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**Research Paper / Makale**

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**Economical One Step Process for Simultaneous Application of Pigment Dyeing And Wrinkle Recovery Finishing of Cotton Fabrics**

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**Abstract:** Main objective of this study is to combining pigment dyeing with N,N'-1,3- dimethylol- 4,5- dihydroxyethylene urea (DMDHEU) durable press finishing in one step pad-dry-cure process. Dry wrinkle recovery angle (WRA), rubbing fastness and colour strength (K/S) values of specimens were measured before and after one/five/ten times washing treatments for determinating optimum process conditions of combining process. The results indicated that good durable press performance and higher dye depth could be obtained with the one step process (80 g/L DMDHEU, 3 g/L pigment dye, 60 g/L binder, 12 g/L catalyst, 10 g/L antimigration agent, cured at 150 °C for 4 min.). It exhibited good abrasion strength and good rubbing fastness values.

**Keywords:** Durable Press, Cotton, Finishing, Fastness, Pigment

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**Pamuklu Kumaşların Buruşmazlık Bitim İşlemi İle Pigment Emdirme İşleminin Aynı Anda Uygulanması İçin Ekonomik Tek Adım Prosesi**

**Öz:** Çalışmanın ana amacı, pigment boyama ile DMDHEU buruşmazlık bitim işlemini tek adımda emdirme-kurutma-fiksaj prosesinde birleştirmektir. Kombine prosesin optimum işlem koşullarını belirlemek için, numunelerin kuru buruşmazlık açısı (WRA), sürtme haslığı, renk verimi (K/S) değerleri; yıkama öncesi ve bir/beş/on tekrarlı yıkamalar sonrası ölçülmüştür. Sonuçlar; tek adım prosesıyla (80 g/L DMDHEU, 3 g/L pigment boya, 60 g/L binder, 12 g/L katalizatör, 10 g/L antimigrasyon maddesi, 150 °C'de 4 dk. kondenzasyon işlemi) iyi buruşmazlık performansı ve daha yüksek boya derinliği elde edilebildiğini göstermiştir. Bu proses, iyi aşınma dayanımı ve iyi sürtme haslığı değerleri sergilemiştir.

**Anahtar kelimeler:** Buruşmazlık, Pamuk, Bitim, Haslık, Pigment

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## 1. Introduction

Cotton is an important natural cellulosic fibre for textile industry because of a lot of characteristics, such as comfortable soft hand, good absorbency, machine washable, dry-cleanable, easy to handle or sew, good strength, colour retention, non-allergenic/toxic for health. One of the main disadvantages of cotton materials is easy wrinkling after washing due to swelling of cellulosic fibres by moisture. When cellulosic fibres are stressed by applied mechanical forces and swelling, the internal polymer chains of the amorphous areas are free to move to relieve stress. Then, the

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stretched/shifted internal molecular chains form new hydrogen bonds in the stretched places so, the fabric holds the creases [1,2].

Conventionally, cotton fabrics are first dyed, then for durable press properties, the textile materials are treated with crosslinking agents in combination with appropriate catalyst in wrinkle recovery finishing processes[3]. One step dyeing and durable press finishing of cotton (OPF) is based on incorporating the dyestuff into the finishing bath. OPF is relatively more economical process due to reduced energy consumption, production time, dyeing and finishing chemicals, and water usage[4-6].

Several reports on simultaneous dyeing and durable press finishing of cotton fabric have appeared in the literature [4-13]. Dong et al. [5] carried out simultaneous dyeing and durable press finishing of cotton fabric with reactive dyes and citric acid finishing agent by using a pad-dry-cure process. Choi [6] studied about OPF with dialdehyde finishing agent and monochlorotriazinyl reactive dye. Raheel et al. [7] investigated the efficacy of polycarboxylic acids (citric acid and 1,2,3,4-butanetetracarboxylic acid) as crosslinkers when used individually and as mixtures of various ratios in conjunction with direct dyes in OPF processes. Chen [8] reported that combined dyeing and finishing treatment on cotton fabric with the mixture of carboxypyridinyltriazine reactive dye and dimethylolethyleneurea (DMEU) was carried out by a pad-bake-wash process. Teli et al. [9] stated that simultaneous dyeing and finishing of cotton fabric was carried out with acid dyes and dimethyloldihydroxyethylene urea (DMDHEU) using ammonium nitrate as catalyst. Gurmu et al. [10] studied about OPF with varying concentration of modified DMDHEU and acid dyes. Ratte [11] described that the Procion-Resin process for cotton fabrics was ensured the simultaneous dyeing and finishing of cotton fabric with reactive dye and some different resin finishing agents. Ibrahim et al. [12] studied about simultaneous dyeing and easy-care finishing of cotton and carbamoylethylated cotton fabrics using reactive dyes and N-methylol finishing agents. The best wrinkle recovery angles were obtained by using DMDHEU finishing agent. Teli et al. [13] carried out simultaneous direct dyeing and resin finishing by using the varying concentration of low formaldehyde based DMDHEU and direct dyes to explore the potential of reduction in energy consumption, time of production as well as dyes and chemicals.

