Mechanical Properties Of Cellulase-Treated Fabrics Analyzed

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Abstract

Studies were intended to show the best parameters for controlling the finishing effects of cellulases as well as propose a new methodology to compare different finishing routes in terms of their mechanical parameters. Wet processing facilities employ this methodology to compare, optimize and implement processes, as well as to select the best parameters for measuring a particular finishing effect.

Introduction

The treatment of cotton with cellulases is a well-known way of gaining fiber surface modification[1, 2]. In fact, a controlled cellulase hydrolysis allows one to obtain clean fiber surfaces from microfibrils. This improves the appearance and the hand of used garments. If selected cellulases activities are used, microfibrils at the fiber surface can be raised and fabrics with a aged look can be obtained. These fiber surface modifications lead to dramatic changes in the hand and to the related low stress mechanical properties.

The low-stress mechanical properties can be measured using the Kawabata Evaluation System (KES), Fabric Assurance by Simple Testing (FAST) or simplified fabric properties[3,4,5]. The use of KES had been widely reported and generally accepted as standard methodology of measuring low stress properties of fabrics. However, simplified fabric properties can be used rather than the KES system if a more technical, rather than scientific, study is being made[5].

The study of the changes of these fabric properties caused by a finishing process is complicated due the large set of data obtained. The so called "snake-diagrams" used by KES and FAST systems poorly overcome these problems and they are just a one dimensional representation of the all parameters used. Recently Bishop and Cox[6] suggested a methodology using multivariate analysis maps. This technique allows the treatment and further analysis of large sets of data with minimal loss of information.

In this article a detailed description of the multivariate analysis maps techniques is used and some simplified properties to measure the fabric changes caused by cellulases are suggested. Using the described techniques, relations between cellulase activities, processing conditions and changes of fabric properties are shown.

Multivariate analysis

The description of a fabric implies the specification of their attributes. In this case, these attributes are the mechanical parameters and fabrics are different from each other because they have different numeric values relative to their attributes (or mechanical parameters). The original data base (Fig. 1) is a matrix of i fabrics with j mechanical parameters with xij values.

The matrix of the original data base can be analyzed by comparison for each parameter with the values for each fabric. Also, from this original matrix a new correlation coefficients matrix can be produced showing the inter-relations between the parameters studied. However, all of this work is tedious and difficult to demonstrate.

Using principal components analysis, linear combinations of all mechanical parameters are made with the maximal variance possible[7]. The first principal component accounts for the largest amount of variance of the data. The second component accounts for the second largest amount of variance, and it is uncorrelated with the first. Successive components are extracted until all of the variance is explained. For the n factor, Fni is the n factor score of the i fabric, x_{ij} is the value of the j parameter relative to the i fabric and a_{nj} is the n factor score coefficient for the j parameter.

$$F_{ni}=\sum_{j} a_{nj}\cdot x_{ij}$$

The estimated factor scores are the new data for the fabrics and the factor scores coefficients are the new data for the mechanical parameters. For a bidimensional map, the first two extracted factors are considered, the first one for the X axis, the second for the Y axis. The mechanical parameters can be represented as vectors where the coordinates are the respective factor scores coefficients. The size of these vectors are the associate variance (or communality) present in the first two factors extracted. The fabrics can be represented as points in the map where the coordinates are the factor scores.

The interdependence between mechanical parameters can be seen by the projection of one vector into the other. If the size of the vectors is I(the variance of the parameter is 100% and it is fully explained by the first two factors extracted) this projection is the correlation coefficient between the two parameters. The differences between fabrics relative to a certain parameter can be seen by the projection of the points in to the respective vector.

As a reduction of data technique it relies on authors' judgments as to how much variance can be discharged, when just the two first factors are considered for a vectors map. Usually, at least 60% of the total variance must be explained by the first two factors. Care must be taken when drawing conclusions from vectors that have low associate variance to their parameter.

The principal components analysis treatment of the original matrix of data can be made in any statistical package. In this article, the SPSS 6.0 for Windows was used.

