# EFFECT OF ADSORBED WATER ON THE SPECIFIC SURFACE AREA OF SOME STANDARDS COTTON C. Kaewprasit Kasetsart University Kampangsaen Campus, Thailand N. Abidi Laboratory of Spectronomie-Physique University of Moulay Ismail Meknes, Marocco J.P. Gourlot Laboratory of Cotton Technology, CIRAD-CA Montpellier, France

### **Abstract**

It is well known that cotton fibers are sensitive to moisture. The interactions of cellulose chains and water molecules could therefore have an influence on the fiber properties. In this presentation, we will investigate the effect of adsorbed water on the specific surface area of standards cotton fibers. The amount of adsorbed water was determined by Thermogravimetric analysis. Adsorption of methylene blue in aqueous phase was used to measure the surface specific area. A numerical relationship of the form  $S_{BM}$ =a +b X has been obtained, where  $S_{BM}$  represents the specific surface area, X the percentage of adsorbed water, a and b are experimental constants.

#### **Introduction**

In previous work (Kaewprasit et al., 1998) a relationship between the specific surface area of International Calibration Cotton Standards (ICCS) fibers, measured by adsorption of methylene blue in aqueous phase, and their fineness, measured with a double compression air-flow instrument, was presented. This very interesting relationship would enable us in the future to propose a new reference method to calibrate specific surface measurements obtained with a double compression air-flow instrument. We are still investigating cotton fiber properties by using physical chemistry techniques. Our goal aims to have a better knowledge on the physical properties of cotton fiber (as natural polymers) and to establish relations between these physical properties and the most important fiber properties such as: fineness, maturity, tenacity, etc.

Cotton fiber is composed of 90% of cellulose and after scouring and bleaching, the cotton fiber is at 99% pure cellulose (Alen, 1998). Cellulose is a natural polymer ( $(C_6H_{10}O_5)_n$ ) made up anhydro-glucose units linked by 1,4 oxygen bridges. The characteristics of cellulose fibers are primarily due to their number of hydroxyl groups. There are three hydroxyls groups presents in each anhydro-glucose unit of cellulose fiber. Therefore, the cellulose fibers are hydrophilic with high moisture regain. The hydroxyl groups of cellulose fiber serve as bonding sites for certain dyes as metylene blue (Kaewprasit et al. 1998) and for adsorption of water through hydrogen bonds. The presence of adsorbed water could affect the physical properties of the cotton fibers. Since the specific surface area is one of the most important physical properties for dyeing, for example, this study was done to find out the effect of adsorbed water on the specific surface area of 6 ICCS cotton fibers.

## Materials and Methods

Six ICCS cotton fiber samples: B26, C36, D5, E4, G17 and I26 were conditioned at  $21 \pm 1$ ?C with  $65 \pm 2\%$  of relative humidity (R.H.) during at least 24 hours at least.

The specific surface area of these cotton fibers was estimated by adsorption of methylene blue in liquid phase (Kaewprasit, 1997). To determine the percentage of adsorbed water on each cotton fiber sample, we used the Thermogravimetric Analysis (TGA).

TGA is a thermal analysis technique used to measure changes in the weight (mass) of a sample as a function of temperature and/or time. TGA is commonly used to determine polymer degradation temperatures, adsorbed water content, etc. A simplified explanation of a TGA sample evaluation may be described as follows. A sample is placed into a tare TGA sample pan which is attached to a sensitive balance. The sample holder is placed into a furnace. The balance measures the initial sample weight at room temperature and then continuously monitors changes in sample weight (losses or gains) as heat is applied to the sample. TGA tests may be run in heating mode at controlled heating rate or isothermally. Typical weight loss profiles are analyzed for the amount or percent of weight loss at any given temperature.

In the present work, the thermal measurements were carried out in air between room temperature and 150°C, and the heating rate is 0.25°C/min.

#### **Results and Discussion**

Figure 1 shows the evolution of the weight loss versus temperature for B26 and E4 cotton samples as examples. The weight loss and the value of specific surface area estimated by adsorption of methylene blue in liquid phase ( $S_{BM}$ ) are given in table 1.

