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An Assessment of Surface Properties and Moisture Uptake of Nonwoven Fabrics from Ginning By-products

Vince Edwards, Paul Sawhney, Alvin Bopp, Alfred French, Ryan Slopek, Michael Reynolds, Chuck Allen, Brian Condon and Joseph Montalvo

Abstract

Greige (raw) cotton by-products resulting from cotton ginning and mill processes have long been bleached for use in absorbent nonwoven products. The potential to use greige cotton by-products as an economical source for absorbent nonwoven blends is explored. The nonwoven hydroentanglement of greige cotton lint with cotton gin motes and comber noils blends was analyzed for fiber surface polarity, swelling, and absorbance to assess properties with potential usefulness in absorbent nonwovens. The electrokinetic analysis of the fabric surface gives a composite picture of the relative hydrophilic/hydrophobic polarity absorbency and swelling properties. Nonwoven fabrics made with cleaned greige cotton lint separately blended with comber noils and ginning motes at 40:60 and 60:40 blend ratios demonstrated charge, swell, and percent moisture uptake profiles that are characteristic of the fabrics’ crystalline/amorphous cellulosic content with some variance in swelling properties. However, cellulose crystallite size varied. X-ray diffraction patterns of the three different cotton constituents displayed similar crystalline cellulose compositions. An electrochemical double-layer analysis of charge based on a pH titration ($\zeta$-plateau) was employed to measure the relative fiber and fabric surface polarity which varied slightly between -21 and -29 mV. A relationship of fiber swelling ($\Delta \zeta$) and percent moisture content is apparent when greige cotton lint and other fibers are blended. The blended nonwoven materials possess absorbent properties characterized by similar moisture uptake (7.1-9.5 %) and fiber polarity, but some variation in swelling is based on the by-product additive and its percent content. The crystallinity, electrokinetic, and water binding properties of the nonwoven by-product materials are discussed in the context of the molecular features water, cellulose, and greige cotton components that enhance potential uses as absorbent nonwoven end-use products.

Keywords: nonwoven cotton, surface properties, cellulose, water of hydration, electrokinetic, by-products
1. Introduction

1.1. Cotton and cotton by-products in nonwovens

In recent years, the preference to use cotton fibers in nonwoven absorbent products has increased. Cotton fiber is naturally renewable and biodegradable. Cotton’s characteristic soft hand, hypoallergenic properties, absorbency, and cellulosic composition have been historically utilized mostly in woven fabric products. Cotton’s current use in nonwovens is estimated to be approximately 2% (by volume/weight) of the total fiber consumption in nonwovens. Most of the cotton used at present in absorbent nonwovens is bleached cotton, including lint, gin motes, linters, comber noils, and the so-called other cotton textile processing wastes. However, the potential to use greige (nonbleached) cotton in nonwoven absorbent products has received increased attention based on innovations in cotton cleaning and nonwovens processes that open and expose the hydrophilic cellulosic component of greige cotton fiber to water absorption [1-3]. This affords an economical source of highly cleaned absorbent greige cotton nonwovens with the retention of properties inherent to the traditional cotton fabrics that generally require costly and eco-sensitive chemical scouring and bleaching processes.

Greige (raw) cotton gin motes are just one of several by-products (viz., cotton seeds, linters, motes, sticks, burrs, gin trash, among others) of the “cotton ginning process” that mechanically separates the (long) cotton fibers (greige lint) from the harvested seed cotton. The cotton ginning by-products are used in numerous applications [4-9]. The cotton gin motes typically have fibers up to ½ inch long and thus can be classified as textile fibers. However, limited use of cotton gin motes is made in traditional textiles made with spun yarns. Although cotton gin motes and textile comber noils have been used in cotton socks, their relatively lower cost and apparent differences in structural and surface properties make these cotton coproducts/fibers of interest to explore a broader nonwoven and high value end use. In addition, the processing innovations of modern nonwovens provide a facile conduit for efficiently blending these types of discounted by-products with greige cotton lint to explore new, value-added cotton-blend nonwoven products.

1.2. Absorbent applications

Highly cleaned greige cotton fiber retains most of its natural, native protective membrane or surface coating of waxes and pectin (native to the greige cotton fiber). In combination with surface-exposed cellulose from nonwoven hydroentanglement process conditions, unique fiber properties are retained when compared to scoured and bleached cotton. The amphiphilic surface character in nonwoven greige cotton, which is a combination of the polarity balance between the hydrophilic and the hydrophobic elements of the cotton material, is suitable for the application to the material layer components of incontinence absorbent products and wipes [10].