Cellulase treatments

Cellulase enzymes used were supplied by Primalco Ltd, Finland. The code names of the enzyme mixtures are: TC -original crude mixture of *Trichoderma reesei*, C-EGs-crude mixtures where EG I and EG II activities were deleted, C-CBHs-crude mixtures where CBH I and CBH II activities were deleted. The fabric used was a 100 % cotton flannelette [21/15 (ends/picks) and 160 g/m^2] after being industrially scoured, bleached and raised. The fabrics were treated in a liquor made from dilution of the crude enzyme 1/50 in acetate buffer (pH=4,8). Different processing conditions were considered: L (low level of mechanical agitation) in a Linitest pot at 65rpm, 50°C and 1gr to 10ml liquor during 30 minutes. H (high level of mechanical agitation) similar to L but 15 steel discs used in various fastness tests. PB (pad-batch simulation) stand the fabric in a beaker 3 days at 20°C.

In a previous report[1], the activities of the cellulase enzymes used in this work were discussed. It confirmed the "endo" activity of C-CBHs and the "exo" activity of C-EGs when compared with TC. The activities of these cellulases were also characterized previously[1] during processing under the same conditions of mechanical agitation described herein.

Table I: Parameters of the cellulase treated fabrics.

Fabric	LT	WT	RT	EMT	G	2HG	2HG5	B	2HB	LC	WC
Untrealed	0.74	13.1	74	5.0	0.64	0.80	2.70	0.053	0.047	0.40	0.44
C-CBHs,LM	0.72	20.4	45	6.3	0.35	0.36	1.20	0.044	0.043	0.42	0.56
C-EGs,IM	0.78	18.1	59	6.7	0.32	0.40	1.30	0.041	0.038	0.42	0.52
C-CBHst,HM	0.63	18.0	57	7.8	0.32	0.30	1.10	0.045	0.050	0.40	0.50
C-EGs,HM	0.64	17.2	64	7.3	0.28	0.30	1.10	0.041	0.043	0.41	0.51
C-CBHs,PB	0.66	19.2	51	8.0	0.32	0.60	1.40	0.048	0.043	0.40	0.52
C-EGs,PB	0.57	18.7	53	8.6	0.34	0.30	1.00	0.038	0.040	0.41	0.54

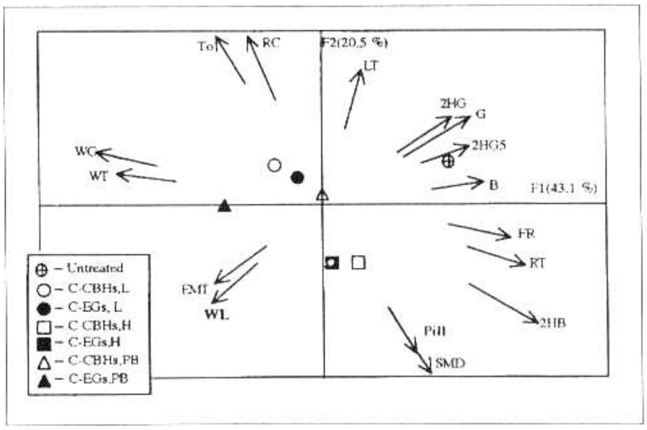
Fabric	RC	To	MIU	MMD	SMD	WL	Pill	BR	EXT	ELAS
Untreated	53	1.00	2.10	1.90	5.9	0.0	1.0	118.00	10.00	0.40
C-CBHs,LM	52	1.00	1.88	1.89	6.0	1.6	1.0	105.00	10.00	0.40
C-EGsLM	52	0.93	2.06	2.58	6.7	2.6	2.0	96.00	10.00	0.50
C-CBHs,HM	49	0.82	2.21	2.60	11.3	2.8	3.0	111.00	14.00	0.29
C-EGs,HM	51	0.72	4.02	1.66	18.3	4.0	5.0	102.00	11.00	0.45
C-CBHs,PB	51	1.00	2.01	1.43	6.0	1.1	1.0	117.00	14.00	0.36
C-EGs,PB	52	1.00	5.48	0.83	6.1	4.6	1.0	81.00	13.50	0.19

All parameters are defined by their abbreviation according to what is described in the text or to what is defined in KES system (3). Unit LT, LC, MIU, MMD, ELAS, Pill are dimension less; WL, RT, EMT, RC, EXT are %; SMD is micron; To is mm; WC is gf.cm/cm²; WT gf.cm/cm; G is gf/cm.degree; 2HG, 2HG5 are gf/cm; B is gf/cm²/cm, 2HB is gf.cm/cm; BR is mg/cm.

