extract method [13]. In this study, silver nanoparticles were prepared by applying green chemistry using skin extract of sweet purple potato (*Ipomoea batatas L.*). Utilization of plants to synthesize nanoparticles can be done based on the ability of these plants to absorb metal ions from the environment. The ions will be reduced through complex metabolic processes and accumulated in certain organs. Plants are known to have organic compounds that serve as reducing agents that can be used to replace or complement inorganic reducers. The use of plant organic compounds for the synthesis of nanoparticles is known as biosynthesis and is an eco-friendly method, as well as simpler and more efficient than chemical procedures [14].

The process of applying nanoparticles to textile fibers is done by composting nanometer-scale particles into textile fibers. The three-dimensional nanostructured surface particles and gel-producing additives produce hydrophobic fabric products that do not reduce the breathability and comfort of the fabric when worn. However, the use of silane compounds causes the impurities attached to the fabric to be easily released when watered, however, the fabric remains dry.

Textile materials developed into self-cleaning textile and antibacterial products in this workare polyester fabrics. Naturally, polyester fibers have hydrophobic properties as well as apparel materials, polyester fibers are also commonly used as materials for sportwear, underwear and sheets [15]. The antibacterial polyester can be developed through modification with silver nanoparticles. Antibacterial textiles have been developed by modifying textile materials with nanoparticles and vegetable oil, such as nylon [16], cotton [17], silk cloths [6], polyurethane [18], leather [19], and wool fiber [20].

Polyester with self-cleaning properties can be developed through modification with silane compounds. Several types of silane compounds used to modify materials are hexamethyldisilazane [21], octyltriethoxysilane [22], and hexadecyltrimethoxysilane [16-17,

23]. The addition of silane-based molecules of HDTMS compounds to textiles can increase water contact angle or hydrophobicity of the fabric [16]. The objectives of our research were to synthesize silver nanoparticle by using peel extract of *Ipomoea batatas L.*, to modifypolyester cloths by coating silver nanoparticles and HDTMS, and to study hydrophobicity property of polyester, and also to determine antibacterial activity of unmodified polyester and modified polyester.

2. MATERIALS AND METHODES

2.1 Materials

Silver nitrate (E-Merck), polyvinyl alcohol (PVA) with molar mass 29,365.96 g/mol (E-Merck), ethanol, acetone, and hexadecyltrimethoxysilane (Aldrich) were purchased as commercial products and used without any further purification. Polyester cloth was purchased from a store in Yogyakarta, Indonesia. Nutrient Agar (NA) and Nutrient Broth (NB) were purchased from Oxoid. *Staphylococcus aureus* ATCC 25923 and *Escherichia coli* 32518 were obtained from a collection of Faculty of Medicine, Universitas Gadjah Mada (UGM) Yogyakarta, Indonesia.

The research was conducted in the following stages: extraction of *Ipomoea batatas L.* peel, synthesis of Ag nanoparticle by using bioreductor, addition of colloidal silver nanoparticle on polyester cloths, modification of polyester cloths by adding HDTMS and colloidal silver nanoparticle, and characterization of unmodified polyester and modified polyester.

2.2 Apparatus

Ultra Violet - Visible (Uv-Vis) spectrophotometer (Shimadzu UV2450, Japan) wasused to study thesuccessful formation of colloid of silver nanoparticle by observing absorption peaks of silver nitrate solution and colloidal silver nanoparticle. Attenuated Total Reflection - Fourier Transfrom Infra-red (FTIR) spectra were recorded on a FTIR - ATR spectrophotometer (Shimadzu prestige 21, Japan). Morphological images of unmodified polyester and modified polyester were observed by using a scanning electron microscope tool (SEM Jeol T300, USA).

2.3 Preparation of Peel Extract

A total of 20 grams of *Ipomoea batatas L.* peel was freshly cleaned with aquadest and inserted it into a 500 mL beaker glass. About 100 mL of distilled water was added into it and then boiled for 15 minutes. Allowing to stand the mixture until room temperature after boiling, the extract of *Ipomoea batatas L.* skin was then filtered using Whatmann no. 42.

2.4 Biosynthesis of Silver Nanoparticle

About 1 mL of peel extract of *Ipomoea batatas L.*in an erlenmeyer was added into 40 mL of 1×10^{-3} M silver nitrate solution. The mixture was allowed to stand for 2 hours to react. To the mixture, 12 mL of 1% PVA solution was added and then it was allowed to stand for 1 hour. Finally, the solution was stirred for 3 days to form colloidal silver nanoparticles. The colloid was then characterized using a UV-Vis spectrophotometer.

2.5 Deposition of Nanoparticle on Polyester Cloths

The sample of polyester fiber (P0) with size 5 cm × 5cmwas soaked in colloidal silver nanoparticles in erlenmeyer 50 mL and shaked using a shaker at 155 rpm for 24 hours. It was then dried at room temperature for one day. Thus, a polyester-nanoparticle sample (P1) was produced.

2.6 Modification of Polyester Cloth with HDTMS

The polyester (P0) and polyester - nanoparticle (P1) were immersed in the solution of HDTMS in 4% ethanol which was stirred for 6 hours prior to use. The immersion of samples in HDTMS solution was performed for 60 minutes at room temperature using a shaker at 155 rpm. Samples of P0 and P1 that have been reacted with HDTMS were dried with a blow dryer and then continued by curing at 110 °C for 60 minutesto obtain the sample of polyester -HDTMS (P2),and of polyester - nanoparticle - HDTMS (P3). Then the sample of P2 was re-immersed in the silver nanoparticle to form polyester - HDTMS - nanoparticle (P4). Each polyester sample was prepared for 5 replications.

2.7 Characterization of Silver Nanoparticle with UV-Vis Spectrophotometer

The spectrum of colloidal silver nanoparticles was analyzed at a wavelength range of 200-600 nm.

2.8 Characterization of Polyester

Characteristic of unmodified polyester and modified polyester was determined by analysis of functional groups, observation of surface images, hydrophobic property, and antibacterial activity. Analysis of functional group of unmodified polyester and modified polyester was performed by using Attenuated Total Reflection (ATR) - FourierTransformInfrared (FTIR) spectrophotometer and observation of surface photo by Scanning Electron Microscopy (SEM).

2.8.1 Test of hydrophobicity

Characterization of hydrophobic properties was perfomed with sessile drop method as has been previously conducted [16]. The water was dropped from a height of 1 cm from a sample with a volume of 0.01 mL. After the water was dripped then a water droplet image on the surface of the cloth was photographed using a camera. The contact angle difference of the five polyester fiber samples was done by using Corel Draw. Each polyester sample was characterized for 3 replications.

2.8.2 Test of antibacterial activity of polyester cloths

The antibacterial activity of P0, P1, P2, P3, and P4 were performed against *Staphylococcus aureus* ATCC 25923 as gram-positive bacteria and against *Escherichia coli* ATCC 35218 as gram-negative bacteria as described in the previous work [16, 17]. *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923 were rejuvenated on an agar medium of nutrient agar (NA). Inoculated *Escherichia coli* ATCC 35218 and *Staphylococcus aureus*ATCC 25923 were rejuvenated into a liquid nutrient broth (NB) medium. Samples of polyester, polyester - nanoparticle, polyester - HDTMS, polyester - nanoparticle - HDTMS, and polyester - HDTMS - nanoparticle were cut off using paper piercing with diameter of 6 mm, and placed on microbial cultures in the petridish. The diameter of the clear zone of samples were measured using a sliding range every 6 hours for 72 hours. Antibacterial activity of each polyester sample was measured for 3 replications. Activity of unmodified polyester and modified polyester in inhibiting the bacterial growth is indicated by the diameter of inhibition zone around the sample pieces.

2.9 Test of Statistic

These results were analyzed using a quantitative descriptive method with

ANOVA, Least Significant Different (LSD), and t-independent. The ANOVA test is conducted to determine the significant differences in antibacterial activity of the five polyester samples, to study the effect of treatment or modification and incubation time toward the antibacterial activity. This was continued with LSD. The LSD test was performed to find out the differences in antibacterial activity of different samples. Tests of t-independent were performed to determine the differences in antibacterial activity of the five samples against *Escherichia coli* and *Staphylococcus aureus* bacteria by using software IBM SPSS.

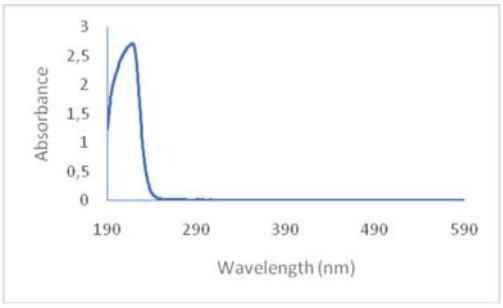
3. RESULTS AND DISCUSSION

3.1 Characteristic of Silver Nanoparticle

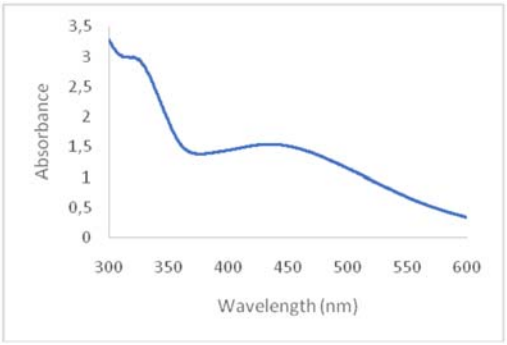
Bioreductor in this study are peel extract of *Ipomoea batatas L.* (Figure 1) which reduced $AgNO_3$ to nanoparticles and polyvinyl alcohol (PVA) as stabilizer agent. The colloids of silver nanoparticle isreddish brown color as in Figure 2. The UV-Vis spectrum (Figure 3 (a)) shows the absorbance of a 10^{-3} M of silver nitrate solution at a wavelength of 218.50 nm. Figure 3 (b) is UV-Vis spectrum of the colloidal silver nanoparticles. A peak appears at a wavelength of 436 nm and diameter of silver particle of 61.8 nm. This absorption peak indicates that the Ag^+ has been reduced to Ag°. The silver nanoparticles have a characteristic of surface plasmon resonance (SPR) peak at 433 nm with a reddish brown colour [24]. Colloidal silver nanoparticles provide a peak at wavelengths of around 400-500 nm which is a typical peak uptake of silver nanoparticles.The UV-Vis spectra of silver nanoparticles showed absorption peak in the range of 400 - 450 nm [6].

Figure 1. *Ipomoea batatas L.*

Figure 2. The colloidal of silver nanoparticle.



(a)



(b)

Figure 3. UV-Vis spectrum of $AgNO_3$ solution of 1×10^{-3} M (a) and silver nanoparticle (b).

Thus, the synthesis of nanoparticles can be done successfully by utilizing plant extracts, especially skin extract of *Ipomoea batatas L.* The process of reduction of Ag^+ ions into nanoparticles occurs outside the cell. The extracellular synthesis of silver nanoparticles could be conducted economically and safely by using leaf extract of *Catharanthus roseus Linn. G. Don* [25]. The extracellular synthesis was done by reacting metal ions with water or plant extracts. The use of plants in the synthesis of silver nanoparticles is relatively simpler and cheaper compared to other microorganisms. The types of plants that have been used for nanoparticle synthesis have been widely practiced by researchers. The biomolecule components contained in the plant are thought to act as a reducing agent, solvents and stabilizers in the formation of silver nanoparticles such as flavonoids, terpenoids, polysaccharides, alkaloids, and other secondary metabolites [25]. Three varieties of *Ipomoea batatas L.* have a major anthocyanin of cyanidin type [26]. However, anthocyanins can be oxidized to quinone. Thus it can be disclosed that in the process of forming silver nanoparticles using peel extracts of *Ipomoea batatas L* occurs reactions between hydroxyl groups (-OH phenolic) in anthocyanin compounds with silver ions to produce silver nanoparticles and quinone that cause the color of the solution to be reddish brown.

Each plant produces a variety of secondary metabolites. One of the most important groups of metabolites is a phenolic compound. Phenolics have at least one aromatic ring (C6) carrying a hydroxyl group. The antioxidant properties of phenolic compounds through their tendency to metal [3]. Based on the standard potential value, reaction (1) can be written as reactions (2) and (3). The sum of reaction (2) and (3) yields a cell potential of -0.445 volts. The negative cell potential value indicates that this redox reaction can't occur spontaneously.

$$Ag^+_{(aq)} + \text{-OH phenolic}_{(aq)} + H_2O_{(l)} \rightarrow Ag^0_{(s)} + \text{quinone}_{(aq)} + H^+_{(aq)} + O_{2(g)} \quad (1)$$

$$Ag^+ + e \rightarrow Ag^0 \quad E^\circ = 0.799 \text{ volt} \quad (2)$$

$$2\,H_2O \rightarrow 4H^+ + O_2 + 4e \quad E^\circ = -1.224 \text{ volt} \quad (3)$$

Although the cell potential of netreaction is negative, the Ag^+can be reduced to Ag^0 in the presence of -OH phenolic from plant extracts (a) (Figure 4). A complex formed (c2) (Figure 4) by the reaction between the -OH group of phenolic and the silver ion has an important role in the reduction and formation of silver nanoparticles. Many researchers concluded that complexes formed from citrate ion and silver ion will catalyze the reduction of Ag^+ to Ag^O even slowly in chemical procedures. The probability of reaction mechanism in extracellular synthesis of silver nanoparticle by supporting peel extract of *Ipomoea batatas L.* is shown in Figure 4. Anthocyanin has a structure as shown in 4(a), and simple structure is shown in 4(b) and 4(c1). When silver ion is reduced to form silver, -OH anthocyanin is oxidized to C=O quinon (c3).

3.2 SEM Image, Functional Groups, and Water Contact Angle of Polyester

SEM images of unmodified and modified polyesters (Figure 5) show that fabrics are composed of fibril. Figure 5(a) shows no contaminants on the surface of unmodified polyester fabrics. The modified polyester shows the presence of silver nanoparticles deposited on the surface of the polyester fabric (Figure 5(b) and 5(c)). Some of the microparticles cover the surface

of the fabric, since the nanoparticles undergo agglomeration to form larger particles [22]. In this study, silver nanoparticles successfully cover the surface of polyester fabric. The addition of silver nanoparticles results in a rougher cloth surface than a cloth without the addition of nanoparticles. However the surface between pure polyester (P0) and the modified polyester (P3) are practically no different. This could be caused by the HDTMS layer covered the silver nanoparticle so that the nanoparticle of silver can't be detected on surface images of P3, whereas in P4 the layer of HDTMS was covered by the silver nanoparticles so that the surface images are different, the silver nanoparticles are very much on the surface of the cloth P4.

