ANTIBACTERIAL FLAME RETARDANT COTTON HIGH LOFT NONWOVENS

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Abstract

Renewable resources for raw materials and biodegradability of the product at the end of the useful life is entailing a shift from petroleum-based synthetics to agro based natural fibers such as cotton, especially for producing high specific volume high loft nonwovens. Cotton is highly flammable and is susceptible to microorganisms. FR is achieved in many cotton fiber blended nonwovens through chemical finishing. Paradoxically, as the fabrics go through the chemical finishing treatment, looses the high loft and soft feel desired for soft furnishings. With nonwoven technology, it is possible to overcome drawbacks of FR treatment and impart desired strength and softness. SRRC flame retardant treated grey cotton fibers were blended with antibacterial treated grey cotton fibers and bicomponent polyester fibers to form high loft fabrics that have high flame resistance (FR) and antibacterial property and these were evaluated for LOI, vertical flame test and antibacterial test.

Introduction

Functional textiles such as antimicrobial, flame retardant and insect repellant have attracted much attention in recent years [Bajaj, 2002; Holme, 2007; Hebeish et al., 2007; Schindler et al., 2004; Ibrahim et al., 2007; and Ibrahim et al., 2005]. Cotton based high-loft nonwovens are universally appreciated for their excellent moisture absorption, soft handle, lightweight, comfort, and are made from a sustainable environmentally friendly fiber. They are used for making wound dressing applications and mattresses used in hospitals [Demitri et al., 2008; Sannino et al., 2003; Lionetto et al., 2005; Schierholz et al., 1998, Schierholz et al., 1999; Bosetti et al., 2002; and Hillyer et al., 2001]. Cotton, being a cellulosic fiber, is highly flammable, and is prone to microorganisms such as mould, bacteria and fungi [Son et al., 2006; and Lim et al., 2004].

Cotton can be treated with various chemicals for antibacterial treatment like Triclosan, titanium dioxide nanoparticles, CuO-chitosan nanocomposite but silver has high biocompatibility, excellent resistance to sterilization conditions, effectiveness on different bacteria and long-term durability of its antibacterial effect. It has strong biocidal effects on many pathogenic bacteria. Nanoparticle silver particles have better functionality due to their small size ranging from 4 to 100 nm [El-tahlawy et al., 2005; Daoud et al., 2005; Li et al., 2006; Lee et al., 2003; Son et al., 2006; Son et al., 2004; Tarimala et al., 2006; Alcamo, 1991; Sondi et al., 2004; Chen et al., 2005, Brady et al., 2003, Slawson et al., 1992, Zhao et al., 1998; Potenza et al., 2004; Inoue et al., 1997; Yang et al., 2003; Lee et al., 2005; Xu et al., 2006; Cho et al., 2006; Hadad et al., 2007; Dubas et al., 2006; and Xu et al., 2006].

In US, more than 4,000 people die each year in 500,000 residential fires, which account for property losses exceeding 4 billion dollars annually, and the long-term emotional damage to victims and their loved ones is incalculable [http//www.cpsc.gov/]. In the recent years, U.S. Consumer Product Safety Commission (CPSC) is enforcing stricter flammability regulations (Chapter 16, Code of Federal Regulations4 - CFR parts 1602 through 1634). The safety of humans and their possessions has become a very important issue due to flammability of textile materials [http//www.noharm.org/details.cfm? ID=1098&type=document]. The demand for flame retardant fibers and fabrics is increasing to comply with the stricter regulations. Cotton goods and nonwoven manufacturers have been seeking methods to develop FR treatments for their products [Alaee et al., 2002; and Parikh et al., 2003]. This highlights the vast market potential for a techno-economically viable product with a desired degree of flame retardancy.

