Antimicrobial activity of cotton fabric pretreated by microwave plasma and dyed with onion skin and onion pulp extractions

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Onion has been extracted to obtain colour compound for dyeing cotton fabric and the antimicrobial property of dyed fabric studied. The fabric is first desiccated with ethanol and then pretreated with low-temperature microwave plasma for 4 s with oxygen pressure of 0.2 Torr and power of 800 W. Thereafter, the plasma-treated samples are grafted for 10, 30, 60 min at 70 °C with onion skin and onion pulp extractions. The best inhibition zone of anti Staphylococcus aureus is found to be 1.1-0.8 cm by 10 min grafting time of onion skin extraction and 0.7-0.5 cm by 30 min grafting time of onion pulp extraction. The samples with 10 and 30 min grafting of both onion skin and onion pulp show anti S. aureus ability even after 5 times test washing, but both the samples lost their anti S. aureus property with 60 min grafting.

The FTIR-ATR spectrum of dyed cotton fabric shows flavonoids’ functional peak at 1624 cm⁻¹ of onion skin that provides cotton fabric brown colour with wash fastness rating of 4.

Keywords: Allium cepa, Antimicrobial activity, Cotton fabric, Microwave plasma, Onion pulp, Staphylococcus aureus

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Onion is one of the earliest cultivated foods with special spicy taste and distinctive smell. Through thousands year onion (Allium cepa) has become a world popular and healthy vegetable. It belongs to Allium family and is mainly composed of water (85-90 g/100 g), fructose, glucose, vitamin B₁, B₂, C, flavonoids, and organosulfur containings. Several studies13 have proved that its flavonoids and organosulfur containing components can provide antibacterial and antifungal properties. In ancient time, before synthetic dye was developed, onion was also used for natural plant dyes. The yellow-brownish colour is primarily derived from the onion’s dry papery skin called Quercetin, which is one kind of flavonoids, also named as C.I. Nature Yellow 10 (3,3',4',5,7-Pentahydroxyflavone, CI 75670).45 It gives the brownish orange colour with mordant alum, reddish brown with mordant chrome, and bright orange with mordant tin.5 Deo and Paul4 successfully used onion extract with natural mordant, such as tannic acid, tartaric acid and combinations, to dye ecru denim and got wash and light fastness ratings of 4 and 5-6 respectively.

Low-temperature plasma modification technique has been successfully applied to change the surface property without destroying the internal structure. Kan et al.6 proved that the low-temperature plasma gases play an important role in affecting the behaviour of chrome dyeing on wool fibre. Later, Poll et al.7 studied the penetration effect of different pressure plasma to textile surface. Yuranova et al.8 then used textile fabrics treated with RF-plasma and vacuum-UV followed by chemical grafting of silver salt to improve the antimicrobial activity.

In this work, the microwave-induced plasma technique has been used to modify the cotton fabric surface with an objective to study the effect of onion skin and onion pulp extractions’ grafting reaction for cotton dyeing and compare the wash fastness and anti S. aureus ability of differently treated cotton fabrics.

Onion (Allium cepa) was purchased from Pin-tong, Taiwan (species number CAL303). Pure cotton fabric having the structure 40×40/110×76 was used for the study. S. aureus (ATCC 6538P supplied by Taiwan Bioreources Collection & Research Center), 95% ethanol (Merck), tryptic soy agar (Merck) were used for antimicrobial test.

Addition-free cotton fabric was first cut into 30×30 cm² each for the experiments. The cotton fabric was first dipped into ethanol for 5 min, washed in a reservoir several times using de-ionized water with ultrasonic vibration for 10 min at 70°C and then dried with nitrogen gas.

Antenna-coupling microwave was distributed by 12×12 array-elements onto a quartz plate and extended to an effective area of ≈ 40×40 cm² in the

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reactive chamber (Fig. 1). The glow discharge microwave plasma was produced using a microwave (≈2.45 GHz) plasma generator (PHILIPS MW-97010) with the maximum power of 2 kW. An experimental value of ≈800 W was applied with low energy reflection. Plasma in the vicinity of the sample was characterized using the Langmuir probe (Fig. 2) connected to an electrometer (Keithley, Model 6514). This invasive, electrostatic probe utilized 10 mm long tungsten wire of 0.25 mm in diameter as the probing tool. A flow rate for O₂ gas (purity of 99.99 %) was maintained by keeping a pressure of ≈0.2 Torr and power of 800 W in the plasma processing chamber. Plasma treatment time was set at about 4 s.

Onion pulp juice was extracted by cold press method. Onion skin liquid was directly extracted with de-ionized water with weight ratio 1:40 for 60 min. The plasma pretreated cotton fabric was separated into two groups for onion pulp extraction and onion.
skin extraction dyeing. The dyeing was carried out at 70°C for 10, 20 and 30 min respectively.

The antimicrobial test method JIS L1902-1998 was used for testing of antibacterial activity and efficacy on textile products. The dyed cotton fabric was cut into 20 mm×20 mm size and put inside tryptic soy agar petri dish at 37°C for 8 h to check the inhibition zone.

