

## Modal wet processing – A novel approach

Abhishek C Jadhav<sup>1</sup>, Navnath Pingale<sup>2</sup> & Sanjeev R Shukla<sup>1,a</sup>

<sup>1</sup> Institute of Chemical Technology, Matunga, Mumbai 400 019, India

<sup>2</sup> DyStar India Pvt. Ltd., Rabale, Mumbai 400 701, India

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In this study, various pretreatment parameters have been optimized for greige woven modal fabric in an industrial set-up of semi-continuous and continuous pretreatment range in order to overcome its processing issues without altering the inherent fabric softness. Various recipes of pad-batch desizing, pad-steam bleaching and cold causticization have been attempted and the results are compared with exhaust pretreatment. The pretreated fabric is then subjected to dyeing with a reactive dye. The performance of the processed fabric has been evaluated in terms of water absorbency, tensile strength, CIE whiteness index, and tegewa rating. Wash and crock fastness of the subsequently dyed fabric are also evaluated. The XRD spectrum indicates marginal increase in the crystallinity of modal fabric post causticization. The results are found encouraging in terms of good and uniform depth of colour with very little deterioration in the desired fabric properties.

**Keywords:** Absorbency, Cold causticization, Continuous pretreatment, Modal fabric, Reactive dyeing

### 1 Introduction

Modal<sup>®</sup> is a regenerated cellulosic fibre, first developed by the Austrian company, Lenzing AG. It is a cellulosic fibre refined through modified conditions of viscose rayon production by using reconstituted cellulose of beech tree as the cellulosic raw material. Lenzing claims to use Edelweiss<sup>®</sup> technology for modal production, i.e. "symbiotic" production process, wherein the raw material (pulp) is produced at the same site as the modal fibre itself. As per Lenzing 2014 annual report, a record volume of total 960,000 tons was produced, of which 68% stands for total textile fibres. Lenzing modal holds 16% of total fibre share in the market, which amounts to 105,000 tons<sup>1</sup>.

Modal is called a new wonder fibre. It combines benefits of natural cellulosic fibre and the incredibly soft feel of the modern micro fibres. It is about 50% more hygroscopic per unit volume than cotton. It can be dyed just like cotton and exhibits similar fastness even after repeated washing. With good softness and good drape, it is comfortable to wear. One more advantage of modal over cotton is its resistance to shrinkage, a notorious problem with cotton. It is less likely to fade or form pills as a result of friction. Its smoothness does not allow adherence of hard water deposits to the fabric surface. It also has high

wet modulus and high breaking strength, which allow greater molecular orientation during stretch coagulation of the fibres<sup>2</sup>.

Modal fibre has numerous applications like bed sheets, towels, robes, etc. In United States, a concept of Bed, Bath & Beyond for modal is quite popularized. In Europe, it is widely used in clothing as a substitute for cotton. Since modal blends well with cotton and other fibres, research activities are carried out mainly on blended textiles and the most prominent among them are discussed hereunder.

Eichinger and Leitner<sup>3</sup> observed that tencel and modal are the ideal partners for blending with cotton as the blended fabric exhibits soft appearance, lustre, moisture management and wear comfort. Both the blends can be processed easily in an environment-friendly manner. The antibacterial properties of modal fabric are not affected by dyeing, finishing or laundering<sup>4</sup>.

Bleaching of modal/AN-g-casein fibre blend using H<sub>2</sub>O<sub>2</sub>/TAED bleaching system was compared to conventional alkaline bleaching system in terms of whiteness and dyeability by Li *et al.*<sup>5</sup> wherein the former system was found to markedly decrease the loss of casein in AN-g-casein fibre due to its acidic conditions, and exhibited the advantages of high efficiency and energy saving.

Dyeing behaviour of cotton and modal fabrics with bifunctional reactive dyes was evaluated in terms of

<sup>a</sup>Corresponding author.  
E-mail: sanjeevshukla19@gmail.com

dye affinity, diffusion co-efficient, K/S values and fastness. It was found that modal have less affinity and diffusion than cotton, although rise in temperature increased the dye diffusion rate<sup>6</sup>.

Manda *et al.*<sup>7</sup> observed that the spun dyed Lenzing black modal fabric requires half the energy and water with 60% lower carbon footprint as compared to the conventionally dyed black modal fabric.

Tao<sup>8</sup> observed that the fine denier modal fibres and their blends are more likely to acquire hairiness, scratch marks and crease marks during dyeing and suggested remedies to avoid/ minimize these problems.

