Biointerface Research in Applied Chemistry

www.BiointerfaceResearch.com

https://doi.org/10.33263/BRIAC0103.592598

ISSN 2069-5837

Original Research Article

Open Access Journal

Received: 01.03.2020 / Revised: 25.03.2020 / Accepted: 26.03.2020 / Published on-line: 29.03.2020

Enhancement of antibacterial properties of various polymers functionalized with silver nanoparticles

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ABSTRACT

A new green deposition of silver nanoparticles (AgNPs) on polymers was proposed in this work. *In-situ* synthesis of AgNPs on polymers was achieved via a green procedure using natural reducing agents, which are *Ageratum conyzoidez* and *Mikania micrantha*. Several characterizations of the treated polymers such as color transformation, surface morphology, elemental contents, and water absorption were comprehensively evaluated. For the application, the treated polymers were then tested against waterborne bacteria, which are *Escherichia coli* and *Bacillus cereus*. Rapid deposition of AgNPs via the presently biological method can be successfully achieved as observed via surface morphology analysis and energy dispersive X-ray investigation. Water absorption capabilities of the polymers can be decreased after attaching with AgNPs, which can also probably contribute to the enhancement of their antibacterial activities. This study observed that the treated polymers showed excellent antibacterial activities against *Escherichia coli* and *Bacillus cereus*. The findings of this study are useful in designing water purifiers to disinfect contaminated water.

Keywords: Silver nanoparticles; solution-immersion; antibacterial.

1. INTRODUCTION

Silver nanoparticles (AgNPs) have been attracting much interest due to their unique properties such as an excellent catalyst, antibacterial, and antifungal [1-5]. Those properties can be explored for medical devices, household products, cosmetic products, and textile industries [6]. Hence, a large production of AgNPs still becomes an interesting topic to be carried out [7,8]. AgNPs can be produced using three methods, which are chemical, physical, and biological. However, chemical and physical methods have the potential to produce negative effects because of the involvement of toxic chemicals and high energy consumption, respectively. Hence, biological method becomes an alternative procedure by using bacteria, fungi, and plant extracts.

Deposition of AgNPs on textiles has been widely studied to improve functionalization of textiles [9]. For instance, cotton and polyester coated with AgNPs proposed to produce antibacterial textiles can reduce about 99% colony of *Staphylococcus aureus* and *Klebsiela pneumoniae* [10]. Antifungal textiles were also fabricated by deposition of AgNPs on cotton and silk fabrics and tested against *Fusarium oxysporum* and *Alternaria brassicicola* [11]. Their study found that the treated silk fabrics offered better inhibition percentage compared to the treated cotton fabrics.

Catalytic properties of AgNPs deposited on cotton fabrics were also studied to degrade methylene blue [12]. For electrical device application, attachment of AgNPs on polyamide fabrics decreased their electrical resistance from 22.5 M Ω /sq to 4.5 Ω /sq for unmodified fabric and AgNPs-modified fabric, respectively [13]. Cotton and wool fabrics were also used to produce sun protection fabric materials and found that the UV transmission of the treated fabrics can be reduced up to 90% [14]. Moreover, a modification of cotton fabrics with AgNPs as a filter for conditioner air application to minimize the bioaerosol concentration was proposed [15]. The filter was able to reduce microorganisms activity present in the airborne particulate matter.

Several methods to fabricate textiles containing AgNPs have been proposed and still continuously revised [9,16]. Deposition of AgNPs on textiles can be carried out by three methods, namely, solution-immersion, layer-by-layer deposition, and sonochemical. The deposition of AgNPs on textiles is not permanent particularly against washing processing. Therefore, application of binders, cross-linkable polymer, and crosslinking agent has been adopted for stabilizing AgNPs on numerous fabrics [17-20]. For instance, the performance of cotton fabrics attached with AgNPs with and without binder (Printofix Binder MTB EG liquid) was evaluated [18]. Their study found that the antibacterial reduction of the treated fabrics decreased by up to 53% (Staphylococcus aureus) and 48.7% (Escherichia coli) after 20 washing cycles. Interestingly, the antibacterial reduction of the treated fabrics with binder can maintain at 94% (Staphylococcus aureus) and 85% (Escherichia coli) after 20 washing cycles.

