Atmospheric Plasma-Aided Biocidal Finishes for Nonwoven Polypropylene Fabrics. II. Functionality of Synthesized Fabrics

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ABSTRACT: Atmospheric plasma-aided graft copolymerization of textile materials provides single or multiple functionality polypropylene (PP) modified fabrics. Biocidal PP's are modified ones to kill or inhibit the growth of microorganisms such as bacteria, molds, and fungi, and insect and tick repelling action. Novel PP biocidal fabrics synthesized by graft copolymerization using plasma-aided technique (see part I of this study) using antibacterial and insect repellent agents have been tested and evaluated and proved to be antimicrobial, tick repellent, and antistatic. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 103: 1911–1917, 2007

Key words: nonwoven PP; atmospheric oxygenated helium plasma; GMA; β-CD; MCT β-CD; HTCC; antistatic; antimicrobial; insect repellent fabrics

INTRODUCTION

Modifying textile materials by plasma-aided techniques has proven to be an efficient, low-cost method in which numerous functionalities could be added.[1–3] While vacuum plasma techniques have shown successful surface modification of textiles, it does not provide on-line treatment, which is important for textile industry. Research and development to generate atmospheric plasmas has shown great success and potential for on-line implementation in the textile industry.[4–9] Atmospheric plasma-aided graft copolymerization of textile materials provide single or multiple functionality modified fabrics. One of the most important functionalization is a biocidal fabric to kill or inhibit the growth of microorganisms such as bacteria, molds, and fungi, and insect repellent action. Biocidal fabrics are very important and have a wide range of applications for human demands because of the appearance of fatal diseases. These fabrics are generally made from nonwoven or woven natural or synthetic fabrics or a blend of them. The modified fabrics are important for medical, hygienic, and home usage applications and have received a great importance because of the recent appearance of fatal diseases like Aids and virus Hepatitis. As a result, there is an increased demand of the biocidal textiles, especially those used in hospitals to prevent or minimize infection or transmission of diseases and protecting health care workers.[10] Novel polypropylene (PP) biocidal fabrics synthesized by plasma-aided graft copolymerization technique (see part I of this study[11]) with antibacterial agents have been tested and evaluated for bacterial and insect repellent activity and have been proved to be antimicrobial, insect repellent, and antistatic. This article includes testing, analysis, and evaluation of the efficiency of the synthesizing biocidal PP fabrics as permanent antistatic, antimicrobial, and insect repellent active agents. A comparison to polyamide fabric (PA) samples, synthesized with same method but has an inclusion of different insect repellent agents, is also included.

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EXPERIMENTAL

Materials and methods

Plasma-treated nonwoven PP fabrics were grafted with glycidyl methacrylate (GMA) and then linked to β-cyclodextrin (CD) or MCT β-CD (MCT-CD) or quaternary ammonium chitosan derivative (HTCC). Atmospheric plasma, generated at 5 kHz audio-frequency using helium (100%) or a mixture of helium and oxygen (99% He + 1% O_2) or helium and forming gas (99% He + 1% N_2 and H_2), provides surface activation for grafting GMA onto PP. The PP/GMA is then reacted with either CD or quaternary ammonium chitosan to produce antimicrobial fabrics. Biocidal guests were introduced onto the CD cavity, which included *p*-hydroxy benzoic acid, $AgNO_3$-ethanolamine mixture, iodine, *N*,*N*-diethyl-metatoluamide (DEET), and perfumed extracts such as citronella, jasmine, and sweat basil. Figure 1 shows a representative schematic of the plasma-aided graft copolymerization and the process sequence to produce antibacterial and insect repellent fabrics. Synthesized PP fabrics were tested for static decay with positive and negative charges of 5 kV; parameters of tested fabrics are summarized in Table I. PP/GMA/CD with inclusion agents such as I_2, $AgNO_3$, or *p*-hydroxy benzoic acid, listed in Table II, were tested for ultraviolet spectral analysis. PP fabrics were plasma treated at two different exposure times, grafted with GMA, and then reacted with antimicrobial agents, listed in Table III, and tested for antimicrobial activity. Plasma-treated fabrics grafted with GMA and then reacted with β-CD and complexed with either DEET, citronella, or sweet basil extract as insect/tick repellent agents, listed in Table IV, were tested for tick repellent activity. Additional to PP, PA samples were also plasma treated, for purpose of comparison to PP, and then grafted with GMA and β-CD as previously described for the inclusion of two proprietary formulations of BioUD30 ® (spray or silicon) (HOMS, LLC., Clayton, NC). Both formulations consist of 30% 2-undecanone by weight.