The crosslinkers used are also known as easy care or durable press finishing agents, which can be basically divided into two main groups: formaldehyde-based chemicals and formaldehyde-free compounds [14]. The most widely used chemical is DMDHEU basis of about 90 % of the easy-care and durable press finish products on the market. It is especially inexpensive and effective chemical that has lots of properties such as low chlorine retention, excellent durability to laundering, medium to very low formaldehyde release [2,3,15].

In this study, the DMDHEU, as a crease resistant agent, is mixed with pigment dye, catalyst, binder, antimigration agent to establish a finishing bath for the OPF. The different concentrations of DMDHEU, dye, binder and softener were picked up in recipes to find out the best process conditions. Despite increasing in WRA results, durable press finishing generally causes loss of abrasion and/or tensile strength, hard handle and shade change of coloured fabrics. So, after pad-dry-cure process, colour strength (K/S values), rubbing fastness as well as WRA results were measured. The obtained test values before and after one/five/ten times domestic washing treatments were interpreted by histogram analyzes. Abrasion resistance values were only evaluated for the fabrics that have better WRA results.

## 2. Experimental

### 2.1 Materials

The desized, bleached, alkaline scoured 100 % cotton fabric was in plain weave structure, 125 g/m<sup>2</sup>. Properties of materials which were used in padding baths were given in Table 1. OPF were carried out in padding machine (Ernst Benz Lab. Fulard). A laboratory scale stenter (ATAÇ) was used for drying and curing. Colour strength values were measured by spectrophotometer (HunterLab UltraScan Pro Spect.) and abrasion resistance were tested by Nu-Mantindale abrasion and pilling tester (Healink).

Table 1: Properties of materials which were used in padding baths.

Materials	Properties of Materials
Dyestuff (Orgaprin red RV, Organik Kimya ve Tic. AS. Turkey)	C.I. Pigment Red 2, is a mono-azo pigment.
Binder (Orgacompound PD, Organik Kimya ve Tic. AS. Turkey)	It is an anionic, acrylic polymer mixture for pigment dyeing.
Catalyst	MgCl <sub>2</sub> .6H <sub>2</sub> O
Anti-migration agent (Orgafix 18, Organik Kimya ve Tic. AS. Turkey)	The chemical structure is not defined.
Softener (Siligen CSP, BASF Co.)	It is an anti-pilling agent with softening effect, is silicone fluid with polyether groups and polyacrylate based. It is compatible with Fixapret AB-9.
Durable press finishing agent (Fixapret AB-9, BASF Co.)	It is DMDHEU based resin.

### 2.2 Methods

#### 2.2.1 One step pigment dyeing and finishing processes

Pad-dry-cure process were used: The pigment dyeing and wrinkle recovery finishing treatment were applied simultaneously in one step. After impregnation with 70% wet pick up, samples were dried at 100°C for 2 min. and cured at 150°C for 4 min. According to the preliminary experiments, for enough dye binding so for good rubbing fastness, it was necessary to choose binder and DMDHEU concentrations higher than 50 g/L. In recipes 1 to 9, only pigment padding process (OP) were carried out for comparison with OPF. Durable press process can cause hard handle problem on fabric, so different concentrations (20-40 g/L) of softener were used in two recipes 19, 20 for OPF processes.

#### 2.2.2 Test methods

Washing treatments were applied with 4 g/L laundry colourmatic detergent at 60 °C, 20 min. WRA values of fabric samples before and of after one/five/ten times washing treatments were measured according to DIN 53890 standard. Rubbing fastness values were determined according to ISO 105 - X12 standard. The colour strength ( K/S ) values of the samples were calculated by using the Kubelka–Munk equation. The samples were measured using an EasyMatch QC software of

Hunterlab Ultrascan Pro color spectrophotometer attached to a personal computer. Abrasion resistance were tested according to ASTM D 4966-98 standard.

Table 2: Properties of recipes which were used for padding baths.

Recipe No	Dye Con. (g/L)	Anti- migration agent (g/L)	Binder (g/L)	DMDHEU (g/L)	Catalyst (g/L)	Softener (g/L)
1	1	10	60	-	9	-
2	1	10	80	-	12	-
3	1	10	100	-	15	-
4	3	10	60	-	9	-
5	3	10	80	-	12	-
6	3	10	100	-	15	-
7	5	10	60	-	9	-
8	5	10	80	-	12	-
9	5	10	100	-	15	-
10	1	10	60	60	9	-
11	1	10	60	80	12	-
12	1	10	60	100	15	-
13	3	10	60	60	9	-
14	3	10	60	80	12	-
15	3	10	60	100	15	-
16	5	10	60	60	9	-
17	5	10	60	80	12	-
18	5	10	60	100	15	-
19	3	10	60	60	9	20
20	3	10	60	60	9	40

### 3. Results and Discussions

#### 3.1 Pigment Dyeing and Finishing of Cotton Fabric

C.I. Pigment Reds 2 is moderately significant commercial product, especially the latter. Red 2 is a relatively low cost mid-red pigment used primarily in printing inks [16]. It which has yellowish red colour, is single azo compound and insoluble in water. Structure of C. I. Pigment Red 2 was given in figure 1 [17].