The potential for the topsheet application of nonwoven greige cotton for incontinence and wound dressing’s materials is based on its suitability as it facilitates a mechanism of optimal polar gradients between the layers surrounding the absorbent core. During fluid transport, an
optimal charge gradient between the layers from topsheet to backsheet helps facilitate better performance of wettable and absorbent materials and is associated with lower rewet, strike-through, and acquisition/distribution to the absorbent core [11]. Nonwoven greige cotton compares well with other commercial materials when analyzed for its performance as an incontinence layer surrounding the absorbent core. Moreover, when combined with synthetic fibers like polyester and polypropylene, greige cotton/synthetic blends possess an added scope of utilization in this regard [10].

1.3. Fabric surface properties and electrokinetic analysis

The zeta (ζ) potential is a measure of the charge and charge density on the surface of a particle or fiber and can be used to gauge the stability of a colloidal or the ability of a material to absorb liquid. The ζ potential can be either positive or negative depending on the surface chemistry. The ζ potential analysis is useful with absorbent materials. The fluid dynamics of the electrochemical double-layer model enables measurement of functional properties similar to those that occur at the solid-liquid interface of incontinence materials [12]. The ζ potential decrease (a change in electrostatic potential at the shear plane of the electrochemical double layer) is caused by the swelling of the fibers and the outward movement of the aqueous shear plane where ions are in contact with the outer Helmholtz plane on the fiber surface [13]. Thus, a concomitant increase in the swelling of the fiber occurs for most fibers as the shear plane moves out to the more diffuse layer of ions, causing a decrease in ζ potential [14].

This chapter examines the electrokinetic properties of hydroentangled nonwoven materials made by blending clean greige cotton lint with greige cotton by-product fibers with a view to understanding the similarities the materials possess. The three constituent cotton fibers employed in the nonwovens were evaluated for their relative cellulosic crystallinity in relation to their moisture uptake properties. Some of the blends also included polyester fibers for comparison as were previously examined [10]. Two different measures of moisture uptake are also reported. One method [15] is simple, portable, easy to use, and involves an infrared lamp to dry the samples. All weight loss is attributed to moisture. The other method is the recent ASTM Karl Fischer titration method for water content developed for lint cotton, raw and processed. Water absorbency tests were also conducted. We report here the preparation, characterization, and electrokinetic analysis of cleaned greige cotton [16] in combination with gin motes and comber noils at two different blend ratios of the greige cotton and the by-products as a measure of the fabric polarity, swelling, and absorbent properties achievable with these fabric blends. The properties of the cotton materials are discussed in light of their water binding properties related to potential absorbent applications.

2. Experimental materials and methods

A commercially available bale of precleaned greige cotton was acquired from T. J. Beall, LLC. A quantity of cotton gin mote fibers was also obtained from T. J. Beall.
2.1. Hydroentanglement of fibrous webs into nonwoven fabric structures

The needle-punched webs of the different fiber blends were uniformly hydroentangled using a Fleissner MiniJet system (Figure 1). The system is equipped with one low water pressure jet head that wets the incoming feed web material on its top face, while two high water pressure jet heads alternatively impact the wetted substrate on either face. For all the fabrics, the low water pressure head was set to inject the water at 50 bars, and the two high water pressure heads were set at 125 bars. The fabric production speed was 5 m per minute. The resulting hydroentangled fabric was dried using a meter-wide, gas-fired drum dryer and wound onto a cardboard tube to form a compact fabric roll. The hydroentangling line was flushed and cleaned after each fabric production trial.

![Diagram of a nonwovens hydroentanglement line. An outline schematic of the Fleissner MiniJet system used in the study.](image)

2.2. Fabric absorbency measurements

The AATCC drop test measures the time it takes for one drop of water applied to a fabric held in an embroidery hoop to be absorbed (when the sheen disappears). The ATSM method uses a sample of fabric that is 76 mm wide and cut to a length that equals 5.0 ± 0.1 g. The sample is rolled into a cylindrical shape, upon itself, and placed in a basket of standardized weight and size. The basket is dropped from a height of 25 mm into a water bath, and the time it takes the sample and basket to sink is measured as sink time. After sinking, the sample is then allowed to remain submerged in water for 10 s. The basket is removed and allowed to drain for 10 s, and the sample is weighed to determine its water content. The weight of the water is reported as the absorptive capacity (grams of water held by 1 g of fabric).

