Indian Journal of Textile Research
Vol. 6, December 1981, pp. 147-150

# Evaluation of Alkali-Treated Cellulosic Materials

K VENKATARAYAN, MRS G R PHALGUMANI & B R MANJUNATHA

Textiles Committee, Bombay 400018

*Received* 4 *July* 1981; *accepted* 4 *November* 1981

A quick and accurate method which can be used effectively for the evaluation of alkali-treated cotton textile goods has been developed based on the ratio of propanol retention values. The ratios of propanol retention values have been classified into specific norms proposed for assessing the degree of mercerization of cotton textile materials. The compatibility of the new method with the barium activity number method is quite good. The applicability of the new method can also be extended to evaluate alkali-treated viscose rayon samples, their blends with cotton cellulose and sulphur-dyed samples.

In the textile industry, mercerization is used as a major process for imparting better finish to textile materials. One of the well known methods for evaluating the efficacy of this process is the determination of barium activity number (BA No.). The present investigation was aimed at developing an alternative method which is quicker and at the same time as reliable as the BA No. method. Different methods[1-5] were considered and among these, propanol retention value (PRV) method has been found to be one of the best practicable methods. The results obtained by the PRV method have been correlated with those of the BA No. method. Apart from this, an in-depth evaluation of alkali-treated cellulosic materials has been carried out.

While working in this direction, various factors like the effect of viscose, polynosic and their blends with cotton on alkali treatment, and the effect of sulphur-dyed materials were also taken into consideration. The study of the effect of viscose, polynosic and their blends with cotton is of immense importance, since of late these materials are being used by the industry in abundance in conjunction with cotton not only to provide balanced performance characteristics to the consumer, but also in line with the mixed fibre policy of the government.

## Materials and Methods

The constructional parameters of fabrics and yarns used are given in Table 1. These samples had previously been scoured before further treatment. All the chemicals used were of commercial grade.

The alkali treatment was given as per the procedure of Malu *et al.*[1].

*Determination of barium activity number*—BA No. was determined using the procedure given in the survey report published by the Textiles Committee[6]. This method differs from the published standards (IS:1689-1973 and AATCC:89-1977) only in that the sample and control in barium hydroxide solution are allowed to stand overnight. This is done for convenience, as there is no evidence to show that a stay period of more than 2 hr in the solution has any effect on the ultimate result.

*Determination of ratio of propanol retention values (RPRV)*—The determination of propanol retention value as a method for the evaluation of alkali treatment to cellulose is based on the method of Andrews and Oberg[7]. This method was modified to suit our requirements and purpose, specially with respect to immersion period and centrifuge speed. The standardized procedure is as follows.

Table 1—Physical Characteristics of Materials Used

| Material | Count | | Threads/in | | BA No. |
|---|---|---|---|---|---|
| | Warp | Weft | Ends | Picks | |
| Poplin control | 42.2s | 41.8s | 126 | 66 | — |
| Voile control | 50.4s | 45.8s | 84 | 72 | — |
| *Commercial fabric samples* | | | | | |
| Mercerized printed fabric | 46.0s | 57.4s | 57 | 40 | 141.2 |
| Mercerized poplin | 41.0s | 36.8s | 126 | 60 | 169.4 |
| Bleached mercerized poplin | 42.0s | 45.8s | 147 | 75 | 108.0 |
| -do- | 42.5s | 37.9s | 92 | 57 | 161.5 |
| -do- | 40.1s | 42.3s | 90 | 70 | 148.8 |
| Mercerized printed cambric | 61.4s | 47.5s | 106 | 80 | 129.9 |
| Printed mercerized poplin | 31.3s | 37.2s | 100 | 56 | 120.4 |
| Cotton-polynosic blended fabrics (commercial) | 40s | 40s | 80 | 70 | — |
| Cotton yarn used for blending | 36s | | | | |
| Viscose yarn | 120D - 40 filaments | | | | |
| Polynosic staple | 1.7D - 38 mm staple | | | | |

The samples before and after alkali treatment (hereinafter referred to as control and sample) were immersed in isopropanol in two different stoppered conical flasks and kept for 30 min. After the specified period, both the control and sample were taken out and put separately in two different 50 ml centrifuge tubes provided with filtering arrangement and then centrifuged at 2000 rpm for 30 min. The centrifuged sample and control were weighed, dried at $105 \pm 2°C$ for 1 hr and again weighed. The propanol retention is calculated on the dry weight for the control and the sample separately and the ratio of the sample to the control is expressed as a percentage. Twelve such determinations were made and the mean value was tabulated. By determining the ratio of propanol retention values, some experimental errors like those arising from variation in centrifugation period, duration of contact in solution and any effect of change in centrifuge speed are eliminated.