TABLE I
Composition of Fabrics Tested for Antistatic

Sample	Sample parameters
PP control	Control PP
PP10	O_2 plasma treated for 5 min, PP/29.7% GMA/2.6% HTCC
PP17A	O_2 plasma treated for 2 min, PP/13.0% GMA/0.96% MCT-CD/1% HTCC
PP22	O_2 plasma treated for 2 min, PP/3.9% GMA
PP24	O_2 plasma treated for 5 min, PP/8.7% GMA
PP31	O_2 plasma treated for 2 min, PP/1.68% GMA

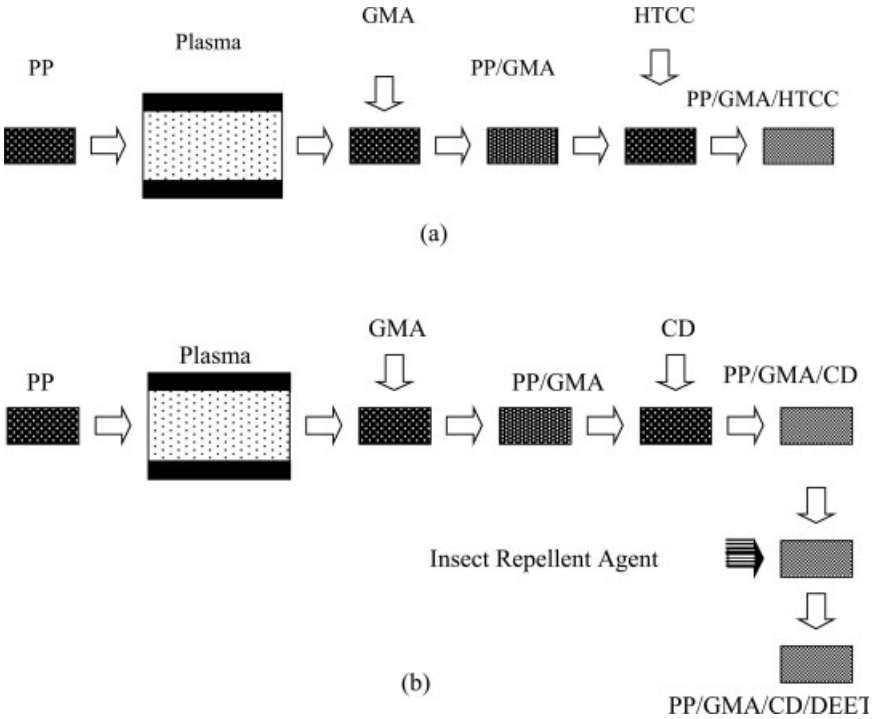

Figure 1 Schematic of the plasma-aided graft copolymerization process. Path one (a) shows PP/GMA/HTCC production. Path two (b) shows PP/GMA/CD production followed by inclusion of insect repellent agent (such as DEET, citronella, sweet basil, or BioUD30®).