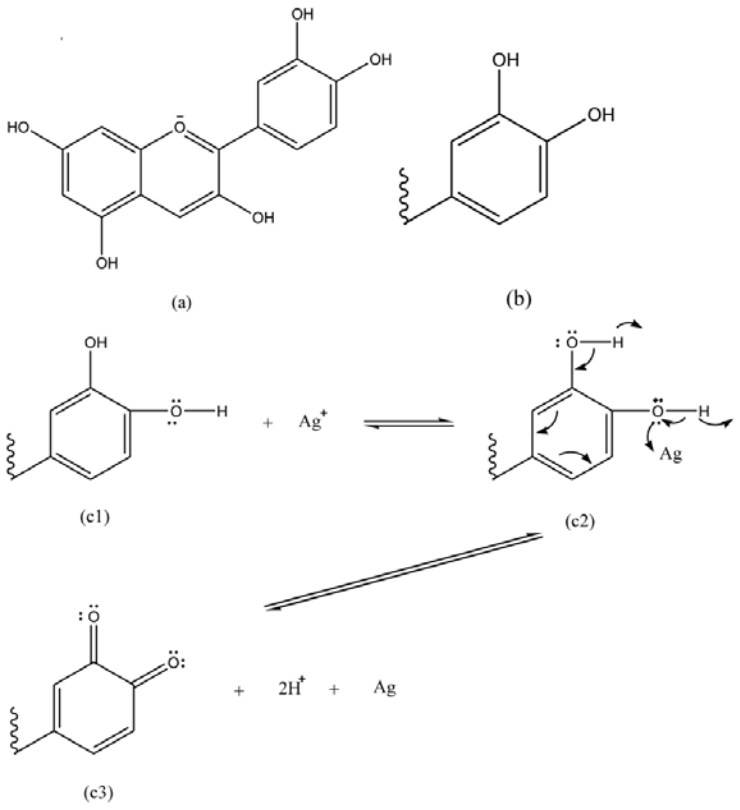


Figure 4. Reaction mechanism of formation the silver nanoparticles from anthocyanin (a) and silver ion.

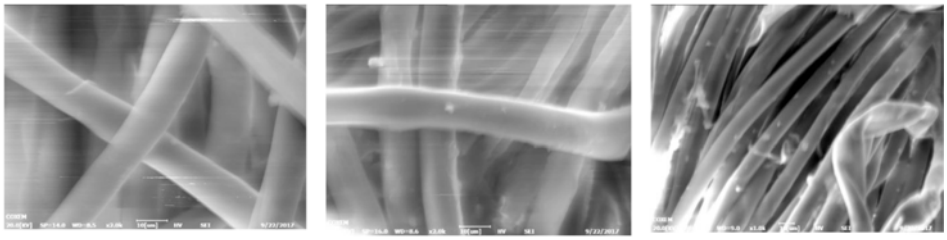


Figure 5. The SEM image of unmodified polyester (P0) (a), modified polyester (P3) (b), and P4 (c).

ATR-FTIR spectra of polyester (Figure 6) shows that polyester contains functional groups of -OH, -C-H, -COO esther, C=O, and C-O. Modification with silver nanoparticle and HDTMS compound does not show a new functional group. Thus, it does not effect the functional groups of polyester. This result is in accordance with the previous studies on the modification of cotton cloth with silver nanoparticles and HDTMS compounds [16]. The addition of the HDTMS compound can decrease absorption band in the spectrum of FTIR of modified Nylon [16].

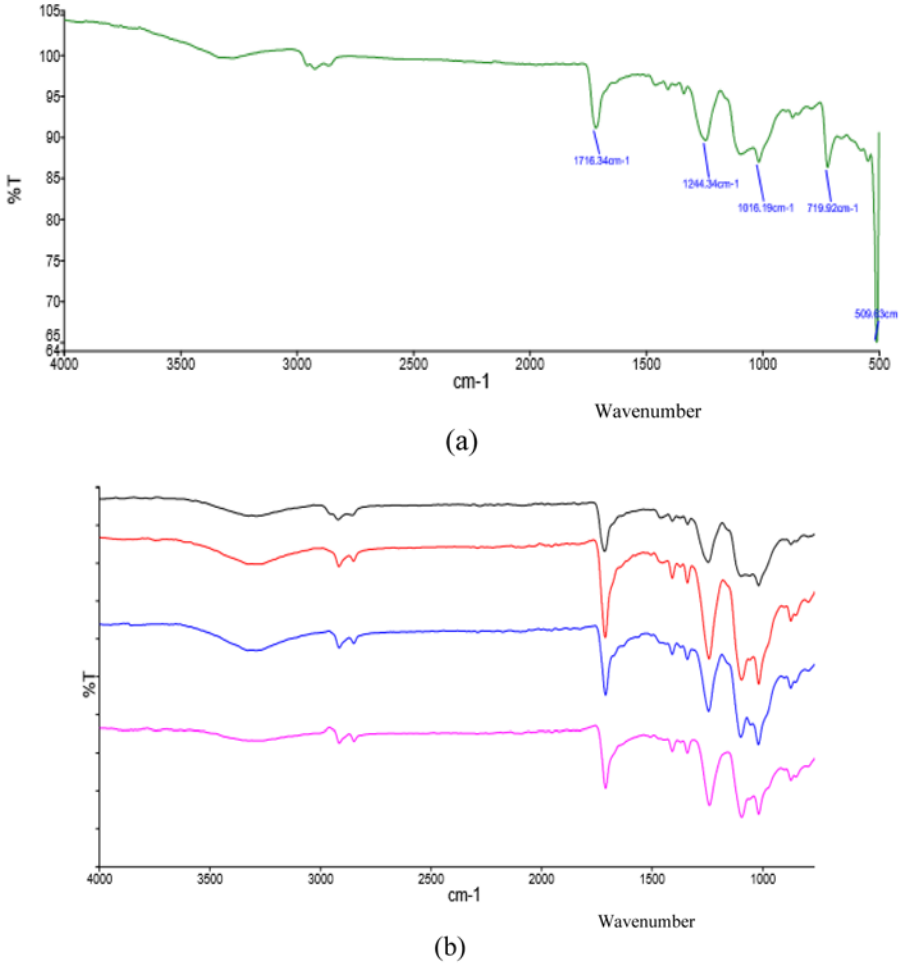


Figure 6. The ATR-FTIR spectra of unmodified polyester (P0)(a) and modified polyester (P1----, P2----, P3----, and P4----) (b).

The data of the contact angle in Figure 7 shows that the addition of one of the silver nanoparticles and HDTMS might increase the contact angle of the polyester fabric. However, the addition of HDTMS and the silver nanoparticles produces the highest contact angle on the resulting polyester. This is in contrast to the previous research [16, 17], that the addition of two types of compounds produced the highest contact angles. Note that the method of modification is different, in the previous research modifications were done with the addition of silver nanoparticle first and then followed

with the addition of HDTMS. While in this study, the addition of HDTMS was firstly performed and then the addition of silver nanoparticle. Thus the modification method seems to affect the properties of the fabric produced. The polyester after modification with HDTMS compound and silver nanoparticles has the largest contact angle value. The presence of a hydrophobic functional group increases the angle contact of water. The method of modification of the polyester fabric, qualitatively does not change the type of functional group present, but that affects the number of hydrophobic groups. Thus, the addition of nanosilver particles to hydrophobic materials, such as polyester composite, caused an increase of the hydrophobic groups and a decrease in surface free energy, so that an increase of the contact angle of P4.

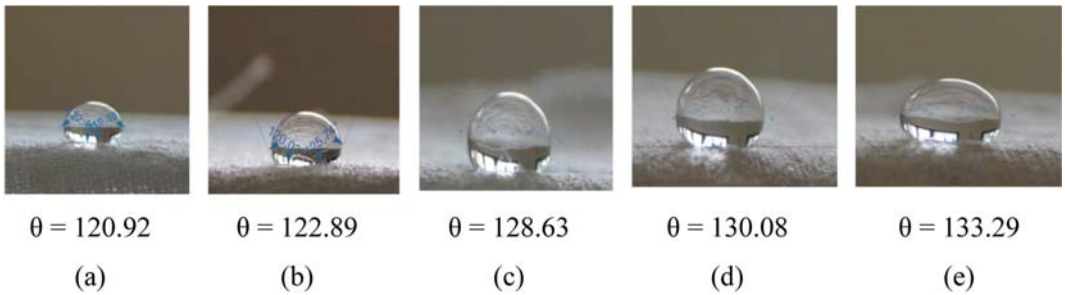


Figure 7. The water contact angle for P0 (a), P1(b), P2 (c), P3(d), and P4(e).

3.3 Antibacterial Activity of Polyester

The lowest inhibition zone of polyester in inhibiting the growth of *E. coli* bacteria is the P0 sample and the highest activity is the modified polyester (polyester - HDTMS) as shown in Figure 8. The highest inhibitory zone diameter of sample P-HDTMS occurs at the 60th hour. Incubation for 60 minutes is the maximum time for modified polyesters to inhibit bacterial growth of *E. coli*. Then the antibacterial activity decreases to the measurement at 72th hour. After 60 min, the ability of polyester in inhibiting bacterial growth decreased.

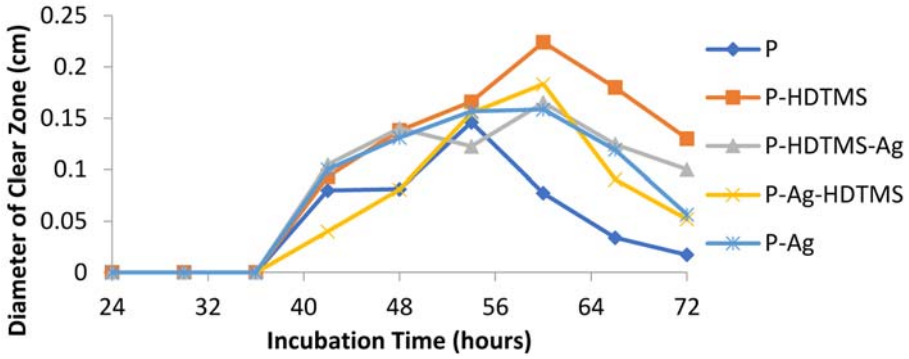


Figure 8. The antibacterial activity of polyester against *Escherichia coli* ATCC 35218.

The P-HDTMS has the highest ability to inhibit the growth of *E. coli* compared to other polyester samples. Thus, gram-negative bacteria are more easily inhibited by modified polyesters by silane compounds. The hydrophobic functional groups are more able to inhibit gram-negative bacteria. Modification of polyester by adding HDTMS compound can increase antibacterial activity of polyester cloths. The interaction between the ester groups and / or the -OH groups in the polyester and the -OH group in HDTMS produces a hydrophobic functional group so that the hydrophobic surface may cause the bacteria to inhibit its growth when interacting with the polyester fabric [27]. Compounds containing hydrophilic and hydrophobic groups are antibacterial agents that can damage cytoplasmic membranes and kill the bacterial cells [16]. The -OH functional groups (hydrophilic) from the polyester cloth and the HDTMS and hydrophobic groups of modified polyester bound to the silver will interact to -S-H on the cytoplasmic membrane by ionic or covalent bonds to form a bond -S-S- and produce S-Ag clusters that cause inhibition of cell respiration.

The lowest inhibition zone of polyesterin inhibiting the growth of *S. aureus* is shown by unmodified polyester (P) and the highest activity is shown by the P-HDTMS-Ag (Figure 9). In general, the graph shows the antibacterial activity of the polyester sample which tends to increase with the time of incubation, which then decreases after 60 hours of incubation. The highest inhibition zone diameter of sample of P-HDTMS-Ag occurs at the 60th hour. Thus, incubation for 60 minutes is the maximum time for modified polyesters to inhibit bacterial growth of *S. aureus*. P-HDTMS-Ag shows the highest antibacterial activity. This is due to the interaction between ester and or -OH groups with -OH groups of HDTMS (Figure 10) resulting in hydrophobic groups which are not favored by bacteria followed with modification by deposition of Ag nanoparticles, which is an antibacterial agent as well, thus increasing activity of antibacterial from polyester fabric. Thus, the polyester modification method using a combination of HDTMS and nanoparticles increases the antibacterial activity of the fabric. The P-HDTMS-Ag has the highest ability to inhibit the growth of *S. aureus* compared to other polyester samples. Thus, gram-positive bacteria are more easily inhibited by modified polyesters by silane compounds and followed by coating of silver nanoparticles. The presence of hydrophobic function groups and silver nanoparticles may inhibit gram-positive bacteria. In other words, gram-positive bacteria can be inhibited by polyester composites, this is because the gram-positive bacteria consist of a thick layer of peptidoglycan and a layer of teicoic acid and also tend to be negatively charged and polar, so that antibacterial material is required that can inhibit its growth in the form of composites polyester with HDTMS and silver nanoparticles.

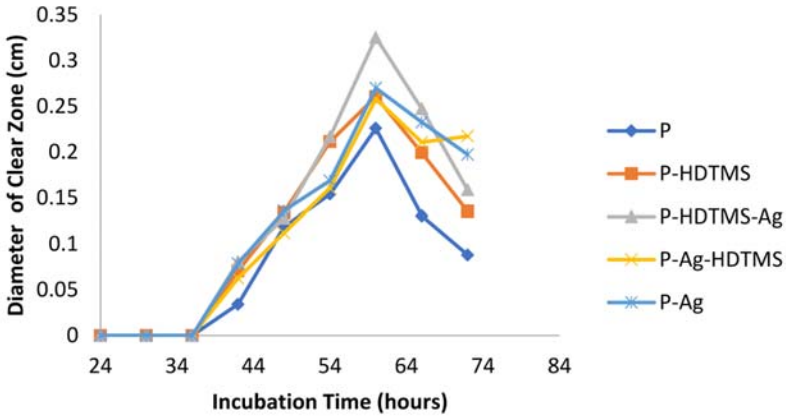


Figure 9. The antibacterial activity of polyester against *Staphylococcus aureus* ATCC 25923.

