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Synthesis and Characterization of Polyvinylimidazole and Investigation of its Antipilling Effect on Different Fabrics

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ABSTRACT

Pilling is one of the major concerns in textile industry. To improve pilling values of the fabrics, some methods have been reported. One of which is chemical finishing. In this study, these chemical finishing methods have been used. A key differentiator of this study is that the polymer employed as an anti-pilling agent was synthesized by the researchers of this study and does not negatively affect the hydrophilicity and brightness of fabrics. As most anti-pilling chemicals, polyvinylimidazole (PVIM) was synthesized and applied to fabrics to reduce the pilling tendency. 1.5-2 degrees improvement was observed in the pilling values ranged 4.5-5.0, which means there were no pills on the fabric surface. It has also been proven that PVIM can be used as an anti-pilling agent without negative effects on fabrics in terms of hydrophilicity, brightness and hand. The PVIM was named RUCO-PLAST EPG 18042 to be included in Rudolf-Duraner's product list.

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KEYWORDS

Pilling, anti-pilling, hydrophilicity, brightness, polyvinylimidazole

1. INTRODUCTION

Pilling is an undesired defect of textile fabrics, consisting of a surface characterized by a number of roughly spherical masses made of entangled fibers [1]. Pilling occurs as a result of the abrasion of fabric surface occurring during washing and wearing of fabrics. Mainly, rubbing is seen in garment areas near pockets and collars, so pills are mostly found in these areas [2]. Anchor fibers that have longer fibers on the fabric surface fracture easily and the pills wear off from the fabric surface [3]. By this reason, cotton (CO), wool, and nylon 6 fibers that have anchor fibers do not have pilling problems. Stronger anchor fibers do not fracture easily, thus their pills do not easily wear off. Pilling occurs on the fabric surface for polyester (PES) or nylon 66 because polyester or nylon 66 staple fibers consist of these stronger anchor fibers areas [4].

As a result of literature review, the most important factors increasing pilling tendency are determined. The type of fibre used in a fabric is a vitual factor [5]. Gintis and Mead have worked at this topic and have ranked different synthetic fibres according to their pilling tendency [6, 7]. When the polyester content increases in a polyester/cotton blended fabric, the pilling increases [8]. It has been determined that the pilling tendency of fabrics produced from viscose fibers is higher than raw fabrics produced from lyocel and modal fibers due to their fiber structural properties. Therefore, among the selected fabrics, there are also blended fabrics containing viscose [9]. One of the crucial factors effecting pilling tendencies is the type of yarn used in the fabric. Candan et al. has reported that a fabric knitted with cotton ring-spun yarns has more pilling performance than fabric with open-ended rotor-spun yarns [10]. The other important factor that affects the pilling

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tendency is the fabric type. Over the last few years, there has been growing interest in treated fabrics due to its simple production technique, low cost, high levels of clothing comfort and wide product range [11, 12].

Yarn sequence modifies the pilling tendency of the fabrics. Because of its loose structure knitted fabrics have more pilling tendency compared to woven fabrics. So, improving anti-pilling values of knitted fabrics is more difficult [13].

Pilling formation is more common in blended fabrics and it is more difficult to improve their pilling values. The most common example is fabrics made of polyester/cotton blends. In these fabrics, weaker cotton fibers are pulled out and separated from the surface as a result of abrasion. Meanwhile, the stronger polyester fibers are only broken and still seized with the other end in the fabric. Both types of fibers form pills that remain strongly associated with the fabric surface as a result of entanglement caused by abrasion [14].

To improve pilling values, some methods have been reported [14-16]. One of which is chemical finishing [17-19] Enzymes are often used as anti-pilling chemicals in this method [20-22]. They can provide sufficient pilling improvement in blended fabrics such as PES/CV, PES/CO, CO/CV. On the other hand, they are not effective enough for all viscose (CV) fabrics. Körlü et al. investigated the effect of cellulase waste on the cellulosic fabrics. The results showed that cellulose enzymes are more effective for cotton fabrics than viscose fabrics [23, 24]. Apart from enzymes, different anti-pilling chemicals have also been used. The negative effects of all these current anti-pilling agents are that they are expensive or give hard hand to the fabrics. In spite of the fact that many different chemicals have been reported to be anti-pilling chemicals [25], having only anti-pilling property is not enough for textile industry. The fabric that has been treated with anti-pilling chemicals should also exhibit other desired properties [26]. It should maintain its hydrophilicity, have a good hand and be non-yellowing. For these reasons, an alternative anti-pilling chemical is required to solve the pilling problem. The difference of the anti-pilling polymer synthesized by the researchers of this study is that it reduces the tendency of pilling without creating negative effect on fabrics in terms of hydrophilicity, hand, and brightness. The name of the synthesized and characterized anti-pilling polymer is polyvinylimidazole. To determine the effect of PVIM as an anti-pilling chemical, applications and tests had been made on many different fabrics. It was also investigated whether PVIM negatively affects the hydrophilicity and brightness of the fabrics or not. In short, PVIM is a functional polymer and has been proven to be used as an anti-pilling agent for different types of fabrics without adverse effects. Thus, the pilling problem of fabrics is eliminated, making it possible to produce better quality products and indirectly reducing energy, production and operating costs [27].