The test methods used in the study are as follows:

- ASTM D6242—mass per unit area (weight) of nonwoven textile fabrics
- ATSM D5035—breaking force and elongation of textile fabrics (strip method)
- AATCC 79—absorbency of textiles (drop test)
2.3. Measurement of fabric polarity, charge, relative composition, swelling, and porosity

Streaming zeta potential experiments are carried out with an electrokinetic analyzer, which is manufactured in Ashland VA, USA, using the cylindrical cell developed for the measurement of fibrous samples. For each measurement, a fiber plug is placed between the Ag/AgCl hollow cylindrical electrodes of the cylindrical cell. The pH dependence of the zeta potential is investigated with the background electrolyte of 1 mM KCl solution. The swelling behavior of the incontinence products is measured using the Anton Paar analyzer with the cylindrical cell template. A 0.65-g sample is loaded into the cell and quickly rinsed with electrolyte solution. The flow rate is adjusted into the range of 60-100 mL/min by compression of the sample to remove trapped air. The pH of the sample is about 5.5 and was adjusted to 9.0 by adding 0.1 N NaOH solution.

2.4. Moisture uptake by ASTM D629-99

The moisture content of the cotton samples is measured using modified ASTM D629-99 and AATCC 20A-2000 methods. (The modification involved using an infrared lamp to dry the materials rather than a laboratory oven as called for in the standard methods.) The sample is conditioned overnight in a humidity chamber with the hygrometer reading at 70 % and a room temperature of 23°C. The moisture measurements are based on weight loss and are taken with an infrared moisture balance (Kett FD 240, manufactured by Kett Electric Laboratory in Tokyo, Japan). The balance is set for automatic wet-based moisture with a drying temperature of 110°C. Approximately, a 1-g sample was used for each measurement on the Kett FD 240.

2.5. Water content by Karl Fischer titration ASTM D7795-12

In order to determine the water content of cotton fibers via Karl Fischer titration (KFT), fiber samples must be conditioned to standard testing conditions, 21.1°C and 65 % relative humidity. The samples were weighed into 0.1-g specimens to four decimal places, placed in the Karl Fischer glass vials, and immediately crimped with septa caps. Each blend was formulated directly in the glass KFT vials by weight basis. All open vials were equilibrated to moisture equilibrium, sealed with a septum cap, and analyzed by standard KFT (ASTM D7785, 2012). The enclosed samples were then placed into mason jars that had been acclimated in the conditioned lab. The samples were then encapsulated in the jar right up until testing to maintain the environment. Prior to KFT testing cotton samples, blank vials were used for quality control measures. During testing, the vial is lowered into a 150°C single sample oven.
for approximately 5 min, while dry nitrogen (< 20 ppb water) is bubbled into the sample at 60 mL per minute. The water from the cotton sample from the vial is released and driven into the titration cell from which the percentage of moisture present is calculated from the volume of reagent consumed. The water content in the blank vial was 0.1 %. After analysis, a visual observation of the containers revealed all samples were white; there was no discoloration or scorching. Additionally, the polyester (PES) fibers had not melted.

2.6. Standard oven drying ASTM D2495-01

The typical method to measuring moisture or weight loss of cotton fibers involves using an oven where samples are heated to 105-110°C for 24 h. Empty sample bottles were weighed and then filled with 1 g samples and reweighed. Cottons were heated in a Yamato DKN 600 mechanical convection oven with a 150-L capacity and a mean flow rate of approximately 1.3 L/s. All weights were made in a standard conditioned laboratory.

2.7. X-ray diffraction

Wiley-milled samples of the comber noils, gin motes, and UltraClean cotton were pressed into pellets and placed in a powder diffractometer with Cu Kα radiation in reflection mode. The sample was secured in a paraffin base, which had a small effect on the comber noil pattern. For comparison, a pattern was simulated with the Mercury software [17] based on a modified crystal information file from the cellulose Iβ crystal structure [18]. The modification was a short increase in the a-axis of the unit cell to 7.906 from the original 7.784 value to better position the (200) peak because of the small differences in the cotton and tunicate unit cell [19]. A peak width at half maximum height of 1.85° 2θ was used. Additionally, the preferred orientation induced by pressing the sample pellet was compensated by a facility in the Mercury software, with a March-Dollase parameter of 1.2. The Scherrer formula was used to convert the peak width at half maximum (pwhm) to crystallite sizes perpendicular to the large (200) peak with a shape constant of 1.0. The Segal Crystallinity Index [20] was used to calculate the degree of relative crystallinity.