TABLE II
Composition of Fabrics Analyzed with Ultraviolet Visible Spectra

Sample	Sample parameters
(a) "Control 13"	Control PP/10.5% GMA/0.74% MCT-CD
(b) "Control 17"	Control PP/13% GMA/0.96% MCT-CD
(c) "17A"	PP/13.0% GMA/0.96% MCT-CD/1% HTCC
(d) "13C"	PP/10.5% GMA/0.75% MCT-CD/1.02% DEET
(e) "13D"	PP/10.5% GMA/0.75% MCT-CD/0.64% *p*-hydroxy benzoic acid
(f) "13A"	PP/10.5% GMA/0.74% MCT-CD/1.2% I_2
(g) "13B"	PP/10.5% GMA/0.75% MCT-CD/6.4% $AgNO_3$

TABLE III
Composition of Fabrics Tested for Antimicrobial Activity

Sample	Sample parameters
Fc	Untreated (control)
F12	O_2 plasma treated for 2 min, PP/18.08% GMA
F21	O_2 plasma treated for 2 min (no grafting)
F23	O_2 plasma treated for 5 min (no grafting)
F10	O_2 plasma treated for 5 min, PP/29.7% GMA/2.6% HTCC
F18	O_2 plasma treated for 2 min, PP/38% GMA/7.2% CD
F19	O_2 plasma treated for 2 min, PP/20.62% GMA/1% CD
F26	H_2/N_2 plasma treated for 2 min (no grafting)
F27	H_2/N_2 plasma treated for 5 min (no grafting)

TABLE IV
Composition of Fabrics Tested for Insect Repellent Activity

Sample	Sample parameters
18A	O_2 plasma treated for 2 min, 38% GMA/7.2% CD/1% DEET
18B	O_2 plasma treated for 2 min, 38% GMA/7.2% CD/1% citronella
19A	O_2 plasma treated for 2 min, 20.6% GMA/1% CD/1% citronella
19B	O_2 plasma treated for 2 min, 20.6% GMA/1% CD/1% sweet basil
3B	O_2 plasma treated for 2 min, 35.4% GMA/1.0% CD/3.13% BioUD30 silicon®[a]
21′A	O_2 plasma treated for 2 min, 81.85% GMA/1.0% CD/0.477% BioUD30 spray®[b]
21′B	O_2 plasma treated for 2 min, 81.85% GMA/1.0% CD/0.84% BioUD30 silicon®[c]

[a] 0.939% 2-undecanone.
[b] 0.143% 2-undecanone.
[c] 0.252% 2-undecanone.

Antistatic test method

Nondestructive static decay was conducted using Electro-tech systems Model 806A test fixture with ETS Model 406 static decay meter inside Model 518 automatically controlled environmental chamber, at 5 kV charging voltage with both positive and negative polarity tests. Static decay was measured at 10 and 50% cut-offs.

Ultraviolet visible spectroscopy

Ultra violet visible Spectrophotometer model Cary 300 version 9.00 was used to scan the UV absorbance of the modified PP fabrics.

Antibacterial activity test method

Bacterial strains and growth media

Escherichia coli HB101, *Lactobacillus plantarum* LA70, and *Staphylococcus aureus* were obtained from the U.S. Food Fermentation Laboratory Culture Collection (USDA-ARS, Raleigh, NC). Bacterial strains were grown overnight on tryptic soy broth or agar (Difco Laboratories, Detroit Mich.) for 14 h. A modified form of American Association of Textile Chemist and Colorist test Method 100 (AATCC-100) was adopted.[12]

The reduction in numbers of bacteria was calculated using the following equation:

$$\text{Reduction rate } (\%) = (A - B)/A \times 100$$

where A is the number of bacterial colonies from untreated fabrics and B is the numbers of bacterial colonies from treated fabrics. One log reduction indicates that finished fabrics were able to kill about 90% of the bacteria.

Lactobacillus plantarum (LA70)

One piece of fabric (1 × 1 in.) was transferred to sterilized plate. A 100 μL containing 10^7 CFU/mL of LA70 was transferred onto the surface of the fabric, which was placed in a Petri plate (100 × 15 mm in diameter; Fisher scientific, Pittsburgh, PA). Plates containing the fabrics were covered and incubated for 3 h at 30°C. After 3 h, the fabrics were transferred into stomacher bags (Spiral Biotech, Norwood, MA) with 10 mL sterilized 8.5 g/L NaCl (Saline), which were treated for 1 min on high in the stomacher (Model TR5T, Tamar Co., Cincinnati, OH). The supernatant was diluted and plated on nutrient agar plates using a spiral platter (Model 4000, Spiral Biotech). After incubation for 24–48 h at 30°C, the bacterial colonies were counted by an automated spiral plate reader (QCount, Spiral Biotech).