2. MATERIAL AND METHOD

2.1. Material

2.1.1 Chemicals

Vinylimidazole (VIM), benzene, 2M AIBN in toluene were used for synthesis of polyvinylimidazole (PVIM). These chemicals were supplied from Merck.

2.1.2 Fabrics

Fabrics used in the study are determined: woven fabric-1 (W-1), woven fabric-2 (W-2), woven fabric-3 (W-3) and knitted fabric (K-1). These fabrics have different fiber composition and colour. Microscopic method was used to determine which fibers are present in fabrics by qualitatively. The brand of the microscope is Olympus and the model is BX51. After the determination of the fibers in the fabrics by microscopic method, their percentages in the fabrics were identified by chemical method according to the ISO-1833-11. The fibers in the fabrics and their percentages are provided in the Table 1. Images of fabric surfaces were taken with the digital surface microscope (LEICA brand, DVM6 model) at x44 magnification. Images are shown in Table 1.

As can be seen from the Table 1, W-1 consists of CV and PES fibers. The percentages of these fibers were determined as 78% CV and 22% PES. For W-2, PES and EL fibers were seen under the light microscope. The percentages of these fibers were determined as 66% PES, 32% CV and 2% EA by chemical method. CV, PES and EL fibers were found for W-3. The percentages of these fibers were determined as 64% PES, 32% CV and 4% EA. Only CO fibers were seen for K-1, it appears that K-1 entirely composed of CO fabrics.

2.2 Method

2.2.1 Synthesis of PVIM

To obtain sufficient polymer and to understand the correctness of the method, reactions were repeated for 7 times. For all experiments polymer can be synthesized in success. Lots of methods were investigated and then polymer was synthesized with a method close to reported in the literature [28-34]. 15 ml of VIM (0.166 mmol) was dissolved in 30 ml of benzene (0.34 mmol). 3 g of 2 M AIBN in toluene (0.016 mmol) was added as a catalyst. Reaction was carried out at 65°C under nitrogen atmosphere. After 4 hours PVIM (Figure 1) was obtained as white solid. Using vacuum distillation benzene was removed at pressures of 500–600 mmHg and temperatures of 50°C-55°C.

Table 1. Properties of fabrics used in the study

Article	Colour	Yarn sequence	Fiber composition	Surface image of fabric under digital surface microscope
W-1 (woven fabric-1)	dark pink	woven	78% CV 22 % PES	
W-2 (woven fabric-2)	green plaid pattern	woven	66% PES 32% CV 2% EA	
W-3 (woven fabric-3)	black	woven	64% PES 32% CV 4% EA	
K-1 (knitted fabric-1)	orange	knitted	100% CO	

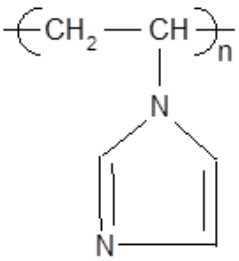

Figure 1. Structure of PVIM

2.2.2 Characterization of polymer

To characterize polymers FT-IR (Shimadzu, IR-Prestige-21), NMR (JEOL-ECZ500R), UV-Visible Spectrophotometer (Shimadzu, UV-1700, PharmaSpec), DSC (Differential scanning calorimetry) (Perkin–Elmer DSC 4000 equipment) and elemental analysis (Perkin Elmer 2400 Series II) were used.

