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Comparative Enactment of Formaldehyde-free and Formaldehyde-based Cross-linkers on Cotton Woven Fabrics

Primerjava učinkovitosti formaldehidnih in neformaldehidnih zamreževal na bombažnih tkaninah

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Abstract
The performances of formaldehyde-based and non-formaldehyde cross-linkers on pretreated cotton woven fabric were assessed and compared in this research. Fixapret CL was considered as the formaldehyde-based resin and Fixapret NF as the formaldehyde-free resin. Dry cross-linking method was adopted for the application of cross-linkers. Different properties of resin treated fabrics investigated and compared were as follows: DP (durable press) rating, wrinkle recovery, stiffness, tensile strength, tear strength, shrinkage, skewness, hydrophobicity, whiteness and yellowness index. Marginally low performances in smoothness appearance and dimensional stability on fabric were exhibited with formaldehyde-free cross-linkers although indicating lower amount of the strength loss percentage. The formaldehyde-based compounds imparted more yellowing tendency to the treated fabric. The formaldehyde-free resins may be a good choice of replacements considering the overall effectiveness on fabric.
Keywords: cross-linkers, cotton woven fabric, durable press, wrinkle recovery, strength

Izvleček
V članku je bila ocenjena in primerjana učinkovitost formaldehidnih in neformaldehidnih zamreževal na predhodno obdelani bombažni tkanini. Kot formaldhidno zamreževalo je bil uporabljen Fixapret CL, kot neformaldehidno pa Fixapret NF. Za nanašanje zamreževal je bila uporabljena metoda suhega zamreženja. Raziskane in primerjane so bile različne lastnosti z zamreževali obdelanih bombažnih tkanin in sicer: stopnja trajne oblike (durable press, DP), razgubanje, togost, natezna trdnost, zatržna trdnost, skrčenje, poševnost, hidrofobnost, stopnja beline in indeks porumentive. Malce slabša učinkovitost, kar zadeva gladkosti in dimenzijske stabilnosti tkanine, je bila ugotovljena pri uporabi neformaldehidnega zamreževala, čeprav je bil pri tem ugotovljen tudi manjši odstotek izgube trdnosti. Tkanine, obdelane s formaldehidnimi mešanicami, so bile bolj nagnjene k porumenitvi. Neformaldehidna zamreževala bi bila dobra zamenjava za formaldehidne, ko gre za splošno učinkovitost na bombažni tkanini.
Ključne besede: zamreževala, bombažna tkanina, trajna oblika, razgubanje, trdnost

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1 Introduction

Cotton is wonderfully versatile fibre either single or blended, it can be washed or dry-cleaned easily [1]. It absorbs moisture readily, hence it is used extensively to produce comfortable end products but it has easy wrinkling properties that often create troubles for the end users [2]. Since a wrinkle occurs from the break or reshaping of hydrogen bonds, preventing the distortions of those bonds could be a solution [3].
Durable press finish or wrinkle free finish is just like that which forms covalent bonds with cellulosic hydroxyl groups of cotton fibres and creates crosslinks among cellulose molecules [4]. These cross-linkers are also known as easy care or wash-n-wear finishing agents. They can be defined as the chemicals used for improving the properties and performance of washable fabrics. According to Tommasino [5], there are two types of durable press finishing agents conventionally used: firstly, those which predominately crosslink cellulose, known as cellulose reactants, and secondly, those which self-polymerize as well as crosslink cellulose, known as aminoplasts. Both of them contain formaldehyde which can be responsible for human cancer [3]. Even the textiles containing high levels of formaldehyde can cause eczema and allergic reactions [6].
The first group of finishing agents is N-methylol compounds, namely urea formaldehyde or melamine-formaldehyde. Fixapret CL, a formaldehyde based resin (Fig. 1) is the concentrated aqueous solution of a reaction product of DMDHEU (dimethylol-4,5-dihydroxyethylene urea) with methanol. The N-methylol groups in DMDHEU react with cellulose forming a crosslinking net which is shown in Figure 2. On the other hand, Fixapret NF is an example of non-formaldehyde resin (Fig. 3) and an aqueous solution of 1,3-dimethyl-4,5-dihydroxyethylene urea (DMeDHEU).
The environmental impact of formaldehyde based resin and the potential danger of formaldehyde led to the introduction of non-formaldehyde finishes later on [2] and according to Can et al. [8], the use of non-formaldehyde resin is inexpensive and hygienic too. But before replacing formaldehyde-based resin by formaldehyde free resins, comparative studies are definitely needed to evaluate their performances from different angles which is the focus of this paper.