Thus, the literature survey indicates more emphasis on spinning/blending issues but none on continuous or semi continuous pretreatment of modal, which is a commercially viable approach for large scale production. Appropriate pretreatment recipes, essential for consistent and reproducible RFD (ready for dyeing) whiteness for pad steam bleaching along with appropriate process flow of pretreatment are not available. Even the exhaust processing of modal is associated with issues like rope marks, abrasion marks, streak marks, occasional uneven dyeing, etc. but overlooked due to ease of operation.

The current work aims at developing an industrially viable practical approach for continuous/ semi-continuous pretreatment of modal fabric by designing appropriate recipes to get better and consistent bleaching and quality dyeing. Greige modal fabric was pretreated carefully in an industrial set up of continuous pretreatment range in order to overcome the processing issues without altering the intrinsic fabric softness. Modal fabric was subjected to variously formulated recipes of pad-batch desizing, pad-steam bleaching and cold causticization followed by reactive dyeing. The effectiveness of process was evaluated by monitoring the fabric characteristics, such as water absorbency (wettability), tensile strength, whiteness index, tegewa, degree of polymerization and fluidity at bleaching stage and wash/crock fastness after subsequently dyeing the fabric. X-ray crystallinity of the fabric after causticization was also evaluated.

## 2 Materials and Methods

### 2.1 Materials

Plain weave modal fabric with 78 g/m<sup>2</sup> and construction 60's x 60's / 92 x 88 was procured from Lenzing AG, India. Hydrogen peroxide (50%) and

caustic lye (50%) were of commercial grade. Various commercial reagents and a reactive dye (Remazol Red RGB) were supplied by DyStar India Pvt. Ltd. The reagents used for the study were (i) Sera<sup>®</sup> Zon Neo (organic peroxide stabilizer based on sodium salt of polycarboxylic acid), (ii) Sera<sup>®</sup> Con M-TC (core alkali neutralizer), (iii) Sera<sup>®</sup> Quest C-PX (sequestering agent based on phosphonic acid derivative), (iv) Sera<sup>®</sup> Zyme C-GEP ( $\alpha$ -amylase base enzyme concentrate), and (v) Sera<sup>®</sup> Wash C-NEC (non-ionic wetting agent based on alkyl polyglycol ester).

### 2.2 Methods

Conventionally, the pretreatment of modal fabric is carried out by exhaust process on soft flow machine in a simple sequence of desizing followed by combined scouring and bleaching. No causticization treatment is given. The fabric is then subjected to dyeing.

#### 2.2.1 Pretreatment by Conventional Exhaust Method

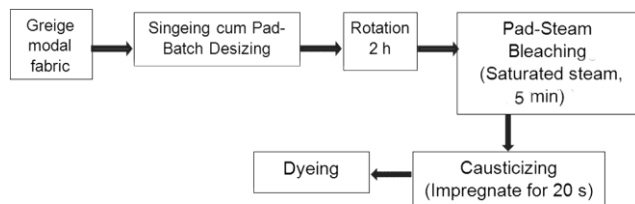
Greige modal fabric was desized in Sclavos HT soft flow dyeing machine, Athena-2, Greece, using bath containing 0.7 % (owf) Sera<sup>®</sup> Zyme C-GEP, 0.5 % Sera<sup>®</sup> quest C-PX and 0.5% Sera<sup>®</sup> Wash C-NEC at 70 °C for 45 min followed by hot wash at 90 °C. The pH of desizing was kept 6.5. The fabric was then bleached with 1.5 % of H<sub>2</sub>O<sub>2</sub>, 1.2 % of soda ash, 0.5 % of Sera<sup>®</sup> Quest C-PX, 0.15 % of Sera<sup>®</sup> Zon Neo and 0.7 % of Sera<sup>®</sup> Wash C-NEC at 85 °C for 1 h followed by hot wash at 90 °C for 10 min. The pH of bleaching was kept 9.5. The rate of heating for both the treatments was kept at 2 °C/min and liquor-to-material ratio was kept at 8:1. After the pretreatment, the fabric was subjected to dyeing without causticization as per the conventional practice.

#### 2.2.2 Pretreatment by Proposed Semi-Continuous and Continuous Scheme

In the proposed scheme, greige modal fabric was subjected to the following sequence of semi-continuous and continuous operations as shown in Scheme 1.

##### 2.2.2.1 Pad-Batch Desizing (PBD)

Greige modal fabric was padded with an aqueous solution containing Sera<sup>®</sup> Zyme C-GEP with concentrations ranging between 1 g/L and 3 g/L, Sera<sup>®</sup> Wash C-NEC (4 g/L) and Sera<sup>®</sup> Quest C-PX (3 g/L) with 80 % expression at 80 °C. The pH of desizing bath was maintained at 6.5. The padded fabric was batched for 2 h under continuous rotation at 22 rpm. The treated fabric was then washed twice with hot water at 90 °C and finally with cold water.



