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Performance of Durable Press Finish on Cotton with Modified DMDHEU, Citric Acid, BTCA and Maleic Acid

Učinkovitost plemenitenja z modificirano DMDHEU, citronsko kislino, BTCA in maleinsko kislino za trajno oblikovanje bombaža

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Abstract
Cotton fabrics possess the inherent property to form wrinkles under stress. To overcome this problem, conventionally, selective cross-linking agents are applied via the pad-dry-cure method at a high curing temperature. Research carried out in this field has identified invariable deterioration in the mechanical properties of finished cotton fabric. In this study, four different cross-linking agents, i.e. modified dimethylol dihydroxy ethylene urea (DMDHEU), citric acid, 1,2,3,4 butanetetracarboxylic acid (BTCA) and maleic acid, were applied on cotton fabric through selection of combinations, using the Box-Behnken experimental design. It was established that DMDHEU shows the best improvement in the crease recovery angle along with the highest durable press (DP) rating with a poor retention of strength. The citric acid shows the average strength retention as well as an acceptable improvement in crease recovery. BTCA shows the best strength retention, but the poorest crease recovery. The maleic acid also shows an average strength retention with a crease recovery superior to that with BTCA.
Keywords: cotton, modified DMDHEU, citric acid, BTCA, maleic acid, DP rating

Izvleček
Bombažno blago se pri obremenitvi mečka. Za zmanjšanje mečkavosti se uporabljajo zamreževala, ki se na blago nanašajo po impregnirnem postopku s suhim zamreženjem pri visoki temperaturi. Raziskave, izvedene na tem področju, so pokazale, da se mehanske lastnosti tako oplemenitenega bombažnega blaga neizogibno poslabšajo. V pričujoči raziskavi so bila uporabljena štiri zamreževala in sicer modificirana dimetilol dihidroksietilen sečnina (DMDHEU), citronska kislina, 1,2,3,4 butan tetkarboksilna kislina (BTCA) in maleinska kislina, ki so bila v različnih kombinacijah, določenih s pomočjo eksperimentalnega modela Box-Behnken nanesena na bombažno tkanino. Ugotovljeno je bilo, da DMDHEU zagotovi najboljše izravnalne kote po gubanju z najvišjo oceno trajnega oblikovanja (DP), a z zelo nizko ohranitvijo trdnosti. Nanos citronske kisline povzroči povprečno ohranitev trdnosti in sprejemljivo izboljšanje izravnave gub, medtem ko prisotnost BTCA zagotovi največjo ohranitev trdnosti, toda omogoča najslabše izravnavanje gub. Tudi z maleinsko kislino je bilo doseženo le povprečno zadrževanje trdnosti, a z veliko boljšo izravnavo gub kot v primeru BTCA.
Ključne besede: modificirana DMDHEU, citronska kislina, BTCA, maleinska kislina, ocena DP

1 Introduction

Cotton is one of the most preferred textile materials due to its superior wearing comfort and excellent wearability. Unfortunately, cotton fabrics wrinkle easily during home laundering and cause considerable inconvenience for the users [1, 2]. It is believed that for a wearer, one of the most influencing factors relating to the fabric quality is the ability of the fabric to recover from induced wrinkles. Wrinkles are categorized

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into desirable wrinkles, i.e. for the smartness of a fabric, and undesirable wrinkles, which need to be prevented [3]. The wrinkling behaviour of cellulosic fabrics may be directly linked to the mobility of free hydroxyl groups in the polymer chain, and the frequent breaking and reforming of hydrogen bonds [4, 5]. As the hydroxyl groups in the amorphous region are far apart and since hydrogen bonds operate at short distances, these hydroxyl groups remain unbound. When a cloth is folded and pressed, some of the hydrogen bonds at the boundary of crystalline and amorphous regions break (hydrogen bonding forces are fairly weak). Simultaneously, free hydroxyl groups in the amorphous region approach other free hydroxyl groups and when they are sufficiently close to each other, they bind. These newly formed hydrogen bonds bind the molecules and prevent unfolding; thus, forming crease marks [6]. The creasing behaviour of a cotton fabric can be reduced by reducing or totally masking the hydrogen bond formation capacity of hydroxyl groups. This is generally done by imparting the crease-resist finish in which the hydroxyl groups of adjacent macromolecules react with multi-functional cross-linking agents as shown in Figure 1 [7, 8].