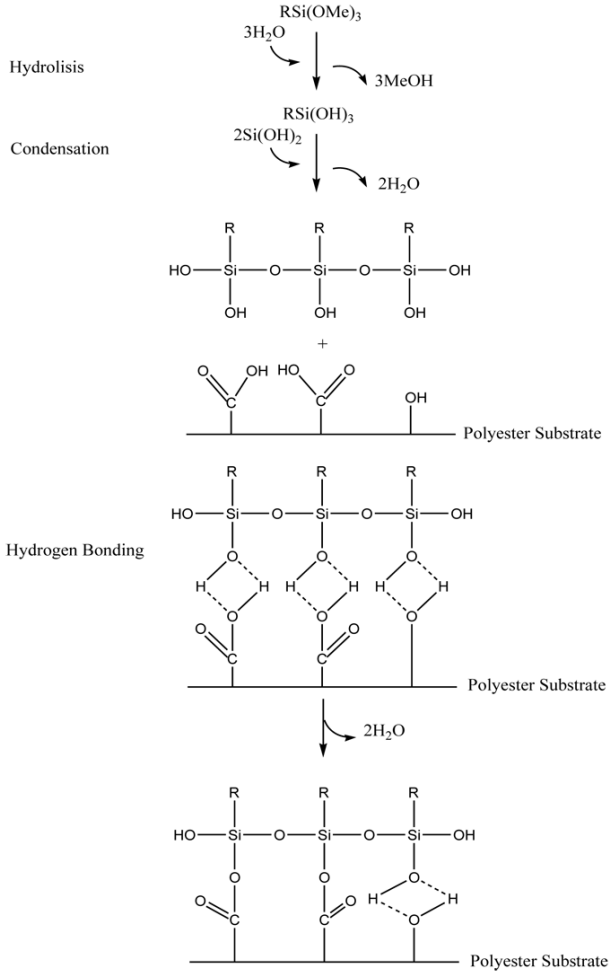


Figure 10. Interaction between the ester / the hydroxyl groups of polyester and the hydroxyl groups of HDTMS.

Based on results of Anova test in Table 1, there are three tests, namely interaction test between incubation time and type of sample, effect of incubation time, and effect of sample type on antibacterial activity of polyester cloths. The significant value of the interaction test between the incubation time and the sample type was 0.000 ($p<0.05$) for both against *E.coli* and *S.aureus*. Interpretation of $p<0.05$ indicates the effect of interaction between incubation time and sample type on antibacterial activity. The significant value of effect test of the incubation time on antibacterial activity was 0.000 ($p <0.05$). The interpretation of $p <0.05$ was the effect of incubation time on antibacterial activity. The significant value of the sample effect test on antibacterial activity was 0.000 ($p< 0.05$). Interpretation of $p<0.05$ indicates the influence of sample type on antibacterial activity. Thus, type of the polyester sample and the incubation time affect the antibacterial activity. The five types of polyester samples have different antibacterial activity significantly. Similarly with incubation time, different incubation times showed different antibacterial activity significantly as well.

Table 1. Significance of antibacterial activity of unmodified polyester and modified polyester against S*taphylococcus aureus* and *Escherichia coli.*

Source of data	Sum	Df	Average	F	Sig.
Escherichia coli ATCC 35218					
Time	0.413	7	0.059	83.652	0.000
Sample	0.053	4	0.013	18.897	0.000
Time* Sample	0.057	28	0.002	2.880	0.000
Staphylococcus aureus ATCC 25923					
Time	0.990	7	0.141	138.091	0.000
Sample	0.035	4	0.009	8.553	0.000
Time* Sample	0.052	28	0.002	1.821	0.000

The results of LSD tests in Table 2 show that the sample was a significant influence amongs the four modified samples with respect to P0 on the antibacterial activity (clear zone) for *Staphylococcus aureus* ATCC 25923, P0-P1, P0-P2, P0-P3 and P0-P4. All of the modified polyester shows significantly higher antibacterial activity than the unmodified polyester. Thus, modification can increase antibacterial activity of polyester cloths.

However, within the modified polyesters (P1-P4) no significant difference in theirs antibacterial activity was observed. Modification of polyester cloths by using silver nanoparticle, HDTMS, silver nanoparticle and HDTMS, and also HDTMS and silver nanoparticle has the similar effect toward antibacterial activity of polyester. Thus, the type of addition material and methods did not effect antibacterial activity of polyester cloths against *S.aureus*.

The results of LSD test in Table 2 shows that there are 7 variations of samples which have significant influence between two samples on antibacterial activity (clear zone) against *Escherichia coli* ATCC 35218, these are P0-P1, P0-P2 , P0-P3, P0-P4, P1-P3, P1-P4 and P2-P3. Therefore, it can be concluded that modification can

affect antibacterial activity of polyester cloths. The addition of silver nanoparticles, HDTMS, and combinations of the two might enhance the antibacterial activity of unmodified polyester fabrics to inhibit the growth of *E.coli*.

Table 2. Antibacterial activity between 2 polyester samples against *Staphylococcus aureus* and *Escherichia coli*.

Type of sample	Conclusion	
	Staphylococcus aureus	*Escherichia coli*
P0 - P1	Significant	Significant
P0 - P2	Significant	Significant
P0 - P3	Significant	Significant
P0 - P4	Significant	Significant
P1 - P2	Not significant	Not significant
P1 - P3	Not significant	Significant
P1 - P4	Not significant	Significant
P2 - P3	Not significant	Significant
P2 - P4	Not significant	Not significant
P3 - P4	Not significant	Not significant

Similarly, the addition method of nanoparticles and HDTMS or a combination of both might affect the antibacterial properties of polyester fabrics on inhibiting growth of *E.coli*. Considering that the differences in antibacterial activity between the following polyester samples P1-P3, P1-P4 and P2-P3 are significantly different, it can be concluded that the modification of fabric with two chemicals in P3 and P4 shows significantly differences compared to modifications using a single material (only nanoparticles or HDTMS) in P1 and P2.

Based on the results of the t-independent test as shown in Table 3, each sample of the polyester fabric shows no difference in antibacterial activity to inhibit the growth of *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923. Thus,the ability each sample to inhibit the *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923 is the same. This can be due to the fact that each type of bacteria has almost the same of chemical content. The chemical composition in cell wall of gram positive bacteria and gram negative bacteria is almost the same, i.e the presence of peptidoglycan and lipopolysaccharide.

Table 3. Interpretation of t-independent test for activity antibacterial of polyester against *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923.

Polyester sample	t-independent
P0	No difference
P1	No difference
P2	No difference
P3	No difference
P4	No difference

4. CONCLUSIONS

Based on the results of the research and discussion it could be concluded that the silver nanoparticles were successfully synthesized using a reducing agent of peel extract of *Ipomoea batatas L.* as confirmed at a wavelength of 436 nm with a reddish brown color. The unmodified polyester fabric

has the lowest contact angle value while the polyester fabric with the modification of HDTMS compound and silver nanoparticles has the largest contact angle value. There is a difference in the antibacterial activity of the fabric between the unmodified polyester and the modified polyester fabric at different incubation times in inhibiting the growth of *Escherichia coli* and *Staphylococcus aureus*. Samples of polyester - HDTMS - silver nanoparticle show the highest antibacterial activity against *S. aureus* with a strength of about 1.4 times greater than unmodified polyester and it is the highest in hydrophobicity properties. Each polyester sample has the same ability in inhibiting the growth of *Staphylococcus aureus* and *Escherichia coli*. Based on the antibacterial properties of the fabric then the modified polyester product of nanoparticles and HDTMS can be used as antibacterial materials for biomedical applications.

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Correction of the manuscript is ok. A little correction on page 4...*aureus*ATCC should be written *aureus* ATCC.....

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Contributed Paper

Application of Silver Nanoparticles Synthesized by Using *Ipomoea batatas L.* Waste to Improve Antibacterial Properties and Hydrophobicity of Polyester Cloths

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ABSTRACT

Synthesis of colloidal silver nanoparticle by using peel extracts of *Ipomoea batatas L.*, its deposition on polyester cloths, and the modification with hexadecyltrimethoxysilane (HDTMS) have been conducted in this work. The silver nanoparticles were characterized by using Uv-Vis spectrophotometer, functional groups of unmodified polyester and those of modified polyester were characterized by ATR-FTIR spectrophotometer, surface images of polyester were observed by Scanning Electron Microscopy tool, antibacterial activity of unmodified polyester cloth and modified polyester against *Staphylococcus aureus* and *Escherichia coli* were determined with a diffusion method, and hydrophobicity of polyester was measured by using asessile drop method. Silver nanoparticles were successfully produced using peel extracts of *Ipomoea batatas L* as indicated by the absorption peaks at 436 nm. SEM images confirmthat the silver nanoparticles coat onto polyester cloths. Modification with nanoparticle and HDTMS do not affect the functional groups of polyester. The polyester fabric with the addition of HDTMS compound and silver nanoparticles showedthe largest contact angle and the antibacterial activity to inhibit *Staphylococcus aureus*. Samples ofpolyester - HDTMS - silver nanoparticle show the highest antibacterial activity against*S. aureus*with a strength 1.4 times greater than unmodified polyester. There is a difference in the antibacterial activity of the polyester among the unmodified polyester and the modified polyester fabric at different incubation times inhibiting the growth of *Escherichia coli* and *Staphylococcus aureus*. Each sample ofthe unmodified polyester and the modified polyester fabric shows the same ability to inhibit the growth of *Staphylococcus aureus* and *Escherichia coli*.

Keywords: Antibacterial Activity, Hydrophobicity, *Ipomoea batatas L.*, Polyester, Silver Nanoparticles

1. INTRODUCTION

Peel of purple sweet potato is an untapped waste, however, the anthocyanin content in a peelof the purple sweet potato is actually still high,being 110.51 mg/100 g. Pigments of anthocyanins in *Ipomoea batatas L.* are more stable than anthocyanins from other sources, such as red cabbage, elderberries, blueberries and red corn [1]. However, anthocyanins contain phenolic hydroxyl groups which are easily oxidized into quinones, and thus skin extract of *Ipomoea batas L.* can be used as a reducing agent to convert silver ions (Ag^{+}) into silver nanoparticles (Ag^{0}). The reduction of particle size to nano-size of silver can be caused by the H radicals formed in phenolic compounds that are antioxidants [2]. Phenolics have hydroxyl and carbonyl groups that can bind metals. The antioxidant properties of phenolic compounds might occur through their tendency to chelate metal [3]. The efficiency of the synthesis of metal nanoparticles depends on the electrochemical potential of the reduced ions. Plant extracts effectively reduce metal ions more significantly for ions that have high positive electrochemical potentials such as Ag^{+} [4].

Nanoparticles can be synthesized using various methods, for examples using microwave irradiation method [5], reduction method [6], photochemical or photosynthesis method [7, 8], ball milling method [9], electrochemical method [10], biosynthesis by using actinomycete or actinobacteria [11, 12], and biosynthesis by using dried fruit extract method [13]. In this study, silver nanoparticles were prepared by applying green chemistry using skin extract of sweet purple potato (*Ipomoea batatas L.*). Utilization of plants to synthesize nanoparticles can be done based on the ability of these plants to absorb metal ions from the environment. The ions will be reduced through complex metabolic processes and accumulated in certain organs. Plants are known to have organic compounds that serve as reducing agents that can be used to replace or complement inorganic reducers. The use of plant organic compounds for the synthesis of nanoparticles is known as biosynthesis and is an eco-friendly method, as well as simpler and more efficient than chemical procedures [14].

The process of applying nanoparticles to textile fibers is done by composting nanometer-scale particles into textile fibers. The three-dimensional nanostructured surface particles and gel-producing additives produce hydrophobic fabric products that do not reduce the breathability and comfort of the fabric when worn. However, the use of silane compounds causes the impurities attached to the fabric to be easily released when watered, however, the fabric remains dry.

Textile materials developed into self-cleaning textile and antibacterial products in this workare polyester fabrics. Naturally, polyester fibers have hydrophobic properties as well as apparel materials, polyester fibers are also commonly used as materials for sportwear, underwear and sheets [15]. The antibacterial polyester can be developed through modification with silver nanoparticles. Antibacterial textiles have been developed by modifying textile materials with nanoparticles and vegetable oil, such as nylon [16], cotton [17], silk cloths [6], polyurethane [18], leather [19], and wool fiber [20].

Polyester with self-cleaning properties can be developed through modification with silane compounds. Several types of silane compounds used to modify materials are hexamethyldisilazane [21], octyltriethoxysilane [22], and hexadecyltrimethoxysilane [16-17,

23]. The addition of silane-based molecules of HDTMS compounds to textiles can increase water contact angle or hydrophobicity of the fabric [16]. The objectives of our research were to synthesize silver nanoparticle by using peel extract of *Ipomoea batatas L.*, to modifypolyester cloths by coating silver nanoparticles and HDTMS, and to study hydrophobicity property of polyester, and also to determine antibacterial activity of unmodified polyester and modified polyester.

2. MATERIALS AND METHODES

2.1 Materials

Silver nitrate (E-Merck), polyvinyl alcohol (PVA) with molar mass 29,365.96 g/mol (E-Merck), ethanol, acetone, and hexadecyltrimethoxysilane (Aldrich) were purchased as commercial products and used without any further purification. Polyester cloth was purchased from a store in Yogyakarta, Indonesia. Nutrient Agar (NA) and Nutrient Broth (NB) were purchased from Oxoid. *Staphylococcus aureus* ATCC 25923 and *Escherichia coli* 32518 were obtained from a collection of Faculty of Medicine, Universitas Gadjah Mada (UGM) Yogyakarta, Indonesia.

The research was conducted in the following stages: extraction of *Ipomoea batatas L.* peel, synthesis of Ag nanoparticle by using bioreductor, addition of colloidal silver nanoparticle on polyester cloths, modification of polyester cloths by adding HDTMS and colloidal silver nanoparticle, and characterization of unmodified polyester and modified polyester.

2.2 Apparatus

Ultra Violet - Visible (Uv-Vis) spectrophotometer (Shimadzu UV2450, Japan) wasused to study thesuccessful formation of colloid of silver nanoparticle by observing absorption peaks of silver nitrate solution and colloidal silver nanoparticle. Attenuated Total Reflection - Fourier Transfrom Infra-red (FTIR) spectra were recorded on a FTIR - ATR spectrophotometer (Shimadzu prestige 21, Japan). Morphological images of unmodified polyester and modified polyester were observed by using a scanning electron microscope tool (SEM Jeol T300, USA).