Scheme 1

The PBD was carried out on singeing-cum-desizing range of Osthoff-Senge GmbH & Co., Germany.

#### 2.2.2.2 Wet- on-Wet Pad-Steam Bleaching (PSB)

The fabric desized by semi continuous method was subjected to pad-steam bleaching (PSB) using an aqueous solution containing H<sub>2</sub>O<sub>2</sub> (4 - 6 mL/kg), caustic lye (2 - 4 mL/kg), Sera<sup>®</sup> Wash C-NEC (3 mL/kg), Sera<sup>®</sup> Quest C-PX (3 mL/kg) and Sera<sup>®</sup> Zon Neo (1/4<sup>th</sup> of peroxide concentration). The ratio of H<sub>2</sub>O<sub>2</sub> to caustic lye concentration was maintained at 2:1. The padding was carried out at 40 °C with 100 % expression. The speed of the machine was kept at 60 m/min. The padded fabric was steamed at 95 °C for 5 min, subsequently washed twice with boiling water and then with cold water. The PSB was carried out on continuous bleaching range of Karl Menzel Maschinenfabrik GmbH & Co, Germany. The fabric was batched on A-frame in wet condition situated at the end of the bleaching range.

#### 2.2.2.3 Causticization

The PSB fabric was causticized with caustic lye (50%) of 8 Be (55 g/L) at room temperature for 22 s, in the impregnation zone of the mercerizing machine and stretched on chain to 15 % of the initial width of fabric before washing. The subsequent washing water flow rate was kept at 4 L/kg so as to achieve weak lye concentrate in second last washer as 1 Be. Further, the neutralization was carried out using Sera<sup>®</sup> Con M-TC to maintain core pH of the fabric as 5.5-6. The causticization was carried out on the open width mercerizing range of Benninger AG, Switzerland. The drying was done gradually across the chambers up to 140 °C for 2 min in five successive chambers of the stenter machine of Harish Tech-Mach Pvt. Ltd., India.

#### 2.2.3 Reactive Dyeing

The modal fabrics pretreated by conventional exhaust method and the PSB followed by causticization was subjected to dyeing with a reactive dye (Remazol Red RGB). The samples were dyed at 4% shade using 70 g/L of Glauber's salt and 20 g/l of

soda ash in an Infra-red beaker dyeing machine of RBE Engineering Pvt. Ltd., India with liquor to material ratio 10:1. The rate of heating was maintained at 2 °C /min. All the fabric samples were subjected to various testing.

### 2.3 Testing and Analysis

#### 2.3.1 Fabric Wettability

The wettability of the fabric samples was measured using the drop penetration test (AATCC 39-1980). The time lapsed (in second) between the contact of a water drop with the fabric and the disappearance of the water drop into the fabric was counted as the wetting time. The time of drop disappearance was averaged out from measurements at different points of the fabric sample.

#### 2.3.2 Tensile Strength

The tensile strength and elongation were measured on Universal tensile tester (Tinius Olsen, H5K-S UTM, USA) using ASTM D 5035 (Strip method) at a gauge length of 5 cm and a strain rate of 1 cm/min.

#### 2.3.3 Whiteness Index

The degree of whiteness (CIE) of the modal fabric after pad-batch desizing and subsequent pad-steam bleaching was measured using DataColor 400 spectrophotometer USA.

#### 2.3.4 XRD study

Fabric sample was cut into powder form, placed in sample holder and wide-angle X-ray diffraction (WAXD) profile was obtained by Lab X, XRD-6100 Shimadzu diffractometer, Japan, and diffraction curves were recorded using Cu (K $\alpha$ ) radiation ( $\lambda=1.54\text{\AA}$ ) generated at 40 kV and 30 mA at a scanning rate of 2.0°/min.

#### 2.3.5 Wash and Crock Fastness

The wash fastness testing on dyed modal fabric was evaluated by ISO 105-C06 90°C washing test method. AATCC grey scale was used to evaluate the change in shade post washing under D65 light source.

The wet and dry rub fastness tests on dyed modal fabric were carried out using ISO 105 X12 test method. AATCC grey scale was used to evaluate the change in shade under D65 light source.

#### 2.3.6 Colorimetric Evaluation

DataColor 400 spectrophotometer, USA was used to evaluate dyed modal fabric in terms of K/S and colour parameters with the specular component of the light excluded and the UV component included using illuminant D65 and 10° standard observer.