The use of a crosslinking agent such as 1,2,3,4butanetetracarboxylic acid can also improve the stabilization of AgNPs on cotton fabrics [19]. Their observation exhibited that the antibacterial reduction of the treated fabrics without the crosslinking agent decreased up to about 60% (*Escherichia coli*) and 50% (*Staphylococcus aureus*) after 30 washing cycles. When the crosslinking agent was used, their antibacterial capabilities were still above 95% against all employed bacteria. The use of crosslinkable polysiloxane was also useful to achieve permanent antibacterial activity without any negative effect on other textile properties such as their comfort and strength [21].

For AgNPs deposition mechanism, it can be explained using three possible rules controlling AgNPs deposition on textiles such as (i) adsorption of silver ions on the fabric fiber surface, (ii) diffusion of AgNPs into fabric fiber, or (iii) interaction of AgNPs with fabric fibers. For instance, the possible interaction between AgNPs and sulfur atoms, which likely appeared as a result of the cleavage of S–S bond in the wool fiber was confirmed by the Xray photoelectron spectroscopy observation [22]. Alternatively, it was reported that there was an interaction between AgNPs with sulfur as well as nitrogen of the amino acids in the wool fabrics

2. MATERIALS AND METHODS

2.1. Materials.

Ageratum conyzoidez and Mikania micrantha leaves were collected from a location in Johor Bahru, Malaysia. Silver nitrate (AgNO₃) was purchased from QReC, Auckland, New Zealand and used as a silver source. Escherichia coli (E. coli) and Bacillus cereus (B. cereus) obtained from the Faculty of Biosciences and Medical Engineering, Universiti Teknologi Malaysia, were employed as bacteria models. The textile fabrics of 100% cotton with its weight of 135 g/m^2 , 100% polyester with its weight of 155 g/m², and 100% nylon with its weight of 102 g/m² were obtained from SBK Textile Trading, Johor Bahru, Malaysia. The culture media for bacterial growth were prepared using the agar powder purchased from Oxoid Ltd (Nutrient Agar CM0003, Oxoid, Basingstoke, UK).

2.2. Extraction procedure.

For this purpose, the extraction procedure was prepared using a method from previous works as basis [26]. Ageratum conyzoidez (15 g) leaves were washed using tap water and ultrapure water three times each to remove any impurities. Extraction was initiated by mixing the cleaned leaves with the ultrapure water (200 mL) and heated at a temperature of 250 °C for 30 min before cooling at room temperature. The extracted leaves were then filtered using a filter paper before storing it at a fridge at a temperature of 7 °C for further use.

2.3. Attachment of AgNPs on textiles.

The textiles were first to cut into a circular shape with a diameter of 13.5 mm. It is then washed using tap water and ultrapure water five times each and dried in an oven for 10 min. AgNO₃ solution was prepared by mixing 0.05 g of AgNO₃ salt with 10 mL of the ultrapure water. Therefore, cotton textiles were added into the solution and stirred using a magnetic stirrer at a speed of 100 rpm for 60 min. Next, *Ageratum conyzoidez* leaves extracts (10 mL) were added slowly into the mixture and then heated at a temperature of 50 °C for 15 min. It then followed by drying the treated textiles in an oven at a temperature of 70 °C for 15 min and cured in an oven at a temperature of 150 °C. A similar procedure was also adopted for *Mikania micrantha* leaves and other textiles (nylon and polyester). It is noted that the cotton,

3. RESULTS

3.1. Color properties.

Figure 1 shows the transformation of color for all untreated and treated textiles. It is obvious that the treated textiles show a different color compared to the pristine textiles. The results [23]. When cotton fibers with vat dye treatment were used, a large amount of AgNPs deposited on the cotton fabrics was mainly due to the electrostatic interaction between AgNPs and vat dyed cotton fabrics [24].

This study aims to propose a simple and green method for deposition of AgNPs without any chemical usages such as binders, cross-linkable polymer, and crosslinking agent. A physical treatment such heating was applied to increase the electrical charging on textile surface [25], so that, it can enhance the interaction between textiles and AgNPs. In addition, the proposed treatments are also expected to penetrate AgNPs into the fabric structure and remained there after the treatment.

nylon, and polyester treated with AgNPs synthesized using *Ageratum conyzoidez* are then abbreviated as the treated-A-C, treated-A-N, and treated-A-P, respectively. In addition, cotton, nylon, and polyester treated with AgNPs synthesized using *Mikania micrantha* are abbreviated as the treated-M-C, treated-M-N, and treated-M-P, respectively. Moreover, untreated cotton, nylon, and polyester are abbreviated as the untreated-C, untreated-N, and untreated-P, respectively.