When cotton burns, it can produce volatile fragments, which can ignite in presence of oxygen. Flame retardant (FR) treatment is one of the most effective methods to improve thermal resistance of cotton to ignition, reduce flame propagation rate, and prevent continuous burning [http//www.inda.org/category/nwn_index.html]. FR chemicals act on one or more components of combustion i.e. heat, fuel and/or oxygen. Either FR reacts to shield the polymer by forming a char layer or inert gas layer or undergo a reaction that cools the system. The aim of FR material is to promote char formation [Kandola et al., 1993; Lawson et al., 2008; and http//www.ides.com/property-descriptions/].

Phosphorous compounds have great market acceptance as they form nontoxic char, which inhibit the pyrolysis process by preventing formation of levoglucosan, which acts as a precursor for flammable volatile formation. Char is a glassy protective film, which acts as a surface barrier to oxidation and shields the base material from burning [Bajaj, 2009; Kozlowski et al., 2007; Weil et al., 2008]. Phosphorus compounds offer resistance to combustion by lowering the decomposition temperature of cellulose that favors dehydration as opposed to depolymerization, thus reducing the formation of combustible volatile fuel compounds.

Diammonium Phosphate (DAP) is a water-soluble ammonium phosphate salt, which contains nitrogen and phosphorous. It is a non-durable FR chemical. DAP acts as an excellent flame retardant for cotton and cotton blended materials. Upon ignition, DAP decomposes at a temperature lower than the degradation temperature of cellulose and leaves a large inert insulating char residue, which prevents further burning [Horrocks et al., 2005].

Cotton Blend High lofts

High loft nonwovens can be defined as fibrous webs with a high ratio of thickness to weight, which are bonded mechanically, thermally or chemically. They are formed by blending the natural fibers with thermoplastic binder fibers. Synthetic fibers of relatively low melting point, such as polyethylene, polypropylene are widely used as binder fiber. Conventional binder fibers are polyolefins, which are not biodegradable, and pose difficulty in disposal. Thus new biodegradable polymers and fibers are gaining ground. Nowadays, core-sheath bicomponent fibers are commonly used as binder fibers. On heating, the sheath of the binder fibers melts at a lower temperature (80 to110°C) and act as a glue to bond the fibers upon cooling at the contact points of the fibers. The core polymer has a higher melting point, which does not melt and hence, provides the cohesive strength required for structural integrity to high loft nonwoven. The proportion of these bonding fibers within the web can be varied from 10 to 25% to achieve the desired performance properties such as strength, drape, and resilience.

Through-air thermal bonding and bi-component fibers enables economical and environmentally safe production of cotton fiber-based high loft nonwovens. Through-air thermal bonding takes place as the web is heated in an oven or passed around perforated drums, where the heated air is blown through or drawn by a vacuum through the web.

Current Research

During the last decade extensive research has been going on to develop new products to enhance FR of cotton. But, as the high loft nonwovens are used for making mattress, which are used indoors for good amount of time, mostly spanning years are bound to provide a good breeding ground for microorganisms in the presence of moisture and in the absence of sunlight. The focus of the present research was to produce nonwoven webs by through-air thermal bonding of FR treated cotton fibers, silver nanoparticle-coated cotton fibers and bicomponent binder fibers and investigate flame resistance and antibacterial properties of the high loft nonwoven fabrics.

Cotton Blend High lofts

High loft nonwovens can be defined as fibrous webs with a high ratio of thickness to weight, which are bonded mechanically, thermally or chemically. They are formed by blending the natural fibers with a thermoplastic binder fiber. The binder fibers may provide the cohesive strength required for structural integrity of the nonwoven fabric. Synthetic fibers of relatively low melting point, such as polyethylene, polypropylene are widely used as binder fiber. Conventional binder fibers are polyolefins, which are not biodegradable, and pose difficulty in disposal. Thus new biodegradable polymers and fibers are gaining ground. Nowadays, bicomponent fibers, which have core-sheath structure, are commonly used as binder fibers. The sheath polymer melts at a lower temperature (80 to110°C) and act as a glue to bond the fibers upon cooling. The core polymer has a higher melting point, which does not melt and hence, provides homogeneity and integrity to high loft nonwoven.