Escherichia coli (HB101) and *Staphylococcus aureus*

Same method was used for LA70 except two pieces were used (1 × 1/2 in. each). One piece of the fabric was transferred to a small (35 × 10 mm in diameter) Petri plate (Fisher brand), 100 μL containing 10^7 CFU/mL of organism transferred onto the surface of fabric, and the fabric was covered by the second piece. The plate was covered and transferred to 100 × 15 mm in diameter Petri plate (Fisher), 1 mL sterilized dH_2O was added to the large plate outside the smaller Petri plate containing the fabric sample, to protect the fabric from dryness. Both plates were covered and incubated for 3 h at 37°C.

Tick repellency assay

American dog ticks, *Dermacentor variabilis* (Say), were obtained from a lab-reared strain maintained on New Zealand White Rabbits, *Oryctolagus cuniculus* L., in the Department of Biological Sciences at Old Dominion University, Virginia, and reared as described by Sonenshine, 1993.[13] Rearing conditions were (26 ± 1)°C, (92 ± 6)% relative humidity, and 14:10 (L:D).

The testing arena was constructed of an upside down 60 mm diameter Petri dish lid (Becton Dickinson, Franklin Lakes, NJ) with a plastic ring glued to the outside top rim to allow approximately 2 mm of space between the dish and the bottom of the arena. The ticks had enough room to freely move about the arena, but could not flip over or completely avoid the testing surface. Figure 2 illustrates a schematic of the test arena.

During the bioassay, two equal size half-circle pieces of fabric (1.4 cm^2) were placed on a plastic surface; six unfed male ticks were placed in the center of the arena on top of the fabrics and the Petri dish lid covered the fabrics and ticks. The fabrics were positioned so that each fabric covered exactly half of the 60 mm diameter arena, thereby forcing the ticks make a choice about which type of fabric on which to reside. All tests were conducted in total darkness at (26 ± 2)°C, (80 ± 3)% relative humidity. Additionally, each arena was separately covered with a lid wrapped with aluminum foil to limit the entrance of light into the arena during the recording of the results. Distribution of the ticks in the arena was recorded at 0.5, 1, 2, and 3 h.

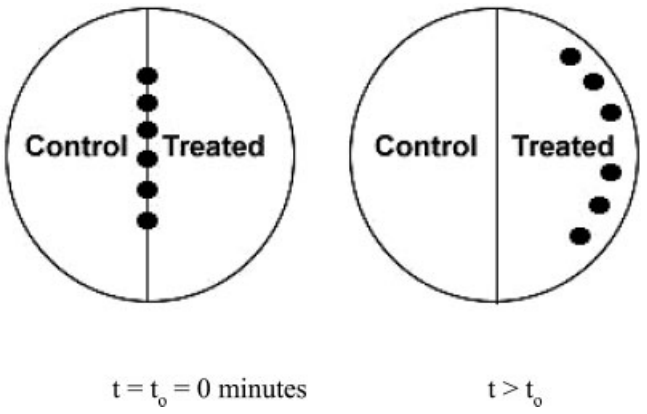

Figure 2 Schematic of the experimental arrangement for tick repellency assay. Two equal size (1.4 cm^2) pieces of fabric were placed in the arena and six ticks were introduced in the center of the arena where the fabrics joined. The distribution of the ticks was recorded at 0.5, 1, 2, and 3 h.

RESULTS AND DISCUSSION

Antistatic test

The static charge tests were repeated four times for each fabric to obtain the average. The 10% cut-off for a positive polarity 5 kV charge is 0.22 s for the PP control fabric, and greater than 90 s for fabric PP17A (PP/13.0% GMA/0.96% MCT-CD/1% HTCC), fabric PP22 (PP/3.9% GMA), and fabric PP24 (PP/8.7% GMA). It appears that GMA graft changes the antistatic property of PP for positive charges; however, it is 0.01 s for the treated fabric (O_2 plasma treated for 5 min, PP/29.7% GMA/2.6% HTCC), indicating that higher percent GMA improves the antistatic property for positive charge. The 50% cut-off is 0.01 for all treated and untreated fabrics. The negative-polarity 5 kV charges showed that both 10 and 50% cut-offs are 0.01 s for all fabrics. This indicates that PP is antistatic and that grafting did not alter its antistatic efficiency for negative charges. It is hypothesized that the treatment method further enhances the antistatic features but not seen on a scale of 0.01 s (Table V).

