

PRODUCTION OF MULTIFUNCTIONAL WOVEN COTTON FABRICS BY A SINGLE STEP FINISHING PROCESS

TEK ADIMDA MULTIFONKSIYONEL PAMUKLU DOKUMA KUMAŞ ÜRETİMİ

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Received: 27.12.2016

Accepted: 15.02. 2017

ABSTRACT

In this study, it was aimed to obtain multifunctional cotton fabrics which would have durable press and antibacterial properties and satisfy the expectations of the consumers in terms of soft touch and easy care. In the scope of this project, more than one function would be given to the cotton fabric in a single step and thus energy, time and water would be saved as well as production costs. As a result of the project, used chemicals were applied in one step by pad-dry-cure method. It was determined that this work showed better performance characteristics and the resulting effects were resistant to 20 times washing when compared with the current applied processes. It was also determined that at least 50% reduction in costs and in the process time was achieved.

Keywords: Cotton, Multifunctional, Antimicrobial, Soft Touch, Easy care

ÖZET

Bu çalışmada tüketicilerin tutum ve kolay bakım açısından beklentileri karşılayacak hem buruşmaz hem antibakteriyel multifonksiyonel pamuklu kumaş elde edilmesi amaçlanmıştır. Proje kapsamında birden fazla fonksiyon bir adımda kazandırılarak enerji, zaman ve su tasarrufu ile birlikte üretim maliyetlerinin de düşürülmesi hedeflenmiştir. Proje çalışması sonucunda kullanılan kimyasallar tek adımda emdirme yöntemine göre applike edilmiş, kurutma yapıldıktan sonra fikse yapılmıştır. Çalışma mevcutta uygulanan proseslerle kıyaslandığında daha iyi performans özellikleri gösterdiği gibi, etkinin 20 yıkamaya dayanıklı olduğu da belirlenmiştir. Ayrıca maliyet ve işlem süresinde %50 oranında tasarruf sağladığı tespit edilmiştir.

Anahtar Kelimeler: Pamuk, Multifonksiyonel, Antibakteriyel, Yumuşak Tutum, Kolay bakım

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1. INTRODUCTION

Textile wet processes generally consist of three steps; pretreatment (or preparation), coloration (dyeing or printing) and finishing. Finishing in the narrow sense is the final step in the fabric manufacturing process, the last chance to provide the properties that customers will evaluate. Finishing completes the fabric's performance and gives it special functional properties including the final "touch" (1).

In this field, the demands and competitions increase day by day and they force the producers to struggle more and make research and development works. How to produce multifunctional fabrics has been an important research field in recent years. For this purpose, increasing knowledge and

improvements in chemistry enable to produce more functional materials.

Textile applications are generally made to give single function to the fabric. When more than one functionality is needed, treatments are performed in more than one step and their costs are very high.

Wrinkle-resistance and soft touch are generally the most demanded features by customers. The importance of antimicrobial textiles and applications has also increased recently. Customers are willing to buy such kind of functional products.

Durable press (DP) or easy care finishing is mostly used for 100 % cotton fabrics or for the blend fabrics with high

contents of cellulosic fibers. This finish provides resistance against shrinkage and improved wet and dry wrinkle recovery of cellulosic textiles. Inhibition of easy movement of the cellulose chains by crosslinking with resins/polymers is the mechanism of a DP finish. Initially, derivatives of urea such as urea-formaldehyde and melamine-formaldehyde resins were used. Environmental concerns and the potential danger of formaldehyde led to the introduction of formaldehyde-free finishes (2).

The growth of microorganisms on textiles during use and storage negatively affects the wearer as well as the textile itself. The detrimental effects can be controlled by durable antimicrobial finishing of the textile material using broad-spectrum biocides or by incorporating the biocide into synthetic fibers during extrusion. Consumers' attitude towards hygiene and active lifestyle has created a rapidly increasing market for antimicrobial textiles, which in turn has stimulated intensive research and development (3).

Chitosan as an antimicrobial agent can be used with Durable Press (DP) finishing agents in a single bath. Cotton finished with different crosslinking agents (BTCA, glyoxal and glutaraldehyde) in the presence of chitosan shows a broad spectrum of antimicrobial activity against gram-positive and gram-negative bacteria and fungi (2).