2.2.3 Finishing process

PVIM solutions were prepared at concentrations of 10, 20, 30, 40, 50 g/l and applied to fabrics by padding method. The brand of the foulard machine used in the applications is Ataç and the model is F-350. In this machine, the treated fabrics were passed through 3 bar pressure to obtain the pick up value of nearly 70%. After this process, the fabrics were dried at 130 °C on the Mathis-stenter frame (Mathis

brand, PTC 96 model). Dried fabrics were kept in the condition room for one day. These fabrics were reserved for hand and hydrophilicity testing. For the pilling test, all the above mentioned processes were repeated and a further batch was prepared.

To observe yellowing effect, PVIM, was applied to white treated fabric. After application process, fabric was exposed to 170 °C. Then, whiteness of the fabric was tested by using Datacolor 600™ method.

2.2.4 Measuring pilling values of fabrics treated with PVIM

To determine pilling values of the fabrics lots of methods can be used [24, 35-41]. Martindale pilling tester device which is one of the mostly preferred was used for this study. All fabrics were tested according to the ISO-12945-2 method and the pilling performance was evaluated after 2000 cycles by using Martindale pilling tester device.

2.2.5 Determination of pilling values of fabrics treated with PVIM

Subjective method was used to predict fabric's pilling tendency. Fabrics treated with PVIM received the score of 5 out of 5. This means no piling in the fabric. For reference, a score of 4 would translate to very little pilling, 3 is reserved for moderate pilling. In a fabric with a pilling score of 2, pilling is clearly visible. The score of fabrics with very intense pilling is 1.

2.2.6 Determination of hydrophilicity values of fabrics treated with PVIM

While determining the hydrophilicity values of the fabrics, the water absorption capacity of the fabrics is evaluated. For this purpose, standard method: AATC 79 is used. In this method, time is meassured by using a stopwatch. Stopwatch starts immediately after the water is dropped on the fabric with a pipette and stopswhen the fabric absorbs the water drop. Elapsed time is recorded and noted as the hydrophilicity value of the fabric. Hydrophilicity measurements were carried out in this way on all applied fabrics. Thus, the effect of anti-pilling chemicals on the water absorbency values of the fabric was investigated.

2.2.7 Evaluation of whiteness values of fabrics treated with PVIM

Datacolor 600™ device was used to perform the whiteness test. To investigate the yellowing effect of PVIM on treated fabrics, white fabric was treated with 50 g/l PVIM. After application, the fabric was exposed to 170 °C for 1 minute in a Mathis-stenter frame. The whiteness value of this fabric was measured in Berger unit using Datacolor device. The whiteness value of the untreated fabric in Berger unit was also measured. The obtained whiteness values of untreated fabric and fabric treated with 50 g/l PVIM were compared.

2.2.8 Evaluation of hand of fabrics treated with PVIM

The hand of treated fabrics with PVIM were compared with their untreated ones. Thus, the effect of anti-pilling polymer on the hand of fabrics was investigated. The hand is evaluated according to the feeling it gives to the person at the momentof taking the fabric between the fingertips. As a result of this feeling, the fabric can be expressed as soft, slippery, bulky or thin. Although the hand is relative and varies from person to person from time to time, the favorite hand is usually the same. When the hand of the treated fabrics were compared according to their untreated ones, in consultation with many people, the hand effect of PVIM was evaluated in similar.

3. RESULTS AND DISCUSSION

3.1 FT-IR Studies

The desired peaks and adsorbents were obtained in the samples in the literature [42] and it was proved that the polymers were successfully synthesized. The FT-IR spectra of the PVIM polymers and the VIM monomer are illustrated in Figure 2.

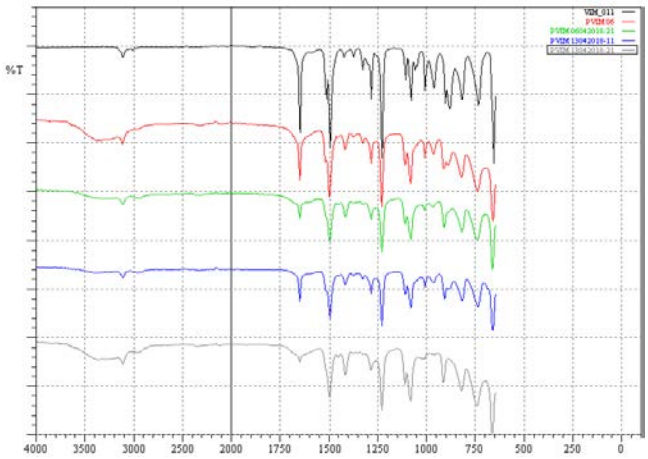

Figure 2. FT-IR spectrum for VIM and PVIM samples

In the FT-IR spectrum of the VIM homopolymer, the following peaks were detected;