## Results and Discussion

*Evaluation of influence of alkali treatment on cotton cellulose*—The cotton samples were treated with alkali of different concentrations for 1 hr at room temperature as per the prescribed procedure. The conditioned samples were then evaluated for the effect of alkali treatment in terms of BA Nos as well as RPRV. The results are projected in Fig. 1. It is seen that RPRV and BA No. show identical trend. Further, the influence of duration of alkali treatment on cotton cellulose was also studied. The samples were treated with 24% (wt/wt) (52° TW) NaOH and their BA No. and RPRV were evaluated after 3,5,10,15 and 30 min. The results are presented in Fig. 2. It is observed that a period of 5 min is sufficient for completion of the alkali treatment. From the initial set of these experiments, it is evident that the values of RPRV and BA Nos are in conformity with each other and as such RPRV can be used effectively as a quick and concise alternative mehtod for evaluating the efficiency of mercerization in place of the usual BA No. determination.

*Application of RPRV method in the evaluation of commercial samples*—The RPRV method was used in the evaluation of commercial cotton fabrics for their mercerization treatment in comparison with BA Nos. The results are given in Table 2. Similarly, certain commercial mercerized yarn samples were subjected to evaluation by the RPRV method in comparison with BA Nos. The results are presented in Table 3.

In both the cases, the samples were assessed in terms of the norms of BA No. range proposed in an earlier report[6]. It is evident that RPRV can ascertain effectively the extent of mercerization or alkali treatment in line with BA Nos.

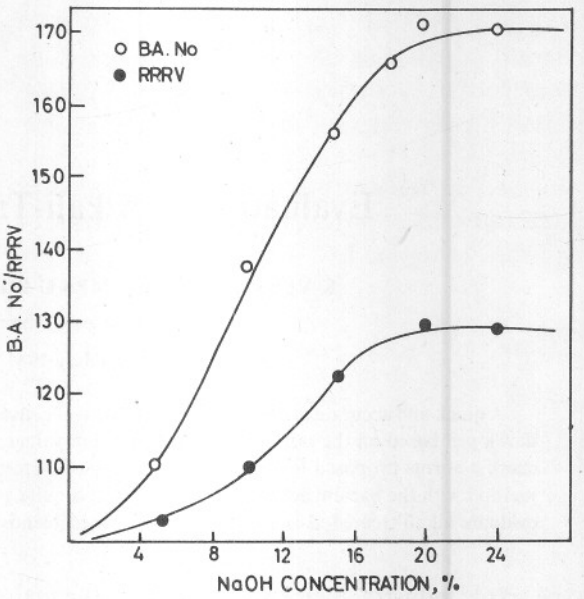


Fig. 1—BA No./RPRV versus concentration of NaOH with which cotton fabrics were treated

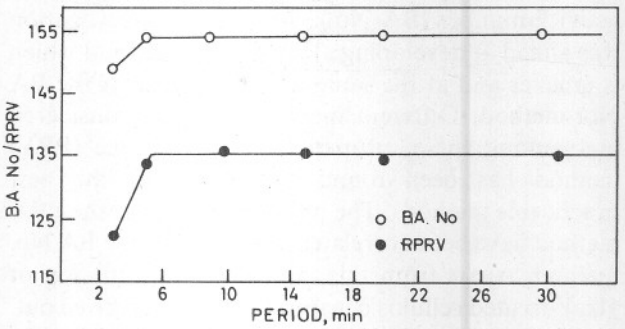


Fig. 2—BA No./RPRV versus period of alkali treatment

Table 2—RPRV for Commercial Mercerized Fabric Samples

| BA No. | RPRV | Assessment |
|---|---|---|
| 108.4 | 100.38 | Unmercerized |
| 120.4 | 104.86 | Causticized |
| 129.9 | 108.12 | Inadequately mercerized |
| 141.2 | 114.18 | Fully mercerized |
| 148.8 | 110.08 | -do- |
| 161.5 | 129.86 | -do- |
| 169.4 | 129.04 | -do- |

Accordingly, the RPRV values were also scrutinized and classified to represent the proposed norms of the corresponding range of BA Nos for assessing the mercerized cotton materials meant for export (Table 4). The tentative norms of RPRV in analogous category of BA Nos will provide a guideline for quick assessment of commercial samples for the degree of mercerization.