In some respects, the softening finish is very important as it can influence the consumer's decision to purchase, as the final hand or touch of the fabrics plays a crucial role. There are many improvements in the field of softening finishing process and the finishing formulations are now modified by taking into account of their applications on a variety of textile blends and the demands for multiple performance properties. Similar to the other finishes, softening also face various challenges during their applications on textiles and there are a lot of research efforts in this field aiming to enhance their final performances, to overcome the problems of thermo migration and adverse effects on crocking fastness, and to reduce environmental impacts (4). About one-third of the softeners used in the textile industry are silicone based as they impart excellent soft handle combined with various other properties such as water repellency, superior smoothness, greasy feel, improved crease resistance, etc. In fact, the silicones were first used by the textile industry primarily as lubricants in fiber and fabric manufacture. Silicone softeners are also applied with permanent press finishes to improve usage life of garments and durability of permanent press finish (5).

In this study, the silk protein which is a natural product was used as a softener in textile applications. Silk protein (sericin&fibroin) is a biodegradable product and is widely

used in skincare for its moisturizing abilities. The structure and the content of amino acids in silk proteins (sericin&fibroin) are similar to the skin of human body. Due to its special chemical structure and chemical composition, it is highly compatible and absorbed easily by the human skin (6, 7).

The main aim of this research is to apply the multifunctional finishes to cotton woven fabrics to meet the expectations of consumers in terms of soft touch, antibacterial property and easy care character. Within the scope of this study, more than one function was given to the cotton fabric in a single step, thus energy, time and water are saved and production costs are reduced. Cotton fabrics were treated with a combination of chemicals by pad-dry and pad-dry-cure methods. Then the treated fabrics samples were tested for multifunctional performances such as antimicrobial, softness and durable press.

2. MATERIAL AND METHOD

2.1 Materials

Desized-bleached-mercerized 100% satin weave cotton fabric was supplied from Söktaş Dokuma İşletmeleri Sanayi ve Ticaret A.Ş., Turkey. Technical information of the fabric is given in Table 1.

In this study, quaternary macro silicone (Rudolf Duraner), quaternary micro-silicone (Rudolf Duraner), macro silicone (Dystar), and micro-silicone (Dystar) softeners are used together with silk protein (sericin&fibroin, Rudolf Duraner) for investigating softening properties on cotton fabric.

Medium molecular weight chitosan (Sigma-Aldrich) was used for both easy care and antibacterial finishing. Imidazole derivative reactive resin with zero formaldehyde (Pulcra Chemicals) was used for easy care finishing. Inorganic salt (AgCl/TiO₂, Rudolf Duraner) was used for antibacterial finishing on cotton fabric. Literature review about silk protein (sericin&fibroin) indicated that this natural product had antibacterial properties, as well as softness. Therefore antibacterial properties of silk protein (sericin&fibroin) and chitosan were examined.

2.2 Method

Finishing applications were done both at laboratory scale and industrial scale. At first all chemicals with different concentrations were applied to the fabrics separately at the laboratory. Then chemicals and determined optimum concentrations were chosen for combined applications to obtain multifunctional cotton fabric at the laboratory scale. After the evaluation of laboratory scale studies, industrial scale studies were planned.

Table 1. Technical information of the cotton fabric

Fabric Type	Weight (g/m ²)	Density (ends/cm)		Yarn Count (Ne)	
		Warp	Weft	Warp	Weft
100% Cotton Satin Fabric	210	84	46	60/1	60/1

2.2.1 Preliminary laboratory test methods

Firstly, each softener was applied as 5 different concentrations, as 20, 30, 40, 50 and 60 g/l by a laboratory type foulard (Mathis) using impregnation technique with liquor pick up %80. After impregnation drying was made at 110°C.

Silk protein (sericin&fibroin) was applied to cotton fabric by a pad-dry method. In order to investigate softness properties, the results were compared with other quaternary macro-silicone, quaternary micro-silicone, macro-silicone and micro-silicone softening agents.

Inorganic salts were applied as 4 different concentrations, as 1, 3, 4 and 5 g/l by the foulard with liquor pick up %65. After impregnation, drying was made at 100° C.

Chitosan solutions were applied as 4 different concentrations, as 0.5, 1.0, 1.5, and 2.0% in at 2% acetic acid solutions then padded to cotton fabrics by foulard using impregnation technique with liquor pick up %90. Drying was made at 80°C and cure was made at 150°C for 5 minutes.