TABLE V
Antistatic Test Results

	Cut-off	
	10%	50%
Blank PP positive polarity	0.22	0.01
Blank PP negative polarity	0.01	0.01
PP 10 positive polarity	0.01	0.01
PP 10 negative polarity	0.01	0.01
PP 17A positive polarity	>90	0.01
PP 17A negative polarity	0.01	0.01
PP 22 positive polarity	>90	0.01
PP 22 negative polarity	0.01	0.01
PP 24 positive polarity	>90	0.01
PP 24 negative polarity	0.01	0.01
PP 31 positive polarity	N/A	N/A
PP 31 negative polarity	0.01	0.01

Ultraviolet visible spectroscopy test

The UV visible spectra of the modified PP fabrics were measured using a Cary 300 version 9.00 UV spectrom-

eter with Scan Software Version 3.00(182) and the absorbance spectrum of the modified fabrics is shown in Figure 3. The control fabrics (a) "control 13" and (b) "control 17" containing MCT-CD show the typical peaks of the triazinyl group at about 210 and 240 nm.[14] The PP fabric (c) "17A", which contains 0.96% MCT-CD/1% HTCC also shows the same two peaks as the control samples. The PP fabric (e) "13D" was modified with MCT-CD/*p*-hydroxy benzoic acid, and thus the characteristic additional peak of benzoic acid at 251 nm is evident from the UV visible spectra. The PP fabric (f) "13A", which contains inclusion of I_2, shows its peak at 295 nm; and that with inclusion of DEET (d) "13C" overlaps with the triazinyl ring and gives a peak at 217 nm. The fabric (g) "13B," which includes $AgNO_3$, shows an additional not well-defined peak at 308 nm. The UV visible spectra proved the grafting of MCT-CD groups onto PP fabrics in addition to the inclusion compounds such as I_2, DEET, benzoic acid, and $AgNO_3$.

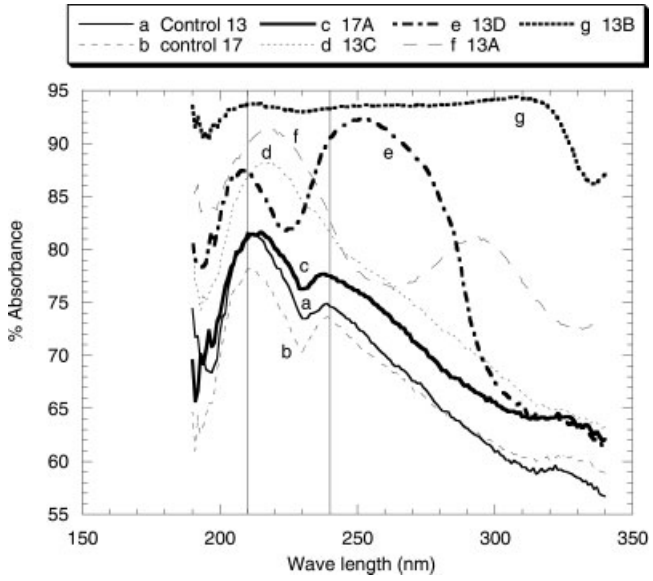

Figure 3 Ultraviolet visible spectra of modified PP fabrics.