Through-air thermal bonding and bi-component fibers enabled economical and environmentally safe production of thermally bonded cotton fiber-based high loft nonwovens. Through-air thermal bonding takes place as the web is heated in an oven or passed around perforated drums, where the heated air is blown through or drawn by a vacuum through the web. On heating, the sheath of the binder fibers melts, and fuses together at the contact points of the fibers. The proportion of these bonding fibers within the web can be varied from 10 to 25% to achieve the desired performance properties such as strength, drape, and resilience.

Current Research

During the last decade extensive research has been going on to develop new products to enhance FR of cotton. But FR treatment may have some adverse effects on few desirable properties of the cellulosic high loft nonwoven fabrics. An ideal FR fabric must be comfortable, eco-friendly, durable and cost effective with minimal lowering of tensile, burst and tear strength, abrasion resistance, softness, and air permeability. The focus of the present research was to produce nonwoven webs by through-air thermal bonding of FR treated cotton fibers, silver nanoparticle-coated cotton fibers and bicomponent binder fibers and investigate flame resistance and antibacterial properties of the high loft nonwoven fabrics.

Materials and Methods

Fibers Used in Construction of Nonwoven Webs

Raw grey cotton fiber from bale was mechanically opened and cleaned through an inclined cleaner and a fine saw-tooth cleaner, thus practically free of undesired foreign matter such as leaves, twigs, stones, seeds, plant debris, seed coat, etc. Grey (unbleached) cotton fiber costs less than the bleached cotton as it saves the cost of bleaching (~$0.70/lb). Commercial grade bicomponent fiber with low-melt polyester sheath and polyester core was obtained from Invista. The melting point of sheath of the bicomponent fiber is 80^0 C.

FR Formulations and Treatment

Fiber was treated with FR formulation SRRC-2 (add-on was 23.6%). The FR formulation imparts non-durable FR to grey cotton fiber; the phosphate-nitrogen based formulation (SRRC-2) contained w/w diammonium phosphate (DAP) 15%, Urea 5%, Citric acid 1.0%, Triton X-100 0.7% (Table 1). DAP is used as a FR chemical for cotton: it lowers the combustion temperature of the material, decreases maximum rate of weight loss and increase char. Urea or urea derivative compounds produces synergism of phosphorous and nitrogen and imparts better flammability resistance. Citric acid is known to impart not only flame resistance but also antibacterial property.

Table 1: SRRC-2 FR Formulation

	(%), (w/w)
Diammonium phosphate	15.0
Urea	5.0
Triton X - 100	0.7
Citric Acid	1.0
Water	78.3
Total	100.0

The fiber was wetted out in FR formulation and loaded into a laboratory centrifuge (Spin X) to obtain the desired wet pick-up, first wetting to produce 70% wet pick-up. The wetting procedure was repeated to wet the fiber with 95-100% wet gain. Twice wetting of grey fiber was adopted with view to have a good penetration of the formulation. The excess solution was captured for use. The wet fiber was opened by hand and dried in the conventional Kenmore 80 series tumbler dryer with setting 'high for cotton' for 75 minutes.

Antibacterial Treatment

Grey cotton fibers were treated with antimicrobial silver nanaoparticles, which were prepared by the "green" synthetic method developed at SRRC. Silver nanoparticles (ca. 3.7 nm in diameter) were produced from silver nitrate precursor in an aqueous solution of poly(ethylene glycol) at 80°C, in which poly(ethylene glycol) acts as both a reducing and stabilizing agent. Grey cotton fibers were immersed in an aqueous silver colloidal solution containing

0.1% Triton® X-100 at room temperature. The excess of the solution was removed using a spin dryer to produce 100% wet pick-up. The fibers were then rinsed with deionized water three times for 15 min, and tumble-dried. The content of silver on the fiber by the elemental analysis was 912 ppm.