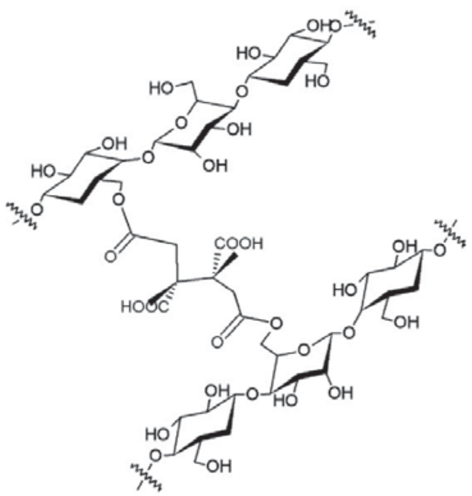

Figure 1: Cross-linking of cellulose using crease resist finish [8]

The mechanical and certain other properties of cellulosic fibres are affected by cross-linking, which is reflected in the performance of fabrics. The stability of fibres in any assembly is a consequence of the combination of internal hydrogen bonding and Van der Waals forces [9]. The introduction of covalent cross-links into cellulosic fibres has two important effects, i.e. i) it reduces the mobility of chain molecules to move laterally [10], and ii) it extends longitudinally under stress [10, 11]. Studies have shown that the unavoidable side effect of cross-linking finishes is a reduction in elasticity and flexibility of cellulosic fibres, next to a considerable decrease in abrasion resistance, tear and tensile strength [12]. A rule of thumb states that an increase in the wrinkle recovery angle of 10° corresponds to the loss in abrasion and tear strength of about 7% [13]. This can be illustrated by considering an imaginary fibre with no lateral forces between chains. It would be mechanically weak, since when under stress, chain molecules would slide past one another. The introduction of cross-links would increase the load required to break the fibre due to the increased inter-chain cohesion. However, after a certain limit, the introduction of more cross-links would restrict the movement of chains to such an extent that the proportion of chains capable of resisting the applied stress would decrease and the tensile strength would fall.

Cotton develops a number of H-bonding during use, which in turn increases its strength. The cross-linking of anti-crease reagents with cellulose replaces a number of H-bonds and imparts stiffness, hence reducing the fibre chain movement on loading [9, 14, 15].

The application of statistical tools has been of paramount importance for a thorough study of the process with a minimum bias. These tools help evaluate a process more comprehensibly with a remarkably smaller number of samples. The Box-Behnken design is one of such statistical tools frequently used.

The Box-Behnken designs usually have fewer design points than the central composite designs and are thus less expensive to run with the same number of factors. For six factors, Box-Behnken requires 54 observations and this is the minimum of the central composite design. In contrast, the central composite designs can fit a full quadratic model. They are often used when the design plan calls for sequential experimentation, as these designs can include information from a correctly planned factorial experiment. Unlike the central composite designs, the Box-Behnken designs never include runs where all factors are at their extreme setting, such as all of the low settings. Therefore, if the experimental region is such that extreme points are a problem, then there are some advantages to Box-Behnken. Otherwise, they both work well.

The Box-Behnken designs can efficiently estimate the first- and second-order coefficients; however, they cannot include the runs from a factorial experiment. The Box-Behnken designs always have 3 levels per factor, unlike the central composite designs, which can have up to 5.

In this study, a pre-treated cotton fabric was finished with four different crease-resist finishes, i.e. modified DMDHEU, citric acid, BTCA and maleic acid, via the Box-Behnken design. These four reagents were selected for the study mainly due to the extensive research on them, showing their promising capability to impart an anti-crease finish on cotton compared to that with other chemicals. DMDHEU being carcinogenic must be eloped from the industries with suitable alternate reagents. A durable press rating as well as the mechanical properties of finished cotton was compared to assess the performance of each finishing process.