Imidazole derivatives without formaldehyde were applied as 4 different concentrations, as 40, 75, 100, 125 g/L using impregnation technique with liquor pick up %65. After impregnation drying was made at 110°C and cure was made at 160°C for 1 minute.

Finishing applications were done both at laboratory scale and industrial scale. At first all chemicals with different concentrations were applied to the fabrics separately at the laboratory. Then chemicals and determined optimum concentrations were chosen for combined applications to obtain multifunctional cotton fabric.

2.2.2 Single step laboratory methods for combination usage of chemicals

As a result of preliminary laboratory trials, chemicals and their optimum concentrations were chosen for combination usage of chemicals to obtain multifunctional cotton fabric in a single step.

Selected chemicals and their optimum concentrations were given in Table 2 for combination usage. Only one optimum concentration was selected for the imidazole derivative because its higher amounts have negative effect on the hand feeling.

Multifunctional finishing was made as follows:

- Treatment without curing: cotton fabrics were padded through solutions containing 2 or 3 chemicals with a wet pick up 70% and dried at 110° for 3min.
- Treatment with curing: cotton fabrics were padded through solutions containing 2 or 3 chemicals with a wet pick up 70%. Drying was made at 110° for 3 min and the fabrics were cured at 100-180°C for 1 min.

To obtain multifunctional effect in a single step, different combinations were tried on the fabric. Experimental design was carried out for combination studies according to selected chemicals and treatment method (Table 3).

Table 2. Chemicals and their optimum concentrations

Chemicals	Concentration (g/L)	Concentration (g/L)
Silk protein (SP)	20	40
Macro silicon (MS)	20	40
Imidazole derivative (RR) ((RR)	75	
Chitosan (CHT)	0,5	1,5
Inorganic salt (AgP)	1	4

Table 3. Used Combinations for experimental design

Method	No	Chemical	Concentration (g/L)
Padding +Drying	1	SP	20
		AgP	1
	2	SP	40
		AgP	1
	3	MS	20
		AgP	1
	4	MS	40
		AgP	1
	5	SP	20
		CHT	0.5
	6	SP	40
		CHT	1.5
Padding +Drying+Curing	7	SP	20
		RR	75
	8	SP	20
		CHT	1.5
	9	SP	20
		CHT	0.5
		RR	75
	10	MS	40
		CHT	0.5
		RR	75
	11	SP	20
		AgP	1
		RR	75
	12	MS	40
		AgP	1
RR		75	
13	SP	40	
	CHT	1,5	
14	SP	20	
	CHT	0,5	
15	SP	40	
	CHT	1,5	
	RR	75	

Silk protein (SP), Macro silicon (MS), Imidazole derivative (RR), Chitosan (CHT), Inorganic salt (AgP)

2.2.3 Industrial Scale Studies

According to the results of laboratory studies, the conditions given in Table 4 were determined for the studies to be carried out at the industrial scale and these results were compared with the present recipes which mill has already applied in min. 2 steps. Table 4 shows the recipes which were decided to use for industrial scale.

Table 4. Industrial scale studies and used chemicals

Method	Industrial Scale Studies No	Chemical	Concentration g/L
Padding +Drying	Mill 1	SP	40
		AgP	1
	Mill 2	SP	40
		AgP	4
Padding +Drying+Curing	Mill 3	SP	20
		AgP	1
		RR	75
	Mill 4	MS	40
		AgP	1
		RR	75
	Mill 5	SP	40
		AgP	1
		RR	75
	Mill 6	MS	40
		CHT	0,5
		RR	75
	Mill 7	SP	40
		CHT	1,5
		RR	75
	Mill 8	SP	40
		AgP	4
	Mill 9	SP	40
		AgP	4
		RR	75
	Two steps	Mill 10 (Mill Real Recipe)	1. Strial
Padding +Drying+Curing	2.		
	3.		
	4.		
	5. tep- RR		
+	4		
Padding +Drying		2. Step- AgP	

2.3. Tests and Analyses

Stiffness properties of fabrics using a Shirley Bending Rigidity Test device were measured according to the ASTM D1388 Standard (8). Bending length was measured and flexural rigidity was calculated.