2.3 Preparation of Peel Extract

A total of 20 grams of *Ipomoea batatas L.* peel was freshly cleaned with aquadest and inserted it into a 500 mL beaker glass. About 100 mL of distilled water was added into it and then boiled for 15 minutes. Allowing to stand the mixture until room temperature after boiling, the extract of *Ipomoea batatas L.* skin was then filtered using Whatmann no. 42.

2.4 Biosynthesis of Silver Nanoparticle

About 1 mL of peel extract of *Ipomoea batatas L.*in an erlenmeyer was added into 40 mL of 1×10^{-3} M silver nitrate solution. The mixture was allowed to stand for 2 hours to react. To the mixture, 12 mL of 1% PVA solution was added and then it was allowed to stand for 1 hour. Finally, the solution was stirred for 3 days to form colloidal silver nanoparticles. The colloid was then characterized using a UV-Vis spectrophotometer.

2.5 Deposition of Nanoparticle on Polyester Cloths

The sample of polyester fiber (P0) with size 5 cm × 5cmwas soaked in colloidal silver nanoparticles in erlenmeyer 50 mL and shaked using a shaker at 155 rpm for 24 hours. It was then dried at room temperature for one day. Thus, a polyester-nanoparticle sample (P1) was produced.

2.6 Modification of Polyester Cloth with HDTMS

The polyester (P0) and polyester - nanoparticle (P1) were immersed in the solution of HDTMS in 4% ethanol which was stirred for 6 hours prior to use. The immersion of samples in HDTMS solution was performed for 60 minutes at room temperature using a shaker at 155 rpm. Samples of P0 and P1 that have been reacted with HDTMS were dried with a blow dryer and then continued by curing at 110 °C for 60 minutesto obtain the sample of polyester -HDTMS (P2),and of polyester - nanoparticle - HDTMS (P3). Then the sample of P2 was re-immersed in the silver nanoparticle to form polyester - HDTMS - nanoparticle (P4). Each polyester sample was prepared for 5 replications.

2.7 Characterization of Silver Nanoparticle with UV-Vis Spectrophotometer

The spectrum of colloidal silver nanoparticles was analyzed at a wavelength range of 200-600 nm.

2.8 Characterization of Polyester

Characteristic of unmodified polyester and modified polyester was determined by analysis of functional groups, observation of surface images, hydrophobic property, and antibacterial activity. Analysis of functional group of unmodified polyester and modified polyester was performed by using Attenuated Total Reflection (ATR) - FourierTransformInfrared (FTIR) spectrophotometer and observation of surface photo by Scanning Electron Microscopy (SEM).

2.8.1 Test of hydrophobicity

Characterization of hydrophobic properties was perfomed with sessile drop method as has been previously conducted [16]. The water was dropped from a height of 1 cm from a sample with a volume of 0.01 mL. After the water was dripped then a water droplet image on the surface of the cloth was photographed using a camera. The contact angle difference of the five polyester fiber samples was done by using Corel Draw. Each polyester sample was characterized for 3 replications.

2.8.2 Test of antibacterial activity of polyester cloths

The antibacterial activity of P0, P1, P2, P3, and P4 were performed against *Staphylococcus aureus* ATCC 25923 as gram-positive bacteria and against *Escherichia coli* ATCC 35218 as gram-negative bacteria as described in the previous work [16, 17]. *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923 were rejuvenated on an agar medium of nutrient agar (NA). Inoculated *Escherichia coli* ATCC 35218 and *Staphylococcus aureus*ATCC 25923 were rejuvenated into a liquid nutrient broth (NB) medium. Samples of polyester, polyester - nanoparticle, polyester - HDTMS, polyester - nanoparticle - HDTMS, and polyester - HDTMS - nanoparticle were cut off using paper piercing with diameter of 6 mm, and placed on microbial cultures in the petridish. The diameter of the clear zone of samples were measured using a sliding range every 6 hours for 72 hours. Antibacterial activity of each polyester sample was measured for 3 replications. Activity of unmodified polyester and modified polyester in inhibiting the bacterial growth is indicated by the diameter of inhibition zone around the sample pieces.

2.9 Test of Statistic

These results were analyzed using a quantitative descriptive method with

ANOVA, Least Significant Different (LSD), and t-independent. The ANOVA test is conducted to determine the significant differences in antibacterial activity of the five polyester samples, to study the effect of treatment or modification and incubation time toward the antibacterial activity. This was continued with LSD. The LSD test was performed to find out the differences in antibacterial activity of different samples. Tests of t-independent were performed to determine the differences in antibacterial activity of the five samples against *Escherichia coli* and *Staphylococcus aureus* bacteria by using software IBM SPSS.

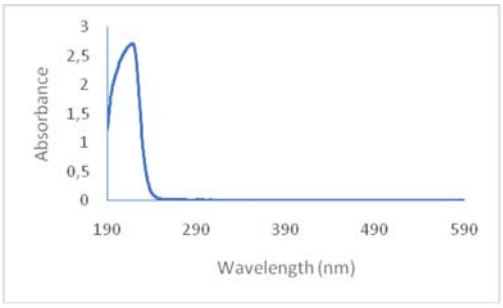
3. RESULTS AND DISCUSSION

3.1 Characteristic of Silver Nanoparticle

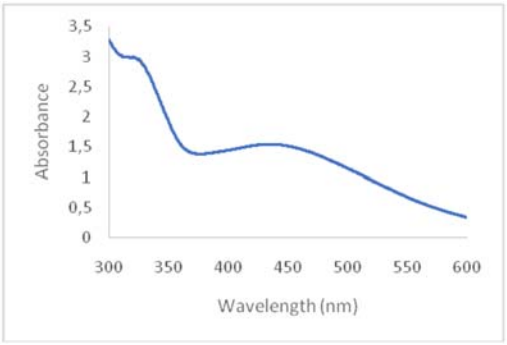
Bioreductor in this study are peel extract of *Ipomoea batatas L.* (Figure 1) which reduced $AgNO_3$ to nanoparticles and polyvinyl alcohol (PVA) as stabilizer agent. The colloids of silver nanoparticle isreddish brown color as in Figure 2. The UV-Vis spectrum (Figure 3 (a)) shows the absorbance of a 10^{-3} M of silver nitrate solution at a wavelength of 218.50 nm. Figure 3 (b) is UV-Vis spectrum of the colloidal silver nanoparticles. A peak appears at a wavelength of 436 nm and diameter of silver particle of 61.8 nm. This absorption peak indicates that the Ag^+ has been reduced to Ag°. The silver nanoparticles have a characteristic of surface plasmon resonance (SPR) peak at 433 nm with a reddish brown colour [24]. Colloidal silver nanoparticles provide a peak at wavelengths of around 400-500 nm which is a typical peak uptake of silver nanoparticles.The UV-Vis spectra of silver nanoparticles showed absorption peak in the range of 400 - 450 nm [6].

Figure 1. *Ipomoea batatas L.*

Figure 2. The colloidal of silver nanoparticle.



(a)



(b)

Figure 3. UV-Vis spectrum of $AgNO_3$ solution of 1×10^{-3} M (a) and silver nanoparticle (b).

Thus, the synthesis of nanoparticles can be done successfully by utilizing plant extracts, especially skin extract of *Ipomoea batatas L.* The process of reduction of Ag^+ ions into nanoparticles occurs outside the cell. The extracellular synthesis of silver nanoparticles could be conducted economically and safely by using leaf extract of *Catharanthus roseus Linn. G. Don* [25]. The extracellular synthesis was done by reacting metal ions with water or plant extracts. The use of plants in the synthesis of silver nanoparticles is relatively simpler and cheaper compared to other microorganisms. The types of plants that have been used for nanoparticle synthesis have been widely practiced by researchers. The biomolecule components contained in the plant are thought to act as a reducing agent, solvents and stabilizers in the formation of silver nanoparticles such as flavonoids, terpenoids, polysaccharides, alkaloids, and other secondary metabolites [25]. Three varieties of *Ipomoea batatas L.* have a major anthocyanin of cyanidin type [26]. However, anthocyanins can be oxidized to quinone. Thus it can be disclosed that in the process of forming silver nanoparticles using peel extracts of *Ipomoea batatas L* occurs reactions between hydroxyl groups (-OH phenolic) in anthocyanin compounds with silver ions to produce silver nanoparticles and quinone that cause the color of the solution to be reddish brown.

Each plant produces a variety of secondary metabolites. One of the most important groups of metabolites is a phenolic compound. Phenolics have at least one aromatic ring (C6) carrying a hydroxyl group. The antioxidant properties of phenolic compounds through their tendency to metal [3]. Based on the standard potential value, reaction (1) can be written as reactions (2) and (3). The sum of reaction (2) and (3) yields a cell potential of -0.445 volts. The negative cell potential value indicates that this redox reaction can't occur spontaneously.

$$Ag^+_{(aq)} + \text{-OH phenolic}_{(aq)} + H_2O_{(l)} \rightarrow Ag^0_{(s)} + \text{quinone}_{(aq)} + H^+_{(aq)} + O_{2(g)} \quad (1)$$

$$Ag^+ + e \rightarrow Ag^0 \quad E^\circ = 0.799 \text{ volt} \quad (2)$$

$$2\,H_2O \rightarrow 4H^+ + O_2 + 4e \quad E^\circ = -1.224 \text{ volt} \quad (3)$$

Although the cell potential of netreaction is negative, the Ag^+can be reduced to Ag^0 in the presence of -OH phenolic from plant extracts (a) (Figure 4). A complex formed (c2) (Figure 4) by the reaction between the -OH group of phenolic and the silver ion has an important role in the reduction and formation of silver nanoparticles. Many researchers concluded that complexes formed from citrate ion and silver ion will catalyze the reduction of Ag^+ to Ag^O even slowly in chemical procedures. The probability of reaction mechanism in extracellular synthesis of silver nanoparticle by supporting peel extract of *Ipomoea batatas L.* is shown in Figure 4. Anthocyanin has a structure as shown in 4(a), and simple structure is shown in 4(b) and 4(c1). When silver ion is reduced to form silver, -OH anthocyanin is oxidized to C=O quinon (c3).

3.2 SEM Image, Functional Groups, and Water Contact Angle of Polyester

SEM images of unmodified and modified polyesters (Figure 5) show that fabrics are composed of fibril. Figure 5(a) shows no contaminants on the surface of unmodified polyester fabrics. The modified polyester shows the presence of silver nanoparticles deposited on the surface of the polyester fabric (Figure 5(b) and 5(c)). Some of the microparticles cover the surface

of the fabric, since the nanoparticles undergo agglomeration to form larger particles [22]. In this study, silver nanoparticles successfully cover the surface of polyester fabric. The addition of silver nanoparticles results in a rougher cloth surface than a cloth without the addition of nanoparticles. However the surface between pure polyester (P0) and the modified polyester (P3) are practically no different. This could be caused by the HDTMS layer covered the silver nanoparticle so that the nanoparticle of silver can't be detected on surface images of P3, whereas in P4 the layer of HDTMS was covered by the silver nanoparticles so that the surface images are different, the silver nanoparticles are very much on the surface of the cloth P4.

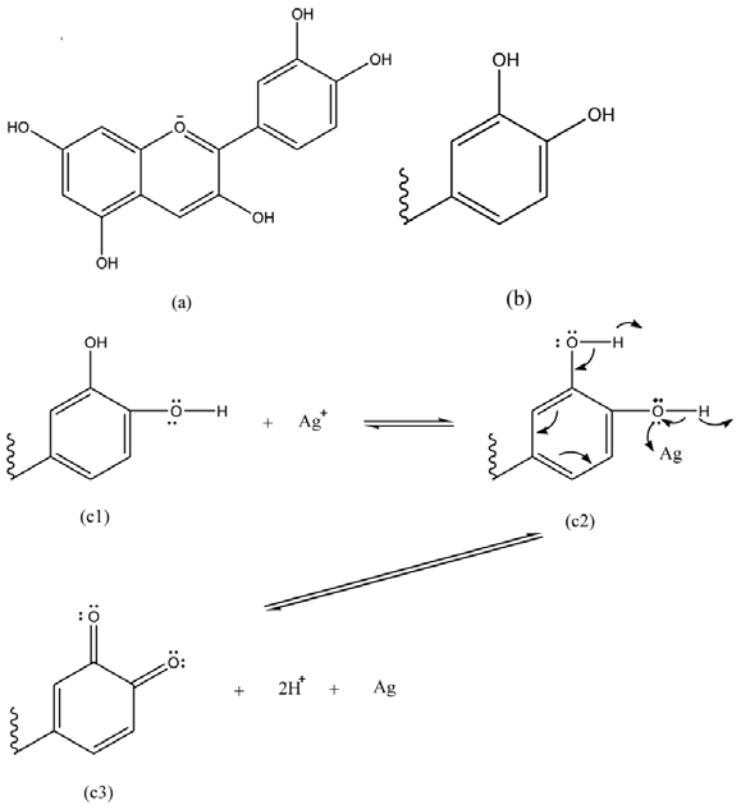


Figure 4. Reaction mechanism of formation the silver nanoparticles from anthocyanin (a) and silver ion.

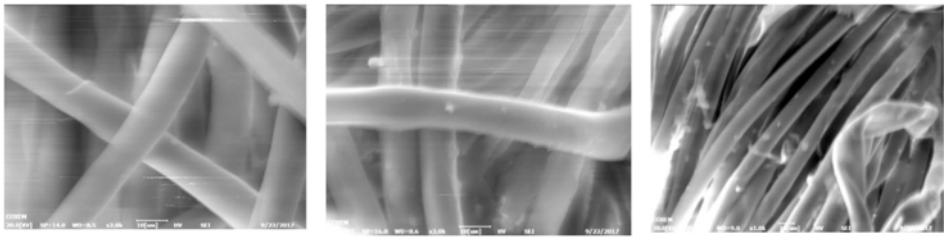


Figure 5. The SEM image of unmodified polyester (P0) (a), modified polyester (P3) (b), and P4 (c).

ATR-FTIR spectra of polyester (Figure 6) shows that polyester contains functional groups of -OH, -C-H, -COO esther, C=O, and C-O. Modification with silver nanoparticle and HDTMS compound does not show a new functional group. Thus, it does not effect the functional groups of polyester. This result is in accordance with the previous studies on the modification of cotton cloth with silver nanoparticles and HDTMS compounds [16]. The addition of the HDTMS compound can decrease absorption band in the spectrum of FTIR of modified Nylon [16].