2.8. Environmental scanning electron microscope

A Philips XL-30 environmental scanning electron microscope (ESEM) was used to image the specimens, operating at 10-13 kV. The samples were mounted on standard Cambridge 1/200 SEM stubs using double-stick photo adhesive tabs. They were coated with 60/40 % gold/palladium in a Technics’ Hummer II sputter coater to a thickness of 20-30 nm.

3. Results and discussion

3.1. Preparation of the various fibrous materials for their respective nonwovens

The fibers utilized in this study were hand blended in ratios, as indicated in Table 1, before being converted into fibrous webs of nominal density (60-90 g/m²). Fiber qualities of materials
used in this study, including fiber length and properties illustrative of the fiber nomenclature, were consistent with those previously reported for greige cotton, gin motes, and comber noils [21]. UltraClean cotton, which is a form of greige cotton [2], was separately combined with the cotton gin motes and comber noils, whereupon the blends were carded, crosslapped, and subjected to light needle punching prior to their separate hydroentanglement at 50 bar wet-out water pressure and 125 bar hydroentangling water pressure. Figure 1 diagrams the process of hydroentanglement. This approach to greige cotton-based nonwoven production has previously been shown to increase absorbency while still retaining some of the cotton’s native waxes and pectin [3]. Thus, depending on the hydroentangling process parameters and conditions [1-3], this approach increases hydrophobicity of the greige cotton nonwoven compared to an equivalent scoured and bleached cotton nonwoven product.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Fiber blend</th>
<th>Fabric weight (g/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>100% UC</td>
<td>55</td>
</tr>
<tr>
<td>2</td>
<td>100% PES</td>
<td>72</td>
</tr>
<tr>
<td>5</td>
<td>60% UC/40% GM</td>
<td>66.5</td>
</tr>
<tr>
<td>6</td>
<td>40% UC/60% GM</td>
<td>59.5</td>
</tr>
<tr>
<td>7</td>
<td>60% UC/40% CM</td>
<td>60.1</td>
</tr>
<tr>
<td>8</td>
<td>40% UC/60% CM</td>
<td>74.8</td>
</tr>
</tbody>
</table>

Fiber ID:
UC = UltraClean—unbleached precleaned cotton staple
PES = polyester staple fiber (1.5 denier, 3.8 cm (1.5") long)
GM = cotton gin motes
CM = cotton textile comber noils

Table 1. Hydroentangled fabrics of different fiber blends

3.2. Fabric surface polarity

The moisture uptake ( % MC) and electrokinetic results, including the fabric surface polarity (ζ<sub>plateau</sub>), swelling (Δζ), rate of swelling (k), isoelectric point (IEP), and related material density for the cotton/by-product blends, are shown in Table 2 for the cotton/by-product blends. The ζ potential is taken from a titration as previously shown [12-14] that assesses the surface charge based on pH and the planar portion along the x-axis of the plot is designated as the zeta plateau value (ζ<sub>plateau</sub>), which is a reflection of the relative hydrophilic versus hydrophobic character of the fabric. The ζ potentials reported here are consistent with the previously reported ζ potential titrations for both absorbent incontinence layers and greige cotton nonwoven incontinence topsheet and acquisition distribution layer prototypes [10,11].

3.3. Swelling and moisture uptake

Figure 2 shows the relationship between % moisture content and swelling behavior (Δζ) of the cotton blends. The swelling of the fiber blends results in the expansion of the electrochemical
shear plane formed near the fibrous surface [11,13] and is manifested as a decrease in the absolute value of $\zeta$. The electrokinetic data presented in Figure 2 includes samples of the cotton blends investigated in this study. The percent moisture versus the $\Delta \zeta$ for all ratios of cotton fiber reflects a correlation between the amount of moisture the material is prone to absorb and its degree of swelling. As the $\Delta \zeta$ value (x-axis) of the cotton fabrics increases, the moisture uptake ( % M) remains between 7.5% and 9%. KFT water content correlates with mean $\Delta \zeta$ with an $R^2=0.88$. These two properties (moisture uptake and swelling) promote fluid transport. The increased swelling and relatively constant value for water uptake shown in Figure 2 can be contrasted with absorbency properties, as shown for the cotton blends in Table 3. As seen in Table 3, the ASTM sink time measurement (an indirect measurement of the material’s swelling properties) principally correlates with material density in contrast with previous observations in that it correlates with swelling in greige cotton/polyester blends [10]. Swelling and rate of swelling of fabric blends are noticeably increased by blending cleaned greige cotton with by-products at a 60/40 ratio. On the other hand, the moisture uptake was the highest in the 100% Gin Motes and the lowest in 60/40 UC/GM. This is consistent with the absorbency capacity (Table 3) being highest in the UC/GM blends. The gin motes have lower density and higher surface area and thus promote hydrophilic transport of water in the fabric. In addition, as discussed below, the smaller cellulose crystallite size of the gin motes is consistent with this and plays a role at a molecular level in the increased water absorption capacity observed in the cotton by-product nonwovens.