Antibacterial activities of the fabric sample were evaluated against *S. aureus* (SA) (as gram positive bacteria) and *E. coli* (EC) (as gram-negative bacteria) by Agar Diffusion Method AATCC Test Method 100-2007(9).

The wrinkle recovery angles (WRA) of fabric samples were measured by M&S P 22 (10). The WRA of specimens in warp (W) and weft (F) directions was tested separately, and the WRA (W+F) values of specimens were evaluated.

Ironability of fabrics was determined by M&S P91 (11). This test method is designed for evaluating the smoothness appearance of fabric specimens after home laundering. Evaluations were performed using a standard light source and viewing area by rating the appearance of specimens in comparison with appropriate reference standards.

The washing process was made in order to check the washing stability of the samples. The washings were made

repeatedly in an Atlas Launder-O-Meter Instrument by ISO 105-C10:2006 Standard (12). The treated fabric samples were washed repeatedly up to 10 and 20 times and the duration of the washing cycles was 30 minutes. In order to prevent any negative effects of detergent, washings were carried out in a soap solution with a concentration of 5 g/l, at 60°C, with a liquor ratio of 50:1. After washing, the samples were rinsed in cold pure water, squeezed and dried at room temperature.

The CIE whiteness index of the samples was measured using Hunter Lab Color Quest II spectrophotometer (Hunter Lab, USA).

The hydrophilicity (absorbency) of fabrics was measured by AATCC 79-2014 standard (13). In this standard, a drop of water was allowed to fall from a fixed height onto the taut surface of a test specimen. The time required for the specular reflection of the water drop to disappear was measured and recorded as wetting time.

One-way analysis of variance (ANOVA) was applied to the test results for the statistical evaluation. The one-way analysis of variance (ANOVA) is used to determine whether there are any significant differences between the means of two or more independent (unrelated) groups (although you tend to only see it used when there are a minimum of three, rather than two groups). The ANOVA produces an F-statistic, the ratio of the variance calculated among the means to the variance within the samples. The null hypothesis will be that all population means are equal; the alternative hypothesis is that at least one means is different (14).

3. RESULTS AND DISCUSSION

3.1 Preliminary tests for determining optimum concentrations

For this group of experiments, several different silicone softeners and natural silk protein were tested separately for obtaining a soft touch finishing. The statistical evaluation of the P value which is smaller than 0.05 shows there is difference between softening chemicals. Natural silk protein (sericin&fibroin) gave better touch finishing when compared with silicones.

For the antibacterial effect, chitosan, inorganic salts (silver) and silk protein (sericin&fibroin) were tried for obtaining a soft touch finishing. An inorganic salt (silver) gave the best antibacterial effect. Silk protein (sericin&fibroin) also was given to get good antibacterial activity.

To get the easy care effect, imidazole derivative without formaldehyde were tested as well as chitosan for WRA. When easy care finishing was analyzed, a significant difference was observed between the chemicals. Therefore, it is necessary to examine the effect of a combination usage of chemicals.

3.2 Results and Discussion of Single Step Laboratory Scale Studies for combination usage of chemicals

The results of studies performed by using different finishing chemicals at laboratory scale as one step process were

examined in terms of stiffness, ironability, crease recovery angle and antibacterial properties. Results are shown in Figure 1, 2, 3 and 4.

3.2.1 Results of Shirley Stiffness test

If Shirley hardness value increases, softness of fabric decreases and hardness of fabric increases. Figure 1 shows that trials 2, 1, 6, 11, 13, 4, 12 show good softness properties. It was concluded that silk protein (trial 2) had positive and best effect on handle of the fabric.

3.2.2 Results of Ironability test

Ironability results show smoothness appearance rate (DP) of fabrics. Most of the brands accept "Ironability value 3" for easy care properties.

Figure 2 shows that commercial product such as imidazole derivate (trials 7, 9, 10, 11, 12) provided required easy care effect and also this effect could be also obtained by using silk protein (trial 8).

3.2.3 Results of Crease Recovery Angle

If the angle is greater, the fabric has fewer tendencies to crease. Crease recovery angle "200" is an acceptable value for easy care properties. When the figure 3 was examined, it could be determined that the highest value for angle was gained with trial 11.

Figure 3 shows that commercial product such as imidazole derivative generally have better crease recovery angle as well as silk protein. Fabric samples showed resistance to crease with trials 10, 11, 12, 15.