2 Experimental

Materials

A plain woven cotton fabric of 1.26 tex warp and weft with 60 ends/cm, 28.35 picks/cm and weight of 128 g/m^2 was used in this study. Modified dimethylol dihydroxy ethylene urea (DMDHE, Resil, India), citric acid (SDFCL, Mumbai), 1,2,3,4 butanetetracarboxylic acid (BTCA, Sigma Aldrich), maleic acid (SDFCL, Mumbai), sodium hypophosphite (NaH_2PO_2, SHP, Thomas Baker, India), magnesium chloride hexahydrate ($MgCl_2.6H_2O$, SDFCL, India), trisodium citrate (TSC, Himedia, India), polyethylene emulsion and silicone softener were procured (Resil, India). The weight of catalysts, i.e. $MgCl_2$, sodium hypophosphite and trisodium citrate, was calculated with respect to the weight of modified DMDHEU, citric acid, BTCA and maleic acid, respectively.

Finishing of cotton fabric

The anti-crease finish liquor, prepared with desired factors (parameters) and levels using the Box-Behnken experimental design as mentioned in Tables 1–4, was imparted to cotton on a laboratory padder with 70–80% expression (owf) with modified DMDHEU, citric acid, BTCA and maleic acid systems. All finished fabrics were dried at 80 °C and cured under stretched conditions at varying curing temperatures and times according to the design. Magnesium chloride, trisodium citrate and sodium hypophosphite were used as catalysts for respective finishes as detailed in Tables 1–4. The finished samples were passed through five cyclic washing and drying processes before the testing for the DP rating.

Evaluation

The finished cotton was evaluated for its DP (durable press)/smoothness appearance rating, using the AATCC DP replica (AATCC test 124:2006). In the test method, a platform with five DP finished cotton replicas are available side by side with five different types of durable press ratings from 1–5, 1 being the poorest and 5 the best DP performance with no crease marks. The finished samples were compared visually with these standards to assess the grade. The finished cotton with the best DP was further evaluated for the total crease recovery angle (TCRA, AATCC Test 66-2003), tensile strength (ASTM D5035) and tear strength (ASTM D 2261).

Experimental design

The Box-Behnken factorial design with six factors and three levels (3^6) was selected for this study. The six factors were the concentrations of reagent, catalyst, silicone softener, polyethylene emulsion along with the time and temperature of curing. The three levels were chosen based on the industrial application parameters with the central point at 0 and equidistant two levels on each side of 0. The Box-Behnken factorial 3^6 research statistical design resulting in 54 runs with six replicates at the central point was used to evaluate the functional characteristics of anti-crease finishes. Various parameters were included in the research design and their actual levels as mentioned in Tables 1–4.

Table 1: Process parameters with respective levels for DMDHEU finish

Parameter	Different levels		
	–1	0	+1
Modified DMDHEU [g/l]	50	60	70
$MgCl_2.6H_2O$ [% of DMDHEU weight]	15	20	25
Silicone softener [g/l]	10	15	20
Polyethylene emulsion [g/l]	15	20	25
Curing temperature [°C]	150	160	170
Curing time [min]	3	4	5

Table 2: Process parameters with respective levels for citric acid finish

Parameter	Different levels		
	–1	0	+1
Citric acid [g/l]	50	60	70
Trisodium citrate [% of citric acid weight]	50	60	70
Silicone softener [g/l]	0	10	20
Polyethylene emulsion [g/l]	0	5	10
Curing temperature [°C]	150	160	170
Curing time [min]	3	4	5

Table 3: Process parameters with respective levels for BTCA finish

Parameter	Different levels		
	–1	0	+1
BTCA [g/l]	50	60	70
Trisodium citrate [% of BTCA weight]	40	50	60
Silicone softener [g/l]	0	10	20
Polyethylene emulsion [g/l]	0	5	10
Curing temperature [°C]	160	170	180
Curing time [min]	2	3	4