Figure 2. Plot of delta Zeta versus percent moisture content for the cotton blends of this study.
It is also notable that 60% UC blended with 40% GM, and CM results in the greatest swelling of the material. The hydrophobicity of the outer greige cotton fiber present in the UltraClean cotton may begin to contribute to the overall swelling of the material at 60% UC present in the blend. There is little data on the presence of waxes and pectin in comber noils and gin motes, so a relative comparison of cotton cuticle contributions is not possible. However, recent contact angle measurements of greige cotton nonwovens have shown that approximately 32% of the water droplet coverage is with wax-coated fiber [22]. Thus, the increased swelling due to increasing the ratio of UC may be a result of an additive contribution of waxes from the greige cotton, which are expected to contribute hydrophobicity to the fiber surface analogous to more hydrophobic fibers like polyester. Consistent with this observation, it is important to note that the greige cotton/polyester blend had the highest water absorption capacity. In addition, the similarity of the isoelectric points (IEPs) among the UltraClean cotton samples is consistent with the composition of the samples being cellulose [13].

<table>
<thead>
<tr>
<th>ID</th>
<th>Composition</th>
<th>KFT water content (%)</th>
<th>Moisture content (%)</th>
<th>IEP pH</th>
<th>Plateau potential (mV)</th>
<th>Swell test, k (min⁻¹)</th>
<th>Δζ</th>
<th>Density (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>100% UC</td>
<td>7.46</td>
<td>8.2</td>
<td>2.3(e)</td>
<td>-26</td>
<td>0.0044</td>
<td>0.065</td>
<td>0.147</td>
</tr>
<tr>
<td>2</td>
<td>100% PES</td>
<td>0.53</td>
<td>1.2</td>
<td>3.8</td>
<td>-23</td>
<td>0.0098</td>
<td>0.133</td>
<td>0.118</td>
</tr>
<tr>
<td>3</td>
<td>60% UC/40% PES</td>
<td>4.77</td>
<td>4.6</td>
<td>2.6</td>
<td>-44</td>
<td>0.0140</td>
<td>0.102</td>
<td>0.110</td>
</tr>
<tr>
<td>4</td>
<td>100% CB</td>
<td>7.54</td>
<td>7.7</td>
<td>2.3</td>
<td>-29.0</td>
<td>0.014</td>
<td>0.082</td>
<td>0.210</td>
</tr>
<tr>
<td>5</td>
<td>60% UC/40% CB</td>
<td>7.50</td>
<td>7.1</td>
<td>2.5</td>
<td>-23.0</td>
<td>0.0059</td>
<td>0.089</td>
<td>0.079</td>
</tr>
<tr>
<td>6</td>
<td>40% UC/60% CB</td>
<td>7.55</td>
<td>8.6</td>
<td>2.3</td>
<td>-22.0</td>
<td>0.0087</td>
<td>0.078</td>
<td>0.115</td>
</tr>
<tr>
<td>7</td>
<td>100% GM</td>
<td>7.58</td>
<td>9.5</td>
<td>2.3</td>
<td>-27.0</td>
<td>0.0150</td>
<td>0.077</td>
<td>0.114</td>
</tr>
<tr>
<td>8</td>
<td>60% UC/40% GM</td>
<td>7.54</td>
<td>7.7</td>
<td>2.5</td>
<td>-23.0</td>
<td>0.0093</td>
<td>0.094</td>
<td>0.103</td>
</tr>
<tr>
<td>9</td>
<td>40% UC/60% GM</td>
<td>7.50</td>
<td>9.0</td>
<td>2.3</td>
<td>-26.0</td>
<td>0.0091</td>
<td>0.063</td>
<td>0.131</td>
</tr>
</tbody>
</table>

Fiber codes: UC = UltraClean; PES = polyester; CB = comber noils; GM = gin motes.

KFT = Kraft Fisher Test; IEP = isoelectric point; plateau potential refers to the flat portion of the zeta potential titration curve; Swell test k refers to the rate of swelling; and Δζ refers to the change in zeta potential.