It can be also observed that angle results of trials 2, 7, 8, 9 were less than the accepted value "200".

3.2.4 Results of Antibacterial Test

According to results shown in Figure 4, the highest antibacterial effect was obtained with trial 11 including inorganic salt and silk protein. Figure 4 shows that silk protein has antibacterial effect as well as softness properties. Silk protein increases antibacterial effect by min. %20.

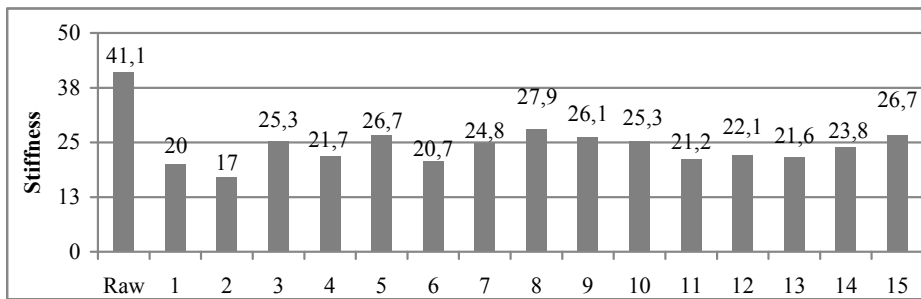


Figure 1. The results of Shirley Stiffness test

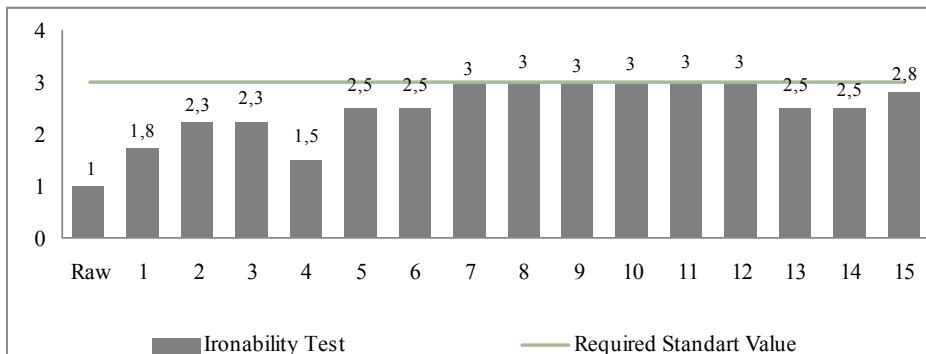


Figure 2. The results of Ironability test



Figure 3. The results of Crease recovery angle

According to results from Figure 4, trials which are treated with inorganic salt had perfect results to *K.Pneumonie* bacteria and acceptable results to *S.Aureus* bacteria.

3.3 Results and Discussion of Industrial Scale Studies

The results of studies performed in an industrial scale were examined in terms of stiffness, ironability, crease recovery angle and antibacterial properties. Results are shown in Figure 5, 6, 7 and 8.

3.3.1 Results of Shirley Stiffness Test

From Figure 5, it is concluded that 40 g/l silk protein (mill 6) has the best value in terms of handle of the fabric.

3.3.2 Results of Ironability Test

Required "top easy care value 3" have been provided with padding + drying + curing steps. Required "easy care value

2,5" is achieved by the use of the silk protein with only padding + drying steps.

With the addition of curing step to silk protein, DP value was increased to 3 (mill 5).

3.3.3 Results of Crease Recovery Angle

Required crease recovery angle value (200) is achieved by padding+drying+curing steps.

Silk protein shows 195 degree angle value (mill 2) with only padding+drying steps. With addition of curing process (mill 5), the angle of the wrinkle was increased to 206 degrees.

3.3.4 Results of Antibacterial Test

Figure 8 shows that silk protein (mill 7) had antibacterial effect but the best value is achieved by antibacterial agent.