1.1 Literature review

Mukthy and Azim [9] observed the effects of DMDHEU on cotton blended woven fabrics. They tested wrinkle recovery, tensile strength, bending length and DP ratings of cotton and cotton blended fabrics on this aspect. Durable press rating was found close to 4 for cotton and cotton-blend fabrics. The influence of resin was far greater on cotton fabrics than cotton-blends in aspect of tensile strength and wrinkle recovery. Wrinkle recovery angle increased approximately by 42% and breaking strength reduced by about 64% after the crease resistant finishing.

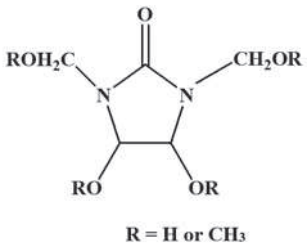

Figure 1: Chemical formula of Fixapret CL [7]

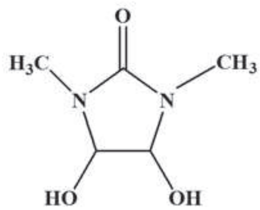

Figure 3: Chemical formula of Fixapret NF [7]

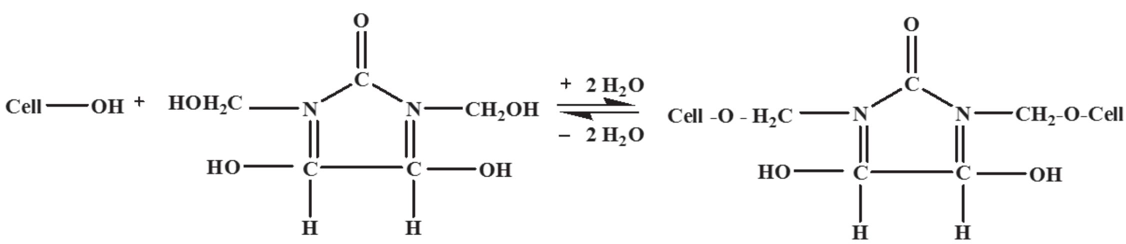

Figure 2: Crosslinking of cellulose with DMDHEU [2]

Thaseen [4] also investigated the effect of formaldehyde based resin (DMDHEU) on cotton, bamboo, lyocell and viscose fabrics with three levels of concentration and four levels of curing temperature. Though the tensile strength was unchanged in the cases of other fibres, the strength of cotton decreased at higher curing temperature and lower concentration of resin.

Saleemuddin *et al.* [10] did their research from a broader aspect to obtain eco-friendly finishes as well as to optimize application parameters for wrinkle free finishing on cotton polyester blend. They applied different pH values and resin concentrations using three types of formaldehyde based resins: dihydroxy ethylene urea, modified dimethylol dihydroxy ethylene urea (DMDHEU) and modified *N*-methylol dihydroxy ethylene urea. They compared the wrinkle recovery and tensile strength of treated fabrics. Formaldehyde extraction was lowest for the first one at 30 g/L concentration at a pH 5.0. The treatment with the first mentioned resin was more eco-friendly but provided low tensile strength. On the other hand, the mechanical properties of the blend fabrics treated with other resins were good with slightly elevated free formaldehyde.

Another group of researchers Lau et al. [11] tested the effect of wrinkle-free treatment on the fabric tear strength and dynamic water absorbency. They considered nitrogen content as a better indicator of the level of wrinkle-free treatment than the swelling index. They found that the swelling index (water retention) had a strong linear relationship with the total nitrogen content in the fabric. The higher the nitrogen content, the lower the swelling index (water retention). High nitrogen content or low swelling index resulted from the wrinkle-free treatment, caused low tear strength and reduced the water absorption rate of the fabric but increased the contact angle when the water drop was in initial contact of the fabric.