Figure 1–Matrix of the original data base.

Fabric/Parameter	1	2	...	j	...
1	X_{11}	X_{12}	...	X_{1j}	...
2	X_{21}	X_{22}	...	X_{2j}	...
...	...	...	...	...	...
i	X_{i1}	X_{i2}	...	X_{ij}	...
...	...	...	...	...	...

Figure 2–Vectors map relative to the study of all parameters.



Mechanical parameters

The matrix of the original data is represented in Table I. All mechanical parameters measured with a KES-FB (Kato, Co) system were considered[3]. Weight loss (WL) was also considered because it is a measure of enzymatic hydrolysis and it is also a function of fabric weight per area (W) and therefore WL was used instead of W.

The simplified mechanical parameters were considered using existing described standard methods: t Flexural Rigidity (FR) was measu according to BS 3356, Extensabi (EXT) was measured according to

4294, Elasticity (ELAS) was calculated by the formula ELAS=(1-RI/EXT) where R_1 is mean residual extension after 1 min defined in the BS 4294 and Pilling level (Pill) was determinated according to ASTM 3512 (level 5—no pills, level 1 —maximum degree of pills). Weight loss and thickness were also considered. Weight loss was considered for the reasons explained. The To (measured in KES-FB3) were also considered a simplified parameter, because the principle of measurement is equal to any other thickness machine. In the view of the author these parameters are the most important. Other parameters could be considered such as fabric drape (highly correlated with flexural rigidity), creasing capacity or dimensional stability, however from the what is known of the cellulase action in textile fibers it does not appear that these parameters will change significantly.

Table II: Matrix of the correlation coefficients.

	B	FR	ELAS	EMT	EXT	G	2HB	2HG	2HGS	L
B	1.000									
FR	0.901	1.000								
ELAS	0.193	0.336	1.000							
EMT	-0.630	-0.480	-0.610	1.000						
EXT	-0.128	-0.040	-0.751	0.834	1.000					
G	0.756	0.424	0.049	-0.758	-0.390	1.000				
2HB	0.618	0.678	-0.220	-0.205	0.257	0.364	1.000			
2HG	0.884	0.660	0.225	-0.621	-0.228	0.826	0.234	1.000		
2HG5	0.845	0.578	0.252	-0.795	-0.420	o.958	0.337	0.924	1.000	
LC	-0.616	-0.581	0.395	-0.107	-0.589	-0.368	-0.715	-0.484	-0.409	1.0
LT	0.409	0.368	0.816	-0.802	-0.785	0.389	-0.154	0.466	0.510	0.3
MIU	-0.661	-0.772	-0.553	0.560	0.295	-0.250	-0.338	-0.462	-0.391	0.0
MMD	0.214	0.380	0.542	-0.407	-0.318	0.016	0.322	-0.024	0.101	0.1
Pill	-0.326	-0.034	0.306	0.162	-0.028	-0.437	0.162	-0.488	-0.352	0.0
RC	0.160	-0.170	0.254	-0.573	-0.660	0.588	-0.496	0.510	0.556	0.3
RT	0.438	0.284	0.299	-0.537	-0.344	0.654	0.298	0.516	0.713	-0.4
SMD	-0.286	-0.006	0.217	0.183	0.021	-0.402	0.221	-0.460	-0.333	-0.0
To	0.302	0.001	-0.246	-0.189	-0.041	0.439	-0.251	0.507	0.366	0.0
WC	-0.717	-0.560	-0.171	0.547	0.157	-0.767	-0.546	-0.706	-0.833	0.6
WL	-0.923	-0.825	-0.356	0.709	0.310	-0.680	-0.367	-0.860	-0.781	0.3
WT	-0.588	-0.364	-0.184	0.613	0.323	-0.818	-0.386	-0.658	-0.844	0.4