The colour fastness to launder test was based on AATCC 61-2A (ref. 11). Samples were washed under 40 rpm at 49°C for 45 min (equal to 5 times washing) and then de-ionized water was used to clean three times for 2 min. The treated samples then used AATCC gray scale for colour change to judge the wash fastness rating from 1 to 5.

Fourier transform infrared attenuated total reflection (FTIR-ATR) spectrophotometer (Bomem, DA8.3 ) is a useful method to obtain surface chemical structural information with a minimum sample preparation. Original (without plasma and dyeing treatment) cotton fabric and plasma pretreated cotton fabrics with 30 min grafting time of onion skin and onion pulp extraction were selected to compare the surface structure difference.

Low-temperature O₂ plasma pretreatment produces functional groups (-OOH, -OH, -C=O) on the polymer surface. These polar functional groups increase polymer hydrophilic properties. The anti S. aureus test of inhibition zone with different onion skin and onion pulp extractions and different grafting time is given in Table 1.

The experimental results reveal that both plasma untreated cotton fabric with different onion grafting time and plasma pretreated cotton fabric without onion grafting show negative anti S. aureus property. But plasma pretreated cotton fabric is helped by both onion skin and onion pulp extractions with grafting reaction and shows positive anti S. aureus ability. The inhibition zone is found to be biggest at 30 min but decreases at 60 min, which might be attributed to the high temperature instability property of onion antimicrobial components.

Plasma-treated and untreated cotton fabrics were grafted with onion skin and onion pulp respectively and the wash fastness of the fabrics tested. As the onion pulp grafted sample shows very light grey colour, their wash fastness is not rated. The 10, 30 and 60 min onion skin grafted samples show brown colour with wash fastness rating of 4 (judged by AATCC grey scale).

Samples with the same washing treatment were tested to determine the change of anti S. aureus property. The test results are shown in Table 2. Both

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Table 1—Anti S. aureus inhibition zone of dyed cotton fabrics with/without microwave plasma pretreatment

<table>
<thead>
<tr>
<th>Onion part</th>
<th>Grafting time min</th>
<th>Inhibition zone, cm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Without plasma</td>
<td>With plasma</td>
</tr>
<tr>
<td>Nil (Control)</td>
<td>0 (Control)</td>
<td>0</td>
</tr>
<tr>
<td>Skin</td>
<td>10</td>
<td>0</td>
</tr>
<tr>
<td>Skin</td>
<td>30</td>
<td>0</td>
</tr>
<tr>
<td>Skin</td>
<td>60</td>
<td>0</td>
</tr>
<tr>
<td>Pulp</td>
<td>10</td>
<td>0</td>
</tr>
<tr>
<td>Pulp</td>
<td>30</td>
<td>0</td>
</tr>
<tr>
<td>Pulp</td>
<td>60</td>
<td>0</td>
</tr>
</tbody>
</table>

Table 2—Anti S. aureus test of cotton fabric with and without microwave plasma pretreatment after 5 times washing

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Skin</th>
<th>Pulp</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 min</td>
<td>+</td>
<td>+</td>
</tr>
<tr>
<td>30 min</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>60 min</td>
<td>+</td>
<td>+</td>
</tr>
</tbody>
</table>

(+) = With anti S. aureus ability; (-) = Without anti S. aureus ability.
the samples with onion skin and onion pulp of 60 min grafting time lost their anti *S. aureus* property since they obtained smaller anti *S. aureus* inhibition zone before washing.

The analytical methods used for the identification of flavonoids in onion have been reported in several papers. High performance liquid chromatography is the most popular technique used. But for this research, FTIR-ATR is the best choice to detect the surface composition of flavonoids’ structure. It is observed from the experimental results (Fig. 3) that the onion skin grafted cotton fabric shows strong flavonoids functional group (C=O) at 1624.05 cm⁻¹. Untreated cotton fabric does not show any absorption peak in this area. This also explains why onion skin grafted cotton fabric expresses deeper brown colour than the other two samples.

It is observed that the cotton fabric with direct grafting of onion skin or onion pulp extractions shows negative results with anti *S. aureus* property. On the contrary, cotton fabric with microwave O₂ plasma pretreatment provides more polar functional groups on the cotton surface and makes onion skin or onion pulp grafting reaction with anti *S. aureus* ability.

Cotton fabric with longer onion grafting time does not show bigger anti *S. aureus* inhibition zone. It might be so because some anti *S. aureus* components of onion are not stable under long high temperature reaction. Cotton, put through 5 times washing test with 10 and 30 min grafting time using onion skin and onion pulp still retains anti *S. aureus* ability. FTIR-ATR peak at 1624.05 cm⁻¹ proves that the onion skin grafting reaction can make flavonoids bonded on the plasma pretreated cotton surface. This brown colour cotton fabric has a wash fastness rating of 4.

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References