Table 2. Electrokinetic data for the hydroentangled fabric samples made with the different fibers and their blends

3.4. Bound versus free water and crystallite size in cotton blends

It is an understatement to say that the nature of the binding of water to cotton plays a role in the swelling of the blended fabrics as are examined here. The microstructure of cotton fibers allows the penetration of water, in the case of the greige cotton nonwovens studied here. Water-accessible sites of cellulose are formed when cotton contacted by the high-pressure water jets.
during the nonwoven hydroentanglement process, which enhances the exposure of the primary and secondary cell wall of the fiber to an aqueous environment and results in an increase in cellulose-bound water. A subsequent loosening of the fiber cuticle resulting in the exposure of the cellulosic portion of the fiber is evidenced in the SEM image of nonwoven greige cotton shown in Figure 3. Thus, the hydroentanglement process promotes the disruption of the fiber cuticle that retains some wax and pectin while exposing cellulose fibrils and microfibrils to water penetration.

Figure 3. SEM image of hydroentangled greige cotton.

The 7-9.5 % moisture uptake observed for the cotton fabrics of this study and correlated in Figure 2 with swelling is consistent with previous studies that show moisture regain of dried cotton in this range [23]. It is also interesting to speculate how variation in cellulose crystallite size may affect binding of water to the cellulosics of this study. The X-ray diffraction patterns for the individual gin motes, comber noils, and greige cotton (UCC) are shown in Figure 4. The spectra all show the profile characteristic of cellulose I [18, 19]. Table 4 gives the percent crystallinity and cellulose crystallite size of the different types of cotton fibers compared with bleached cotton. The crystallinity index for each sample is as high as can be expected [20] for cotton and reflects nearly complete crystallinity. The calculated X-ray diffraction pattern is shown in Figure 3. The calculated pattern matches the observed patterns [28], especially that of the gin motes, fairly well despite the lack of any consideration of amorphous material in the calculation. The Segal Crystallinity Index values arise almost completely from the overlap of the wide observed peaks (20).

On the other hand, the crystallite size of cellulose in the gin motes and comber noils was larger than cellulose crystallites in greige cotton linters. It is important to note that bleached cotton also was found to have larger crystallite size, but the much higher density of the sample tested excludes it from comparison with the other samples. The relationship between crystallinity
and moisture uptake has received some attention in the literature over the years (24-26). Relevant to crystallinity, crystallite size, and moisture content, Nelson and O’Connor have previously noted that for two celluloses of the same crystallinity but different crystallite size, the cellulose with the smaller crystallite size will exhibit higher moisture regain (27). Observation of this phenomenon is based on more accessible water binding sites, i.e., hydroxyls on smaller crystallites, and is consistent with what is observed in this study (Table 4) in samples of similar density (Table 1) that exhibit differences in crystallite size, i.e., there is a parallel of crystallite size to absorption capacity in the cellulosic greige cotton, gin motes, and comber noils. Thus, as the crystallite size decreases, all things being equal, the absorption capacity of the fabrics consisting of 100 % greige cotton, gin motes, and comber noils increases. The calculated surface-to-volume ratio of smaller cellulose crystallites, as observed with the cotton by-products, is higher than the greige cotton, which infers more accessible hydroxyls for bound water. Thus, an increased capacity to hold water is observed as well in the relatively higher absorption capacity of the cotton by-product nonwovens, which have smaller cellulose crystallite size than the greige cotton (Table 4).

3.5. Moisture determinations

The water content results via Karl Fischer titration (KFT), following ASTM 7795, track the moisture results based on the Kett moisture determination balance that utilizes an infrared lamp (Table 3). The differences between KFT and Kett are due, in part, to the different nature of the two methods. The Kett measures moisture weight loss after drying under a large infrared heat lamp which, minimizes scorching. On the other hand, scorching may contribute to oxidation, thus increasing weight loss (29). Because KFT is specific to water and is carried out under nitrogen, oxidation is eliminated. As a check, standard oven drying was used on the UC
cotton and polyester fibers, generating a moisture content of 7.3 % and 0.43 %, respectively. The polyester water content value is consistent with that found in literature of fiber at standard testing conditions, around 0.4 % (30). Also, note that the KFT water content (Table 3) for polyester is 0.53 %, which is expected since the carrier gas is dry nitrogen. In addition, the small range of KFT values of the cellulosic blends is due to the specificity of KFT to water compared to the weight loss as measured with the Kett infrared heating method. The tight range of crystallinities (Table 4) of the cellulosic is consistent with the KFT findings.