Table 4: Process parameters with respective levels for maleic acid finish

Parameter	Different levels		
	–1	0	+1
Maleic acid [g/l]	50	60	70
NaH_2PO_2 [% of maleic acid weight]	40	50	60
Silicone softener [g/l]	0	10	20
Polyethylene emulsion [g/l]	0	5	10
Curing temperature [°C]	160	170	180
Curing time [min]	2	3	4

3 Results and discussion

3.1 Durable press ratings of finished cotton

Cotton finished with a set of parameters according to the respective experimental design was evaluated for its DP rating. The DP rating of the unfinished fabric was found to be as poor as 1.5. Fabrics finished with anti-crease finishes showed an improvement in the DP rating. However, some sets of parameters lagged in either optimum temperature, or the concentration of catalyst or reactant, and could not produce the desired DP ratings. For further discussions, all the sets of runs with DP rating ≥ 3.5 were selected. Tables 5–8 highlight the set of parameters showing DP ratings equal to 3.5 or higher.

Table 5: Selective sets of parameters showing DP rating at 3.5 or higher in modified DMDHEU finish

Sr. no.	DMDHEU [g/l]	$MgCl_2.6H_2O$ [% wt of DMDHEU]	Silicone softener [g/l]	Polyethylene emulsion [g/l]	Curing temperature [°C]	Curing time [min]	DP rating
1	50	20	15	25	150	4	3.5
2	50	25	15	15	160	5	3.5
3	50	20	10	20	160	5	3.8
4	50	15	15	15	160	4	4.5
5	50	15	15	25	160	4	4.2
6	50	25	15	25	160	4	3.7
7	50	20	15	15	170	4	3.75
8	60	25	15	20	150	5	4
9	60	15	15	20	150	3	3.8
10	60	15	20	20	150	4	3.7
11	60	20	10	15	160	5	3.8
12	60	20	15	20	160	4	3.7

13	60	20	20	25	160	5	3.5
14	60	20	15	20	160	4	4
15	60	20	10	25	160	3	3.6
16	60	20	20	15	160	3	4
17	60	20	15	20	160	4	3.5
18	60	15	15	20	170	5	3.48
19	60	25	15	20	170	3	3.6
20	60	15	20	20	170	4	3.2
21	60	25	10	20	170	4	3.6
22	70	20	15	15	150	4	3.6
23	70	20	15	25	150	4	4
24	70	20	20	20	160	5	3.8
25	70	25	15	15	160	4	3.8
26	70	15	15	25	160	4	3.75
27	70	15	15	15	160	4	3.7
28	70	25	15	25	160	4	3.6
29	70	20	15	15	170	4	3.8

Table 6: Selective sets of parameters showing DP rating at 3.5 or higher in citric acid finish

Sr. no.	Citric acid [g/l]	Trisodium citrate [% wt of citric acid]	Silicone softener [g/l]	Polyethylene emulsion [g/l]	Curing temperature [°C]	Curing time [min]	DP rating
1	50	60	10	25	150	4	4
2	50	60	0	20	160	5	3.5
3	50	50	10	25	160	4	4.25
4	50	60	10	25	170	4	3.5
5	60	50	10	20	150	3	3.5
6	60	60	0	15	160	5	3.5
7	60	60	10	20	160	4	3.5
8	60	50	20	20	170	4	3.8
9	60	60	10	25	170	4	3.5
10	60	70	10	20	170	5	3.8
11	60	50	20	20	170	4	3.8
12	70	70	10	15	160	4	3.5

Table 7: Selective sets of parameters showing DP rating at 3.5 or higher in BTCA finish

Sr. no.	BTCA [g/l]	Trisodium citrate [% wt of citric acid]	Silicone softener [g/l]	Polyethylene emulsion [g/l]	Curing temperature [°C]	Curing time [min]	DP rating
1	50	50	0	5	170	2	3.5
2	50	50	10	10	180	3	3.6
3	60	40	0	5	160	3	3.5
4	60	60	10	5	160	4	3.5
5	60	60	0	5	160	3	3.6
6	60	50	10	5	170	3	3.7
7	60	50	20	10	170	4	4
8	60	50	20	10	170	2	3.5
9	60	50	10	5	170	3	3.6
10	70	50	20	5	170	4	3.5
11	70	60	10	0	170	3	3.8
12	70	50	10	10	180	3	4

