Specific Surface Area of Cotton Measured by Methylene Blue Absorption and Relation to its Fineness

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ABSTRACT

The specific surface area of six cotton fiber samples from ICCS was estimated by means of methylene blue adsorption in the liquid phase. The adsorption kinetic of the dye on these cotton fibers was determined to obtain the conditions of adsorption isotherm. This was held at 25°C with shaking rotation at 180 rpm. for 24 hours. The concentration of methylene blue was in the range 0.004 - 0.32 g l-1 for each isotherm. Spectrophotometry technique at the wavelength 660 nm was used to analyze the concentration of methylene blue solution after 24 hours. The surface areas of these cottons, B-26, C-36, D-5, E-4, G-17 and I-26 were found to be 32.3, 32.4, 34.4, 52.72, 43.9 and 29.9 m2 g-1 respectively. There are relationships between these surface areas and their fineness analyzed by gravimetric method, the Shirley Development Ltd. Fineness Maturity Tester 3. (FMT 3), and the Advance Fiber Information System (AFIS). A numerical relationship of the form a + bH-1 was obtained through nonlinear regression computer software.

Introduction

The surface area of cotton fibers influences its printing characteristics. Various methods are used to study and measure printing characteristics such as: moisture adsorption (Assaf *et al.*, 1944), nitrogen adsorption (Assaf *et al.*, 1944; Merchant, 1957), NMR (Froix and Nelson, 1975) etc. but each method presents inconveniences and difficulties because of modifications in the surface area caused by the surrounding phase.

Methylene blue was chosen in this study because of its known strong adsorption onto solids and its recognized usefulness in characterizing adsorptive material (Froix and Nelson, 1975 ; Giles and De Silva, 1969 ; Barton, 1987). Its application to cotton fiber had not previously been tested. Methylene blue or 3, 7 bis (dimethylamino) phenothiazin-5-ium ion (Ardizzone *et al.*, 1993), molecular weight 373.9 g mol-1 that corresponds to methylene blue hydrocloride with three groups of water, was purchased from Carlo Elba. The structure of this dye is shown in the Fig. 1.

The objective of this study is to propose a simple method of analyzing cotton fiber specific surface area and to establish the relationship between the specific surface area and its mechanical properties.

Materials and methods

The ICCS (International Calibration Cotton Standard) samples B-26, C-36, D-5, E-4, G-17, and I-26 were used to estimate the specific surface area by adsorption of methylene blue because they differ in physical characteristics. All the samples were conditioned at 21±1°C, 65±2% R.H. for at least 24 h, the same conditions as those used for normal fiber testing.

The intent of the experiment was to determine the value of the surface area, specific for each cotton and to verify the sensitivity of this method by using the effect of cotton fiber preparation and of temperature on the cotton before analysis. Two cotton fiber samples, C-36 and B-26, were used and prepared in different ways: a) hand opening of fibers of raw cotton; b) single carded fibers; c) triple carded fibers; and d) drawn fibers after triple carding. These cotton were also subjected to different temperature, 21°C and 60°C, before analysis.

Absorption of methylene blue

The absorption kinetics were studied first to determine the equilibrium time. This time was determined through a series of measurements extending from 2 to 72 h at 25°C on cotton C-36 with shaking rotation of 180 rpm. In presence of the solid, adsorbing solutions reached complete equilibrium in about 24 h. The adsorption measurements were then carried out as follows: 2 gms of cotton were put in 250 ml methylene blue solution in a flask, maintained at 25°C and continuously shaken at 180 rpm for 24 h. The concentration of methylene blue was is in the range 0.004 - 0.32 g l-1. The methylene blue uptake in cotton fibers was calculated from the difference between concentration of methylene blue concentration before and after adsorption on the cotton fibers. Four

replications were carried out for each type of cotton. There was negligible methylene blue adsorption onto the glassware.

Determination of methylene blue concentration in solution

The concentrations of methylene blue solution after adsorption were analyzed by measuring their absorbency at 660 nm on a Pye Unicam spectrophotometer. This wavelength corresponds to the maximum absorption peak of methylene blue monomer (Bergmann and O'Konski, 1963). A calibration curve of absorbency against methylene blue concentrations was obtained, using standard methylene blue solutions of known concentrations. The data fitted a straight regression line with a high correlation coefficient (r = 0.9992) (Figure 2).

Result and discussion

The typical adsorption isotherm of methylene blue on cotton fibers represented by E-4 is shown in the Figure 3. These isotherms are of type I generally associated with monolayer adsorption, however their initial slopes do not lie very near to the y-axis, showing that the affinity of methylene blue to the cotton fibers is moderate. Nonlinear regression computer software was used to calculate the methylene blue adsorption at the monolayer of the fibers (χMB). And the surface area was calculated by the following equation:

$$S_{MB} = \underline{\hspace{4cm}}$$

where ; S_{MB} specific surface area in m^2 g^{-1}; χ_{MB} methylene blue adsorption at the monolayer of fibers in g g^{-1}; a_{MB} occupied surface area of one molecule of methylene blue $=197 A^{\circ 2}$ (Graham 1955) ; N Avogadro number 6.02×10^{23} mol^{-1} ; M molecular weight of methylene blue 373.9 g mol^{-1}.

The methylene blue adsorption at monolayer and surface areas of the six standard cottons, are shown in Table 1.

Table 1. Surface area of 6 studied cottons.