2.4. Surface characterization and elemental analysis.

Scanning electron microscopy (SEM, S-3400N, HITACHI) was used to characterize surface morphology of the textiles before and after the attached with AgNPs. Elemental analysis of the textiles was analyzed using energy dispersive X-ray (EDX).

2.5. Water absorption test.

For this test, all treated and untreated textiles were used. The treated textiles were first soaked in the ultrapure water for 1 h. Water absorption characteristics for all treated textiles were estimated using the following equation:

$$WA = \frac{W_w - W_d}{W_d} \times 100\%$$
(1)

where WA is the water absorption (%), W_d is the initial weight of the textiles before absorption (g), and W_w is the weight of the textiles after absorption (g).

2.6. Antibacterial investigation.

Antibacterial properties of all untreated and treated textiles were evaluated against *B. cereus* and *E. coli* using the previous method as basis. Nutrient agar (NA) solution was prepared from the mixture of 10 g NA powder and 500 mL of ultrapure water before sterilizing at a temperature of 125 °C for 25 min. The sterilized solution (25 mL) was then poured into a petri dish and cooled at room temperature. Furthermore, 0.1 mL of bacterial culture (105 colonies) was spread onto a petri dish using a spread glass rod. Next, the treated-A (cotton, nylon, and polyester) and treated-M (cotton, nylon, and polyester) were placed on the NA surface. It was then stored in an incubator at a temperature of 36 °C for 24 h.

showed that the treated textiles exhibited a darker color than those of the untreated textiles. The effect of the interaction of AgNPs on the property of the fabric surfaces alters the color of the textiles into darker because AgNPs can excite resonance effect of light trapping when pairing with dielectric materials of cotton, polyester, and nylon [27].

Basically, color transformation for all textiles after exposing with AgNPs depends highly on textile types, size and shape of AgNPs, and deposition procedure. For instance, the pristine cotton fabric changed its color from white to dark green after treating with AgNPs [28]. The colors of the merino wool treated with AgNPs ranged from yellow/brown to red/brown and then to brown/black because of the surface plasmon resonance effect of AgNPs [23]. The effects of AgNPs shape on textiles colors were also investigated [29]. Their study observed that the wool fabrics exposed to AgNPs nanoprisms and two types of differently synthesized AgNPs nanodisks became blue, red, and yellow colored, respectively.



Figure 1. Transformation of color of (i) cotton, (ii) nylon, and (iii) polyester at different treatments. It is noted that a, b, and c refer to untreated, treated-A, and treated-M, respectively.

3.2. Surface morphology.

Surface morphologies of cotton, nylon, and polyester before and after deposited with AgNPs are shown in Figures 2-4. Figures 2a-2c show the surface morphology of untreated cotton and treated with AgNPs. It can be observed from Figure 2a for the pristine cotton fabric that the image demonstrates smooth longitudinal fibril structure of the fibers without any contaminating particles on the textile surfaces. In addition, the cotton textiles treated with AgNPs synthesized using *Ageratum conyzoidez* and *Mikania micrantha* as seen in Figures 2b-2c, respectively, showed rough surface structure, demonstrating AgNPs were formed on the cotton textiles. It was obvious from Figures 2b-2c that AgNPs were agglomerated and they formed a larger nanoparticle size.

As a comparison, the surface morphology of nylon before and after treated with AgNPs can be seen in Figures 3a-3c. The pristine nylon shows smooth longitudinal fibril structure of the fibers without any agglomerating particles. After treated with AgNPs synthesized using *Ageratum conyzoidez*, the surface morphology of the textile was rougher, indicating the presence of AgNPs on the textile surface. The presence of AgNPs on textiles can also be observed after treated with AgNPs synthesized using *Mikania micrantha*.





Figure 2. Surface morphology of (a) untreated-C, (b) treated-A-C, and (c) treated-M-C.





Figure 3. Surface morphology of (a) untreated-N, (b) treated-A-N, and (c) treated-M-N.