Table 8: Selective sets of parameters showing DP rating at 3.5 or higher in maleic acid finish

Sr. no.	Maleic acid [g/l]	Sodium hypophosphite [% wt of maleic acid]	Silicone softener [g/l]	Polyethylene emulsion [g/l]	Curing temperature [°C]	Curing time [min]	DP rating
1	50	50	0	5	170	2	4
2	50	50	20	5	170	4	3.8
3	50	50	20	5	170	2	3.8
4	50	50	10	10	180	3	3.5
5	60	40	20	5	160	3	3.5
6	60	50	0	10	170	4	3.5
7	60	50	20	0	170	4	3.7
8	60	50	10	5	170	3	3.5
9	60	50	10	5	170	3	3.5
10	60	40	20	5	180	3	3.75
11	70	50	0	5	170	2	3.6
12	70	50	20	5	170	3	3.5

The samples with a higher concentration of the finish and higher temperature requirements are not suitable from the industrial viewpoint and cost. Moreover, a higher temperature and a higher concentration of the finish may lead to a greater degradation of cellulose. Since the runs with lower energy and chemical requirement also show good DP ratings, the samples prepared with more energy and chemical requirement were eliminated for further study. The combinations requiring less energy and a lower concentration of the finish were hence evaluated for a further test to evaluate the total crease recovery angle, tensile strength and tear strength. The test values for the unfinished cotton (control) were thus: tensile strength 580 N, tear strength 10.4 N, total crease recovery angle 132° and DP rating 1.5 (Tables 9–12).

Table 9: Physical properties of cotton finished with DMDHEU

Sr. no.	DMDHEU [g/l]	$MgCl_2.6H_2O$ [% wt of DMDHEU]	Silicone softener [g/l]	Polyethylene emulsion [g/l]	Curing temperature [°C]	Curing time [min]	Tensile strength [N]	Tear strength [N]	Crease recovery angle [°]			DP rating
									Warp wise	Weft wise	Total	
1	50	20	10	20	160	5	427.3	6.43	105	101	206	3.8
2	50	15	15	20	160	4	474.13	8.08	100	95	195	4.5
3	50	15	15	15	160	4	413.9	7.67	95	90	185	4.2
4	50	25	15	25	160	4	358.56	6.06	118	113	231	3.7
5	50	20	15	15	170	4	370.8	4.81	115	110	225	3.75
6	60	25	15	20	150	5	414.23	6.21	125	123	248	4
7	60	20	15	20	160	4	403.48	5.68	107	103	210	4
8	60	20	20	15	160	3	423.94	5.62	105	100	205	4

Table 10: Physical properties of cotton finished with citric acid

Sr. no.	Citric acid [g/l]	TSC [% of citric acid]	Silicone softener [g/l]	Polyethylene emulsion [g/l]	Curing temperature [°C]	Curing time [min]	Tensile strength [N]	Tear strength [N]	Crease recovery angle [°]			DP rating
									Warp wise	Weft wise	Total	
1	50	60	10	25	150	4	535.18	6.5	96	94	190	4
2	50	60	0	20	160	5	494.44	7.5	104	100	204	3.5
3	50	50	10	25	160	4	502.33	7	105	101	206	4.25
4	50	60	10	25	170	5	487.65	7.8	110	106	216	3.5

Table 11: Physical properties of cotton finished with BTCA

Sr. no.	BTCA [g/l]	TSC [% of BTCA]	Silicone softener [g/l]	Polyethylene emulsion [g/l]	Curing temperature [°C]	Curing time [min]	Tensile strength [N]	Tear strength [N]	Crease recovery angle [°]			DP rating
									Warp wise	Weft wise	Total	
1	50	50	0	5	170	2	556.5	8.88	95	90	185	3.5
2	50	50	10	10	180	3	424.9	6.26	101	96	197	3.6
3	60	50	20	10	170	4	457.1	5.67	98	92	190	4
4	60	60	10	0	170	3	477.4	5.89	95	91	186	3.8
5	70	50	10	10	180	3	405.59	5.46	102	93	205	4