Safdar et al. [12] compared the effectiveness of three different types of resin finishes for improving the dimensional stability of cotton knitted fabrics. They used two formaldehyde based resins: dimethylol dihydroxy ethylene urea (DMDHEU) and a methylolation product of glyoxal monourein; the third one, modified dihydroxy ethylene urea (DHEU), was formaldehyde free. From their results, it was found that the DHEU, the non-formaldehyde resin, had better bursting strength as well as higher shrinkage problem.

Can et al. [8] used glyoxal reactive, a non-formaldehyde resin as the finishing agent to examine the effect of wrinkle free finish on cotton fabric properties. The result shows that after applying glyoxal reactive, the fabric achieved better crease recovery angle and also improved in pilling performance (decreased pilling). But the strength properties such as breaking strength and tearing strength were decreased by the resin application.

Tusief et al. [3] also took formaldehyde free finish (dihydroxy ethylene urea and dimethyl dihydroxy urea) for investigating the impact on wrinkle recovery of cotton fabric under different variables like concentrations and methods. A high concentration like 120 mg/L using pad-flash-cure method gave the best wrinkle recovery.

Talebpour and Holme [13] worked on easy care finished cotton fabric to see the effect on physical properties of bleached cotton. They used the DMeDHEU based resin and observed that static and kinetic friction was also increased along with crease recovery. They also applied a silicone based softener on an easy care finished fabric which reduced the friction as well as bending length but further increase in the crease recovery angle was observed.

There was also an initiative to design the factors affecting the printing with reactive dye combined with crease resistance finishing on cotton fabrics by Asim and Mahmud [14]. They took DMDHEU as a cross-linker and found that the crease recovery angle depended on the interaction effect of factors as well as individual factors.

Though there are a lot of works on resin finishing of cotton, there is no specific comparison done between the performances of formaldehyde based and non-formaldehyde resin, excluding Safdar et al. [12]. Even in that research, evaluations were done on a knitted fabric (shrinkage and bursting strength only), whereas a woven fabric tends to be more wrinkled due to its intensive structural nature and needs more treatment from cross-linkers than a knitted fabric.

Therefore, this paper aims to investigate different properties of 100% cotton woven fabric including DP rating, wrinkle recovery, stiffness, skewness, hydrophobicity, abrasion resistance, shrinkage and strength (both tensile and tear) using formaldehyde containing and formaldehyde free resins. Moreover, it focuses on the comparative study of the treated samples with the non-treated one as reference which can guide the scientists to make a better decision about the substitution of the formaldehyde based resin with the non-formaldehyde one.

2 Materials and methods

2.1 Fabrics and chemicals

100% cotton woven fabrics (enzyme-desized, scoured with non-ionic detergents and bleached with hydrogen peroxide) were used for this experiment to apply different types of crosslinking agents by padding method. The specifications of fabrics are given in Tab. 1. Trade names and types of all the commercial grades of chemicals for finishing supplied by BASF are listed in Tab. 2.

Table 1: Fabric specifications

Parameters	Desized-scoured-bleached fabric
Structure	Woven (twill)
Composition	Cotton
Ends [cm^{-1}]	45.67
Picks [cm^{-1}]	22.05
Warp linear density [tex]	29.53
Weft linear density [tex]	59.05

Table 2: Trade names and types of chemicals and auxiliaries used

Sl no	Chemicals (trade names)	Types
1	Fixapret CL	Cross-linking agent/ resin (formaldehyde based)
2	Fixapret NF	Cross-linking agent/ resin (formaldehyde free)
3	Condensol FM	Catalyst
4	Siligen PEP	Polyethylene based additive
5	Siligen SIS	Polysiloxane containing silicone softener
6	Kieralon JET-B Conc.	Non-ionic surfactant

2.2 Resin application by padding in finishing (dry-crosslinking method)

Fabrics were treated with different resins (at concentrations: 50g/L, 60g/L, 70g/L, 80g/L, 90g/L and 100g/L) from a separate bath with the same finishing bath conditions. The process was adopted as recommended by the supplier (BASF). The bath setup is given in Tab. 3.