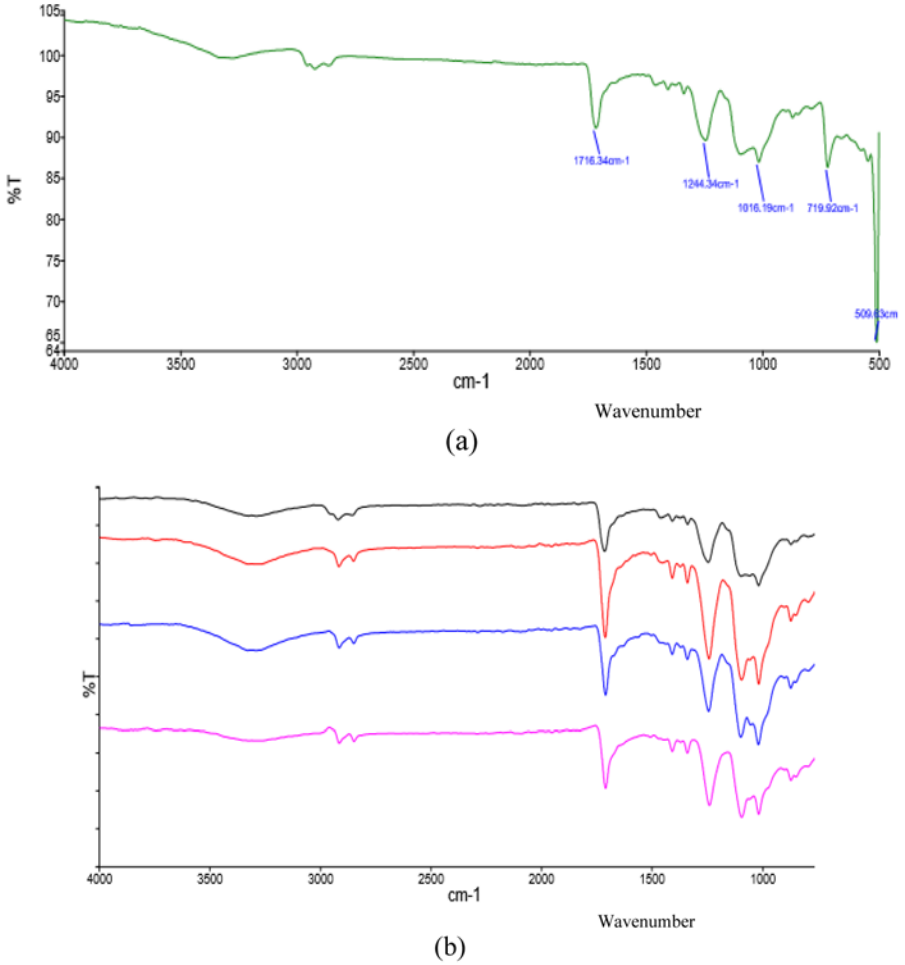


Figure 6. The ATR-FTIR spectra of unmodified polyester (P0)(a) and modified polyester (P1----, P2----, P3----, and P4----) (b).

The data of the contact angle in Figure 7 shows that the addition of one of the silver nanoparticles and HDTMS might increase the contact angle of the polyester fabric. However, the addition of HDTMS and the silver nanoparticles produces the highest contact angle on the resulting polyester. This is in contrast to the previous research [16, 17], that the addition of two types of compounds produced the highest contact angles. Note that the method of modification is different, in the previous research modifications were done with the addition of silver nanoparticle first and then followed

with the addition of HDTMS. While in this study, the addition of HDTMS was firstly performed and then the addition of silver nanoparticle. Thus the modification method seems to affect the properties of the fabric produced. The polyester after modification with HDTMS compound and silver nanoparticles has the largest contact angle value. The presence of a hydrophobic functional group increases the angle contact of water. The method of modification of the polyester fabric, qualitatively does not change the type of functional group present, but that affects the number of hydrophobic groups. Thus, the addition of nanosilver particles to hydrophobic materials, such as polyester composite, caused an increase of the hydrophobic groups and a decrease in surface free energy, so that an increase of the contact angle of P4.

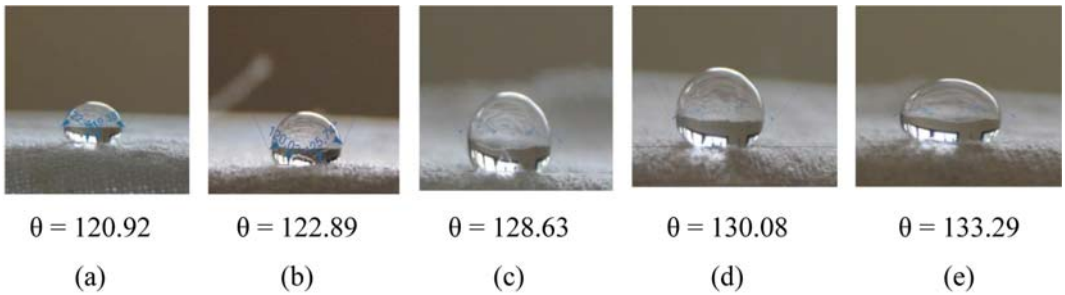


Figure 7. The water contact angle for P0 (a), P1(b), P2 (c), P3(d), and P4(e).

3.3 Antibacterial Activity of Polyester

The lowest inhibition zone of polyester in inhibiting the growth of *E. coli* bacteria is the P0 sample and the highest activity is the modified polyester (polyester - HDTMS) as shown in Figure 8. The highest inhibitory zone diameter of sample P-HDTMS occurs at the 60th hour. Incubation for 60 minutes is the maximum time for modified polyesters to inhibit bacterial growth of *E. coli*. Then the antibacterial activity decreases to the measurement at 72th hour. After 60 min, the ability of polyester in inhibiting bacterial growth decreased.

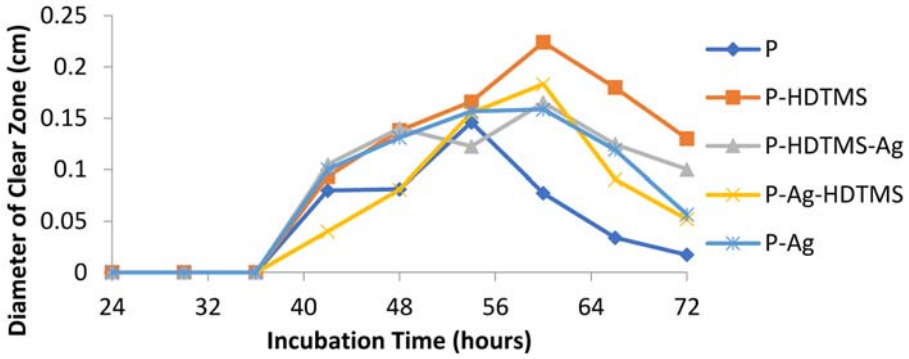


Figure 8. The antibacterial activity of polyester against *Escherichia coli* ATCC 35218.

The P-HDTMS has the highest ability to inhibit the growth of *E. coli* compared to other polyester samples. Thus, gram-negative bacteria are more easily inhibited by modified polyesters by silane compounds. The hydrophobic functional groups are more able to inhibit gram-negative bacteria. Modification of polyester by adding HDTMS compound can increase antibacterial activity of polyester cloths. The interaction between the ester groups and / or the -OH groups in the polyester and the -OH group in HDTMS produces a hydrophobic functional group so that the hydrophobic surface may cause the bacteria to inhibit its growth when interacting with the polyester fabric [27]. Compounds containing hydrophilic and hydrophobic groups are antibacterial agents that can damage cytoplasmic membranes and kill the bacterial cells [16]. The -OH functional groups (hydrophilic) from the polyester cloth and the HDTMS and hydrophobic groups of modified polyester bound to the silver will interact to -S-H on the cytoplasmic membrane by ionic or covalent bonds to form a bond -S-S- and produce S-Ag clusters that cause inhibition of cell respiration.

The lowest inhibition zone of polyesterin inhibiting the growth of *S. aureus* is shown by unmodified polyester (P) and the highest activity is shown by the P-HDTMS-Ag (Figure 9). In general, the graph shows the antibacterial activity of the polyester sample which tends to increase with the time of incubation, which then decreases after 60 hours of incubation. The highest inhibition zone diameter of sample of P-HDTMS-Ag occurs at the 60th hour. Thus, incubation for 60 minutes is the maximum time for modified polyesters to inhibit bacterial growth of *S. aureus*. P-HDTMS-Ag shows the highest antibacterial activity. This is due to the interaction between ester and or -OH groups with -OH groups of HDTMS (Figure 10) resulting in hydrophobic groups which are not favored by bacteria followed with modification by deposition of Ag nanoparticles, which is an antibacterial agent as well, thus increasing activity of antibacterial from polyester fabric. Thus, the polyester modification method using a combination of HDTMS and nanoparticles increases the antibacterial activity of the fabric. The P-HDTMS-Ag has the highest ability to inhibit the growth of *S. aureus* compared to other polyester samples. Thus, gram-positive bacteria are more easily inhibited by modified polyesters by silane compounds and followed by coating of silver nanoparticles. The presence of hydrophobic function groups and silver nanoparticles may inhibit gram-positive bacteria. In other words, gram-positive bacteria can be inhibited by polyester composites, this is because the gram-positive bacteria consist of a thick layer of peptidoglycan and a layer of teicoic acid and also tend to be negatively charged and polar, so that antibacterial material is required that can inhibit its growth in the form of composites polyester with HDTMS and silver nanoparticles.

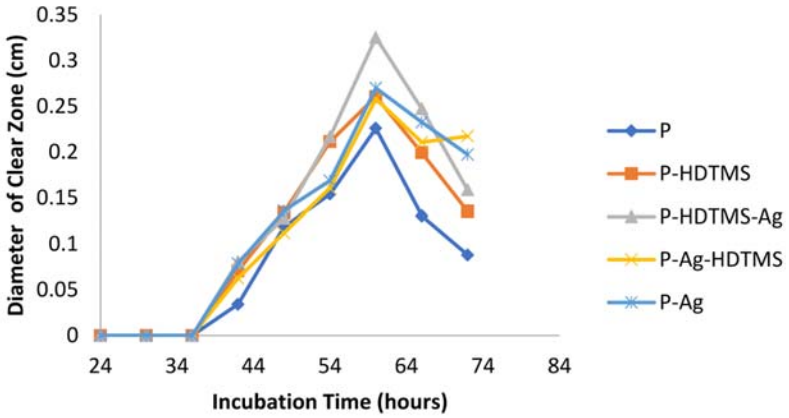


Figure 9. The antibacterial activity of polyester against *Staphylococcus aureus* ATCC 25923.

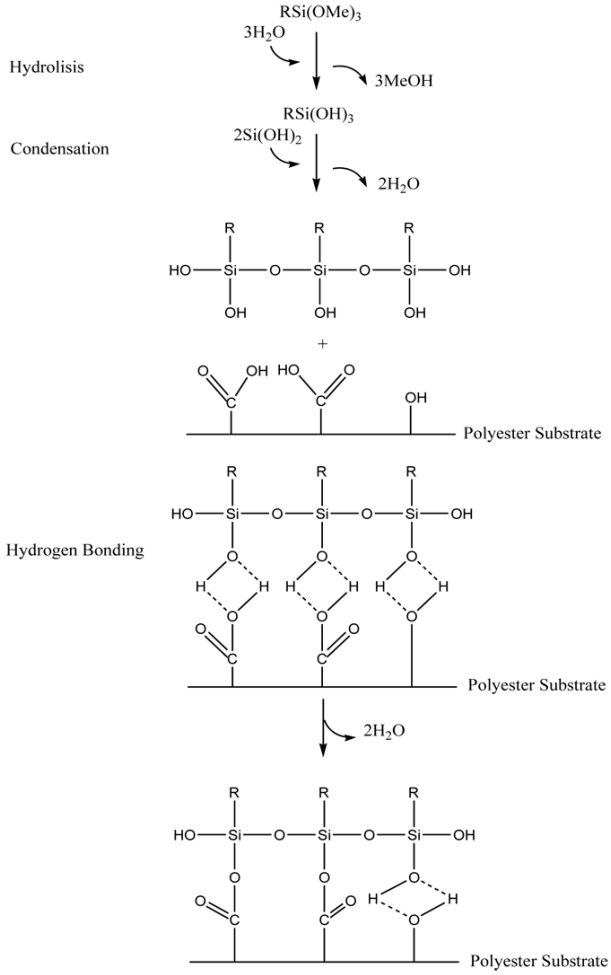


Figure 10. Interaction between the ester / the hydroxyl groups of polyester and the hydroxyl groups of HDTMS.

Based on results of Anova test in Table 1, there are three tests, namely interaction test between incubation time and type of sample, effect of incubation time, and effect of sample type on antibacterial activity of polyester cloths. The significant value of the interaction test between the incubation time and the sample type was 0.000 ($p<0.05$) for both against *E.coli* and *S.aureus*. Interpretation of $p<0.05$ indicates the effect of interaction between incubation time and sample type on antibacterial activity. The significant value of effect test of the incubation time on antibacterial activity was 0.000 ($p <0.05$). The interpretation of $p <0.05$ was the effect of incubation time on antibacterial activity. The significant value of the sample effect test on antibacterial activity was 0.000 ($p< 0.05$). Interpretation of $p<0.05$ indicates the influence of sample type on antibacterial activity. Thus, type of the polyester sample and the incubation time affect the antibacterial activity. The five types of polyester samples have different antibacterial activity significantly. Similarly with incubation time, different incubation times showed different antibacterial activity significantly as well.

Table 1. Significance of antibacterial activity of unmodified polyester and modified polyester against S*taphylococcus aureus* and *Escherichia coli.*

Source of data	Sum	Df	Average	F	Sig.
Escherichia coli ATCC 35218					
Time	0.413	7	0.059	83.652	0.000
Sample	0.053	4	0.013	18.897	0.000
Time* Sample	0.057	28	0.002	2.880	0.000
Staphylococcus aureus ATCC 25923					
Time	0.990	7	0.141	138.091	0.000
Sample	0.035	4	0.009	8.553	0.000
Time* Sample	0.052	28	0.002	1.821	0.000

The results of LSD tests in Table 2 show that the sample was a significant influence amongs the four modified samples with respect to P0 on the antibacterial activity (clear zone) for *Staphylococcus aureus* ATCC 25923, P0-P1, P0-P2, P0-P3 and P0-P4. All of the modified polyester shows significantly higher antibacterial activity than the unmodified polyester. Thus, modification can increase antibacterial activity of polyester cloths.