Antimicrobial test

Lactobacillus plantarum (LA70) test

Test results using *L. plantarum* (LA70) has shown that fabric F10 (PP/29.7% GMA/2.6% HTCC) has the highest antimicrobial effect with about 1 log reduction (90% kill), followed by F19 (PP/20.62% GMA/1% β-CD) and F18 (PP/38% GMA/7.2% β-CD). Fabric samples Fc, F12, F21, F23, F26, and F27 have no antimicrobial effect, indicating that un-grafted samples do not incorporate antimicrobial effect. It appears from the results of fabrics F19 and F18 that lower percent of GMA and β-CD is more effective. Figure 4 shows the

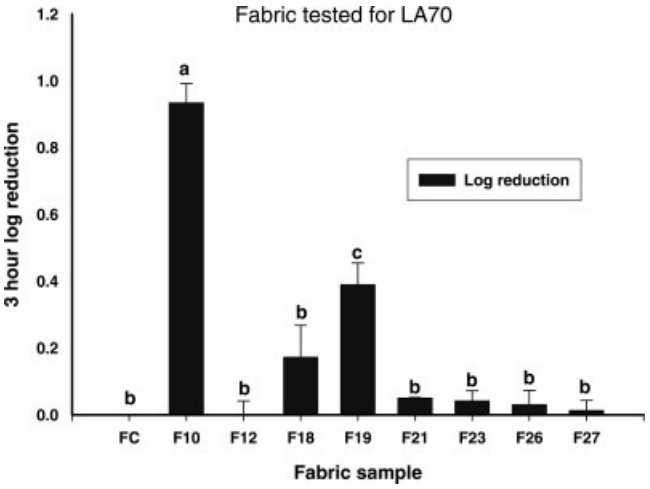

Figure 4 Reduction in viable cells of *L. plantarum* LA70 on PP fabrics. Fabrics are coded as listed in Table I. The log reduction in cell numbers after 3 h incubation at 30°C is shown. The error bars represent the standard deviation for three replications. All bars with different letters are significantly different ($P < 0.05$).

3-h log-reduction graph of the *L. plantarum* (LA70) test results.

As shown in Figure 4, fabric F10, PP/29.7% GMA/2.6% quaternary ammonium chitosan (HTCC), shows the greatest reduction in cell counts, with just 0.94 log reduction in cell counts for *L. plantarum* LA70. Fabric F19, which was grafted with PP/20.62%/GMA/1% β-CD, showed a 0.39 log reduction. All other tested samples showed less than a 0.1 log reduction.

E. coli test

Test results using *E. coli* are similar to the LA70 test results. Fabric F10 was the most effective followed by F19 and F18, with F18 been the least effective. Fc, F12, F21, F23, F26, and F27 have shown no antimicrobial effect. Figure 5 shows the 3-h log-reduction graph of the *E. coil* test results.

As shown in Figure 5, fabric F10, PP/29.7%/GMA/2.6 quaternary ammonium chitosan (HTCC), shows the best effect with 1.36 log reduction. Fabric F19, PP/20.62% GMA/1% β-CD, shows 1.1 log reduction. Fabric F18, PP/38% GMA/7.2% β-CD, has only 0.54 log reduction. All other tested samples are either 0 or slightly less than 0.1 log reduction.

Staphylococcus aureus test

Test results using *S. aureus* are similar to the LA70 and *E. coli* test results. Fabric F10 was the most effective followed by F19 and F18, with F18 been the least effective. Fc, F12, F21, F23, F26, and F27 have shown no antimicrobial effect. Figure 6 shows the 3-h log-reduction graph of the *S. aureus* test results.

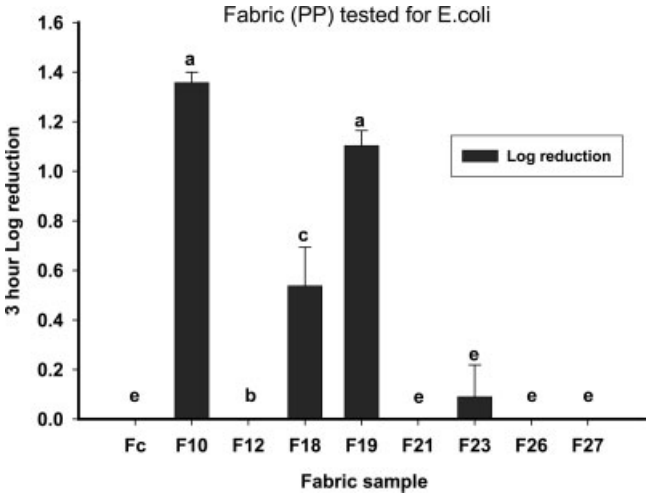

Figure 5 Reduction in viable cells of *E. coli* on PP fabrics. Fabrics are coded as listed in Table I. The log reduction in cell numbers after 3 h incubation at 30°C is shown. The error bars represent the standard deviation for three replications. All bars with different letters are significantly different ($P < 0.05$).