The analysis of the evolution of the weight loss versus temperature (figure 1) shows that the curves could be decomposed into two regions: the first one is from room temperature to  $100^{\circ}$ C degrees, and the second from  $100^{\circ}$ C to  $150^{\circ}$ C.

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For the cottons B26, C36, D5 and I26 the loss of weight is rapid in the first region. As they reach 100°C, these cotton samples have lost in the range of 5.8% and 6.4% of their initial weight. This is ascribed primarily to the loss of free water or weakly bonded water. Above 100°C, no significant loss is observed (or in the second region). We can concluded that when B26, C36, D5 and I26 are conditioned at  $21 \pm 1$ °C with  $65 \pm 2$ % of relative humidity only free water is present.

For the cotton samples E4 and G17 at 100°C the weight loss is about 7.4 - 7.6%, and we observe that weight lost is still continue until the temperature reach at 150°C. The total loss is considerable and is estimated to be nearly 8%. We will ascribe the weight loss between 100°C and 150°C to bonded water. We assume that the water molecules are bonded to cellulosic chains via hydroxyls groups.

The analysis of table 1 shows a relationship between specific surface area and the weight loss between room temperature and 150°C. The evolution of surface area versus the amount of water is represented in figure 2. We observe an increase of specific surface area when the amount of adsorbed water increases. The following numerical relationship was obtained:

$$S_{MB} = a + b X$$
,

where  $S_{BM}$  represents the specific surface area of cotton fiber in m<sup>2</sup>g<sup>-1</sup>, X represents the amount of adsorbed water in %, a = -8.7970, b = 6.6825 with R<sup>2</sup> = 93.4%.

The increase of specific surface area with increasing adsorbed water could be explained by the fact that the water molecules enter the amorphous regions and force the polymeric chains to move. The water molecules remain bonded to cellulosic chains through hydrogen bonds. When we measure the specific surface area by adsorption in liquid phase, methylene blue molecules have, therefore, a high accessible internal surface area. In this case, water molecules serve as vehicle for these dye molecules. In addition, nitrogen sorption measurement shows that the surface area of cotton fiber is in the range  $0.6-1m^2g^{-1}$ (Kaewprasit, 1997). This effect could be explained by the fact that in dry state, strong inter-hydrogen bond (bond between cellulosic chains) could be established. This phenomena decrease the accessibility of the internal surface and, therefore, only external surface is measured.

It is important to point out that the increase of adsorbed water leads to an increase of intermolecular hydrogen bonding between cellulosic chains. Since cellulosic chains become orderly, there is an increase in the degree of cristallinity. And the strength of cotton fiber increases with increased moisture. This phenomenon could affect the cotton fiber tenacity. Kaewprasit et al.1997, has established a relationship between specific surface area and fineness. It seems interesting to plot the evolution of fineness against percentage of adsorbed water (figure 3). As it can be observed, there is a decrease of fineness as the amount of adsorbed water increases. However, at present we can not concluded it exactly and further experiments are necessary before establishing of numerical relationship between fineness and adsorbed water. Figure 4 shows the evolution of maturity ratio versus percentage of adsorbed water. From this result, there is no significant correlation.

In this study we tried to explain the variation of specific surface area of 6 ICCS. However, other parameters could affect also the value of the surface such as the variation of perimeter from one type of cotton to another one. In the future, we will investigate the relationship between surface and tenacity and adsorbed water for one type of cotton fiber and confirm the existing relationships with other cottons.

### **Conclusion**

This study leads to establish a relationship between the specific surface area and the percentage of adsorbed water (free water or bonded water). High specific surface area is associated with high amount of adsorbed water. This phenomenon is the consequence of formation of intermolecular hydrogen bonds that could be moved by dyeing molecules in liquid phase.

#### **References**

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Table 1: Specific surface area  $(m^2g^{\text{-}1})$  and loss of weight during heat between  $25^\circ C$  and  $150^\circ C.$ 



Figure 1: Evolution of loss of weight against heat temperature for cottons B26 and E4.



Figure 2: Relationship between specific surface area and percentage of adsorbed water.



Figure 3: Evolution of fineness against percentage of adsorbed water.



Figure 4: Evolution of maturity ratio against percentage of adsorbed water.