- C-H (ring) stretch, 3113 cm^{-1};
- C-H and CH_2 (main chain) stretch, 3008 cm^{-1};
- C-C ve C-N stretch, 1494 cm^{-1};
- (Ring) stretch, 1416 cm^{-1};
- C-H (ring) vibration in plane and C-N (ring) stretch, 1281 ve 1226 cm^{-1}
- C-H (ring) vibration in plane, 1107 cm^{-1};
- C-H (ring) vibration in plane and (ring) stretch, 1080 cm^{-1};
- C-H (main chain) vibration in plane and C-C stretch 1045 cm^{-1};
- (Ring) stretch and vibration in plane, 912 cm^{-1};

- C-H vibration out of plane and (ring) vibration in plane, 818 cm^{-1};
- C-N stretch and (ring) vibration out of plane, 737 cm^{-1}
- (Ring) vibration out of plane 656 cm^{-1}

Peaks shown in the figure prove that PVIM is synthesized successfully.

3.2 NMR Studies

H atoms in PVIM structure are numbered and illustrated in Figure 3.

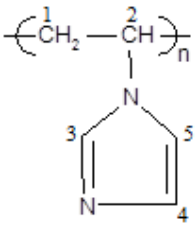

Figure 3. Numbered representation of H atoms in PVIM structure

The ^1H NMR spectra of the polymer is provided in Figure 4. Peaksprove that PVIM was synthesized successfully. When ^1H NMR spectrum of PVIM taken in deuterated dimethyl sulfoxide (DMSO-d6) is examined, peaks are obtained as below. Methylene (-CH_2) protons in the main chain are multiplet between 2.49-2.50 ppm and 2H (1), (-CH) proton in the main chain is between 3.24-3.39 ppm as a triplet and 1H (2), (-CH) protons of the imidazole ring are doublet between 4.85-4.87 ppm and 1H (5), (-CH) protons of the imidazole ring are doublet between 5.45-5.48 ppm and 1H (4).

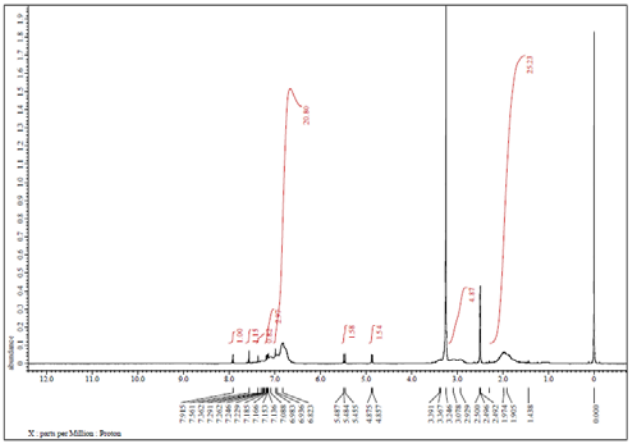

Figure 4. NMR spectrum for PVIM

These characteristic peaks seen in figure indicate that homopolymerization has taken place and PVIM synthesis is conducted successfully.

3.3 UV-Visible Spectrum

Solutions of PVIM, which was synthesized in toluene medium, were prepared in deionized H_2O to obtain UV-visible region spectra and the electronic transitions of polymers were examined. The UV-Visible region of the polymers was observed with a sharp band. λ_{max} value of the polymer is determined as 216.8 nm (Figure 5). In the literature, some researchers which are synthesized and characterized PVIM are reported λ_{max} value of the polymer in deionized water is approximately 216 nm. By the way, only one peak (216.8 nm) is observed for the UV-VIS spectrum and it corresponds to the $\pi \rightarrow \pi^*$ transitions of C = C and C = N groups in the imidazole rings in the polymer structure.

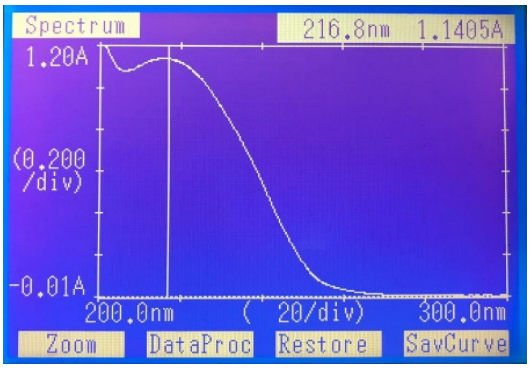

Figure 5. UV-visible spectrum of PVIM in H_2O solution

Considering that λ_{max} matches the results recorded in the literature, it is clear that the polymer was successfully synthesized.