Figure 4. Surface morphology of (a) untreated-P, (b) treated-A-P, and (c) treated-M-P.

Moreover, Figures 4a-4c displays the surface morphology of the untreated polyester and treated with AgNPs. Similar morphology as observed for the pristine cotton and polyester fabrics can be found for the pristine nylon fabric, demonstrating a smooth longitudinal structure of the fibers without any particles on their textile's surfaces. When the nylon textiles were treated with AgNPs, their surface morphology demonstrated a rough surface indicating that AgNPs were successfully deposited on the polyester textiles (see Figures 4b-4c).

In general, the aforementioned observation indicated that AgNPs can be succesfully deposited on the textiles. Rough fiber surface can be observed after binding with AgNPs because the deposition of AgNPs may make a modification of the surface morphology of the textile fabrics. The formation of particles and agglomerates in different sizes of the microparticles can be observed because every fabric surface has a unique interaction between AgNPs and textile fiber [30].

3.3. Elemental analysis.

Table 1 lists the elemental analysis of the textiles untreated and treated with AgNPs. The carbon, oxygen, and silver can be detected on the textiles treated with AgNPs. For the untreated textiles, there was no silver element detected on the samples. Percentages of carbon, oxygen, and silver for the cotton treated with AgNPs synthesized using Ageratum conyzoidez were 58.99, 34.80, and 6.21%, respectively. For the nylon treated with AgNPs synthesized using *Ageratum conyzoidez*, the corresponding percentages were 43.07, 46.76, and 10.17%.

In addition, a slight difference can be observed when the polyester was treated with AgNPs synthesized using Ageratum conyzoidez by performing the corresponding percentages of 54.13, 37.99, and 7.88%. For cotton treated with AgNPs synthesized using *Mikania micrantha*, the percentages of the carbon, oxygen, and silver elements detected on the samples were 52.73, 45.40, and 1.87%, respectively. For the nylon treated with AgNPs synthesized using *Mikania micrantha*, the corresponding percentages were 38.03, 45.18, and 16.79%. Moreover, the polyester treated with AgNPs synthesized using *Mikania micrantha* performed the corresponding percentages of 42.28, 52.56, and 5.16%.

In general, the EDX analysis as listed in Table 1 indicated that the percentages of silver element detected on the samples ranged from 1.87 - 16.79%, depending on the textile types and synthesized AgNPs. AgNPs can be successfully attached to all textiles with the nylon deposited with AgNPs synthesized using *Ageratum conyzoidez* performed the highest silver percentages compared to other treated textiles (nylon > polyster > cotton). A similar finding was also observed for textiles treated with AgNPs synthesized using *Mikania micrantha*.

This fact confirmed that the nylon has some free electron pair higher compared to the polyester and cotton. Nylon has a -CONH group with free electron pairs on O and N atoms, as a result, nylon can bind more Ag+ ions compared to cotton and polyester [31]. The deposition of AgNPs on the cotton because it has a hydroxyl group (-OH) of cellulose so that the free electron pair O can bind to Ag+. For the polyester, it has free electron pairs in the ester (-COO) groups resulting in Ag+ ions that can bind to O to form AgO [32].

Type of	Treatment	Elemental composition (%)			
textiles		Carbon	Oxygen	Silver	
Cotton	untreated	71.82	28.18	-	
	treated-A-C	58.99	34.80	6.21	
	treated-M-C	52.73	45.40	1.87	
Nylon	untreated	65.24	34.76		
	treated-A-N	43.07	46.76	10.17	
	treated-M-N	38.03	45.18	16.79	
Polyester	untreated	63.48	36.52	-	
	treated-A-P	54.13	37.99	7.88	
	treated-M-P	42.28	52.56	5.16	

3.4. Water absorption analysis.

The ability of treated and untreated textiles towards absorption of water is shown in Table 2. It can be observed from the table that the water absorption of the untreated textiles was higher compared to the treated textiles. Attachment of AgNPs synthesized using *Ageratum conyzoidez* on cotton, nylon, and polyester performed their water absorption capabilities of 76.15, 84.49, and 78.39%, respectively, compared to before treated with the synthesized AgNPs, which are 83.52, 87.69, and 83.80%, respectively. When cotton, nylon, and polyester were treated with AgNPs synthesized using *Mikania micrantha*, they showed the water absorption capability of 66.93, 73.12, and 59.70%, respectively.