As seen from Figure 4, sample F10, PP/29.7%/GMA/2.6% quaternary ammonium chitosan (HTCC), shows the best effect with 1.3 log reduction. Fabric F19, PP/20.62% GMA/1% β-CD, shows 1.06 log reduction. Fabric F18, PP/38% GMA/7.2% β-CD, has only 0.59 log reduction. All other tested samples are either 0 or slightly less than 0.2 log reduction.

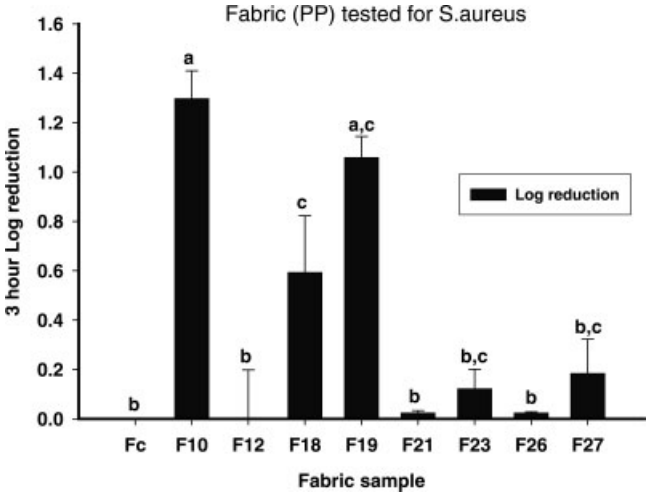

Figure 6 Reduction in viable cells of *S. aureus* on fabrics. Fabrics are coded as listed in Table I. The log reduction in cell numbers after 3 h incubation at 30°C is shown. The error bars represent the standard deviation for three replications. All bars with different letters are significantly different ($P < 0.05$).

Comparison between LA70, *E. coli*, and *S. aureus* test results

A comparison between the antimicrobial effectiveness of grafted PP fabrics for three different microorganisms (gram positive LA70 and *S. aureus* and gram negative *E. coli*) is shown in Figure 7. It is obvious that fabrics grafted with quaternary ammonium chitosan HTCC (fabric F10) have the best antimicrobial effect on all microorganisms, followed by F19, which has less β-CD than F18.

It is apparent from test results that PP fabrics grafted with lower percent GMA and β-CD is more effective than higher percentages. This is in agreement with reported observation that highest antimicrobial activity is with lowest concentration.[15,16] The effect of lower concentration on antimicrobial activity was obvious from the test results of fabric F19 (PP/20.62% GMA/1% β-CD) when compared with sample F18 (PP/38% GMA/7.2% β-CD). Here in these two specific fabrics, F19 is about a factor of 2 less GMA and a factor of 7 less CD. Fabrics with quaternary ammonium chitosan (HTCC), PP/29.7% GMA/2.6% HTCC, have shown the best effectiveness as an antimicrobial fabric.