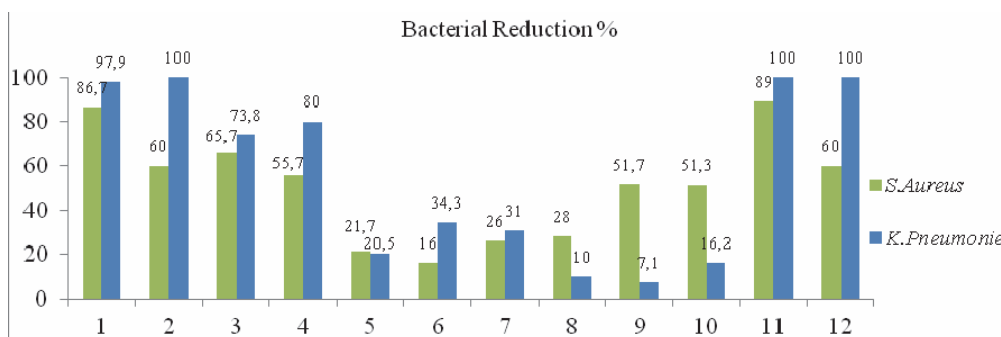


Figure 4. The results of the antibacterial test

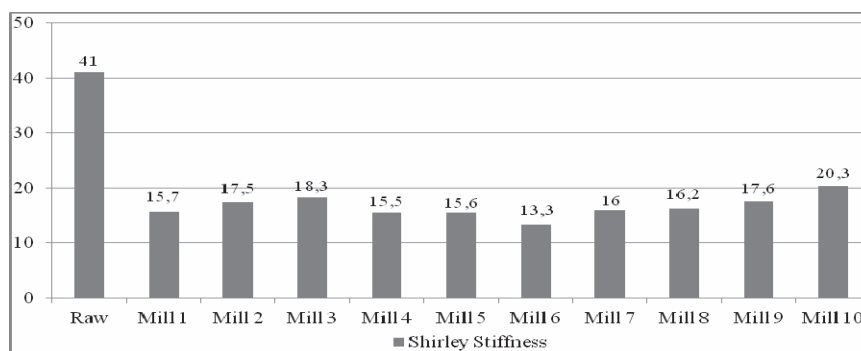


Figure 5. Results of Shirley Stiffness test

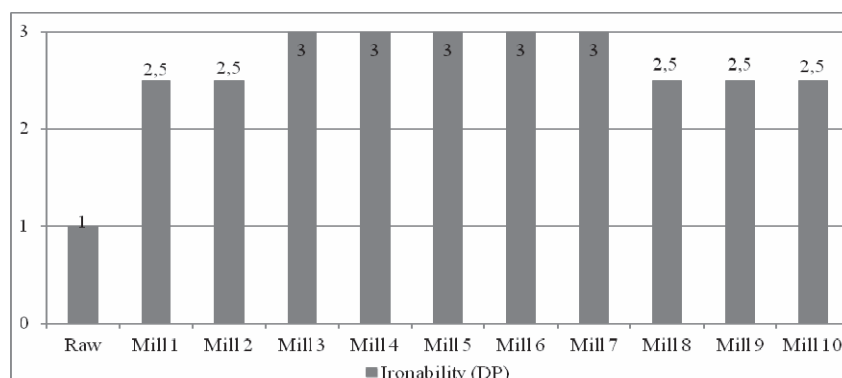


Figure 6. The results of Ironability test

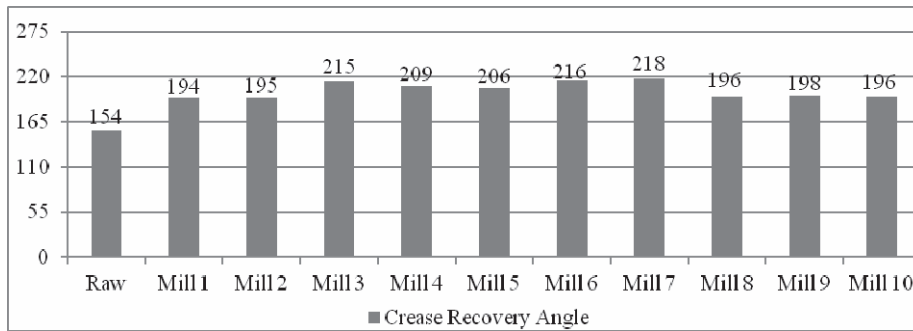


Figure 7. The results of Crease recovery angle

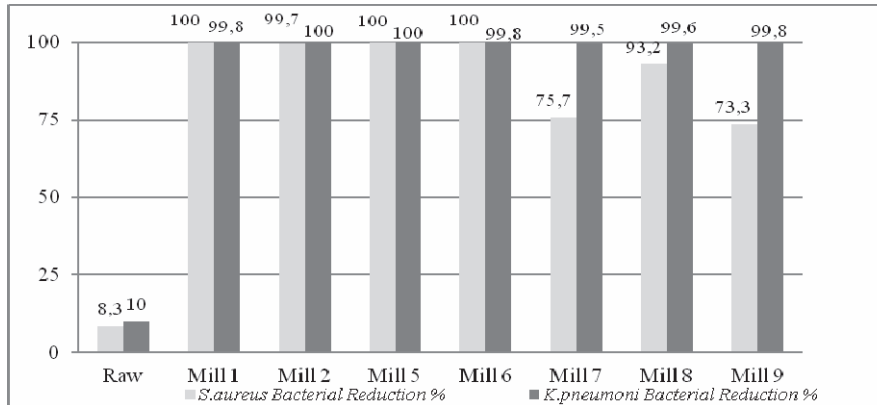


Figure 8. The results of the antibacterial test

3.3.5 Whiteness and Hydrophilicity Test

Hydrophilicity results are changing between 11-15 seconds. All hydrophilicity results are acceptable and almost the same.

When the whiteness index values were considered, it was seen that fabrics treated by chitosan had 80 Berger degrees. On the other hand, there were noticed no significant differences between other recipes which were changed 134-145 Berger degrees.

3.3.6 Results of Industrial Scale Studies After Multiple Washings

It is so important that durability of multifunctional effect so washing tests were performed to the recipes which have better results. For this purpose, durability properties such as Shirley stiffness and antibacterial effect were examined after 20 times washings.

If Shirley hardness value increases, softness of fabric decreases and hardness of fabric increases. Results show that when number of washes increases, softness of fabric decreases. The best value was obtained with Mill 6 in terms of softness. The recipe of Mill 6 included 40 g/l silk protein. After 10 times washing, softness value decreased approximately %7 in Mill 6. After 20 times washing, softness value decreases approximately %19 in Mill 6.