Table 3: Resin application bath

Finishing bath set-up		Amount
Chemicals	Resin [g/L]	50, 60, 70, 80, 90, 100
	Condensol FM* [g/L]	12.5, 15, 17.5, 20, 22.5, 25
	Siligen PEP [g/L]	10
	Siligen SIS [g/L]	5
	Kieralon JET-B Conc. [g/L]	1
Application parameters	pH	5.5
	Pick-up [%]	60
	Drying temperature [°C]	110
	Drying time [s]	60
	Curing temperature [°C]	170
	Curing time [s]	40

* Catalysts were taken as 25% of the amount of resin at each concentration.

2.3 Evaluation of smoothness

Smoothness appearance of fabrics was tested according to the standard method AATCC 124. DP (durable press) rating was evaluated using 3-D standard replicas on a rating of 1 to 5 where 1 and 5 indicated severely wrinkled appearance and very smooth appearance respectively

2.4 Measurement of wrinkle recovery properties

Wrinkle recovery angle (in degrees) was measured using Shirley crease recovery tester taking the average values of warp and weft results.

2.5 Stiffness measurement

Stiffness was measured by bending length (in cm) using Shirley stiffness tester.

$$\text{Strength loss} = \frac{\text{Strength before finishing} - \text{Strength after finishing}}{\text{Strength before finishing}} \times 100\ [\%] \quad (1)$$

2.6 Strength evaluation: tensile and tear

Tensile strength & Tear strength were evaluated according to ISO 13934-1: 1999 (strip strength test using universal strength tester) and ISO 1397-1:1999 (using Elmendorf tear strength tester) respectively. Both warp and weft way strength were determined and loss of strength percentages were calculated using Equation 1.

2.7 Dimensional properties measurement: shrinkage and skewness

Shrinkage and skewness percentage were calculated adopting the standard methods BS EN 26330-2A (60°C, tumble dry) and AATCC 179 respectively.

2.8 Assessment of hydrophobicity (water resistance)

Hydrophobicity of the samples was determined by the absorbency test. A drop test was performed according to AATCC 79 method where time required (in s) of a drop of water to be absorbed was recorded. Water absorbency of the treated samples was evaluated according to the wicking test method (column test). Each sample of 2.5cm width was hung vertically immersing 1cm length into 0.1% direct dye solution for 1min. The height of the liquid (in cm) absorbed by the sample was taken as a measure of absorbency.

2.9 Measurement of abrasion resistance

Abrasion resistance was evaluated by the method ISO 12947-3, with 9kPa loading for 10.000cycles. The weight loss percentage was calculated (after 10.000cycles) comparing to the initial weight of each sample according to the Equation 2.

2.10 Reflectance evaluation: whiteness and yellowness

The effect of cross-linkers on whiteness and yellowness were evaluated after applying resins and also in combination with Optical Brightening Agent (only whiteness with nonionic Fluorescent brightener by BASF -5g/L) for each sample from a separate bath following Tab. 3. The degree of whiteness (Berger) and yellowness (CIE) of the samples was measured using a spectrophotometer with 10° observer and standard illuminant D65. All the values were measured in four different places making four folds of the samples and their average was taken for the analysis of the results.

3 Results and discussion

3.1 Crease recovery properties: DP rating (smoothness) and wrinkle recovery angle

Durable press performance and wrinkle recovery angle of the samples treated with formaldehyde containing resins were pronouncedly improved more than those of the samples treated with formaldehyde free resins with increasing concentrations. As the bath concentration approached 70–80g/L, smoothness appearance and wrinkle recovery power began to level-off. Beyond this level the rate of increment was less rapid for both types of resin. Massive amounts of cross-linkers produced only modest gains of crease recovery properties in every case. The results of DP rating and wrinkle recovery angle in degrees (both warp and weft way) are listed in Tab. 4.

3.2 Stiffness: bending length

Stiffness was evaluated measuring bending length in cm which increased from a level of 3.25cm bending length for both types of the treated samples with the resin add-on but the formaldehyde-based resin treated sample was comparatively stiffer with a maximum of 3.65cm as compared to 3.51cm at 100g/L with the non-formaldehyde reactants. Stiff fabrics were torn more easily than softer ones. The values of bending length are listed in Tab. 4.