<table>
<thead>
<tr>
<th>Fiber/fiber blend</th>
<th>WT (g/m²)</th>
<th>AATCC Drop Test (sec.)</th>
<th>ASTM Sink time (s)</th>
<th>ASTM Absorbency Capacity (g H₂O per g of fabric)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100% UC</td>
<td>54.6</td>
<td>&lt;1</td>
<td>40.8</td>
<td>4.97</td>
</tr>
<tr>
<td>100% GM</td>
<td>51.9</td>
<td>2.4</td>
<td>32.1</td>
<td>6.88</td>
</tr>
<tr>
<td>100% CB</td>
<td>73.9</td>
<td>6.6</td>
<td>45.0</td>
<td>6.71</td>
</tr>
<tr>
<td>100% PES</td>
<td>68.6</td>
<td>&gt;60</td>
<td>&gt;300</td>
<td>0.14</td>
</tr>
<tr>
<td>60% UC/ 40% CB</td>
<td>60.1</td>
<td>&lt;1</td>
<td>69.4</td>
<td>6.87</td>
</tr>
<tr>
<td>40% UC/ 60% CB</td>
<td>74.8</td>
<td>&lt;1</td>
<td>79.0</td>
<td>6.12</td>
</tr>
<tr>
<td>60% UC/ 40% GM</td>
<td>66.5</td>
<td>5.4</td>
<td>26.4</td>
<td>7.25</td>
</tr>
<tr>
<td>40% UC/ 60% GM</td>
<td>59.5</td>
<td>&lt;1</td>
<td>40.2</td>
<td>7.13</td>
</tr>
<tr>
<td>60% UC/ 40% PES</td>
<td>75.4</td>
<td>&gt;60</td>
<td>113.8</td>
<td>7.05</td>
</tr>
<tr>
<td>40% UC/ 60% PES</td>
<td>66.6</td>
<td>&gt;60</td>
<td>241.0</td>
<td>7.97</td>
</tr>
<tr>
<td>100% BC</td>
<td>103.6</td>
<td>&lt;1</td>
<td>2.25</td>
<td>6.46</td>
</tr>
</tbody>
</table>

Fiber codes: UC = UltraClean; GM = gin motes; CB = comber noils; PES = polyester staple; BC = bleached cotton. (A sample of bleached cotton prepared by similar process was not available for the testing. This sample is provided for information and not comparison due to its higher density).

Table 3. Absorbency characteristics of the various hydroentangled fabrics

cotton and polyester fibers, generating a moisture content of 7.3 % and 0.43 %, respectively. The polyester water content value is consistent with that found in literature of fiber at standard testing conditions, around 0.4 % (30). Also, note that the KFT water content (Table 3) for polyester is 0.53 %, which is expected since the carrier gas is dry nitrogen. In addition, the small range of KFT values of the cellulosic blends is due to the specificity of KFT to water compared to the weight loss as measured with the Kett infrared heating method. The tight range of crystallinities (Table 4) of the cellulosic is consistent with the KFT findings.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Crystallinity index</th>
<th>Crystallite size (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>UltraClean cotton</td>
<td>88.77</td>
<td>53.48</td>
</tr>
<tr>
<td>Comber noils</td>
<td>87.25</td>
<td>47.08</td>
</tr>
<tr>
<td>Gin motes</td>
<td>88.73</td>
<td>46.06</td>
</tr>
<tr>
<td>Bleached cotton</td>
<td>87.95%</td>
<td>65.30</td>
</tr>
</tbody>
</table>

Table 4. Crystallinity index and crystallite size of the different fibers
3.6. Cellulose-water binding considerations

The binding of water to cotton has been characterized in three states, including (1) strongly bound or nonfreezing water, (2) anisotropically constrained or perturbed water, and (3) unperturbed water, or water undergoing isotropic motion (24-26). Consideration of the phenomena of water binding to cellulosic fiber, from crystalline to fibrillar state, crystalline cellulose (crystallites of 36 cellulose chains or more) has been characterized as low water binding (24-26, 31). However, ordered microfibrillar cellulose, which is composed of cellulose crystallites, possesses surface hydroxyls that present accessible water binding sites where penetrating water may form a monolayer (termed nonfreezing water) at a level of (0.1 g/g cotton) (24,31).