	LT	MIU	MMD	Pill	RC	RT	SMD	To	WC	WL	W
LT	1.000										
MIU	-0.745	1.000									
MMD	0.615	-0.708	1.000								
Pill	-0.210	0.220	0.267	1.000							
RC	0.466	0.069	-0.340	-0.515	1.000						
RT	0.250	0.002	0.201	0.300	0.253	1.000					
SMD	-0.318	0.289	0.125	0.981	-0.498	0.282	1.000				
To	0.239	-0.184	-0.326	-0.992	0.604	-0.282	-0.969	1.000			
WC	-0.252	0.234	-0.256	-0.087	-0.134	-0.917	-0.083	0.103	1.000		
WL	-0.647	0.811	-0.281	0,.506	-0.321	-0.224	0.498	-0.497	0.490	1.000	
WT	-0.258	0.059	-0.133	-0.085	-0.342	0.966	0.089	0.076	0.956	0.381	1.0

Correlation matrix

A matrix of the correlation coefficients between all pararneters is shown in Table II, from the data of Table I. The analyses of this table allow us to know the parameters that are correlated between each other and further verify the information from the vectors maps. In Table II, the values that appear in bold are higher than 0.801, i.e., for seven observations which corresponds to a verified correlation between two parameters at a level of 95%.

It is shown (Table II) that simplified and KES parameters are correlated:

- Flexural (FR) and bending (B) rigidities
- Both extensabilities: EMT and EXT
- Linearity of the curve tension-extensability (LT) with elasticity (ELAS)
- Thickness(To), fabric surface roughness (SMD) and pilling level (Pill) are highly intercorrelated.

Vectors maps

In our study, three treatments were done from the original matrix of data: the extraction of the two first factors from all data and the extraction considering only the KES pararneters or just the simplified parameters. Parameters that have less than 50% of variance associate (communality) for the first two factors extracted were not represented in the vector maps.

In Tables III, IV and V are displayed the variance associate (communality) to each parameter. In Figures 2, 3, 4 the vectors map are shown for the three treatments performed. The total variance for the two first extracted factors increases with a decrease of the size of the data set included. The analysis of the vector maps can be accomplished by observation of the relative positions of the vectors (parameters) and after by the observation of the relative positions of the points (fabrics) relative to each other and relative to the vectors.

The vectors map of Figure 2 is also relative to all data of Table I a accounts with a total variance of 6 %. Parameters can be joint in gro that are positively inter-related:

Group 1: WL, EMT
Group 2: WC, WT
Group 3: To, RC
Group 4: G, 2HG, 2HG5, B
Group 5: FR, RT, 2HB
Group 6: Pill, SMD

The following groups are negativ

intra-related: Groups 1 & 4, Groups 2 & 5, and Groups 3 & 6. The relation between KES parameters and simplified parameters can only be seen for B, FR and Pill, To, SMD. The communality of EXT and ELAS were low and therefore can't be seen in Figure 2. This fact indicates that care must be taken when imformation is developed after a study with the vectors maps is made.

The vectors map of Figure 2 also shows that the cellulase treatment causes an increase of parameters in group 1, a decrease of parameters in group 4. In fact, the weight loss (WL) caused by cellulase treatment increases fabric extensability (EMT) and (decreases bending rigidity (B) and shear parameters (G, 2HG, 2HG5).

The positions of points in the vector map of Figure 2 indicates that the treated fabrics relative to the untreated one have a higher weight loss and variation of the related properties. The different conditions of processing make a dispersion of the points along the parameters of groups 2 and 5 and groups 3 and 6. This means that the degree of mechanical agitation present during a cellulase treatment will change properties like the final pilling level (Pill), fabric roughness (SMD), thickness (To) bending hysterisis (2HB). It is clear that a cellulase treatment with a high mechanical agitation will remove the pills (increase of pilling level) decreasing the thickness (To). The removal of pills will show the woven fabric structure increasing the surface roughness (increase of SMD). A decrease of 2HB and FR is verified for the process with lower mechanical agitation, while for the process with higher mechanical agitation an increase of these parameters, is verified.

It appears that C-EGs produces a higher fabric weight loss (WL) from their position in the vector maps, as verified in Table II. This enzyme also has a better dipilling action than C-CBHs. Previously it was verified that C-EGs[1], despite the deletion of EG I and EG II activities have a high hydrolytic action due to the synergism of CBHs with the remaining EG III and EG V[1]. C-CBHs have mainly an "endo" and with high levels of mechanical agitation seems to increase bending hysterisis (2HB). This could be due to raising of fiber material on the fiber surfaces as verified before[1,2].

Table III: Communality for the first two factors extracted (Total Variance=63.6 % relative to the study of all parameters.