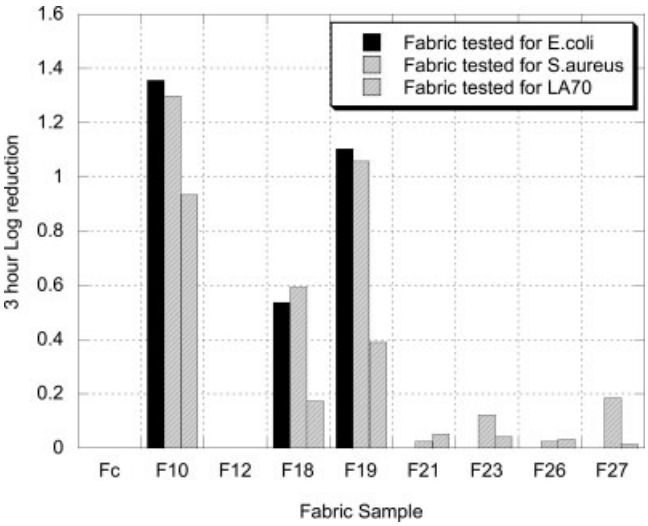

Figure 7 Comparison between experiments with *E. coli*, *S. aureus*, and LA70.

Tick repellency assay results

The percentage of ticks on either the untreated control fabric or the treated fabric was arcsine transformed using the formula of Freeman and Tukey,[17] and the transformed data were analyzed using the general linear model (PROC GLM, SAS Institute).[18] Ticks that were touching both the control and treated fabric were removed from the analysis. Test results are tabulated in Table VI.

Ticks that were subjected to a choice test of two untreated identical fabric samples (controls) or a control sample and 19.8% GMA, 1.0% CD treated fabric, did not significantly choose either sample ($P > 0.05$).

TABLE VI
Response of *D. variabilis*, American Dog Tick, 3 h After Exposure to a Choice Test of Untreated and Treated Fabrics Containing Citronella, Sweet Basil, DEET, or BioUD30[a] (Spray or Silicon Formulation)

Treated fabric	% choosing control ± 1 SEM	DF	*F*-value[b]	*P*-value[b]
Untreated fabric only[c]	61.11 ± 22.22	1,4	0.50	0.519
19.8% GMA, 1.0% CD	50.00 ± 9.62	1,4	0.00	1.00
20.6% GMA, 1.0% CD, 1.0% citronella	88.89 ± 5.56	1,4	36.88	0.0037
20.6% GMA, 1.0% CD, 1.0% sweet basil	88.89 ± 5.56	1,4	36.88	0.0037
38.0% GMA, 7.2% CD, 1.0% DEET	100.00 ± 0.00	1,4	Infty	<0.0001
35.4% GMA, 1.33% CD, 3.13% BioUD30 silicon	100.00 ± 0.00	1,4	Infty	<0.0001
82.0% GMA, 1.0% CD, 0.477% BioUD30 spray	38.89 ± 24.22	1,4	0.60	0.4808
82.0% GMA, 1.0% CD, 0.84% BioUD30 silicon	33.33 ± 25.46	1,4	0.83	0.4126

[a] BioUD30 consists of 30% 2-undecanone by weight (HOMS, LLC., Clayton, NC).
[b] Calculated with arcsine transformed percentages using the formula from Freeman and Tukey.[17]
[c] Two individual pieces of untreated PP were used; statistics represent percentage of ticks that chose one side over the other.

The samples with the inclusion of 1.0% citronella, 1.0% sweet basil, 1.0% DEET, and 3.13% BioUD30 all significantly repelled the ticks ($P < 0.05$); however, only the DEET and 3.13% BioUD30 silicon formulation resulted in 100% repellency. It is important to note that the active ingredient in both formulations of BioUD30 against ticks is 2-undecanone, which is 30% by weight; therefore, the sample containing 3.13% BioUD30 consists of 0.939% 2-undecanone. Two other samples with the inclusion of BioUD30 contained 0.477% (spray formulation) and 0.84% (silicon formulation), consisted of 0.143 and 0.25% 2-undecanone, respectively, did not effectively repel ticks significantly.

CONCLUSIONS

Atmospheric pressure plasma-aided graft copolymerization of nonwoven PP fabrics has proved to be successful. Static decay testing has shown that these modified fabrics are antistatic. Ultraviolet visible spectroscopy confirmed the success of grafting and has shown the typical peaks of grafted agents. Fabrics grafted with GMA and either HTCC or β-CD with additional inclusion of insect/tick repellent agents in CD cavities has shown antimicrobial activity and tick repellent efficiency.

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