### 3 Results and Discussion

#### 3.1 Exhaust Pretreatment

Table 1 shows that conventional exhaust pretreated (post bleached) modal fabric exhibits an average whiteness index  $75 \pm 2$ , absorbency 2-3 s, tegewa rating of 7 and strength loss of 19%.

#### 3.2 Pad Batch Desizing

At greige desizing stage, the concentration of enzyme plays very important role in degrading the size applied during weaving of fabric. The size on the greige modal fabric was identified to be a mixture of polyvinyl alcohol and starch by drop test method using a mixture of iodine and boric acid<sup>9</sup>. The enzyme degrades the starch size, while simultaneously removing PVA size by mechanical loosening from the fabric.

The concentration of Sera<sup>®</sup> Zyme C-GEP was varied from 1g/L to 3 g/L and the results on important parameters are given in Table 2.

It is apparent from the results that with the increase in enzyme concentration up to 2 g/L, the tegewa rating improves significantly, as more size on the fabric is degraded. The tensile strength is found to reduce insignificantly, which is also due to the removal of size from the yarns. The concentration of 2 g/L Sera<sup>®</sup> Zyme C-GEP is hence considered as optimum based on the absorbency (2-3 s) and lower loss of tensile strength. The whiteness index is also increased from 52.27 for greige to 65.8 due to removal of size.

Table 1— Properties of conventional exhaust pretreated modal fabric

Treatment	Tegewa	Whiteness index	Tensile strength, kgf	Absorbency s
Greige	1—2	52.27	24.22	>2 min
Desizing	4	62.19	23.85	<5
Bleaching	7	77.8	19.61	2-3

Table 2 — Optimization of enzyme conc. in pad-batch desizing and H<sub>2</sub>O<sub>2</sub> conc. in pad-steam bleaching of modal fabric

Chemical conc.	Tegewa	Whiteness index	Tensile strength, kgf	Absorbency s
Sera <sup>®</sup> Zyme C-GEP, g/L				
0	1-2	52.27	24.22	>2 min
1	4-5	63.09	23.85	<5
2	6-7	65.8	23.26	2-3
3	6-7	66.58	21.9	<2
H <sub>2</sub> O <sub>2</sub> , mL/kg				
4	8	73.92	22.36	<1
5	8	75.82	20.13	<1
6	8	76.05	19.36	<1

#### 3.3 Pad-Steam Bleaching (PSB)

Table 2 also gives results on optimization of H<sub>2</sub>O<sub>2</sub> concentration in continuous pad-steam bleaching (PSB) process to achieve whiteness on modal fabric. The H<sub>2</sub>O<sub>2</sub> concentration, when increased from 4 mL/kg to 6 mL/kg, gives the whiteness index 75.82 at 5 mL/kg H<sub>2</sub>O<sub>2</sub> due to increase in the perhydroxyl ion concentration. Since no significant enhancement is observed beyond 5 mL/kg of H<sub>2</sub>O<sub>2</sub>, it is considered as an optimum dosage. The tensile strength decreases with increasing H<sub>2</sub>O<sub>2</sub> concentration, whereas the absorbency and tegewa ratings remain constant at <1 s and 8 respectively.

Two intermediate hot washes are given to this fabric at 90 °C, batched on an A-frame and then subjected to subsequent causticization. The fabric in PSB should strictly pass through rollers and should even bypass drying range as it would cause shine marks.

#### 3.4 Causticization

Cellulosic fibre swells in caustic solution and as the NaOH concentration increases, the swelling also increases. In post mercerization washing range, the cellulosic fibre tends to contract. During washing, if the fabric is allowed to shrink without tension, running crease marks are formed in the warp direction of the fabric, which are visible only after dyeing. Hence, the fabric after stabilizing zone is allowed to stretch up to 15% of the initial width of the fabric. This tension does not allow fabric to shrink unevenly, exhibiting better dimensional stability.

From Table 3, it is evident that the CIE whiteness index reduces with the increase in the NaOH concentration and at 12 Be alkali concentration, it reduces to 71.11. The tensile strength improves and it is little higher than the greige at 8 Be caustic lye. From this point of view, 8 Be caustic lye is considered optimum as the absorbency improves to instant even at 6 Be.

#### 3.5 XRD Study

Wide angle X-ray diffraction profiles are obtained for the greige and causticized modal fabric samples to explicate the changes in the crystalline structure (Fig. 1). The pretreated (PSB) modal causticized with 8 Be caustic lye shows two distinct peaks at 21.44° and 19.72° corresponding to the crystalline plane spacing of 4.14Å, 4.41Å. The later peak is prominently associated with cellulose II polymorph, a characteristic of caustic lye treatment on cellulosic material. These peaks are associated with  $\alpha$  phase.