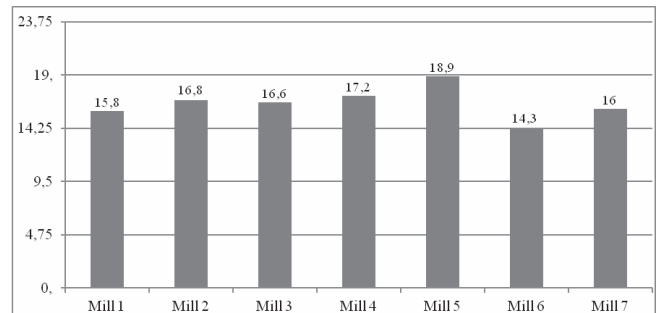


Figure 9. The results of the Shirley stiffness results after 10 times washings

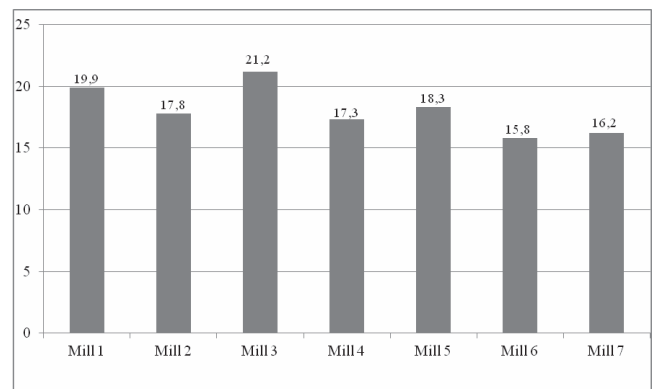


Figure 10. The results of the Shirley stiffness results after 20 times washings

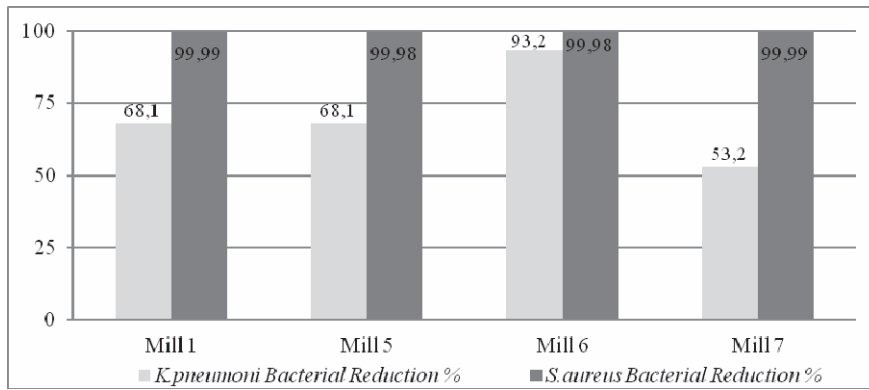


Figure 11. Antibacterial result after 20 times washing

Wash-resistant antimicrobial effect was achieved with recipe mill 6.

4. AN EVALUATION IN TERMS OF COST AND PERFORMANCE PROPERTIES

As a results of industrial scale studies, it was observed that recipe "mill 6" provided desired effect in terms of softness, antibacterial and easy care. Technical details of the mill 6 recipe are presented in Table 5.

Chemicals were impregnated as combination together by only one step method then dried and then finally cured. Used chemicals are compatible with each other. This recipe has been shown that better performance in terms of softness, easy care and antibacterial properties when it is compared with the present recipe (mill 10) which is used in the mill as two steps impregnation method. Furthermore it was determined that all properties have at least 20 wash-resistant effects.

The cost of the mill 6 which carried out in the industrial scale is 0.18 euro /m. The cost of company present recipe is 0,39

euro /m. When recipe mill 6 is compared with present recipe mill 10 in terms of cost, it was found that 50% of cost savings. The cost details of finishing processes are given in Table 6.

There is also at least 50% reduction in processing time. Total processing time is 80 minutes for present finishing processes of the company.

Company process time for 1000 m. fabric (one batch):

Easy care finishing including curing: 40 minutes

Antibacterial finishing = 20 minutes

Silk protein finishing = 20 minutes

After this work it was given multifunctional properties to the fabrics with a single step impregnation and curing time. Total process time was reduced to 40 minutes.

Table 5. Technical details of the studied recipe "mill 6" and company present recipe "mill 10"

Method	Mill No	Used chemicals	Concentration (g/L)
One Step	6	Silk protein (SP)	40
Padding+Drying+Curing		Inorganic salt (AgP)	4
		Imidazol derivative (RR)	75
Two steps	Mill 10 (Mill Present Recipe)	1. Step- RR	75
Padding +Drying+Curing		+	
Padding +Drying		2. Step- AgP	4

Table 6. Cost comparison of the studied recipes and company present recipes

Finishing	Chemical Cost (euro/m)	Workmanship Cost (euro/m)	Energy cost (euro/m)	Total cost (euro/m)	Total (euro/m)
Easy care finishing	0,06	0,02	0,06	0,14	Present Recipe 0,39
Antibacterial finishing	0,08	0,01	0,03	0,12	
Silk protein finishing	0,09	0,01	0,03	0,13	
Mill 6	0,14	0,01	0,03	0,18	
Mill 7	0,1	0,01	0,03	0,14	

5. CONCLUSION

Statistical analysis of the P value which is smaller than 0.05 shows there is difference between the new cost and time, single step method was found to be advantageous according to the present method.

In this study, different types of silicones and silk protein (sericin&fibroin) which is natural product were used as softeners. Literature review about silk protein (sericin&fibroin) indicated that the natural product has antibacterial properties, as well as softness. Therefore antibacterial and easy care properties of silk protein (sericin&fibroin) were also examined. As a result of softening process, silk protein (sericin&fibroin) showed much better softening properties compared with silicones. Silk protein (sericin&fibroin) was increased the wrinkle recovery angle by approximately 20 degrees and increased

ironability value by approximately "1" scale. Antibacterial test results of silk protein (sericin&fibroin) showed that it decreased *K. pneumoniae* bacteria by 93%.

The combination of chemicals used in the present study successfully resulted in multifunctional finishes such as antimicrobial, easy care and soft hand. This attempt of applying multi-functional finishes to woven fabrics will pave a new way to textile industry in both satisfying the needs and requirements of their customers while providing cost savings and competitive advantages.

ACKNOWLEDGEMENTS

The authors would like to thank Ministry of Science, Industry and Technology for their support within 0345.STZ.2013-2 coded Santez project that was carried out with Söktaş Dokuma A.Ş.

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