3.3 Strength: tensile and tear

The strength (tensile and tear) of the treated sample was greatly reduced but the formaldehyde cross-linker resulted in higher strength loss. The tensile strength loss tended to level off up to 70–80g/L with improved DP properties. The tensile

$$\text{Weihht loss} = \frac{\text{Weight of untreated fabric} - \text{Weight of treated fabric}}{\text{Weight of untreated fabric}} \times 100\ [\%] \quad (2)$$

Table 4: DP rating, wrinkle recovery angle and bending length

Sample	Concentration [g/L]	DP rating	Wrinkle recovery angle [°] (warp + weft)	Bending length [cm]
Non-treated	–	2	206	3.25
R1 treated	50	3.25	248	3.52
	60	3.25	253	3.55
	70	3.5	260	3.58
	80	3.5	268	3.61
	90	3.75	273	3.63
	100	3.75	279	3.65
R2 treated	50	2.25	223	3.37
	60	2.25	229	3.4
	70	2.5	230	3.43
	80	2.5	238	3.46
	90	2.75	242	3.48
	100	2.75	245	3.51

strength loss percentage was ranged within 20 to 42% and 14 to 32% of original fabric values for the formaldehyde containing and formaldehyde-free resin treated samples respectively. The rate of improvement is more rapid with increasing resin level. The tear strength also indicated in sharp decrease with increasing amounts of resin. The percentage loss of tear strength amounted to the range from about 26 to 44% and 19 to 37% of the non-treated samples for formaldehyde containing

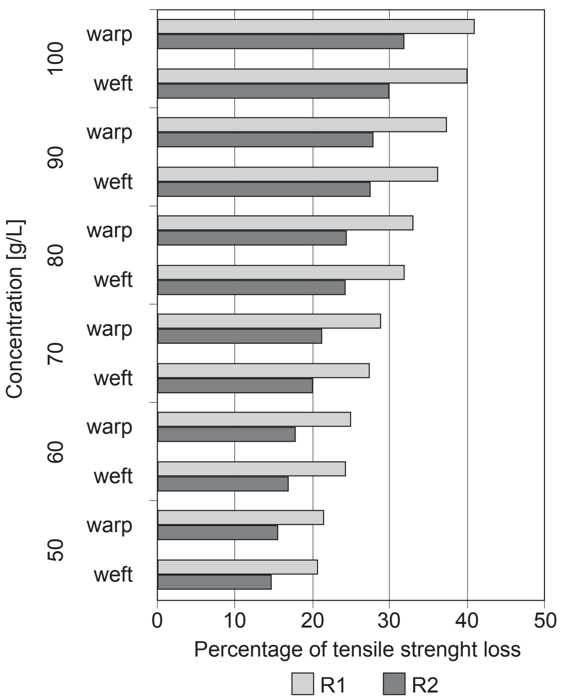

Figure 4: Loss of tensile strength of fabrics treated with cross-linkers

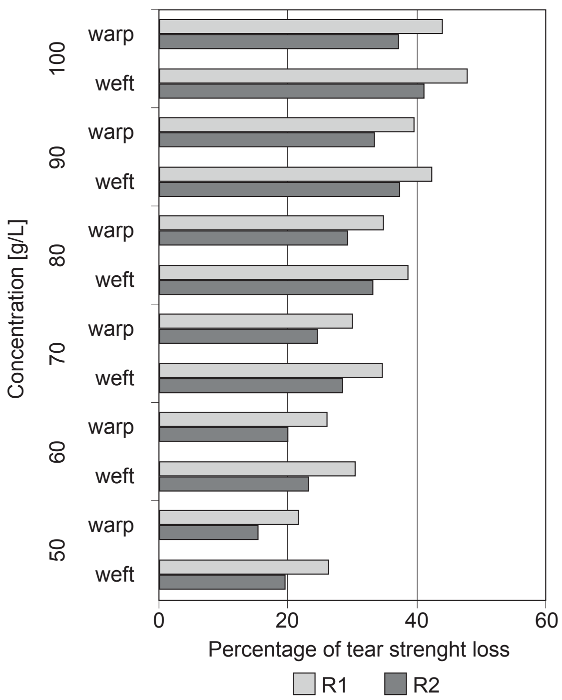

Figure 5: Loss of tear strength of fabrics treated with cross-linkers

and non-formaldehyde product. The tear loss percentage was obtained somewhat more as compared to the tensile loss percentage due to fibre rigidification or stiffness. A comparative profile of tensile and tear strength loss percentage with both types of resins are shown in Fig. 4 and Fig. 5 respectively.