Table 12: Physical properties of cotton finished with maleic acid

Sr. no.	Maleic acid [g/l]	SHP [% of maleic acid]	Silicone softener [g/l]	Polyethylene emulsion [g/l]	Curing temperature [°C]	Curing time [min]	Tensile strength [N]	Tear strength [N]	Crease recovery angle [°]			DP rating
									Warp wise	Weft wise	Total	
1	50	50	0	5	170	2	455.3	6.64	83	80	163	4
2	50	50	20	5	170	4	437.2	5.67	108	101	209	3.8
3	50	50	20	5	170	2	459.3	6.19	97	92	189	3.8
4	50	50	10	10	180	3	410.81	5.42	105	105	210	3.5

The concentration of the anti-crease finish, catalyst, curing temperature and time are important parameters for crosslinking with cellulose to obtain better fabric properties. The combinations (runs) showing the desired durable rating (≥ 3.5) with their corresponding physical properties for all finishes are summarized in Tables 9–12. The increase in intermolecular crosslinking with cellulose resulted in the restriction of the cellulose chain movement and in the loss of mobility of cellulosic macromolecular network, thus reducing the equalized stress distribution, ultimately leading to the loss in mechanical strength. The addition of polyethylene emulsion and silicon softener provided lubrication and yarn slippage and helped retaining strength.

The results show that high curing temperatures of 170°C or 180°C had an adverse effect on tensile strength as compared to lower curing temperatures. The loss in tensile strength also depends on the type of finish. DMDHEU showed the maximum loss in tensile strength, while the citric acid finished samples retained 80–90% of tensile strength. The tensile strength retention of BTCA and maleic acid lies in between the citric acid and DMDHEU. The combination of BTCA showed around 95% of tensile strength retention but a substantially smaller improvement in the total crease recovery angle (TCRA), which is the sum of the crease recovery angle in warp and weft directions. The TCRA of finished fabrics was notably higher than that of the unfinished fabric, showing substantial crosslinking. The increase in either curing time or resin concentration increased TCRA of most samples, while the catalyst at a higher concentration decreased the tensile and tear strength retention of the finished fabric.

As crosslinking diminishes fibre extensibility and restricts chain slippage, it tends to produce decreased tear strength of the finished fabric. A higher curing temperature and/or a high catalyst concentration caused a fall in tear strength. The citric acid finish showed the highest tear strength retention of about 85‑90% in most samples, while other finishes showed 50–65% retention in most samples.

Table 13: Best selective combinations among all finishes

Finish type	Finish [g/l]	Catalyst [%]	Silicone softener [g/l]	Polyethylene emulsion [g/l]	Curing temperature [°C]	Curing time [min]	Tensile strength [N]	Tear strength [N]	Crease recovery angle			DP rating
									Warp	Weft	Total	
DMDHEU	60	25	15	20	150	5	414.23	6.21	125	123	248	4
Citric acid	50	60	10	25	170	5	487.65	7.8	110	106	216	3.5
BTCA	60	50	20	10	170	4	457.1	5.67	98	92	190	4
Maleic acid	50	50	20	5	170	4	437.2	5.67	108	101	209	3.8

This selection was based mainly on the improvement in the crease recovery angle. Further, it was considered that the selected combination should not show a severe loss in the strength. Consequently, a compromise for crease recovery was conducted in the case of some finishes to ensure acceptable physical properties.

4 Conclusion

The final selection shows that DMDHEU showed the best improvement in the crease recovery angle along with the highest DP rating, whereas the strength retention was poor in the case of DMDHEU. BTCA showed the best strength retention but the poorest crease recovery. The maleic acid also showed average strength retention with crease recovery superior to BTCA. The citric acid showed the average strength retention as well as an acceptable improvement in crease recovery. To conclude, the best combinations of parameters for different alternative finishes have been suggested in this work and the physical characteristics of finished cotton presented.

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