COTTON SPECIFIC SURFACE AREA MEASUREMENT BY ADSORPTION OF METHYLENE BLUE

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Abstract

Specific surface areas of 6 coton fibers from ICCS were estimated by the methylene blue adsorption. The adsorption isotherm was done at 25°C during 24 h. This condition was obtained from the kinetic study of this dye on the cotton fibers previously. The concentration of methylene blue used, in the range 0.004 - 0.32 g l^{-1}, was analysed by spectrophotometer at the wavelength 660 nm. The surface areas of these cottons, B-26, C-36, D-5, E-4, G-17 and I-26 were found 32.3, 32.4, 34.4, 5.72, 43.9 and 29.9 m^2 g^{-1} respectively. There is relationships between these surface areas and their fineness analysed by FMT 3 (Fineness Maturity Tester 3, by Shirley Dev. Lim.). A numerical relationship of the form $a+bH^{-1}$ has been obtained by nonlinear regression computerized software.

Introduction

Surface area of cotton fibers is important for printing characteristics. It is studied and measured by methods such as : moisture adsorption (Assaf et. al., 1944), nitrogen adsorption (Assaf et. al., 1944 ; Merchant 1957), NMR (Froix and Nelson 1975) etc. But there are a lot of inconveniences and difficulties for each method due to the modification of 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). Methylene blue or 3, 7 bis(dimethylamino)phenothiazin-5-ium ion (Ardizzone et. al., 1993), molecular weight 373.9 g mol^{-1}, which corresponds to methylene blue hydrocloride with three groups of water, was purchased from Carlo Elba. The structure of this dye is shown in **figure 1**.

The purpose of this study is to propose a simple method to analyse the cotton fibre specific surface area.

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Materials and Methods

The ICCS (International Calibration Cotton Standard) named B-26, C-36, D-5, E-4, G-17, and I-26 were used because the mechanical properties are different among them. All of the samples were conditioned at 21"1°C, 65"2% R.H. during at least 24 h, at the same conditions as for measurement of mechanical properties. Different preparations of two cotton fibers (C-36 and B-26) as follows : a) open fibers by hand named raw cotton, b) carded fibers for 1 time, c) carded fibers for 3 times and d) drawn fibers after carding for 3 times, were also studied in order to obtain higher surface area.

Adsorption of Methylene Blue

First of all, adsorption equilibrium conditions were studied. The equilibrium time was determined by series of measurements extending from 2 to 72 h at 25°C on cotton C-36. In presence of the solid, adsorbing solutions reached complete equilibrium in about 24 h. Then, the adsorption measurements are carried out as follows : 2 g of cotton were put in 250 ml methylene blue solution contained in a flask, maintained at 25°C and continuously shaken at 180 rpm during 24 h. The concentration of methylene blue used 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 the methylene blue concentration before and after adsorption on the cotton fibers. Four replications were carried out for each type of studied cotton. We found that there was negligible methylene blue adsorption on the glassware.

Determination of Methylene Blue Concentration in Solution

The concentrations of methylene blue solution after adsorption were analysed by measuring their absorbance 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 absorbance against methylene blue concentrations was obtained by using standard methylene blue solutions of known concentrations. The experimental data reported in **figure 2** are fitted by a straight line with a high correlation coefficient (r = 0.9992).

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 this show that the affinity of methylene blue to the cotton fibers is moderate. Nonlinear regression computerized software was used to calculate the methylene blue adsorption at the monolayer of the fibers (χ_{MB}). And the surface area was calculated by the following equation:

$$S_{MB} = \frac{\chi_{MB} * a_{MB} * N * 10^{20}}{M}$$

where ; S_{MB} specific surface area in m^2g^{-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 D^2 (Graham 1955) ; N Avogadro number $6.02x10^{23}$ mol^{-1} ; M molecular weigth of methylene blue 373.9 g mol^{-1}.

The methylene blue adsorption at monolayer and surface areas of the 6 standard cottons, are shown in the **table 1**. From these results we find that, firstly, cotton C-36 gives the surface area of 32.42 m^2g^{-1} which is around 30% larger than those obtained by method of nitrogen adsorption (Brunauer, Emmet and Teller or BET) (Kaewprasit 1997). Secondely, there are the difference of these values among them and we can express the relationship between these surface areas and their fineness analysed by FMT3 (Fineness Maturity Tester3, Shirley Dev. Lim.) which shown in the **figure 4**. This relationship is perhaps related to the building of the cellulose network during plant growing. By nonlinear regression computerized 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^2g^{-1} ; H represents the fineness of cotton in mTex; a = 4, b = 5432 with R^2 = 79%.