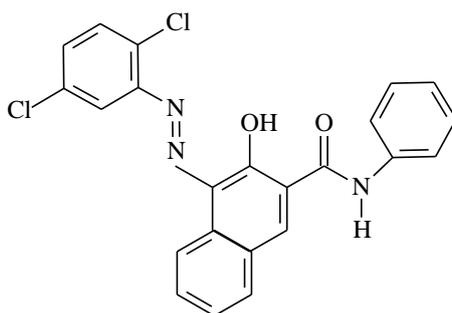


Figure 1: Structure of C.I. Pigment Red 2 [17].

Pigments for textiles have no substantivity for fibers and are not generally found in monomolecular (solution) form but rather in the form of very finely divided water dispersible particles after pigment grinding process with convenient dispersant. So, we needs binders which are polymeric materials for sticking pigment particles to fiber surface as a few microns thick film with help of adhesion forces. Binders are in form of dispersions of water insoluble droplets of composition containing low

molecular weight prepolymers. They bring out macropolymer network due to crosslinking themselves in each other at stage of curing. Pigment colouration has some limitations/drawbacks such as: being not appropriate for dark, deep or very bright shades; speedily colour fading if not desired like denim; having unsatisfactory bad rubbing fastness, fabric handle, aesthetic feelings and low abrasion resistance in associated with binder, pigment and softener selection. Although pigment padding procedure is easy applicable way for textile colouration, the procedure does not need washing treatment and can be applied all type of fibres and blends continuously. Compatibility of chemical structures of materials including in padding bath is important due to causing coagulation [18]. The pigment was added in bath last after the catalyst in the processes for preventing coagulation. The cotton fabrics had uniformly coloured surface with tint of pink after the OP and OPF pad-dry-cure processes.

DMDHEU contain N-methylol groups that can react with cellulose or self-polymerize in presence of an acid catalyst ( $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ) and heat (at curing,  $150^\circ\text{C}$ ). Mechanism of crosslinking of DMDHEU with cellulose was given in figure 2.

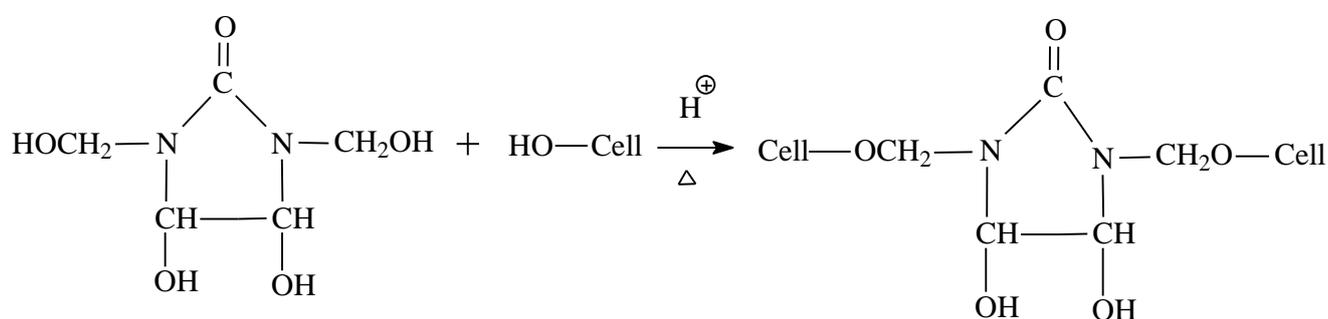


Figure 2: Mechanism of crosslinking of DMDHEU with cellulose [1,2].

In durable press finishing formulations, it is common to use Lewis acids as the catalyst for the crosslinking step such as magnesium chloride that generate acid conditions during curing process, thus providing neutral liquors and good finish bath stability [2,4,19]. The associated Lewis acid mechanism of this crosslinking reaction was given in figure 3.

### 3.2 Wrinkle Recovery Angle ( Total, Warp + Filling)

OP processes as 1-9 recipes were carried out with three different binder concentration 60-80-100g/L. The effect of binder concentration on WRA, K/S and rubbing fastness was investigated. According to WRA results given in Figure 4, the higher binder concentrations ensured the higher WRA values at the same dye concentrations. But, slightly increasing in WRA results was observed within increasing binder concentration from 60g/L to 100g/L at the same dye concentrations. Therefore constant 60g/L binder concentration was used in OPF processes.

In 10-18. recipes, the binder concentrations was constant (60 g/L) and three different DMDHEU concentration as 60-80-100g/L were used in OPF processes. WRA values of fabric samples treated with recipes including DMDHEU addition ensured the significant increase in in comparison with WRA values of fabric samples treated with recipes without DMDHEU addition (recipe 1,10 - 4,13 - 7,16) (figure 5).