 Table 2. Water absorption percentages for all textiles

Type of textiles	Untreated (%)	Treated-A (%)	Treated-M (%)
Cotton	83.52	76.15	66.93
Nylon	87.69	84.49	73.12
Polyester	83.80	78.39	59.70

It was found that the textiles treated with AgNPs synthesized using *Ageratum conyzoidez* can reduce their water absorption ranging from 3 to 7.3% compared to untreated textiles with the highest reduction achieved for the treated cotton. For the textiles treated with AgNPs synthesized using *Mikania micrantha*, their water absorption reduction ranged from 14 to 24% compared to untreated textiles with the highest reduction achieved for the treated polyester. In general, attachment of AgNPs synthesized using *Mikania micrantha* on all textiles models was better in terms of water absorption reduction compared to textiles treated with AgNPs synthesized using *Mikania micrantha* on all textiles models was better in terms of water absorption reduction compared to textiles treated with AgNPs synthesized using *Ageratum conyzoidez*.

Findings of the present work are in agreement with the previous study who observed that the treated cotton performed better water resistance compared to the untreated cotton [33]. After dropping water to the treated and untreated cotton, the bulb of water can be formed on their treated textiles surface while the water can be perfectly absorbed in untreated textile. A similar result was also obtained by the previous work who examined the water absorption of polyester and viscose [34].

Their study reported that the deposition of AgNPs on the textiles can form a barrier between textile fibres and water, which can affect the water absorption characteristics. It is well known that the textile's fibre has a large surface area enabling it to maintain its moisture. Therefore, the deposition of AgNPs on textile surface can reduce their moisture properties.

3.5. Antibacterial capability against E. Coli.

The present study demonstrated the antibacterial capability of textiles treated with AgNPs synthesized using *Ageratum conyzoidez* and *Mikania micrantha* against *E. coli*. It is noted that there is no inhibition zone of untreated textiles against *E. coli*. The inhibition zones of cotton, nylon, and polyester treated with AgNPs synthesized using *Ageratum conyzoidez* were 15.50 ± 0.50 , 14.83 ± 0.29 , and 14.83 ± 0.58 mm, respectively. In addition, the cotton, nylon, and polyester treated with AgNPs synthesized using *Mikania micrantha* also performed the antibacterial capability with inhibition zones of 15.00 ± 0.00 , 14.83 ± 0.29 , and 15.67 ± 0.58 , respectively. For a comprehensive overview, inhibition zone of all treated and untreated textiles against *E. coli* is summarized in Table 3.

It is noted from Table 3 that all untreated textiles show no inhibition zone against all employed bacteria. For a cotton case, the inhibition zone of the treated textiles with AgNPs synthesized using *Ageratum conyzoidez* was slighlty higher compared to the treated with AgNPs synthesized using *Mikania micrantha*. For nylon and polyester cases, the inhibition zone of the treated textiles with AgNPs synthesized using *Mikania micrantha* can be enhanced compared to the treated with AgNPs synthesized using *Ageratum conyzoidez*.

Table 3. Inhibition zone of all untreated and treated textiles	against	E.
coli and B. cereus.		

Type of	Treatment	Bacteria (mm)	
textiles		E. coli	B. cereus
Cotton	untreated-C	0	0
	treated-A-C	15.50±0.50	16.50 ± 0.50
	treated-M-C	15.00 ± 0.00	15.50 ± 0.50
Nylon	untreated-N	0	0
	treated-A-N	14.83±0.29	16.33±1.15
	treated-M-N	14.83±0.29	17.00 ± 0.00
Polyester	untreated-P	0	0
	treated-A-P	14.83±0.58	16.00 ± 0.87
	treated-M-P	14.83±0.29	15.67 ± 0.58

3.6. Antibacterial capability against B. cereus.

The inhibition zones of cotton, nylon, and polyester treated with AgNPs synthesized using Ageratum conyzoidez against *B. cereus* were 16.50 \pm 0.50, 16.33 \pm 1.15, and 16.00 \pm 0.87 mm, respectively. For cotton, nylon, and polyester treated with AgNPs synthesized using *Mikania micrantha*, their inhibition zones were 15.50 \pm 0.50, 17.00 \pm 0.00, and 15.67 \pm 0.58, respectively. For a comprehensive overview, the inhibition zone of all treated and untreated textiles against *B. cereus* is summarized in Table 3.