Nonwoven Web Formation, Carding and Through-air Bonding Process

Grey FR Cotton fibers, grey antibacterial cotton fibers and bicomponent binder fibers were intimately blended at SRRC in the desired percentages to produce prepare five different blends of nonwoven webs (Table 2). The proportion of bicomponent binder fiber was maintained at 20% for all the blends. The proportion of SRRC FR grey cotton fiber was decreased from 65% to 55% and the proportion of antibacterial cotton fiber was increased from 15 to 25% in blend number 1 to 3, respectively. Blend number 4 had 80% SRRC FR grey cotton and Blend number 5 had 80% grey cotton. Hence, blend number 4 and 5 are considered as control blends.

SDS Atlas carding machine was used to individually open and intimately blend the fibers. After the carding process, the web was bonded by through-air thermal bonding in an in the Mathis oven to form the high loft nonwoven fabrics. In previous Cotton FR research the process conditions of through-air bonding to impart good strength, loftiness and appearance were optimized. It was found that the optimum bonding conditions for cotton-based nonwovens should be at 150^0 C for 3 minutes. The sheath provides the fusing point while the core preserves the integrity of the nonwoven. Low melt bicomponent fiber sheath melts at ~80°C and the core melts at ~250°C.

Table 2:Percentage composition for different blends

Blend #	Fiber	Blend %
1	SRRC FR grey cotton Fiber	65
	Antibacterial grey cotton Fiber	15
	Bicomponent binder Fiber	20
2	SRRC FR grey cotton Fiber	60
	Antibacterial grey cotton Fiber	20
	Bicomponent Binder Fiber	20
3	SRRC FR grey cotton Fiber	55
	Antibacterial grey cotton Fiber	25
	Bicomponent Binder Fiber	20
4	SRRC FR grey cotton	80
	Bicomponent Binder Fiber	20
5	Grey cotton	80
	Bicomponent Binder Fiber	20

Characterization methods

The high loft nonwoven fabrics samples were conditioned for at least 24 hours under standard laboratory conditions (21°C ± 1°C and 65% ± 10% relative humidity) and were tested for physical properties, flammability and antibacterial properties.

Basis weight of bonded webs was measured according to ASTM D3776-96. The weight of 76mm x 76mm samples was measured. Basis weight was expressed as grams per square meter (gsm).

The samples, having size of 76 mm X 76 mm, were set on a horizontal table and a 76 mm X 76 mm Plexiglass square platen, weighing 72 (±1) grams, was placed on the samples and the thickness of the samples was measured in mm. Average 16 readings (four readings from four sides) were taken for evaluation.

FR tests are designed to determine the fire hazard of materials and products for the application, which they are required. The most popular test for textile materials is limiting oxygen index (LOI). It is a method to determine the minimum oxygen concentration in an oxygen/nitrogen mixture that will sustain the flame. It is a convenient, reproducible and inexpensive way of determining tendency of a material to sustain a flame.

LOI testing was done according to ASTM 2863. A test sample 150 mm x 50mm, placed in a transparent test chamber, is ignited at the top and the oxygen concentration in the mixture of oxygen and nitrogen is increased until the sample sustains burning. The volume fraction of the oxygen in the gas mixture is defined as the LOI. Since air compromises about 20.95% oxygen, so it can be considered as a threshold value while grouping the materials.
The vertical flame test was conducted using a flame control module (Govmark organization, Inc.) according to ASTM standard method D6413-99. The flame is applied to the bottom of the vertically placed specimen. After 12 sec of flame exposure, after-flame time, after-glow time, and char length were measured. Photographs of the samples were taken with a digital camera (NV3, Samsung).

The antibacterial properties of high-loft samples against S. Aureus (ATCC 6538), a Gram-positive bacterium and K. pneumonia (ATCC 4352), Gram-negative bacterium were evaluated using the AATCC test method 100-2004. All plates were incubated at 35±1°C for approximately 24 hours.