Cotton	χ_{MB} g g^{-1}	S_{MB} m^2 g^{-1}
B-26	0.0102	32.32
C-36	0.0102	32.42
D-5	0.0109	34.48
E-4	0.0166	52.72
G-17	0.0139	43.96
I-26	0.0094	29.91

These results show: 1) cotton C-36 has a surface area of 32.42 m^2 g^{-1} which is around 30% larger than that determined by the nitrogen adsorption method (Brunauer, Emmet and Teller or BET) (Kaewprasit 1997); and 2) there are differences between these values that can be used to express the relationship between surface area and fineness, determined by gravimetric method, FMT 3 and AFIS (Figure 4-6). This relationship is possibly related to the building of the cellulose network during plant growing. The nonlinear regression computer software shows a numerical relationship of the form $S_{MB} = a + bH^{-1}$ where; S_{MB} represents the specific surface area of cotton in m^2 g^{-1}; H represents the fineness of cotton in mTex; a and b are constant with R^2 = 75%, 79% and 67% for measurement by gravimetric method, FMT3 and AFIS respectively.

Table 2 shows the effect of different sample preparations and different temperature treatments on the cotton fiber before adsorption to the specific surface of cotton fibers. The carded cotton fibers give a higher surface area than the other types of the preparation. There is no direct evidence from the SEM photos for the different preparation of cotton fibers. This discrepancy can be explained by the fact that the fibers are perfectly parallel, and the methylene blue molecules can easily diffuse through the fibers. With different temperature treatments the dried cotton fibers, held at 60°C for 2 h, give a higher surface area than fibers that were conditioned at 21±1°C and 65±2% R.H. This phenomenon may be due to a higher area in dried cotton fibers. In addition, this method is sensitive to differences in both cotton fiber preparation and temperature treatment.

Conclusion

The adsorption of methylene blue is useful in determining the surface area of natural cotton fibers. The method is simple and requires less elaborate apparatus and time than the other methods. This specific surface area may be used directly as a cotton fiber characteristic. In the future, we will try to link these results to the moisture percentage and X-ray fiber structure of each cotton type.

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Table 2. Effect of different ambient and different preparations of sample to the specific surface area of cotton fibers B-26 and C-36.

	B-26		C-36	
	χ_{MB} g g^{-1}	S_{MB} m^2 g^{-1}	χ_{MB} g g^{-1}	S_{MB} m^2 g^{-1}
Conditioning :				
21±1°C, 65±2% R.H., at least 24 h				
raw	0.01019	32.32	0.01022	32.42
carded 1 time	0.01057	33.53	0.01411	44.75
carded 3 time	0.00993	31.50	0.01348	42.76
drawn	0.00633	20.08	0.01016	32.23
Dried at 60°C, 2h				
raw	0.01492	43.32	0.02092	66.35

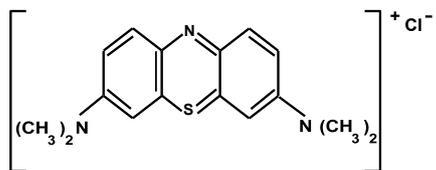**Figure 1. Structure of methylene blue.**

Figure 2. Calibration curve of the spectro-photometer absorbance vs methylene blue concentration.
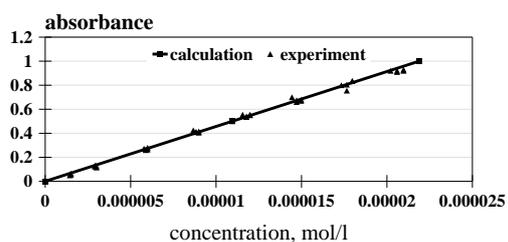

Figure 3. Typical adsorption isotherm of cotton at 25°C, during 24 h presented by cotton E-4.
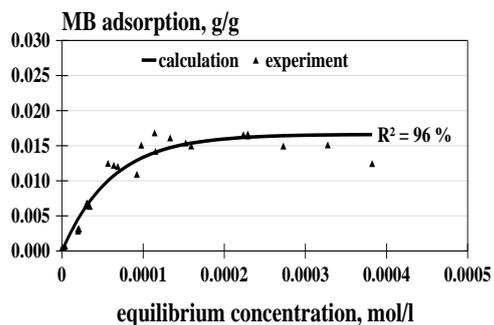

Figure 4. Relationship curve of cotton fiber specific surface area and fineness by gravimetric method.
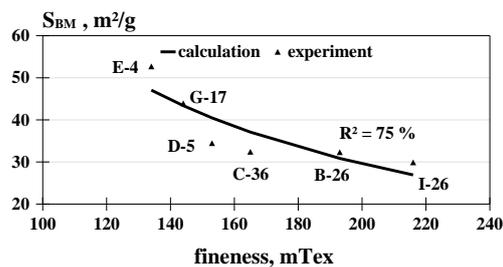

Figure 5. Relationship curve of cotton fiber specific surface area and fineness by FMT3.
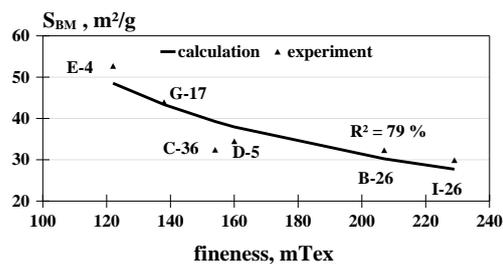

Figure 6. Relationship curve of cotton fiber specific surface area and fineness by AFIS.