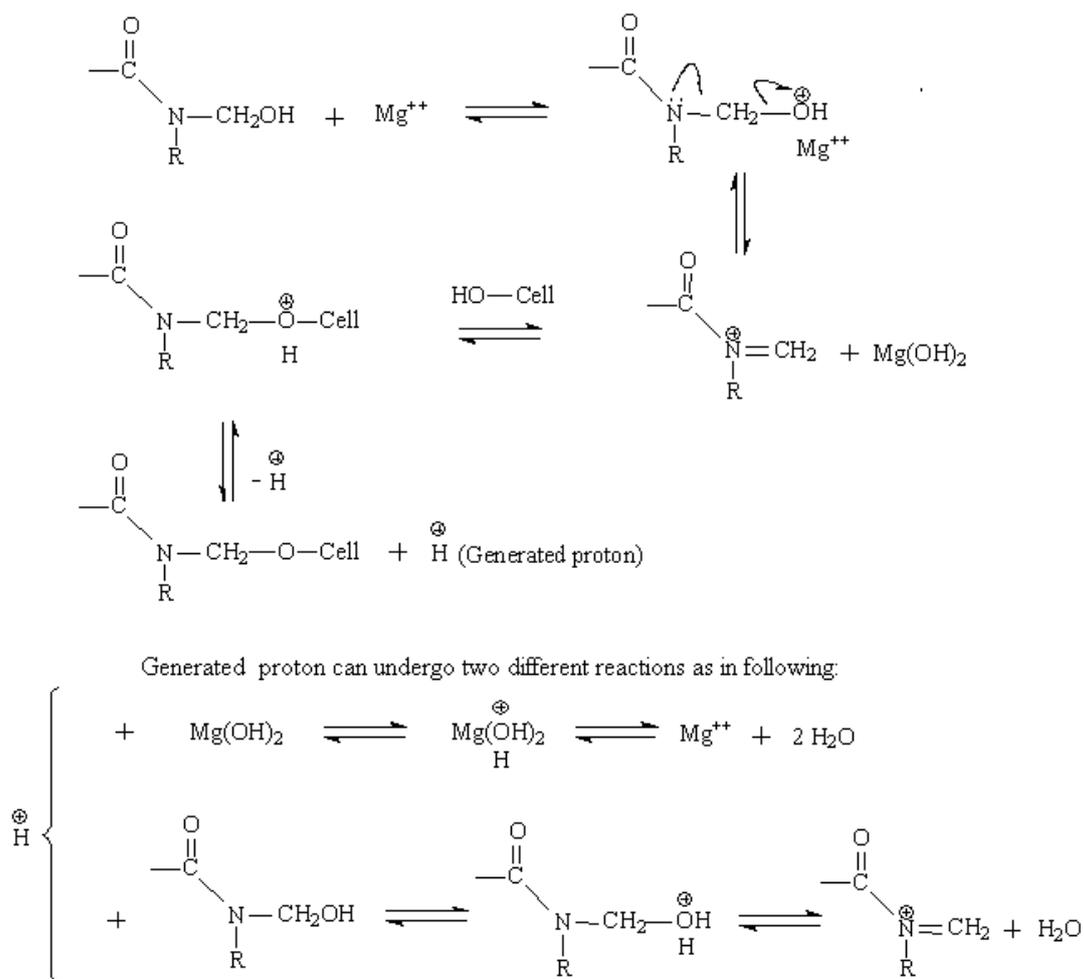


Figure 3: Lewis acid mechanism of DMDHEU crosslinking reaction.

In OPF processes for all dye concentrations, WRA values of fabric samples increased while DMDHEU concentrations in bath were increasing (Figure 5). Softener addition in OPF processes (recipe 13, 19, 20, in figure 5) caused slightly decreasing in WRA results. Repeated washing treatments caused decreasing in WRA results in all OP and OPF processes (Figure 4,5).

The results indicated that 80g/L DMDHEU concentration was optimum and ensured good WRA values in OPF processes. The risk of showing the drawbacks on fabrics and the cost of chemicals for processes increase with increasing mixtures of DMDHEU in OPF processes.