3.4 Dimensional stability: shrinkage and skewness percentage

Dimensional stability was measured in terms of the shrinkage and skewness percentage of the treated samples. The results indicated that the degree of stabilization of the treated samples was increased with the increased number of crosslinkings. Crosslinking reduced the shrinkage and skewness more in case of formaldehyde-containing resins compared to the other. The percentage of both physical parameters dropped with the increased concentrations of resins. The comparison of the shrinkage and skewness percentage for different resins was shown in Fig. 6 and Fig. 7 respectively.

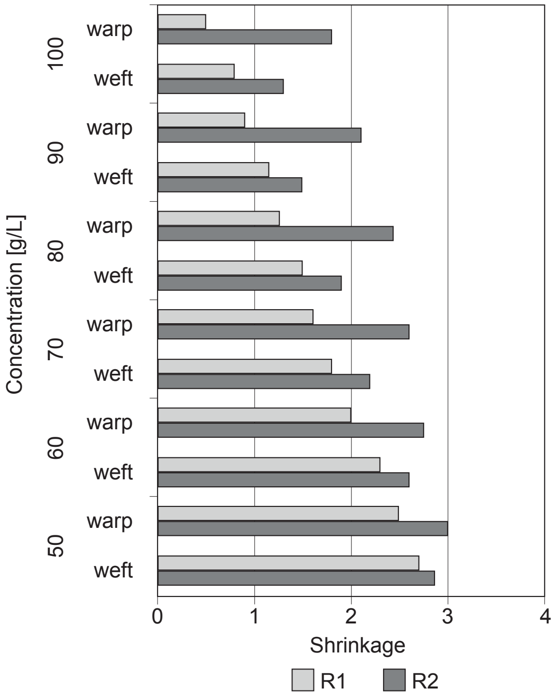

Figure 6: Shrinkage of fabrics treated with cross-linkers

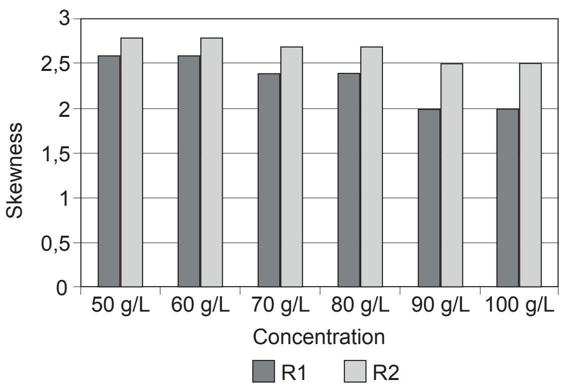

Figure 7: Skewness of treated fabrics with cross-linkers

3.5 Effect on hydrophobicity: absorbency test

Absorbency by drop test and column test of the treated samples indicated that the resins imparted hydrophobicity to the treated samples in both cases. This might be explained that because of the participation of hydroxyl groups of cellulose in crosslinking with resins, there was less availability of such groups to attract water molecules which eventually compensated to reduce water absorbency. As the fabrics became less absorbent the time of absorption of a drop of water was delayed and correspondingly found lower wicking distance on the treated fabric with formaldehyde-free crosslinking agent. Tab. 5 is showing the results of drop test in seconds and column test in centimeters.