Table 2 shows the effect of different preparations of sample and different temperature treated on the cotton fibre before adsorption to the specific surface of cotton fibers. We find that carded cotton fibers give 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 discripancy can be explained the fibers are perfectly parallel, and the methylene blue molecules can easily diffuse through the fibers. For the different temperature traited on cotton fibre, we found that the dried cotton fibers, at 60°C during 2 h, give higher surface area than those which were conditioned at 21"1°C and 65"2% R.H. This phenomenon may be dued to more disposable area of the dried cotton fibers.

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.

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Table 1. Surface area of 6 studied cottons.

Cotton	χ_{MB} g g^{-1}	surface m^2g^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

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

	χ_{BM} g g^{-1}		S_{BM} m^2g^1	
	B-26	C-36	B-26	C-36
Conditioning : 21"1°C, 65"2% R.H.				
raw	0.01019	0.01022	32.32	32.42
carded 1 time	0.01057	0.01411	33.53	44.75
carded 3 times	0.0099	0.01348	31.5	42.76
drawn	0.0063	0.01016	20.08	32.23
Dried at 60°C, 2h				
raw	0.01492	0.02092	43.32	66.35

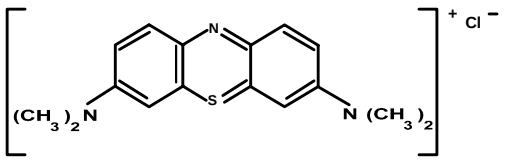

Figure 1. Structure of methylene blue.

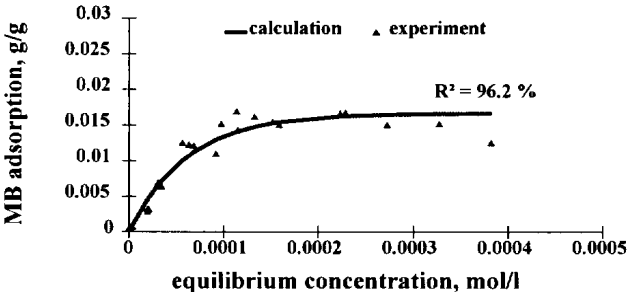

Figure 3. Typical adsorption isotherm of cotton at 25°C, during 24 h presented by cotton E-4.

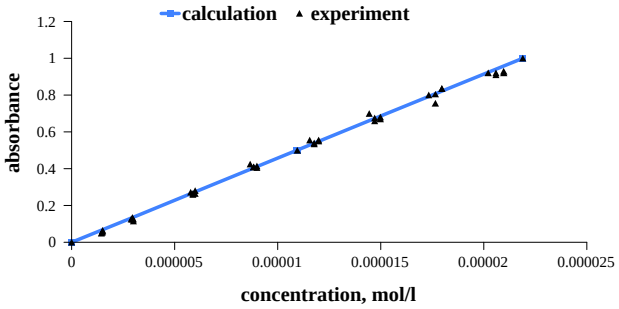

Figure 2.Calibration curve of the spectrophotometer absorbance vs methylene blue concentration.

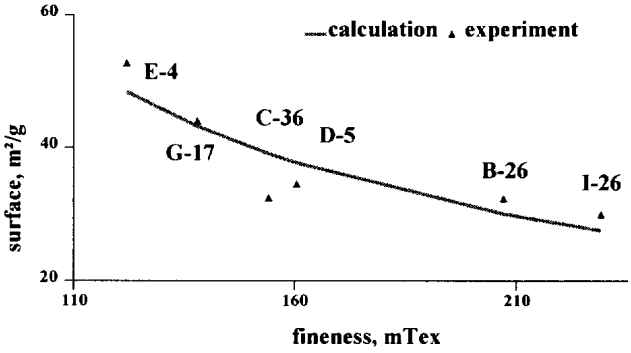

Figure 4. Relationship curve of cotton fiber specific surface area and fineness.