The inhibition zone of the treated cotton textile with AgNPs synthesized using *Ageratum conyzoidez* against *B. cereus* was higher compared to the treated with AgNPs synthesized using *Mikania micrantha*. In addition, the treated nylon by AgNPs synthesized using *Mikania micrantha* performed inhibition zone

4. CONCLUSION

The present study was aimed to propose a new green method for the deposition of AgNPs on textiles and investigate their antibacterial capability against *E. coli* and *B. cereus*. Surface morphology analysis showed the different properties between untreated textiles and treated textiles. The treated textiles revealed the presence of AgNPs covering the textile fibres as observed from the scanning electron microscopy images. Water absorption analysis showed that the absorbency of untreated textiles was higher than that of treated textiles. The treated textiles with AgNPs synthesized using *Ageratum conyzoidez* performed the absorption

better than that of treated with AgNPs synthesized using *Ageratum* conyzoidez. Moreover, the inhibition zone of the treated polyester textile with AgNPs synthesized using *Ageratum conyzoidez* against *B. cereus* was higher compared to the treated with AgNPs synthesized using *Mikania micrantha*.

3.7. Antibacterial mechanism.

Tables 3 indicated that the treated textiles exhibited the antibacterial capability better against *B. cereus* compared to *E. coli*. As a Gram-negative bacterium, *E. coli* has a higher resistance compared *B. cereus* as a Gram-positive bacterium. This agrees with those results obtained from the previous study who observed that the inhibition zone of AgNPs against *B. cereus* was about 32 mm whereas for *E. coli* was 28 mm [35]. A similar finding was also reported in another study [36]. Their study observed that the inhibition zone of AgNPs against *E. coli* was 9.0±1.0 mm and the inhibition zone of 12.0±0.4 mm was achieved when tested against *S. aureus* as a Gram-positive bacterium. It was also stated that the antibacterial action of nanoparticles was more effective against Gram-positive compared to Gram-negative bacteria [37].

Antibacterial action of AgNPs is common ly associated with four basic mechanisms as follows. The first mechanism is probably by the adhesion of AgNPs on the surface of the bacterial cell membrane. AgNPs have a role in ruining lipopolysaccharide molecules and changing the properties of the bacterial cell membrane [38]. By attaching AgNPs to the cell membrane, they can increase the permeability of bacterial cell membrane [39]. AgNPs can form a gap of bacterial cell membranes resulting in leakage of cell contents and damage to cell membrane transport activity [40]

The second mechanism is possibly due to the penetration of AgNPs into bacterial cells, which contains mitochondria, proteins, ribosomes, and deoxyribonucleic acid [40]. It is also possible for the antibacterial action of AgNPs by the formation of reactive oxygen species (ROS). The interaction between ROS and AgNPs can release Ag+, which cause damage to DNA and proteins of microorganism [41]. Moreover, antibacterial action of AgNPs can also be viewed from the possible interaction between the released Ag+ and a thiol group that is a part of enzymes in bacterial cells. This interaction can change metabolism in cell and lead to the death of bacterial cells [42].

Moreover, it is also found that *Ageratum conyzoides* and *Mikania micrantha* have antioxidant features. Antioxidants have antimicrobial properties against bacteria [43,44]. It was also possible that their surface properties also affect the bacteria growth as reported in the previous work who found that antioxidant compounds can inhibit *Staphylococcus aureus* [43].

reductions ranging from 3 to 7.3% compared to untreated textiles with the highest reduction achieved for the treated cotton. For the textiles treated with AgNPs synthesized using *Mikania micrantha*, their water absorption reduction ranged from 14 to 24% with the highest reduction achieved for the treated polyester. All treated textiles showed an excellent antibacterial capability against *E. coli* and *B. cereus* depending on the textile types, synthesized AgNPs, and bacteria types. Overall, the treated textiles with antibacterial properties have the potential application for wound dressings, bandages, and coating for medical devices.

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6. ACKNOWLEDGEMENTS

All authors thank the Institut Teknologi Sepuluh Nopember (ITS) for providing the International Cooperation Research Grant 2018 under the contract number: 1258/PKS/ITS/2018. The authors also thank the Universiti Teknologi Malaysia for this collaboration to conduct the present study.



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