Antimicrobial Activity or Percent Reduction of bacteria, R, was calculated using the following equation:
Percent Reduction = 100(B-A)/B, where:
A = Average number of viable cells on the inoculated treated high-lofts post 24 hour incubation period.
B = Average number of viable cells on the inoculated untreated control high-lofts immediately after inoculation ("0" contact time).

Results and Discussion

Thickness and Basis Weight

The loftiness of the control blend 5 with 80% untreated grey cotton was found to be highest (Table 3). The thickness of the blended high loft nonwovens was found to be similar but was lesser than that for control blend number 5.

The basis weight of the control blend 5 with 80% untreated grey cotton was found to be lowest (Table 3). The specific gravity of the SRRC FR cotton is higher than that of grey cotton, which may account for the increase in the basis weight of the antibacterial FR high loft nonwovens. The basis weight of the antibacterial FR high loft nonwovens was found to be not very much affected by the proportion of SRRC FR cotton in the range of 65% to 80%. The specific gravity of the SRRC FR treated cotton and antibacterial treated cotton might be similar, so varying the proportions of two may not affect the basis weight of the antibacterial FR high loft nonwovens.

Table 3:Physical properties of the high loft nonwoven blended samples

Property		Blend #				
		1	2	3	4	5
Thickness (cm)	Average	5.0	4.6	4.6	4.1	5.3
	SD	0.14	0.17	0.17	0.07	0.26
Basis weight (GSM)	Average	436.9	333.8	292.5	496.4	252.2
	SD	0.7	1.5	0.3	0.5	1.3

Limiting Oxygen Index (LOI) and Vertical flame tests

The LOI of the control blend 5 with 80% untreated grey cotton was found to be the lowest (Table 4). LOI of the antibacterial FR high loft nonwovens tend to increase with the increase in the proportion of the SRRC FR cotton in the blends (Figure 1). We found that SRRC FR cotton has high LOI and hence, has very high flame resistance. An increase in the proportion of SRRC FR cotton is bound to improve the LOI of antibacterial FR high loft nonwovens.

All four blends pass the vertical flame test (Table 4). The char length increases with the decrease in the decrease in the proportion of the SRRC FR cotton in the blends. SRRC FR cotton has very high flame resistance and helps in reducing the char length. Blend 3, although its char length is small (less than 2 inch), flame propagated on the surface of the high-loft to a half of the sample length, so, blend 3 may be considered marginal (Figure 2). Blend 5 samples have dent after the test, which was due to bicomponent fiber. The polyester binder fibers melt on heating and shrank to hold cotton fibers to form the dent (Figure 2). After-flame and Char length were highest for blend 5 because it had 80% untreated grey cotton. An after glow of the control high loft nonwovens (blend 5) was observed after vertical flame test (Figure 2).

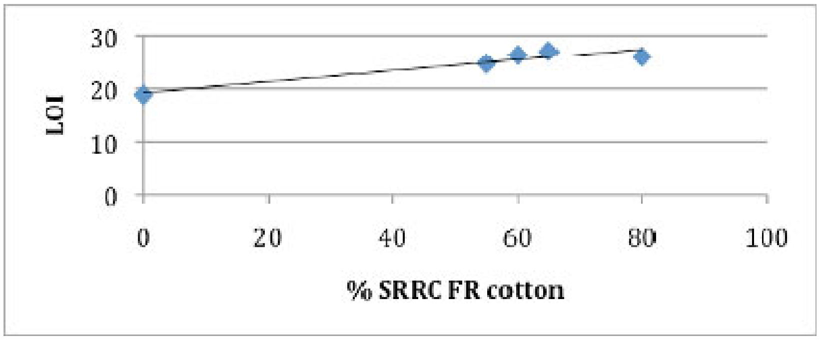

Figure 1: LOI profile of the antibacterial FR high loft nonwovens

Table 4:Flame resistance properties of the high loft nonwoven blended samples.