*Compatibility of BA Nos with RPRV*—The congruity of these two methods of assessment of alkali-treated cotton goods was statistically established in terms of high correlation coefficient data presented in Table 5.

To see that the observed correlation coefficient is significantly different from zero, a significance test was performed using the following formula:

$$\frac{r\sqrt{n-2}}{\sqrt{1-r^2}}$$

According to the values of correlation coefficients presented above

$$\frac{r\sqrt{n-2}}{\sqrt{1-r^2}} = \frac{0.9007}{0.4344} \times \sqrt{23}$$

$$= \frac{0.9007}{0.4344} \times 4.7958$$

$$= 9.9438$$

Table 3—RPRV for Commercially Mercerized Yarn Samples

| BA No. | RPRV | Assessment |
|---|---|---|
| 139.5 | 113.50 | Inadequately mercerized |
| 114.49 | 103.43 | Unmercerized |
| 125.0 | 107.94 | Causticized |
| 134.36 | 108.96 | Inadequately mercerized |
| 138.0 | 112.00 | -do- |
| 142.2 | 114.77 | -do- |
| 168.7 | 121.92 | Fully mercerized |
| 163.6 | 122.83 | -do- |
| 128.0 | 105.52 | Causticized |
| 132.6 | 109.41 | Inadequately mercerized |

Table 4—Proposed Norms of RPRV to Indicate Corresponding BA No. Range

| Assessment | Range of BA No. for fabrics | Range of RPRV for fabrics | Range of BA No. for yarns | Range of RPRV for yarns |
|---|---|---|---|---|
| Unmercerized | Below 110 | Below 101 | Below 120 | Below 104 |
| Causticized | 110 and below 120 | 101 and below 104 | 120 and below 130 | 104 and below 108 |
| Inadequately mercerized | 120 and below 135 | 104 and below 110 | 130 and below 150 | 108 and below 116 |
| Fully mercerized | 135 and above | 110 and above | 150 and above | 116 and above |

Thus, the correlation between BA No. and RPRV is significant at 5% level.

*Evaluation of RPRV for alkali-treated viscose/polynosic samples*—The viscose rayon/polynosic samples were treated with alkali of different concentrations for 5 min at 27 ± 2°C. The retention values were determined for these samples against viscose/polynosic controls. The values are presented in Table 6 along with their corresponding BA Nos. It is obvious that the trend in the values showing the behaviour of rayons after alkali treatment shows good agreement. It is, however, very interesting that both the retention values and BA Nos decrease after 5% alkali treatment and remain constant, implying that there is no effect of further concentration of alkali on viscose rayon samples.

Similarly, the blended cellulosic textiles were also well characterized by RPRV in agreement with the respective BA Nos. in assessing the samples for mercerization or alkali treatment. Viscose-cotton

Table 5—Correlation Coefficient Data

| BA No. $x$ | RPRV $y$ | BA No. $x$ | RPRV $y$ |
|---|---|---|---|
| 168.9 | 137.51 | 151.9 | 128.68 |
| 140.5 | 123.07 | 166.0 | 128.23 |
| 108.0 | 100.38 | 172.2 | 128.27 |
| 120.4 | 104.86 | 170.8 | 130.23 |
| 129.9 | 103.12 | 149.3 | 122.69 |
| 141.2 | 114.12 | 153.8 | 131.39 |
| 148.8 | 110.08 | 153.8 | 135.92 |
| 161.5 | 129.86 | 153.8 | 135.47 |
| 169.4 | 125.04 | 153.8 | 135.38 |
| 108.3 | 95.23 | 164.9 | 129.18 |
| 117.5 | 103.70 | 141.2 | 118.68 |
| 145.8 | 122.59 | 182.6 | 142.41 |
| | | 169.6 | 133.88 |

$\Sigma x = 3743.9$
$\Sigma y = 3070.03$
$\Sigma xy = 465461$
$\Sigma x^2 = 570382.21$
$\Sigma y^2 = 381136$
$r_{xy} = 0.9007$

Table 6—BA No. and RPRV of Viscose and Polynosic Samples Treated with Different Concentrations of NaOH at 27±2°C for 5 min against Viscose/Polynosic Controls

| NaOH % | Viscose | | Polynosic | |
|---|---|---|---|---|
| | BA No. | RPRV | BA No. | RPRV |
| 5 | 96.13 | 93.91 | 95.43 | 96.58 |
| 10 | 86.08 | 95.60 | 86.80 | 93.79 |
| 15 | 87.11 | 93.10 | 87.82 | 95.80 |
| 20 | 88.40 | 92.05 | 87.06 | 91.80 |
| 25 | 85.30 | 92.09 | 87.58 | 93.82 |