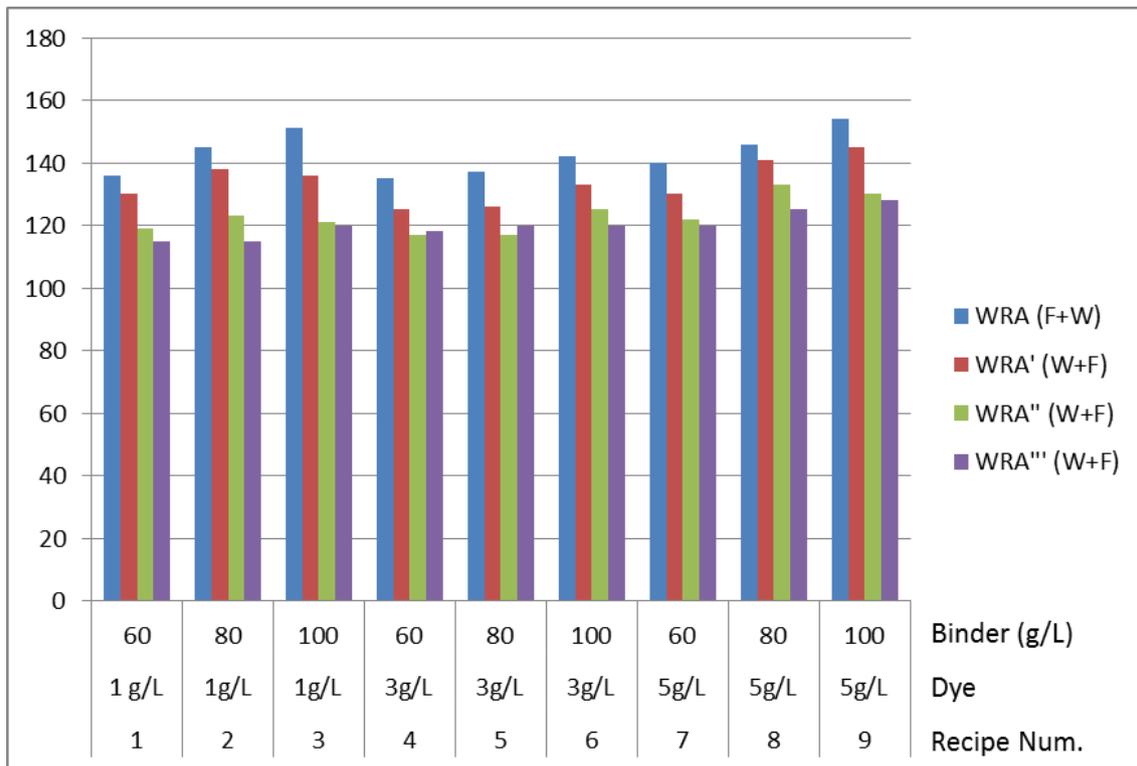


Figure 4: Effects of binder concentrations on WRA results of only pigment padding processes. (WRA: pre-washing, WRA': after one times washing, WRA'': after five times washing, WRA''': after ten times washing).

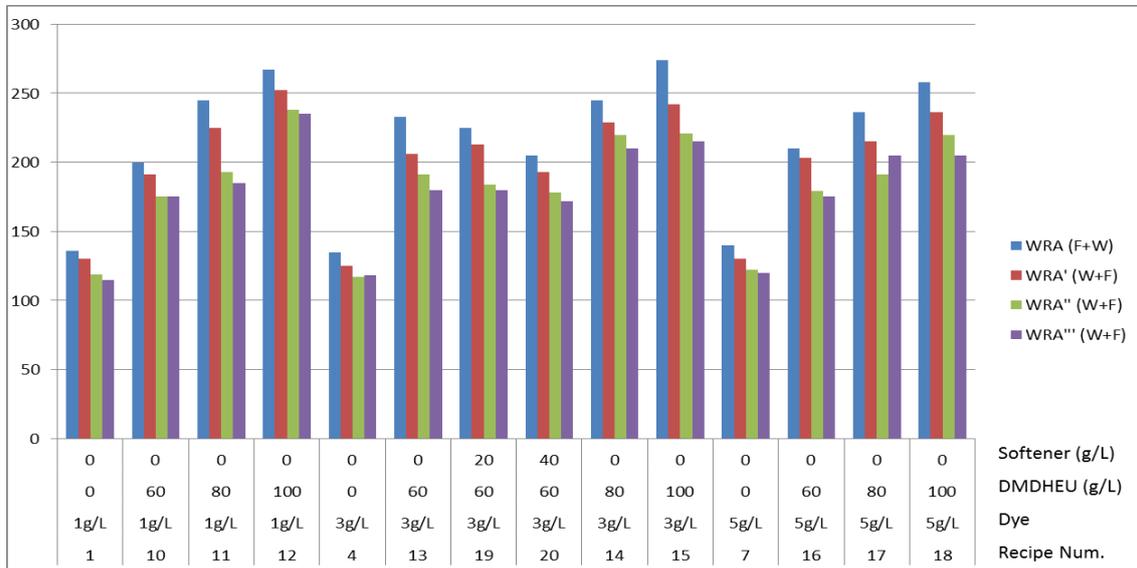


Figure 5: Effects of DMDHEU concentrations on WRA results of OPF processes. (WRA: pre-washing, WRA': after one times washing, WRA'': after five times washing, WRA''': after ten times washing).

### 3.3 Colour Strength

The higher binder concentration for all dye concentrations led to the higher K/S values of fabric samples treated with OP processes due to the better sticking dye to fibre (figure 6). The higher dye concentrations result in the higher K/S values at the same binder concentrations (such as recipe 1, 4, 7 in figure 6). K/S values of fabric samples treated with OP processes at same binder and dye concentrations decreased after one/ five/ten times washing treatments (figure 6). DMDHEU addition in OP baths (such as recipe 1,10 or 4,13 or 7, 16 ) gave rise to increasing in K/S values due to its possible bonding ability between dye and cellulose (figure 7). The K/S result which was obtained from adding 20 g/L softener in OPF process in recipe 19, was lower than the result that was obtained from OP process without DMDHEU in recipe 4 in figure 7. So, adding softener caused too much downfall in the K/S values (such as recipes 13, 19, 20 in figure 7). Repeated washing treatments caused decreasing in K/S values for all recipes in Figure 7.