Table 5: Hydrophobicity of the treated fabrics

Sample	Concentration [g/L]	Absorbency test	
		Drop test [s]	Column test [cm]
Non-treated	–	1	43
R1 treated	50	3	32
	60	3	30
	70	3	27
	80	4	25
	90	4	24
	100	4	23
R2 treated	50	2	37
	60	2	35
	70	2	33
	80	3	31
	90	3	29
	100	3	27

3.6 Abrasion resistance: weight loss measurement

Abrasion damage had a rapid increase with the resin add-on as the loss was obvious due to the number of crosslinks but the trends appeared to level off at 70–80g/L which was obtained as same as in case of strength loss and improved DP performance. The damage occurred on a slower rate after the specific concentration level for both types of cross-linkers applied. Abrasion resistance was evaluated in terms of the weight loss percentage and the formaldehyde-containing resin suffered weight loss most as is shown in Fig. 8.

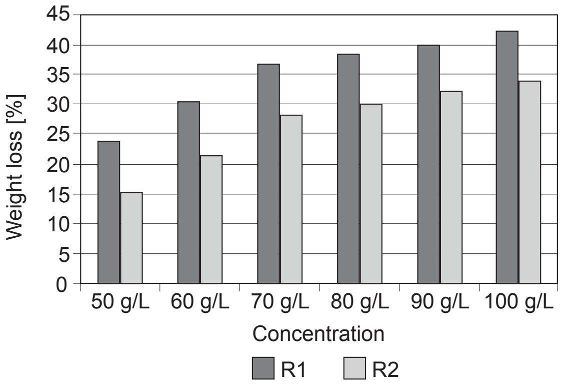

Figure 8: Weight loss for abrasion of treated fabrics by cross-linkers

3.7 Reflectance: whiteness and yellowness index

The formaldehyde based cross-linker treated fabric surfaces appeared duller and hence, whiteness (with and without OBA) was affected most and for the same reason the yellowness values were increased gradually with concentrations. The brightness was least affected by the formaldehyde free compound. Tab. 6 shows the reflectance data of all types of samples. Yellowing and deterioration of whiteness as well might be the consequences of using the formaldehyde reactants in combination with the catalysts and elevated curing temperature.

4 Conclusion

The objective of applying resins to textile fabrics is to impart specifically wrinkle free smooth surface to cellulosic fabrics as well as apparels so as to eliminate the daily pressing or ironing of garments after wash on usage. In this study, an investigation was performed comparing the effectiveness on a woven fabric between basically two types of resins which are commercially very common. The crease recovery properties in terms of durable press rating and smoothness appearance were increased with concentrations for both formaldehyde and non-formaldehyde reactants. It was found that the formaldehyde based resin imparted best smoothness appearance to

Table 6: Whiteness and yellowness index of treated fabrics

Sample	Concentration [g/L]	Berger whiteness index	CIE yellowness index	Berger whiteness index (with OBA)
Non-treated	–	70.2	12.3	137.5
R1 treated	50	64.2	13.2	133.6
	60	63.3	13.3	132.2
	70	62.8	13.5	131.5
	80	62.1	13.7	130.3
	90	61.3	13.9	129.7
	100	60.4	14.1	129.0
R2 treated	50	68.1	12.8	136.0
	60	67.2	12.9	135.2
	70	66.4	13.0	134.5
	80	65.3	13.1	133.8
	90	64.0	13.3	132.6
	100	63.2	13.4	132.0

the fabric as compared with the other resin. Stiffer fabrics were produced with the resin add-on but the formaldehyde-free containing samples produced comparatively softer handle with an increased bending length of only 8% as compared to the other types with 12% increment. Higher loss in fabric tensile strength was accompanied by the formaldehyde reactants which reached to as much as 42% with higher concentrations applied. The fabric tear strength was also impaired with gradual increase of concentrations and maximum loss in percentage of 44% was obtained for formaldehyde- based products. Both types of cross-linkers produced dimensionally stable fabric structures in terms of shrinkage and skewness after wash. Shrinkage and skewness were more or less similar in case of both. Cross-linked sites participated in lowering fabric absorbency in both cases. The fabric treated with formaldehyde containing crosslinking agents contributed in higher weight loss percentage but showed a tendency to get a slower rate of increment beyond a certain concentration in case of both. A considerable rise in yellowness and the reduction in whiteness values resulted due to gradual increment of resin levels for both. Although the target of performance was achieved in most cases of formaldehyde based compounds, the use of formaldehyde free cross-linkers as a replacement is of much concern due to ecological aspects and safety issues.

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