French et al. illustrated a cotton fiber moisture model based on the cotton fiber as a solid cylinder of cellulose with a central lumen (length 28 mm, diameter 15 μm) having crystallites at a size of 4 nm by 28 mm, a crystallite density of 1.63 g/cm³, and a water monolayer of 0.25 nm thickness (25, 28). This model is contrasted with experimental models, including one based on TEM images of a water swollen cotton fiber to image water-accessible surfaces. In the cotton fiber water model at a moisture level of 5 %, a monolayer of water only covers 30 % of the surface, and this scenario accounts for one water molecule per cellobiose unit in the cellulose chains of the crystallite. This is somewhat consistent with a recent report on cotton water sorption based on sorption isotherms of cotton by Yakumin et al., showing a capacity of 41.5 g water/g of cellulose (32). They report that 37 % of the dry cotton cellulose is accessible to water binding at equilibrium moisture adsorption. These authors also point out that it is insufficient to use the cellulose crystallinity alone in calculating accessible water surfaces since water molecules can also enter defective regions in the crystallites formed during drying. This feature is also implicated in the incomplete removal of water being located in regions of the crystalline phase that are disorganized or “defective crystallites” upon drying of cotton. Nonetheless, the presence of a strongly bound monolayer of water on cotton is consistent historically with thermal calculations (90 cal/g), approximating that of the heat of fusion for ice and validating the hydrogen bonding forces of water to cellulose (31,33). From this state, further water sorption then assumes the character of a capillary-like condensation and has been characterized as free water, i.e., perturbed and unperturbed water. Thus, based on numerous past studies that characterize the role of water binding to moisture in cotton, the results of this study suggest that the cotton blends, which possess an average of 8 % moisture content, probably have most of the water strongly bound as nonfreezing water under ambient conditions. This is likely as well as it has previously been shown that the density of interfacial water (strongly bound water) on cellulose is increased when it is perturbed. This property improves the wettability of the cotton blends and may be seen as contributing synergistically to improving the swelling properties in the 60 % UC blends.

4. Conclusion

This study has shown that the ability of cotton gin mote fibers to modulate swelling and moisture uptake is beneficial in absorbent products. Depending on the end-use application, a
hydroentangled nonwoven fabric made by using a blend of cotton mote fibers and greige (bleach-less) cleaned cotton lint gave optimal swelling and reasonably good moisture uptake. We compare properties of the nonwoven fabrics made with precleaned greige cotton that were blended in different proportions with cotton gin motes and comber noils. This approach opens up considerations for a more eco-friendly and economical use of cotton by-products, i.e., for wettable topsheet use and fluid transport layer in absorbent nonwovens. Here we contrast cotton by-product blends in greige cotton nonwovens with a similar previous study where polyester was blended with precleaned greige cotton lint (10). The results have shown that the cotton gin mote fibers, compared to the polyester fibers, yield improved moisture uptake while giving comparable swelling attributes. The cellulose-water interactions have been discussed in light of these findings. This moisture uptake/swelling property could assist toward promoting greater utilization of cotton and cotton motes in absorbent nonwoven products where end-use applications require comparable moisture uptake to cotton and similar swelling properties to polyester. Also, the water content by KFT seems to correlate with the ζ potential. Balanced material surface polarity, swelling, density, and moisture uptake is key to optimizing absorbent nonwovens for use in hygiene, incontinence, and even wound care applications, and the results of this study illustrate how these properties may be tuned in with cotton by products used in combination with greige cotton lint. Thus, it is demonstrated that the use of less expensive cotton gin motes—the by-product of cotton ginning process—in blends with cleaned greige cotton lint can potentially be useful and competitive for many nonwoven end-use products where absorbency or moisture uptake, swelling, biodegradability, and sustainability are desirable. This study demonstrates the versatility of nonwoven greige cotton when combined with cotton by-products as putative economical substitutes for synthetic fibers in absorbent applications. It further demonstrates the merit of focusing on material construction and analysis of fiber surface properties with novel by-product fibers at solid-liquid interfaces and the value of considering the molecular factors that influence properties of wettability and fluid transport as they exist in topsheet and layer components useful in absorbent prototypes, i.e., fibers that present green alternatives to petro-based approaches while demonstrating structure/function value. Future studies will focus on specific functional applications for incontinence hygiene and wound care nonwovens.

Author details

Vince Edwards1*, Paul Sawhney1, Alvin Bopp2, Alfred French1, Ryan Slopek1, Michael Reynolds3, Chuck Allen1, Brian Condon1 and Joseph Montalvo1

*Address all correspondence to: vince.edwards@ars.usda.gov

1 Southern Regional Research Center, US Department of Agriculture, USA
2 Chemistry Department, Southern University of New Orleans, USA
References


