# ENVIRONMENTALLY BENIGN ANTIBACTERIAL AGENTS FOR NONWOVENS

**T. L. Vigo, D. V. Parikh and G. F. Danna**
**USDA, ARS, SRRC**
**New Orleans, LA**

## Abstract

Environmentally benign, peroxide-based antibacterial agents were bound to cotton, cotton/polyester and polyester nonwovens before and after fabrication. Bound peroxide was 0.2-1.5% after process washing and drying and varied with the nature of the fibrous substrate, curing conditions and additives in the treating formulation. Magnesium hydroperoxyacetate (MHPA or [HOO-Mg-OAc]) is the active antibacterial agent. At comparable peroxide contents, antibacterial activity is usually superior for all cotton fabrics and cotton/polyester blend fabrics compared to all polyester fabrics due to the better moisture transport properties of hydrophilic natural fibers. Optimum results for cotton/polyester nonwovens were achieved when they contained a core/low-melting sheath bicomponent polyester binder fiber and were subsequently cured with a formulation containing an alkaline polyethylene softener at temperatures that melt or soften the binder fiber component. These blend fabrics had peroxide contents greater than 1% after leaching and gave complete reduction of both gram-positive and gram-negative bacteria.

## Introduction

Initial studies demonstrated that woven cotton and cotton blends with high cotton content could be treated with aqueous dispersions of environmentally benign antibacterial agents (magnesium hydroperoxyacetate--MHPA/magnesium dihydroperoxide--MDHP) to bind these agents to fiber surfaces by subsequent curing (Vigo and Danna, 1996). The scope of the treatment was first expanded to fabricated nonwovens of varying fiber type (Vigo, Danna and Parikh, 1999) and later to incorporation of these magnesium-peroxide-based antibacterial agents into nonwovens at earlier stages of formation (such as binding at the web stage prior to conversion into a nonwoven fabric) for treated cotton webs blended with other fibers or alternatively treatment of a cotton/synthetic fiber blend prior to formation of nonwoven fabrics (Vigo, Parikh and Danna, 2000). More recently, cotton/polyester and all polyester nonwovens with the polyester part comprised of a core/sheath bicomponent fiber were treated with these agents in the presence and absence of specific softeners (Vigo, Parikh and Danna, 2001). A critical comparison of all these studies on nonwovens is the topic of this paper.

## Materials and Methods

For incorporation of agents prior to nonwoven fabrication, bleached cotton fibers were obtained from Veratec, Inc. with an average micronaire value of 5.38 (standard deviation 0.03) and an average staple length of 0.89 in. (standard deviation 0.015). Staple polyester fibers (Trevira P121) were obtained from Hoechst-Celanese and had an average staple length of 1.33 in. (five replications, standard deviation 0.01). Resultant blended, untreated carded webs were 10/90, 20/80 and 30/70 cotton/polyester. The following fabricated nonwovens were used in this study: 50/50 cotton/polyester hydroentangled gauze--40 g/m$^2$ (obtained from Johnson and Johnson) ; bleached 100% cotton needle-punched--160 g/m$^2$; polyester (D271 bicomponent core/sheath polyester fiber from Dupont with binder fiber as sheath--137.4 g/m$^2$; and 50/50 cotton/polyester with polyester part as D271 bicomponent fiber described above--136 g/m$^2$. The latter two fabrics were fabricated by blending (for cotton/polyester), carding and needle-punching twice.

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Potassium iodide, hydrogen peroxide (30%), hydrochloric acid (7%), standardized 0.1N sodium thiosulfate (J. T. Baker, Inc.) and magnesium acetate tetrahydrate (Fluka) were all reagent grade. Alkaline polyethylene-based softener, Discosoft 1566, 30% solids, pH 8.0-8.5 was obtained from Callaway Chemical Co.

The MHPA was prepared as previously described (Vigo and Danna, 1996) by carefully heating (at or below 90$^o$C) excess 30% aq. hydrogen peroxide and solid magnesium acetate tetrahydrate. Peroxide (active oxygen) contents of the MHPA and modified nonwovens were determined by iodometric titration. Melting points and thermal behavior of $Mg(OAc)_2.4H_2O$ and the MHPA were determined by differential scanning calorimetry; both procedures are described in a previous publication (Vigo and Danna, 1996). Physical tests for untreated and treated nonwoven blend fabrics were conducted by standard test methods (stiffness, Federal Laboratory Test Method 5202; air permeability, ASTM Method 737-96; bursting strength, Method D3786-87) at our laboratory. The antibacterial activity of untreated and treated nonwovens were determined by AATCC Method 100 by NAMSA Labs, Marietta, Ga.

Blended, untreated carded webs of 10/90, 20/80 and 30/70 cotton/polyester content were each sprayed with a 4% aq. dispersion of MHPA to a wet pickup of 140%, air-drying for 2 hr, then oven-drying for 30 min at 100$^o$C. Each of the modified and carded webs were then needlepunched once on each side (12 mm needling depth, 18.1 needles per cm width, 23 penetrations per cm$^2$ with two passages through a Morrison Berkshire needleloom equipped with a single 31.8 cm board containing 575 needles). The antibacterial treatment did not cause any difficulty in the needlepunching process, i. e., the needles did not break. All needlepunched nonwovens had a wt. of 65g/m$^2$ + /- 5.