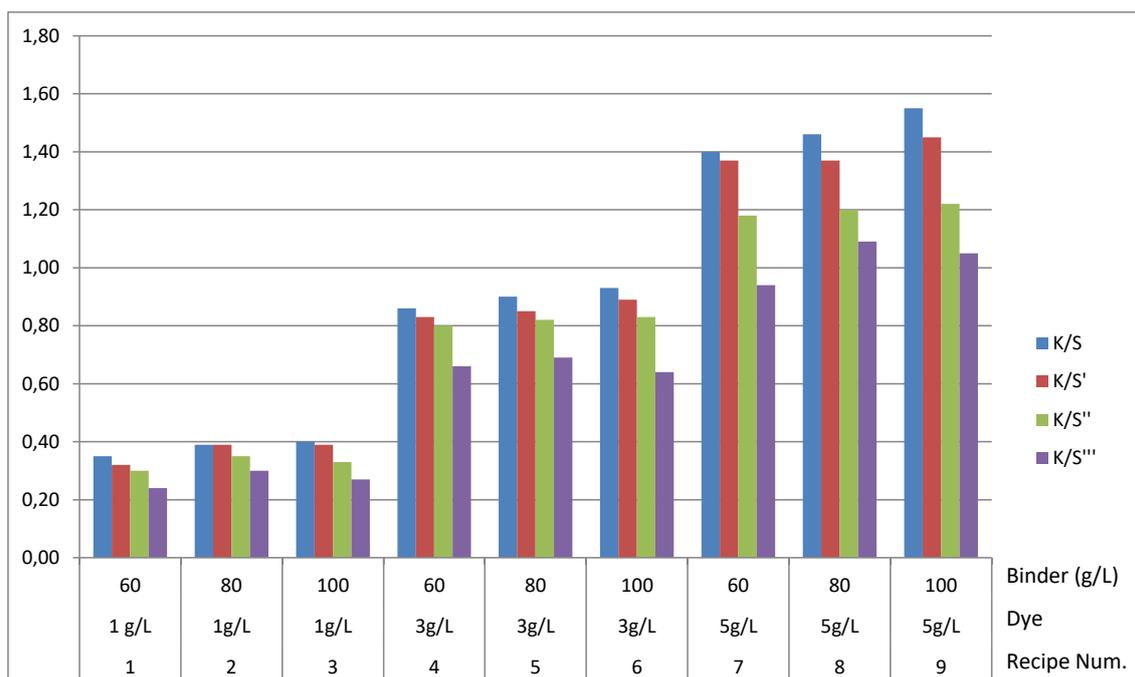


Figure 6: Effects of binder concentrations on K/S value results of only pigment padding processes. (K/S: pre-washing, K/S':after one times washing, K/S'': after five times washing, K/S''': after ten times washing).

### 3.4 Rubbing Fastness

Dry rubbing fastness values (DR) and wet rubbing fastness values (WR) of fabric samples treated with OP and OPF processes were commercially acceptable levels (3/4 – 5), given in figure 8-11. DR and WR values of fabric samples treated with OP and OPF processes decreased with increasing dye concentration (1 g/L to 5 g/L) (figure 8-11).

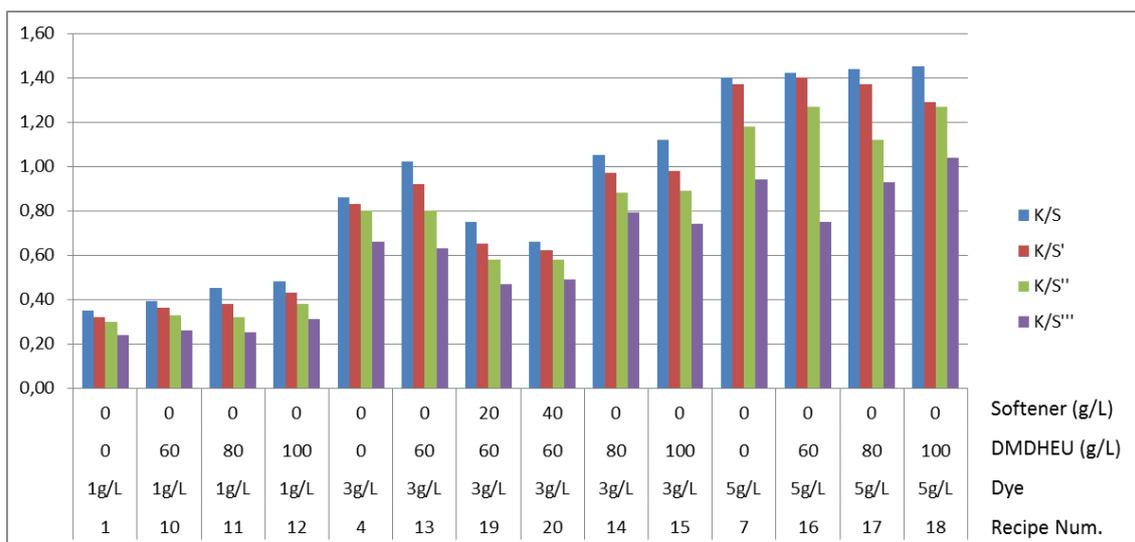


Figure 7: Effects of binder concentrations on K/S value results of OPF processes. (K/S: pre-washing, K/S':after one times washing, K/S'': after five times washing, K/S''': after ten times washing).

It was deduced that rubbing fastness values of fabric samples changed mainly depending on their dye concentrations. Repeated washing treatments generally caused increasing in DR and WR results of all fabric samples treated with OP and OPF processes due to decreasing of colour strength. DMDHEU addition in baths caused decreasing on DR and WR values of fabric samples in figure 10,11 (recipe 1,10 - 4,13 - 7,16). Softener addition (20 g/L) in OPF process caused slightly increasing in DR values in spite of no changing in their WR values in figure 10,11 (recipe 13,19).