Blend #	LOI (Average)	After-flame (sec)	After-glow (min)	Char length (inch)
1	27	nil	Nil	0.6
2	26.3	Nil	Nil	0.8
3	24.7	Nil	Nil	1.4
4	26	nil	Nil	0.6
5	18.9	3.25 (0.35)	3.88 (1.59)	Dent: 4.5 (0.7)

Figures in parenthesis are standard deviation of two measurements.

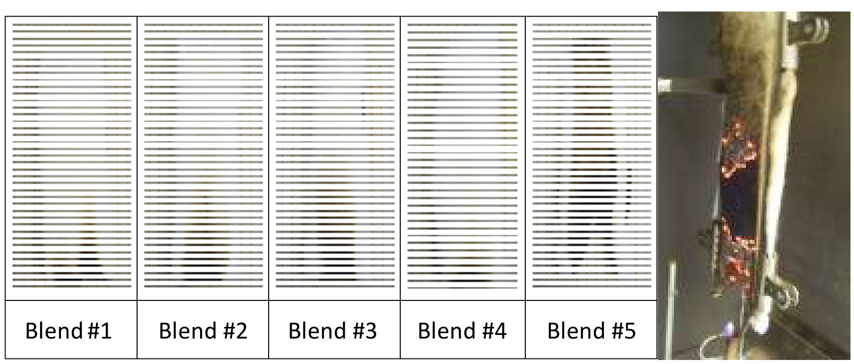

Figure 2: Char length of the antibacterial FR high loft nonwovens after vertical flame test and after glow of the high loft nonwovens (blend #5) after vertical flame test

Antibacterial Property

Cotton, being a cellulosic fiber, the high-loft control samples provided an excellent environment for the growth of both types of bacteria, S. Aureus (ATCC 6538) and K. pneumonia (ATCC 4352). In the absence of antibacterial treated cotton or FR treated cotton in the control samples may not effect reduction of bacteria, as may be expected. All blends 1, 2, 3, and 4, showed good antibacterial properties against both Gram positive and negative bacteria. The reduction of bacteria for high loft blends with antibacterial cotton fiber 99.995% for S. Aureus (ATCC 6538), a Gram-positive bacterium and was greater than 99.993% for K. pneumonia (ATCC 4352), Gram-negative bacterium. Slight difference between the data in bar chart probably comes from experimental details. The results show that the antibacterial cotton fiber was very effective in eliminating both types of bacteria. The silver nanoparticles may interact with the bacterial peptidoglycan layer, form pits in the cell wall, change membrane polarity to damage the membrane to effect antimicrobial property. Silver nanoparticles in the range of 1-10 nm attach to HIV proteins and inhibit the virus from binding to cells. On account of these antimicrobial properties, silver nanoparticle technology has been incorporated into surgical instruments, hospital wound dressings, and a wide variety of consumer products, such as washing machines, food storage containers, bandages, and clothing. FR treated cotton (Blend # 4) too has good antibacterial properties to show 99.9% reduction, and even without adding antibacterial grey cotton.

Conclusions

Grey FR cotton fibers, grey antibacterial cotton fibers and bicomponent binder fibers were intimately blended at SRRC in different percentages to produce three blends of nonwoven webs. Blend number 4 had grey FR cotton fibers blended with bicomponent binder and blend number 5 had untreated grey cotton blended with bicomponent binder fibers. The blended high loft nonwovens fabrics, except the control blend number 5, had high LOI values, passed the vertical flame test and were effective in reducing the bacteria by 99.9% for S. Aureus (ATCC 6538), a Gram-positive bacterium and K. pneumonia (ATCC 4352), a Gram-negative bacterium. FR grey cotton fiber obtained from the treatment of SRRC 2 formulation (of grey cotton fiber) shows antibacterial properties.

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