Each fabricated nonwoven was immersed in a 16% aq. dispersion of MHPA to give wet pickups respectively of 119% (bleached, needle-punched cotton); 277% (double treatment, pad-cure; pad-cure for the gauze for comparable add-on); 194% (100% bicomponent polyester); and 162% (50/50 cotton/bicomponent polyester). The latter two fabrics were also padded respectively to wet pickups of 102% (polyester) and 108% (cotton/polyester) with formulations containing 16% aq. dispersion of MHPA/4%Discosoft 1566 polyethylene softener. Fabrics were then cured 3-6 min/125$^o$C, tap water-rinsed 10 min/50$^o$C to remove excess, unbound antibacterial agents, then dried 3-10 min/125$^o$C. Leaching was conducted by placing treated fabrics in 2 gallons of warm tap water (40$^o$C) for 1 hr. (with intermittent agitation every 10 min for a duration of 5 seconds), and repeating this process for an additional hour, followed by blotting the samples on paper towels, then drying them under the same conditions described above after their cure.

## Results and Discussion

The efficiency (amount of antibacterial agent retained after initial padding, curing and in most cases process washing and drying) of various nonwovens varied markedly with fiber type, method of nonwoven bonding and method of agent application (Table 1). 4% aq. dispersions were used on the predominantly polyester/cotton blends (first three fabrics in the table) prior to needle-punching because these fabrics would not be subjected to process washing and drying. Although the 30/70 cotton/polyester blend had extraordinary efficiency (96%) compared to the other two blends (26 and 39%), these fabrics were not processed washed and dried and all would retain significantly less agent after these operations. The remainder of the fabrics were treated with the agent after fabrication and all were process washed and dried. Irrespective of type of nonwoven and fiber type (hydroentangled 50/50 cotton/polyester gauze, needle punched all cotton and thermally bonded 50/50 cotton /polyester or all polyester), efficiency of binding (% theoretical amount that could be retained based on wet pickup) ranged from 14-33% for fabrics not containing alkaline softener in the formulation. When this softener was used, the absolute amounts and % efficiency were much greater (58% for the blend and 51% for the polyester). This is probably due to the encapsulation of the agent by synergistic melting of the bicomponent polyester fiber and the polyethylene softener.

This marked improvement was also noted after leaching of the blend and all polyester fabrics with and without softener (Table 2). It was possible to obtain excellent antibacterial activity (99.5%) against representative gram-positive (*S. aureus*) and gram-negative (*K.. pneumoniae)* bacteria at active oxygen or peroxide content as low as 0.22% (Table 2) for all unleached fabrics. However, only the cotton/polyester blends retained this excellent antibacterial activity after leaching (with or without softener in the formulation). The hydrophobicity of all polyester fabrics may account for their poorer performance than the more hydrophilic cotton/polyester blends. Additional experiments and evaluation techniques are planned (particularly scanning electron microscopy) to determine the location of the bound agent and new processing methods will be studied to optimize its binding and efficiency to a variety of nonwoven fabrics.

Antibacterial nonwovens could find use in filtration in food and pharmaceutical processing and in air-conditioning filters to remove undesirable microbes. They may also be useful in hygenic materials, bedding, insulation, disposable blankets, automotive components, home furnishings, apparel interlinings and other applications where bacterial control is required or useful.

## Summary

Nonwovens of diverse fiber type and method of fabrication were modified with environmentally benign antibacterial agents (MHPA). It was determined that optimum efficiency and absolute amount of agent bound was achieved by using thermal bonded cotton/polyester and all polyester nonwovens using a bicomponent binder fiber and alkaline polyethylene softener. Under these conditions, the antibacterial activity was excellent both after processing washing and drying and after leaching.

## References

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Table 1. Efficiency of Binding MHPA on Various Nonwovens.

| Fiber Type | % Active $O_2$ | % Efficiency |
|---|---|---|
| 10/90 C/PET Needlepunch | 0.22 | 26 |
| 20/80 C/PET Needlepunch | 0.33 | 39 |
| 30/70 C/PET Needlepunch | 0.81 | 96 |
| 50/50 C/PET Gauze | 0.74 | 14 |
| Cotton Needlepunch | 0.76 | 33 |
| 50/50 C/Bicomponent PET | 0.88 | 23 |
| 50/50 C/ Bicomponent PET Softener | 1.50 | 58 |
| Bicomponent PET | 0.48 | 21 |
| Bicomponent PET Softener | 1.26 | 51 |

Table 2. Antibacterial activity of various.

| Fiber Type | % Active | | |
|---|---|---|---|
| $g^+$ $g^-$ | $O_2$ | Antibacterial | Activity[a] |
| 10/90 C/PET Needlepunch | 0.22 | 99.9 | 99.9 |
| 20/80 C/PET Needlepunch | 0.33 | 99.9 | 99.5 |
| 30/70 C/PET Needlepunch | 0.81 | 99.9 | 100 |
| 50/50 C/ Bicomponent PET | 0.88 | 100 | 100 |
| 50/50C/ Bicomponent PET Leach | 0.62 | 100 | 100 |
| 50/50 C/Bicomponent PET Softener | 1.50 | 100 | 100 |
| 50/50C/Bicomponent PET Softener/Leach | 1.14 | 100 | 100 |
| Bicomponent PET | 0.48 | 100 | 100 |
| Bicomponent PET Leach | 0.27 | 97.4 | 97.5 |
| Bicomponent PET Softener | 1.26 | 100 | 100 |
| Bicomponent PET Softener/Leach | 0.90 | 81.8 | 100 |

[a] $g^+$ = *S. aureus* and $g^-$ =*K. pneumoniae*. Values are % reduction bacterial growth--AATCC Method 100; All control fabrics gave NR= no reduction in bacterial growth.