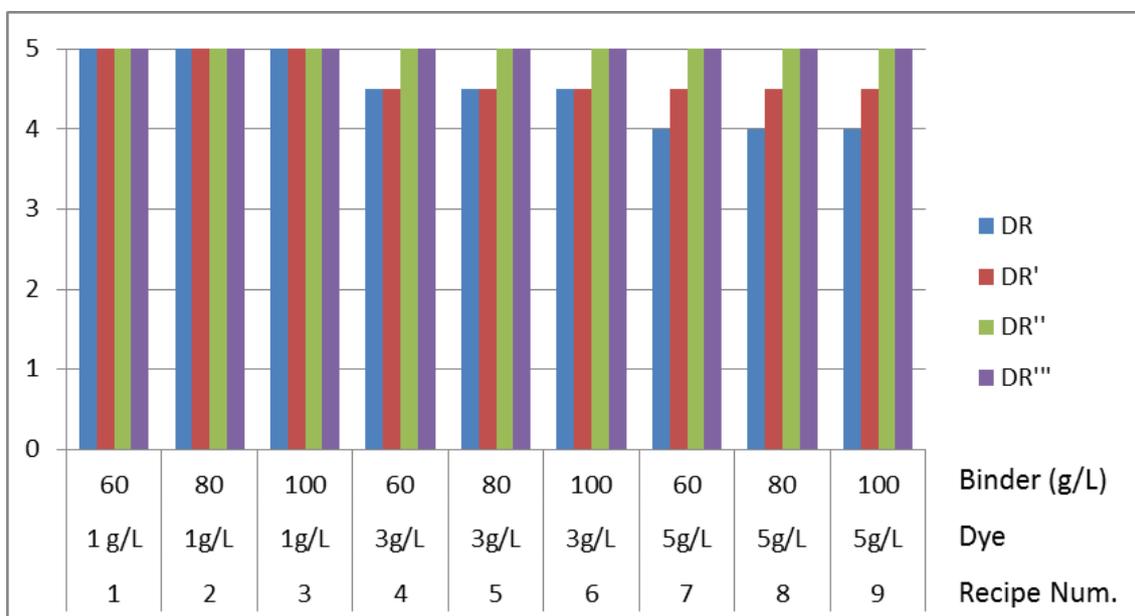


Figure 8: Effects of binder concentrations on dry rubbing fastness (DR) results of only pigment padding processes. (DR: pre-washing, DR':after one times washing, DR'': after five times washing, DR''': after ten times washing).

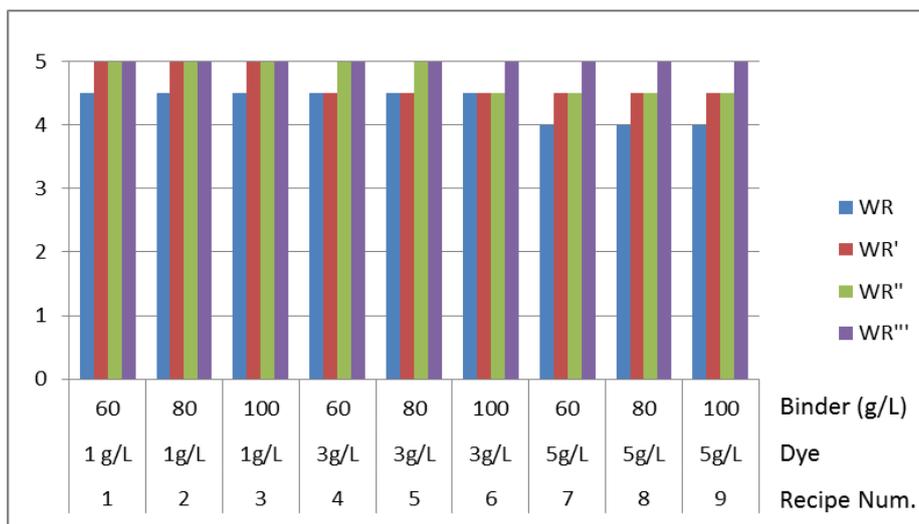


Figure 9: Effects of binder concentrations on wet rubbing fastness (WR) results of only pigment padding processes. (WR: pre-washing, WR':after one times washing, WR'': after five times washing, WR''': after ten times washing.)