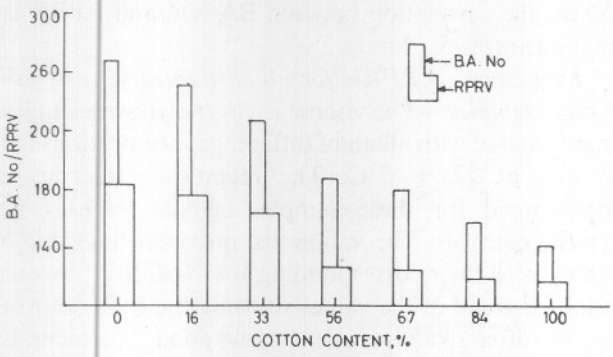


Fig. 3—BA No./RPRV versus cotton content in commercial cotton-polynosic blended fabric

Table 7—BA No. and RPRV of Cotton Blended with Viscose/Polynosic Treated with 52°TW NaOH for 5 min at 27±2°C against Cotton control

| Cotton | Viscose | | Polynosic | |
|---|---|---|---|---|
| % | BA No. | RPRV | BA No. | RPRV |
| 80 | 147.5 | 133.45 | 148.1 | 139.73 |
| 60 | 156.2 | 145.37 | 168.1 | 148.34 |

blends as well as polynosic-cotton blends were treated with 24% (wt/wt) (52° TW) alkali for 5 min at 27±2°C and were evaluated for their RPRV values against cotton control. The results are presented in Table 7. The results of the analysis of commercial cotton-polynosic blend fabric samples with increasing polynosic content are illustrated in Fig.3, which shows the influence of polynosic content on RPRV and BA No. of the alkali-treated blend sample. It is seen that as the polynosic content increases, the retention values also increase in line with BA Nos.

*Effect of sulphur dyes on the evaluation of retention values*—According to Edelstein[8] BA No. is not affected by the different classes of dyes. But as brought out in the earlier report[6] on assessment of mercerization of textile fabrics and yarns, among the different dyes investigated, sulphur dyes show an unusually high BA No. as compared to the undyed mercerized samples. When these samples were subjected to the evaluation of retention values, a similar trend as in the case of BA Nos was observed (Table 8). However, vat dyes were found not to interfere in the respective evaluations. It is also seen from Table 8 that the presence of sulphur dyes on the sample increases BA No/RPRV, even though no alkali treatment or mercerization has been effected on the sample. This may be mainly due to the alkaline reducing agent/dye entrapped in the sample during sulphur dyeing.

Table 8—Effect of Sulphur Dyes on BA No. and RPRV

| Material | Before dyeing | | After dyeing | | After stripping | |
|---|---|---|---|---|---|---|
| | BA No. | RPRV | BA No. | RPRV | BA No. | RPRV |
| Poplin | 164.9 | 129.18 | 182.6 | 142.41 | 168.9 | 137.51 |
| Voile | 141.2 | 118.68 | 169.6 | 133.88 | 140.5 | 123.07 |

Therefore, while evaluating the sulphur-dyed cotton textiles for the extent of mercerization it is recommended that the dye should be stripped off from the samples before determining either RPRV or BA Nos. While stripping off the dye from the sample, the control specimen is also to be given a similar treatment.

## References

1 Carl J Malu, Glegg R E & Margaret Luce, *Tappi*, **44** (1961) 102.
2 Kulshreshtha A K & Dweltz N E, *Proceedings, 15th joint technological conference of ATIRA, BTRA & SITRA* (BTRA, Bombay) 1974, 22.1.
3 Nelson M L, Rouselle M A, Congemi S J & Trouand P, *Text Res J*, **40** (1970) 872.
4 Ferns R & Pages P, *Cellul Chem Technol*, **11** (1977) 633.
5 Chandler J & Zeronian S H, *Text Chem and Color*, **11** (1979) 56-61.
6 *Survey on the assessment of the degree of mercerization of textile fabrics and yarn* (Textiles Committee, Bombay) 1978.
7 Andrews C M & Oberg A R, *Text Res J*, **33** (1963) 330.
8 Edelstein S M, *Am Dyestuff Reptr*, **25** (1936) 186.