However, within the modified polyesters (P1-P4) no significant difference in theirs antibacterial activity was observed. Modification of polyester cloths by using silver nanoparticle, HDTMS, silver nanoparticle and HDTMS, and also HDTMS and silver nanoparticle has the similar effect toward antibacterial activity of polyester. Thus, the type of addition material and methods did not effect antibacterial activity of polyester cloths against *S.aureus*.

The results of LSD test in Table 2 shows that there are 7 variations of samples which have significant influence between two samples on antibacterial activity (clear zone) against *Escherichia coli* ATCC 35218, these are P0-P1, P0-P2 , P0-P3, P0-P4, P1-P3, P1-P4 and P2-P3. Therefore, it can be concluded that modification can

affect antibacterial activity of polyester cloths. The addition of silver nanoparticles, HDTMS, and combinations of the two might enhance the antibacterial activity of unmodified polyester fabrics to inhibit the growth of *E.coli*.

Table 2. Antibacterial activity between 2 polyester samples against *Staphylococcus aureus* and *Escherichia coli*.

Type of sample	Conclusion	
	Staphylococcus aureus	*Escherichia coli*
P0 - P1	Significant	Significant
P0 - P2	Significant	Significant
P0 - P3	Significant	Significant
P0 - P4	Significant	Significant
P1 - P2	Not significant	Not significant
P1 - P3	Not significant	Significant
P1 - P4	Not significant	Significant
P2 - P3	Not significant	Significant
P2 - P4	Not significant	Not significant
P3 - P4	Not significant	Not significant

Similarly, the addition method of nanoparticles and HDTMS or a combination of both might affect the antibacterial properties of polyester fabrics on inhibiting growth of *E.coli*. Considering that the differences in antibacterial activity between the following polyester samples P1-P3, P1-P4 and P2-P3 are significantly different, it can be concluded that the modification of fabric with two chemicals in P3 and P4 shows significantly differences compared to modifications using a single material (only nanoparticles or HDTMS) in P1 and P2.

Based on the results of the t-independent test as shown in Table 3, each sample of the polyester fabric shows no difference in antibacterial activity to inhibit the growth of *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923. Thus,the ability each sample to inhibit the *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923 is the same. This can be due to the fact that each type of bacteria has almost the same of chemical content. The chemical composition in cell wall of gram positive bacteria and gram negative bacteria is almost the same, i.e the presence of peptidoglycan and lipopolysaccharide.

Table 3. Interpretation of t-independent test for activity antibacterial of polyester against *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923.

Polyester sample	t-independent
P0	No difference
P1	No difference
P2	No difference
P3	No difference
P4	No difference

4. CONCLUSIONS

Based on the results of the research and discussion it could be concluded that the silver nanoparticles were successfully synthesized using a reducing agent of peel extract of *Ipomoea batatas L.* as confirmed at a wavelength of 436 nm with a reddish brown color. The unmodified polyester fabric

has the lowest contact angle value while the polyester fabric with the modification of HDTMS compound and silver nanoparticles has the largest contact angle value. There is a difference in the antibacterial activity of the fabric between the unmodified polyester and the modified polyester fabric at different incubation times in inhibiting the growth of *Escherichia coli* and *Staphylococcus aureus*. Samples of polyester - HDTMS - silver nanoparticle show the highest antibacterial activity against *S. aureus* with a strength of about 1.4 times greater than unmodified polyester and it is the highest in hydrophobicity properties. Each polyester sample has the same ability in inhibiting the growth of *Staphylococcus aureus* and *Escherichia coli*. Based on the antibacterial properties of the fabric then the modified polyester product of nanoparticles and HDTMS can be used as antibacterial materials for biomedical applications.

ACKNOWLEDGEMENT

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Contributed Paper

Application of Silver Nanoparticles Synthesized by Using *Ipomoea batatas L.* Waste to Improve Antibacterial Properties and Hydrophobicity of Polyester Cloths

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ABSTRACT

Synthesis of colloidal silver nanoparticle by using peel extracts of *Ipomoea batatas L.*, its deposition on polyester cloths, and the modification with hexadecyltrimethoxysilane (HDTMS) have been conducted in this work. The silver nanoparticles were characterized by using Uv-Vis spectrophotometer, functional groups of unmodified polyester and those of modified polyester were characterized by ATR-FTIR spectrophotometer, surface images of polyester were observed by Scanning Electron Microscopy tool, antibacterial activity of unmodified polyester cloth and modified polyester against *Staphylococcus aureus* and *Escherichia coli* were determined with a diffusion method, and hydrophobicity of polyester was measured by using a sessile drop method. Silver nanoparticles were successfully produced using peel extracts of *Ipomoea batatas L* as indicated by the absorption peaks at 436 nm. SEM images confirm that the silver nanoparticles coat onto polyester cloths. Modification with nanoparticle and HDTMS do not affect the functional groups of polyester. The polyester fabric with the addition of HDTMS compound and silver nanoparticles showed the largest contact angle and the antibacterial activity to inhibit *Staphylococcus aureus*. Samples of polyester - HDTMS - silver nanoparticle show the highest antibacterial activity against *S. aureus* with a strength 1.4 times greater than unmodified polyester. There is a difference in the antibacterial activity of the polyester among the unmodified polyester and the modified polyester fabric at different incubation times inhibiting the growth of *Escherichia coli* and *Staphylococcus aureus*. Each sample ofthe unmodified polyester and the modified polyester fabric shows the same ability to inhibit the growth of *Staphylococcus aureus* and *Escherichia coli*.

Keywords: Antibacterial Activity, Hydrophobicity, *Ipomoea batatas L.*, Polyester, Silver Nanoparticles

1. INTRODUCTION

Peel of purple sweet potato is an untapped waste, however, the anthocyanin content in a peel of the purple sweet potato is actually still high, being 110.51 mg/100 g. Pigments of anthocyanins in *Ipomoea batatas L.* are more stable than anthocyanins from other sources, such as red cabbage, elderberries, blueberries and red corn [1]. However, anthocyanins contain phenolic hydroxyl groups which are easily oxidized into quinones, and thus peel extract of *Ipomoea batas L.* can be used as a reducing agent to convert silver ions (Ag^{+}) into silver nanoparticles (Ag^{0}). The reduction of particle size to nano-size of silver can be caused by the H radicals formed in phenolic compounds that are antioxidants [2]. Phenolics have hydroxyl and carbonyl groups that can bind metals. The antioxidant properties of phenolic compounds might occur through their tendency to chelate metal [3]. The efficiency of the synthesis of metal nanoparticles depends on the electrochemical potential of the reduced ions. Plant extracts effectively reduce metal ions more significantly for ions that have high positive electrochemical potentials such as Ag^{+} [4].

Nanoparticles can be synthesized using various methods, for examples using microwave irradiation method [5], reduction method [6], photochemical or photosynthesis method [7, 8], ball milling method [9], electrochemical method [10], biosynthesis by using actinomycete or actinobacteria [11, 12], and biosynthesis by using dried fruit extract method [13]. In this study, silver nanoparticles were prepared by applying green chemistry using peel extract of sweet purple potato (*Ipomoea batatas L.*). Utilization of plants to synthesize nanoparticles can be done based on the ability of these plants to absorb metal ions from the environment. The ions will be reduced through complex metabolic processes and accumulated in certain organs. Plants are known to have organic compounds that serve as reducing agents that can be used to replace or complement inorganic reducers. The use of plant organic compounds for the synthesis of nanoparticles is known as biosynthesis and is an eco-friendly method, as well as simpler and more efficient than chemical procedures [14].

The process of applying nanoparticles to textile fibers is done by composting nanometer-scale particles into textile fibers. The three-dimensional nanostructured surface particles and gel-producing additives produce hydrophobic fabric products that do not reduce the breathability and comfort of the fabric when worn. However, the use of silane compounds causes the impurities attached to the fabric to be easily released when watered, however, the fabric remains dry.

Textile materials developed into self-cleaning textile and antibacterial products in this work are polyester fabrics. Naturally, polyester fibers have hydrophobic properties as well as apparel materials, polyester fibers are also commonly used as materials for sportwear, underwear and sheets [15]. The antibacterial polyester can be developed through modification with silver nanoparticles. Antibacterial textiles have been developed by modifying textile materials with nanoparticles and vegetable oil, such as nylon [16], cotton [17], silk cloths [6], polyurethane [18], leather [19], and wool fiber [20].

Polyester with self-cleaning properties can be developed through modification with silane compounds. Several types of silane compounds used to modify materials are hexamethyldisilazane [21], octyltriethoxysilane [22], and hexadecyltrimethoxysilane [16-17,

23]. The addition of silane-based molecules of HDTMS compounds to textiles can increase water contact angle or hydrophobicity of the fabric [16]. The objectives of our research were to synthesize silver nanoparticle by using peel extract of *Ipomoea batatas L.*, to modify polyester cloths by coating silver nanoparticles and HDTMS, and to study hydrophobicity property of polyester, and also to determine antibacterial activity of unmodified polyester and modified polyester.

2. MATERIALS AND METHODES

2.1 Materials

Silver nitrate (E-Merck), polyvinyl alcohol (PVA) with molar mass 29,365.96 g/mol (E-Merck), ethanol, acetone, and hexadecyltrimethoxysilane (Aldrich) were purchased as commercial products and used without any further purification. Polyester cloth was purchased from a store in Yogyakarta, Indonesia. Nutrient Agar (NA) and Nutrient Broth (NB) were purchased from Oxoid. *Staphylococcus aureus* ATCC 25923 and *Escherichia coli* 32518 were obtained from a collection of Faculty of Medicine, Universitas Gadjah Mada (UGM) Yogyakarta, Indonesia.

The research was conducted in the following stages: extraction of *Ipomoea batatas L.* peel, synthesis of Ag nanoparticle by using bioreductor, addition of colloidal silver nanoparticle on polyester cloths, modification of polyester cloths by adding HDTMS and colloidal silver nanoparticle, and characterization of unmodified polyester and modified polyester.

2.2 Apparatus

Ultra Violet - Visible (Uv-Vis) spectrophotometer (Shimadzu UV2450, Japan) was used to study the successful formation of colloid of silver nanoparticle by observing absorption peaks of silver nitrate solution and colloidal silver nanoparticle. Attenuated Total Reflection - Fourier Transfrom Infra-red (FTIR) spectra were recorded on a FTIR - ATR spectrophotometer (Shimadzu prestige 21, Japan). Morphological images of unmodified polyester and modified polyester were observed by using a scanning electron microscope tool (SEM Jeol T300, USA).

2.3 Preparation of Peel Extract

A total of 20 grams of *Ipomoea batatas L.* peel was freshly cleaned with aquadest and inserted it into a 500 mL beaker glass. About 100 mL of distilled water was added into it and then boiled for 15 minutes. Allowing to stand the mixture until room temperature after boiling, the extract of *Ipomoea batatas L.* peel was then filtered using Whatmann no. 42.

2.4 Biosynthesis of Silver Nanoparticle

About 1 mL of peel extract of *Ipomoea batatas L.*in an erlenmeyer was added into 40 mL of 1×10^{-3} M silver nitrate solution. The mixture was allowed to stand for 2 hours to react. To the mixture, 12 mL of 1% PVA solution was added and then it was allowed to stand for 1 hour. Finally, the solution was stirred for 3 days to form colloidal silver nanoparticles. The colloid was then characterized using a UV-Vis spectrophotometer.

2.5 Deposition of Nanoparticle on Polyester Cloths

The sample of polyester fiber (P0) with size 5 cm × 5 cm was soaked in colloidal silver nanoparticles in erlenmeyer 50 mL and shaked using a shaker at 155 rpm for 24 hours. It was then dried at room temperature for one day. Thus, a polyester-nanoparticle sample (P1) was produced.

2.6 Modification of Polyester Cloth with HDTMS

The polyester (P0) and polyester - nanoparticle (P1) were immersed in the solution of HDTMS in 4% ethanol which was stirred for 6 hours prior to use. The immersion of samples in HDTMS solution was performed for 60 minutes at room temperature using a shaker at 155 rpm. Samples of P0 and P1 that have been reacted with HDTMS were dried with a blow dryer and then continued by curing at 110 °C for 60 minutes to obtain the sample of polyester -HDTMS (P2), and of polyester - nanoparticle - HDTMS (P3). Then the sample of P2 was re-immersed in the silver nanoparticle to form polyester - HDTMS - nanoparticle (P4). Each polyester sample was prepared for 5 replications.

2.7 Characterization of Silver Nanoparticle with UV-Vis Spectrophotometer

The spectrum of colloidal silver nanoparticles was analyzed at a wavelength range of 200-600 nm.

2.8 Characterization of Polyester

Characteristic of unmodified polyester and modified polyester was determined by analysis of functional groups, observation of surface images, hydrophobic property, and antibacterial activity. Analysis of functional group of unmodified polyester and modified polyester was performed by using Attenuated Total Reflection (ATR) - Fourier Transform Infra Red (FTIR) spectrophotometer and observation of surface photo by Scanning Electron Microscopy (SEM).

2.8.1 Test of hydrophobicity

Characterization of hydrophobic properties was perfomed with sessile drop method as has been previously conducted [16]. The water was dropped from a height of 1 cm from a sample with a volume of 0.01 mL. After the water was dripped then a water droplet image on the surface of the cloth was photographed using a camera. The contact angle difference of the five polyester fiber samples was done by using Corel Draw. Each polyester sample was characterized for 3 replications.

2.8.2 Test of antibacterial activity of polyester cloths

The antibacterial activity of P0, P1, P2, P3, and P4 were performed against *Staphylococcus aureus* ATCC 25923 as gram-positive bacteria and against *Escherichia coli* ATCC 35218 as gram-negative bacteria as described in the previous work [16, 17]. *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923 were rejuvenated on an agar medium of nutrient agar (NA). Inoculated *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923 were rejuvenated into a liquid nutrient broth (NB) medium. Samples of polyester, polyester - nanoparticle, polyester - HDTMS, polyester - nanoparticle - HDTMS, and polyester - HDTMS - nanoparticle were cut off using paper piercing with diameter of 6 mm, and placed on microbial cultures in the petridish. The diameter of the clear zone of samples were measured using a sliding range every 6 hours for 72 hours. Antibacterial activity of each polyester sample was measured for 3 replications. Activity of unmodified polyester and modified polyester in inhibiting the bacterial growth is indicated by the diameter of inhibition zone around the sample pieces.