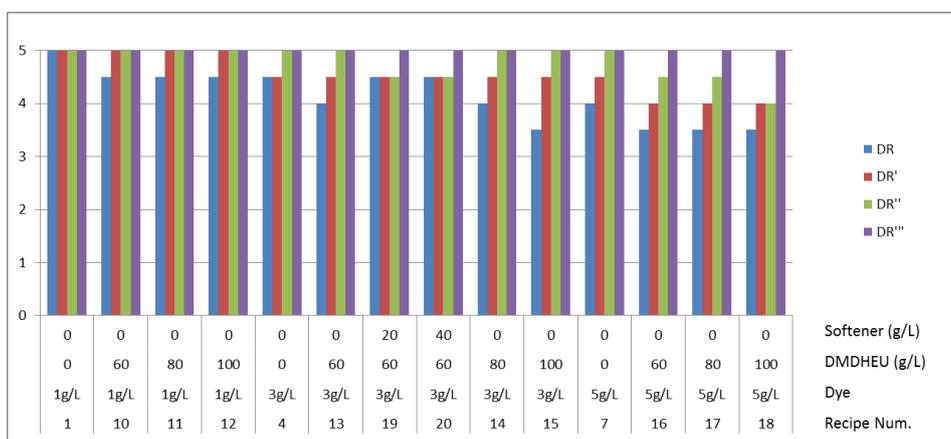


Figure 10: Effects of DMDHEU concentrations on dry rubbing fastness results of OPF processes. (DR: pre-washing, DR':after one times washing, DR'': after five times washing, DR''': after ten times washing).

### 3.5 Abrasion Strength

Crosslinking of the cellulose at the fiber/fabric surface, which may be acerbated by migration of the reactant to the surface during the drying and curing resulting in increased crosslinking at the surface, results in increased embrittlement of the fiber surface and a decreased abrasion resistance [20].

Abrasion resistance values were determinated only for the sample treated with recipe 5, 11, 14 by Martindale Methods. Abrasion strength values of fabric samples were in range of 9000-10000 rpm (10g/L antimigration agent, drying at 100°C, 2 min., cured at 150 °C for 4 min.) for all specimens.

The end point for the woven fabric specimens was determinated when two yarns had broken in Martindale Methods. The increase in repeated times of washing treatments caused the higher abrasion level for all fabric samples at the 10000 rpm. The abrasion strength values of the fabric samples treated with recipe 5 were slightly higher than that of the fabric samples treated with recipe

14. Therefore the abrasion strength result of OP processes was better than OPF processes and that DMDHEU caused decreasing in abrasion strength of fabric.

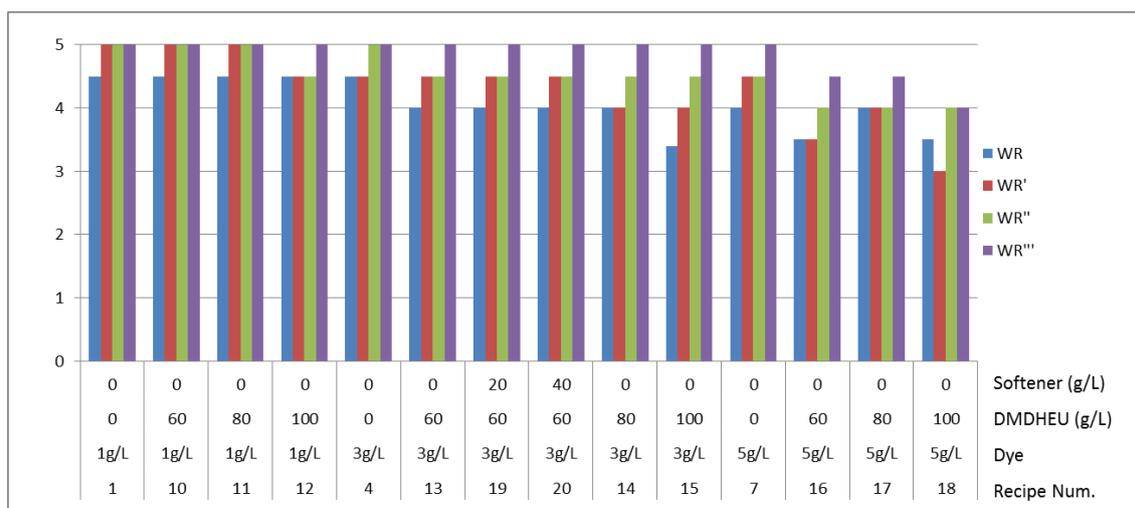


Figure 11: Effects of DMDHEU concentrations on wet rubbing fastness results of OPF processes. (WR: pre-washing, WR': after one times washing, WR'': after five times washing, WR''': after ten times washing).

#### 4. Conclusion

In this study, combining pigment padding with DMDHEU durable press finishing in one step pad-dry-cure process was achieved. Simultaneously colouration with finishing is the economical, easy application way for textiles, especially in colouration of cellulosic fabrics in lighter shades. Homogenous coloured fabric surfaces and commercially acceptable levels of WRA, rubbing, colouration strength values were obtained from OPF processes. The results indicated that good durable press performance and higher dye depth could be obtained with the optimum conditions of one step process (80g/L DMDHEU, 3 g/L pigment dye, 60g/L binder, 12g/L catalyst, 10g/L antimigration agent, drying at 100°C, 2 min., cured at 150 °C for 4 min.). The significant increase in WRA results were obtained with adding DMDHEU in OP processes. With increase in times of washing treatments, the WRA results and colour strength values were decreased, whereas rubbing fastness values were increased for all recipes. In the darker shade colourations with OPF processes (5 g/L dye), the rubbing fastness values were relatively decreased about one degree. With the repeated washing treatments abrasion strength of fabrics were decreased. The abrasion strength result of OP processes was slightly better than OPF processes at 10000 rpm.

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