B	84	2HG	84	RC	67
FR	65	2HG5	93	RT	62
ELAS	16	LC	49	SMD	73
EMT	75	LT	55	To	78
EXT	32	MIU	39	WC	86
G	78	MMD	18	WT	70
2HB	61	Pill	70	WL	82

Table IV: Communality for the first two factors extracted (Total Variance=68.4 relative to the study of KES parameters.

B	83	LC	56	SMD	71
EMT	70	LT	54	To	74
G	84	MIU	34	WC	93
2HB	60	MMD	10	WT	80
2HG	86	RC	61	WL	79
2HG5	96	RT	70		

Table V: Communality for the first two factors extracted (Total Variance=78.4 %) r ative to the study of the simplified parameters.

FR	53	EXT	53	TO	92
ELAS	85	PILL	95	WL	93

The vectors map with just the KES parameters confirm the observations verified for the map with all parameters. The vector and fabric positions are similar as well as the information contained.

The vector map for the simplified parameters accounts with 78.4 of the total variance (Fig. 4). The verified negative inter relations in Table II can be seen in the vector map of Figure 4:

- Flexural Rigidity and Weight Loss (FR & WL)
- Thickness and Pilling Level (To & Pill)
- Extensability and Elasticity (EXT & ELAS)

All cellulase processes leads to some weight-loss and a decrease of FR. The process with high mechanical agitation decreases To and removes Pills, while the process with no mechanical agitation (PB) increases extensability (EXT) decreasing (ELAS). It see that the woven flannelettes used lo their elastic behavior when they a submitted to higher tensile deform tions.

The enzyme C-EGs seems emphasize all of these changes verif in Figure 4, relative to C-CBHs.

Conclusions

The changes caused by cellul treatment on fabrics can be sho treating the original data of the mech ical parameters with principal com nents analyses. The new data can represented in a vectors map. The u of simplified parameters like flexu rigidity, thickness, extensability, elast ty, weight loss and pilling level can

Figure 3- Vectors map for the study with KES parameters.

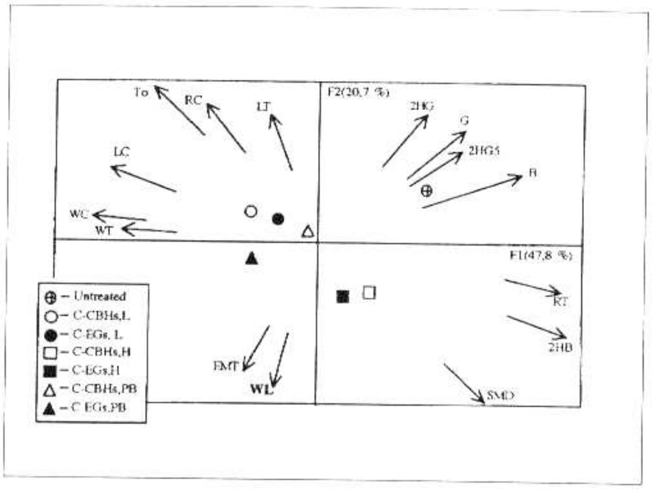
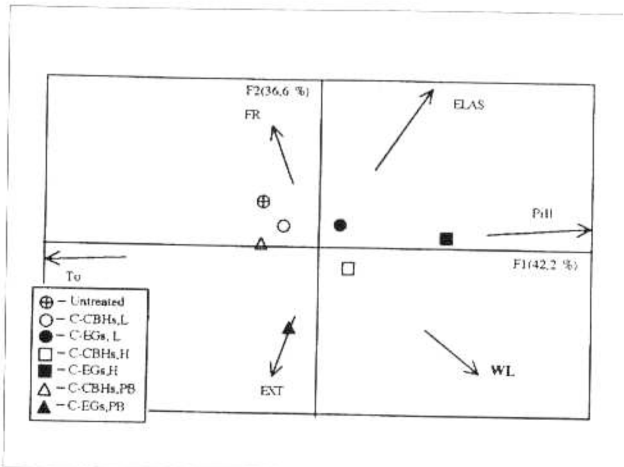


Figure 4- Vectors map for the study with simplified param
ters.



ferentiate the changes caused by "endo" or "exo" rich cellulase activities and different levels of mechanical agitation during processing of flannelette fabrics. □ □ □

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