2.9 Test of Statistic

These results were analyzed using a quantitative descriptive method with

ANOVA, Least Significant Different (LSD), and t-independent. The ANOVA test is conducted to determine the significant differences in antibacterial activity of the five polyester samples, to study the effect of treatment or modification and incubation time toward the antibacterial activity. This was continued with LSD. The LSD test was performed to find out the differences in antibacterial activity of different samples. Tests of t-independent were performed to determine the differences in antibacterial activity of the five samples against *Escherichia coli* and *Staphylococcus aureus* bacteria by using software IBM SPSS.

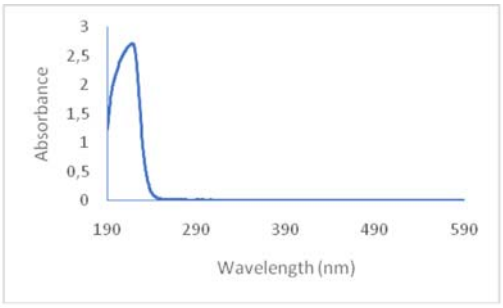
3. RESULTS AND DISCUSSION

3.1 Characteristic of Silver Nanoparticle

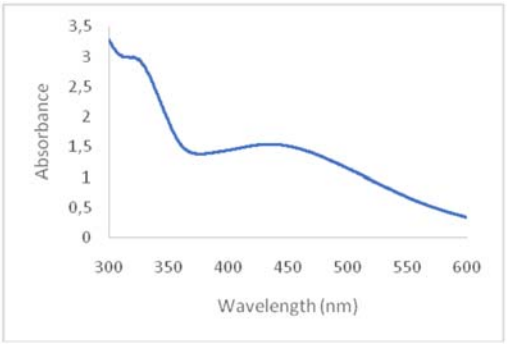
Bioreductor in this study are peel extract of *Ipomoea batatas L.* (Figure 1) which reduced $AgNO_3$ to nanoparticles and polyvinyl alcohol (PVA) as stabilizer agent. The colloids of silver nanoparticle is reddish brown color as in Figure 2. The UV-Vis spectrum (Figure 3 (a)) shows the absorbance of a 10^{-3} M of silver nitrate solution at a wavelength of 218.50 nm. Figure 3 (b) is UV-Vis spectrum of the colloidal silver nanoparticles. A peak appears at a wavelength of 436 nm and diameter of silver particle of 61.8 nm. This absorption peak indicates that the Ag^+ has been reduced to Ag°. The silver nanoparticles have a characteristic of surface plasmon resonance (SPR) peak at 433 nm with a reddish brown colour [24]. Colloidal silver nanoparticles provide a peak at wavelengths of around 400-500 nm which is a typical peak uptake of silver nanoparticles.The UV-Vis spectra of silver nanoparticles showed absorption peak in the range of 400 - 450 nm [6].

Figure 1. *Ipomoea batatas L.*

Figure 2. The colloidal of silver nanoparticle.



(a)



(b)

Figure 3. UV-Vis spectrum of $AgNO_3$ solution of 1×10^{-3} M (a) and silver nanoparticle (b).

Thus, the synthesis of nanoparticles can be done successfully by utilizing plant extracts, especially peel extract of *Ipomoea batatas L.* The process of reduction of Ag^+ ions into nanoparticles occurs outside the cell. The extracellular synthesis of silver nanoparticles could be conducted economically and safely by using leaf extract of *Catharanthus roseus Linn. G. Don* [25]. The extracellular synthesis was done by reacting metal ions with water or plant extracts. The use of plants in the synthesis of silver nanoparticles is relatively simpler and cheaper compared to other microorganisms. The types of plants that have been used for nanoparticle synthesis have been widely practiced by researchers. The biomolecule components contained in the plant are thought to act as a reducing agent, solvents and stabilizers in the formation of silver nanoparticles such as flavonoids, terpenoids, polysaccharides, alkaloids, and other secondary metabolites [25]. Three varieties of *Ipomoea batatas L.* have a major anthocyanin of cyanidin type [26]. However, anthocyanins can be oxidized to quinone. Thus it can be disclosed that in the process of forming silver nanoparticles using peel extracts of *Ipomoea batatas L* occurs reactions between hydroxyl groups (-OH phenolic) in anthocyanin compounds with silver ions to produce silver nanoparticles and quinone that cause the color of the solution to be reddish brown.

Each plant produces a variety of secondary metabolites. One of the most important groups of metabolites is a phenolic compound. Phenolics have at least one aromatic ring (C6) carrying a hydroxyl group. The antioxidant properties of phenolic compounds through their tendency to metal [3]. Based on the standard potential value, reaction (1) can be written as reactions (2) and (3). The sum of reaction (2) and (3) yields a cell potential of -0.445 volts. The negative cell potential value indicates that this redox reaction can't occur spontaneously.

$$Ag^+_{(aq)} + \text{-OH phenolic}_{(aq)} + H_2O_{(l)} \rightarrow Ag^0_{(s)} + \text{quinone}_{(aq)} + H^+_{(aq)} + O_{2(g)} \quad (1)$$

$$Ag^+ + e \rightarrow Ag^0 \quad E^\circ = 0.799 \text{ volt} \quad (2)$$

$$2\,H_2O \rightarrow 4H^+ + O_2 + 4e \quad E^\circ = -1.224 \text{ volt} \quad (3)$$

Although the cell potential of net reaction is negative, the Ag^+can be reduced to Ag^0 in the presence of -OH phenolic from plant extracts (a) (Figure 4). A complex formed (c2) (Figure 4) by the reaction between the -OH group of phenolic and the silver ion has an important role in the reduction and formation of silver nanoparticles. Many researchers concluded that complexes formed from citrate ion and silver ion will catalyze the reduction of Ag^+ to Ag^O even slowly in chemical procedures. The probability of reaction mechanism in extracellular synthesis of silver nanoparticle by supporting peel extract of *Ipomoea batatas L.* is shown in Figure 4. Anthocyanin has a structure as shown in 4(a), and simple structure is shown in 4(b) and 4(c1). When silver ion is reduced to form silver, -OH anthocyanin is oxidized to C=O quinon (c3).

3.2 SEM Image, Functional Groups, and Water Contact Angle of Polyester

SEM images of unmodified and modified polyesters (Figure 5) show that fabrics are composed of fibril. Figure 5(a) shows no contaminants on the surface of unmodified polyester fabrics. The modified polyester shows the presence of silver nanoparticles deposited on the surface of the polyester fabric (Figure 5(b) and 5(c)). Some of the microparticles cover the surface

of the fabric, since the nanoparticles undergo agglomeration to form larger particles [22]. In this study, silver nanoparticles successfully cover the surface of polyester fabric. The addition of silver nanoparticles results in a rougher cloth surface than a cloth without the addition of nanoparticles. However the surface between pure polyester (P0) and the modified polyester (P3) are practically no different. This could be caused by the HDTMS layer covered the silver nanoparticle so that the nanoparticle of silver can't be detected on surface images of P3, whereas in P4 the layer of HDTMS was covered by the silver nanoparticles so that the surface images are different, the silver nanoparticles are very much on the surface of the cloth P4.

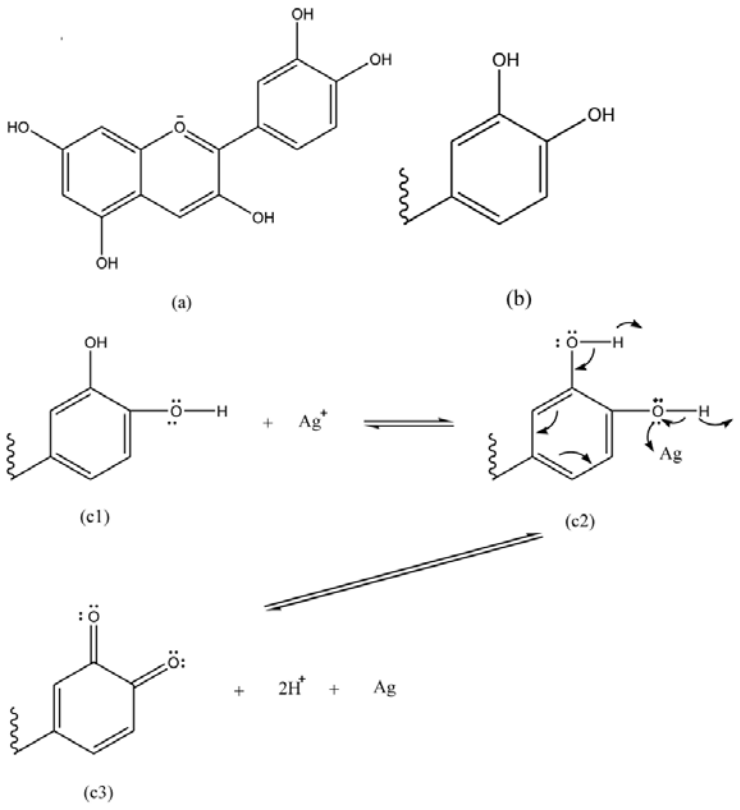


Figure 4. Reaction mechanism of formation the silver nanoparticles from anthocyanin (a) and silver ion.

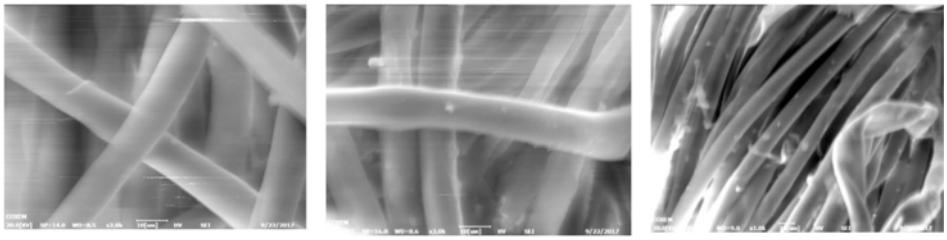


Figure 5. The SEM image of unmodified polyester (P0) (a), modified polyester (P3) (b), and P4 (c).

ATR-FTIR spectra of polyester (Figure 6) shows that polyester contains functional groups of -OH, -C-H, -COO esther, C=O, and C-O. Modification with silver nanoparticle and HDTMS compound does not show a new functional group. Thus, it does not effect the functional groups of polyester. This result is in accordance with the previous studies on the modification of cotton cloth with silver nanoparticles and HDTMS compounds [16]. The addition of the HDTMS compound can decrease absorption band in the spectrum of FTIR of modified Nylon [16].

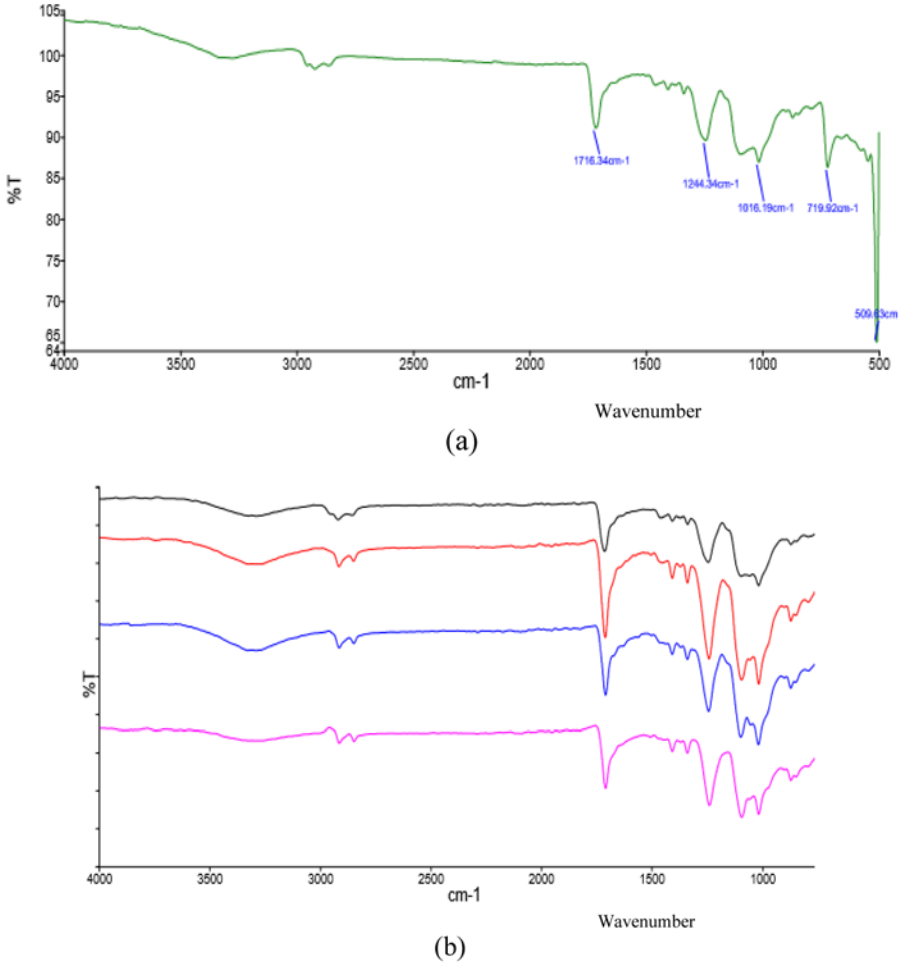


Figure 6. The ATR-FTIR spectra of unmodified polyester (P0)(a) and modified polyester (P1----, P2----, P3----, and P4----) (b).

The data of the contact angle in Figure 7 shows that the addition of one of the silver nanoparticles and HDTMS might increase the contact angle of the polyester fabric. However, the addition of HDTMS and the silver nanoparticles produces the highest contact angle on the resulting polyester. This is in contrast to the previous research [16, 17], that the addition of two types of compounds produced the highest contact angles. Note that the method of modification is different, in the previous research modifications were done with the addition of silver nanoparticle first and then followed

with the addition of HDTMS. While in this study, the addition of HDTMS was firstly performed and then the addition of silver nanoparticle. Thus the modification method seems to affect the properties of the fabric produced. The polyester after modification with HDTMS compound and silver nanoparticles has the largest contact angle value. The presence of a hydrophobic functional group increases the angle contact of water. The method of modification of the polyester fabric, qualitatively does not change the type of functional group present, but that affects the number of hydrophobic groups. Thus, the addition of nanosilver particles to hydrophobic materials, such as polyester composite, caused an increase of the hydrophobic groups and a decrease in surface free energy, so that an increase of the contact angle of P4.