Table 3 — Optimization of caustic lye conc. in causticization of PSB modal fabric

Caustic lye (Be)	[Impregnation time 22 s, and temperature 35 °C]			
	Whiteness index	Tensile strength kgf	Absorbency s	Crystallinity index, %
0 (Greige)	52.27	24.22	>2 min	23.42
0 (Exhaust)	77.8	19.61	2-3 s	26.59
0 (PSB)	75.82	20.13	<1	23.42
8	74.1	25.27	Instant	27.7
10	73.29	28.9	Instant	29.64
12	71.11	32.22	Instant	29.12

Table 4— Effect of alkali lye conc. in causticization on colorimetric data of dyed modal fabric

Caustic lye (Be)	K/S	Colour values			
		L*	a*	b*	$\Delta E$
0 (Exhaust)	5.55	38.6	60.31	8.97	-
0 (PSB)	4.53	40.62	61.17	6.94	3.52
8	5.32	39.18	60.68	8.49	0.62
10	5.48	38.82	60.43	8.71	0.4
12	5.74	38.25	59.83	8.97	0.78

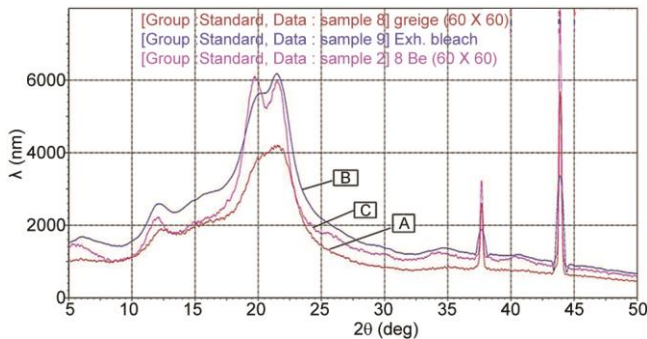


Fig. 1 — XRD spectra of modal [(A) greige, (B) exhaust pretreated, and (C) continuous pretreated followed by causticization (8Be)]

The XRD pattern of greige fabric shows a single broad peak at  $2\theta$  position of  $21.52^\circ$  with crystalline plane spacing at 4.12Å, whereas the exhaust pretreated modal shows slight appearance of a shoulder at  $19.72^\circ$ , not much different appearance from that of the greige modal. Thus, no major changes are observed in the prominent peaks of the greige and treated modal fabric samples, which confirm that the crystalline structure is only marginally affected by the causticizing treatment.

The crystallinity index shows little increase in the greige modal after performing the pretreatment. The values for the exhaust pretreated and for the proposed scheme after causticization with 8 Be caustic lye are found nearly similar.

### 3.6 Dyeing

Pretreated modal fabric is dyed with a reactive dye Remazol Red RGB at 4% shade. The  $K/S$  and colour

values of dyed modal after different treatments are given in Table 4.

It may be observed that the  $K/S$  values of the continuous pretreated modal matched with the exhaust pretreated only after causticization. No tonal variations are observed among the samples, irrespective of the pretreatment scheme or the level of causticization. This indicates that the role of causticization is mainly to maintain original strength and improve the absorbency of the fabric. Moreover, it is observed that with further increase in caustic lye concentration, the fabric feel becomes increasingly harsh, and therefore 8 Bé concentration is found to be the most appropriate in maintaining a balance between the feel and the dyeing properties of the fabric.

The wash fastness of the fabric pretreated by the exhaust and by the proposed continuous scheme and then dyed with reactive dye is found identical with rating 5. The dry rub fastness (5) is extremely good than the wet rubbing (3-4) for the dyed fabric and it is found same irrespective of the pretreatment procedure.

## 4 Conclusion

In textile wet processing, production process route which furnishes increased meterage is always followed without impeding optimum quality of the processed fabric. Thereby the continuous process route will always be preferred than the exhaust process, provided the associated quality issues are resolved. The drawbacks associated with the exhaust

pretreatment of the modal fabric can be successfully overcome by the proposed pad-batch desizing – pad-steam bleaching – causticization scheme. Although an additional step of causticization is necessary, it is justified from the quality dyeing produced and hand feel achieved at the end of the process. The fastness ratings of dyed modal are also at par for both routes. The utility savings in terms of time and water are found to be substantial. Thus, the continuous wet processing of modal through recommended route holds an edge over the exhaust.

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