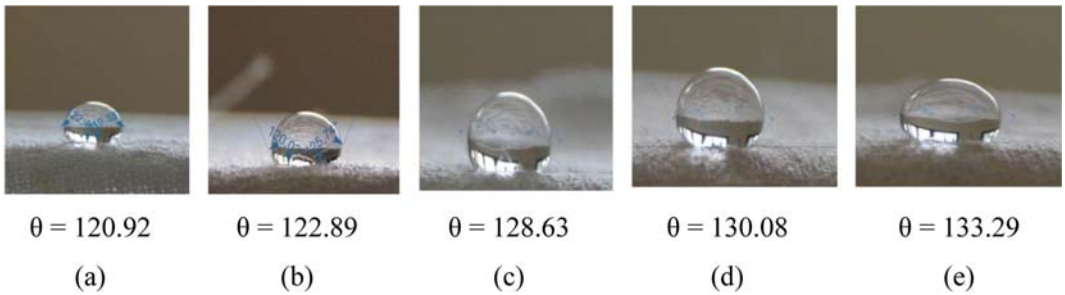


Figure 7. The water contact angle for P0 (a), P1(b), P2 (c), P3(d), and P4(e).

3.3 Antibacterial Activity of Polyester

The lowest inhibition zone of polyester in inhibiting the growth of *E. coli* bacteria is the P0 sample and the highest activity is the modified polyester (polyester - HDTMS) as shown in Figure 8. The highest inhibitory zone diameter of sample P-HDTMS occurs at the 60th hour. Incubation for 60 minutes is the maximum time for modified polyesters to inhibit bacterial growth of *E. coli*. Then the antibacterial activity decreases to the measurement at 72th hour. After 60 min, the ability of polyester in inhibiting bacterial growth decreased.

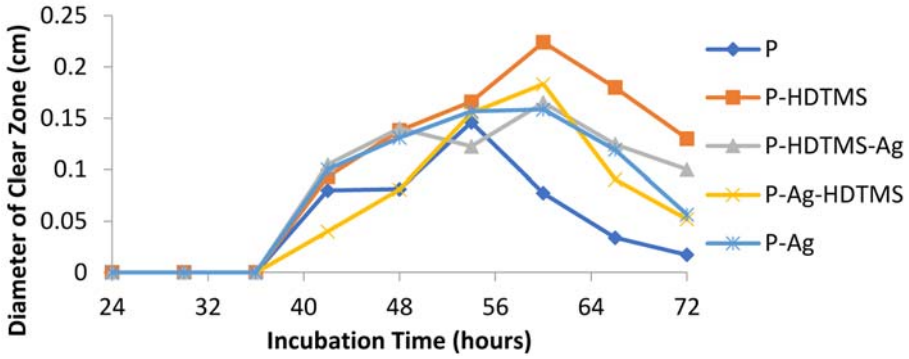


Figure 8. The antibacterial activity of polyester against *Escherichia coli* ATCC 35218.

The P-HDTMS has the highest ability to inhibit the growth of *E. coli* compared to other polyester samples. Thus, gram-negative bacteria are more easily inhibited by modified polyesters by silane compounds. The hydrophobic functional groups are more able to inhibit gram-negative bacteria. Modification of polyester by adding HDTMS compound can increase antibacterial activity of polyester cloths. The interaction between the ester groups and / or the -OH groups in the polyester and the -OH group in HDTMS produces a hydrophobic functional group so that the hydrophobic surface may cause the bacteria to inhibit its growth when interacting with the polyester fabric [27]. Compounds containing hydrophilic and hydrophobic groups are antibacterial agents that can damage cytoplasmic membranes and kill the bacterial cells [16]. The -OH functional groups (hydrophilic) from the polyester cloth and the HDTMS and hydrophobic groups of modified polyester bound to the silver will interact to -S-H on the cytoplasmic membrane by ionic or covalent bonds to form a bond -S-S- and produce S-Ag clusters that cause inhibition of cell respiration.

The lowest inhibition zone of polyester in inhibiting the growth of *S. aureus* is shown by unmodified polyester (P) and the highest activity is shown by the P-HDTMS-Ag (Figure 9). In general, the graph shows the antibacterial activity of the polyester sample which tends to increase with the time of incubation, which then decreases after 60 hours of incubation. The highest inhibition zone diameter of sample of P-HDTMS-Ag occurs at the 60th hour. Thus, incubation for 60 minutes is the maximum time for modified polyesters to inhibit bacterial growth of *S. aureus*. P-HDTMS-Ag shows the highest antibacterial activity. This is due to the interaction between ester and or -OH groups with -OH groups of HDTMS (Figure 10) resulting in hydrophobic groups which are not favored by bacteria followed with modification by deposition of Ag nanoparticles, which is an antibacterial agent as well, thus increasing activity of antibacterial from polyester fabric. Thus, the polyester modification method using a combination of HDTMS and nanoparticles increases the antibacterial activity of the fabric. The P-HDTMS-Ag has the highest ability to inhibit the growth of *S. aureus* compared to other polyester samples. Thus, gram-positive bacteria are more easily inhibited by modified polyesters by silane compounds and followed by coating of silver nanoparticles. The presence of hydrophobic function groups and silver nanoparticles may inhibit gram-positive bacteria. In other words, gram-positive bacteria can be inhibited by polyester composites, this is because the gram-positive bacteria consist of a thick layer of peptidoglycan and a layer of teicoic acid and also tend to be negatively charged and polar, so that antibacterial material is required that can inhibit its growth in the form of composites polyester with HDTMS and silver nanoparticles.

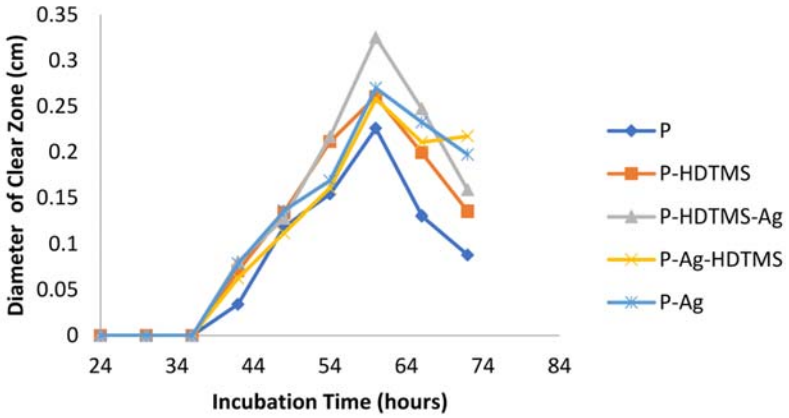


Figure 9. The antibacterial activity of polyester against *Staphylococcus aureus* ATCC 25923.

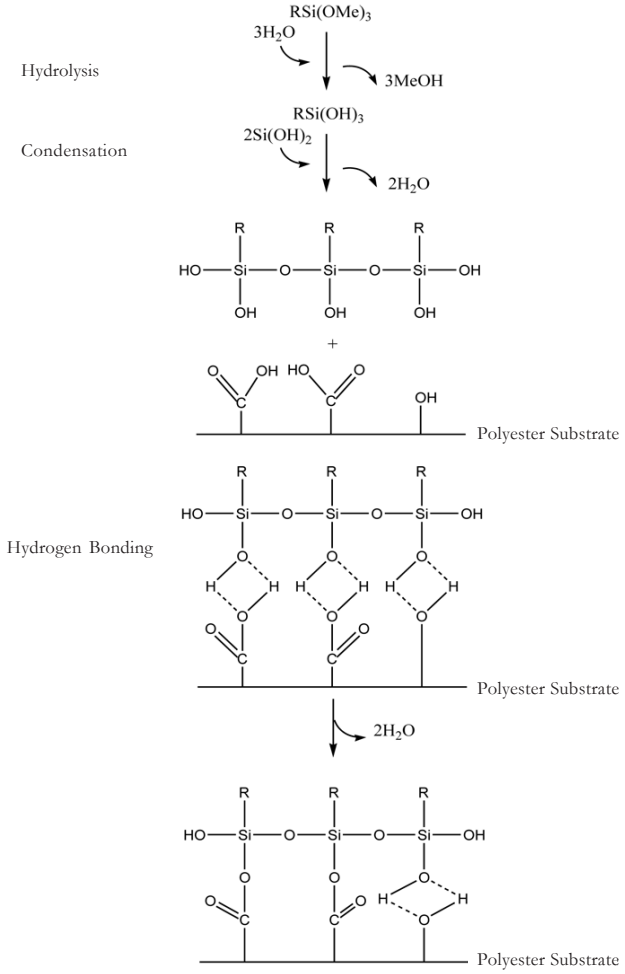


Figure 10. Interaction between the ester / the hydroxyl groups of polyester and the hydroxyl groups of HDTMS.

Based on results of Anova test in Table 1, there are three tests, namely interaction test between incubation time and type of sample, effect of incubation time, and effect of sample type on antibacterial activity of polyester cloths. The significant value of the interaction test between the incubation time and the sample type was 0.000 ($p<0.05$) for both against *E.coli* and *S.aureus*. Interpretation of $p<0.05$ indicates the effect of interaction between incubation time and sample type on antibacterial activity. The significant value of effect test of the incubation time on antibacterial activity was 0.000 ($p <0.05$). The interpretation of $p <0.05$ was the effect of incubation time on antibacterial activity. The significant value of the sample effect test on antibacterial activity was 0.000 ($p< 0.05$). Interpretation of $p<0.05$ indicates the influence of sample type on antibacterial activity. Thus, type of the polyester sample and the incubation time affect the antibacterial activity. The five types of polyester samples have different antibacterial activity significantly. Similarly with incubation time, different incubation times showed different antibacterial activity significantly as well.

Table 1. Significance of antibacterial activity of unmodified polyester and modified polyester against S*taphylococcus aureus* and *Escherichia coli.*

Source of data	Sum	Df	Average	F	Sig.
Escherichia coli ATCC 35218					
Time	0.413	7	0.059	83.652	0.000
Sample	0.053	4	0.013	18.897	0.000
Time* Sample	0.057	28	0.002	2.880	0.000
Staphylococcus aureus ATCC 25923					
Time	0.990	7	0.141	138.091	0.000
Sample	0.035	4	0.009	8.553	0.000
Time* Sample	0.052	28	0.002	1.821	0.000

The results of LSD tests in Table 2 show that the sample was a significant influence amongs the four modified samples with respect to P0 on the antibacterial activity (clear zone) for *Staphylococcus aureus* ATCC 25923, P0-P1, P0-P2, P0-P3 and P0-P4. All of the modified polyester shows significantly higher antibacterial activity than the unmodified polyester. Thus, modification can increase antibacterial activity of polyester cloths.

However, within the modified polyesters (P1-P4) no significant difference in theirs antibacterial activity was observed. Modification of polyester cloths by using silver nanoparticle, HDTMS, silver nanoparticle and HDTMS, and also HDTMS and silver nanoparticle has the similar effect toward antibacterial activity of polyester. Thus, the type of addition material and methods did not effect antibacterial activity of polyester cloths against *S.aureus*.

The results of LSD test in Table 2 shows that there are 7 variations of samples which have significant influence between two samples on antibacterial activity (clear zone) against *Escherichia coli* ATCC 35218, these are P0-P1, P0-P2 , P0-P3, P0-P4, P1-P3, P1-P4 and P2-P3. Therefore, it can be concluded that modification can

affect antibacterial activity of polyester cloths. The addition of silver nanoparticles, HDTMS, and combinations of the two might enhance the antibacterial activity of unmodified polyester fabrics to inhibit the growth of *E.coli*.

Table 2. Antibacterial activity between 2 polyester samples against *Staphylococcus aureus* and *Escherichia coli*.

Type of sample	Conclusion	
	Staphylococcus aureus	*Escherichia coli*
P0 - P1	Significant	Significant
P0 - P2	Significant	Significant
P0 - P3	Significant	Significant
P0 - P4	Significant	Significant
P1 - P2	Not significant	Not significant
P1 - P3	Not significant	Significant
P1 - P4	Not significant	Significant
P2 - P3	Not significant	Significant
P2 - P4	Not significant	Not significant
P3 - P4	Not significant	Not significant

Similarly, the addition method of nanoparticles and HDTMS or a combination of both might affect the antibacterial properties of polyester fabrics on inhibiting growth of *E.coli*. Considering that the differences in antibacterial activity between the following polyester samples P1-P3, P1-P4 and P2-P3 are significantly different, it can be concluded that the modification of fabric with two chemicals in P3 and P4 shows significantly differences compared to modifications using a single material (only nanoparticles or HDTMS) in P1 and P2.

Based on the results of the t-independent test as shown in Table 3, each sample of the polyester fabric shows no difference in antibacterial activity to inhibit the growth of *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923. Thus,the ability each sample to inhibit the *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923 is the same. This can be due to the fact that each type of bacteria has almost the same of chemical content. The chemical composition in cell wall of gram positive bacteria and gram negative bacteria is almost the same, i.e the presence of peptidoglycan and lipopolysaccharide.

Table 3. Interpretation of t-independent test for activity antibacterial of polyester against *Escherichia coli* ATCC 35218 and *Staphylococcus aureus* ATCC 25923.

Polyester sample	t-independent
P0	No difference
P1	No difference
P2	No difference
P3	No difference
P4	No difference

4. CONCLUSIONS

Based on the results of the research and discussion it could be concluded that the silver nanoparticles were successfully synthesized using a reducing agent of peel extract of *Ipomoea batatas L.* as confirmed at a wavelength of 436 nm with a reddish brown color. The unmodified polyester fabric

has the lowest contact angle value while the polyester fabric with the modification of HDTMS compound and silver nanoparticles has the largest contact angle value. There is a difference in the antibacterial activity of the fabric between the unmodified polyester and the modified polyester fabric at different incubation times in inhibiting the growth of *Escherichia coli* and *Staphylococcus aureus*. Samples of polyester - HDTMS - silver nanoparticle show the highest antibacterial activity against *S. aureus* with a strength of about 1.4 times greater than unmodified polyester and it is the highest in hydrophobicity properties. Each polyester sample has the same ability in inhibiting the growth of *Staphylococcus aureus* and *Escherichia coli*. Based on the antibacterial properties of the fabric then the modified polyester product of nanoparticles and HDTMS can be used as antibacterial materials for biomedical applications.

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