3.4 Differential scanning calorimetry (DSC)

By using Perkin–Elmer DSC 4000 equipment, differential scanning calorimetry was performed. Measurements were taken by using 5 mg PVIM sample. Results are shown in Figure 6.

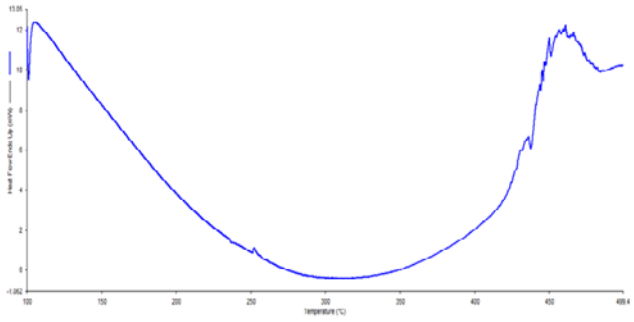

Figure 6. DSC results for PVIM

According to the DSC result, the Tm value was found to be around 440°C. In the literature, the Tm value for PVIM is reported as 440°C [43, 44]. Hence, it is proved that PVIM was synthesized successfully.

3.5 Elemental Analysis

Elemental analysis results of PVIM are shown in Table 2.

Table 2. Elemental analysis results of PVIM

Composition of the initial reaction mixture (mol%)			Elemental analysis (%)		
VIM	Benzene	2 M AIBN in toluene	Carbon	Hydrogen	Nitrogen
31.80	65.13	3.07	58.32	7.50	21.15

3.6 Effect of Polymer on Pilling Performance

Martindale pilling device was used to understand pilling tendency of treated fabrics. After that, these treated fabrics were evaluated by subjective method to determine pilling values. Concentrations of PVIM solutions were determined according to type and improvement of fabrics. PVIM applications on different fabrics has been done. The results of pilling values in these fabrics are provided in the tables below.

Table 3. Pilling results of fabric W-1 treated with different concentrations of PVIM

Artical	PVIM amount (gram/liter)	W-1 Pilling degree*
A1	0	2-3
A2	10	3
A3	20	3
A4	30	3
A5	40	4
A6	50	4-5

*Degree of pilling 5: means no pilling; 1 means very severe pilling

As can be seen from Table 3, untreated W-1 fabric has a pilling value of: 2-3. This value is significantly low and needs improvement. Because of its fiber composition made from PES and CV fiber, low pilling value is expected. PVIM treated fabrics has better pilling value. 40 g/l PVIM application is enough for W-1 fabric in order to achieve acceptable pilling degree.

Table 4. Pilling results of fabric W-2 treated with different concentrations of PVIM

Artical	PVIM amount (gram/liter)	W-2 Pilling degree
A1	0	3
A2	10	3.5
A3	20	3.5
A4	30	4-5
A5	40	5
A6	50	5

It is clearly demonstrated from Table 4, that PVIM contributes anti-pilling properties of W-2 fabric. When untreated W-2 fabric has a pilling value of: 3, 10 g/l and 20 g/l PVIM solutions improve pilling values and 30 g/l, 40 g/l and 50 g/l PVIM solutions have very positive effect on treated fabrics. 30 g/l PVIM application is sufficient for W-2 fabric to achieve acceptable pilling degree. On the other hand, with increasing amount of PVIM concentration, better values were obtained. And for fabrics treated with 40 g/l and 50 g/l PVIM solutions, the best pilling degree of 5 was achieved.

Table 5. Pilling results of fabric W-3 treated with different concentrations of PVIM

Artical	PVIM amount (gram/liter)	W-3 Pilling degree
A1	0	2-3
A2	10	3
A3	20	3
A4	30	4
A5	40	4-5
A6	50	4.5-5

Like W-1 and W-2 fabrics, W-3 untreated fabric has a pilling degree of 2-3. This low value is expected, but needs to be improved. All woven fabrics (W1, W2 and W3) used in this study, are composed of PES and CV fiber. These fiber blends cause pilling problem and it is difficult to increase their pilling values. Even so, with functional polymer PVIM, desired pilling results were achieved. As can be seen in the Table 4, 30 g/l PVIM application is enough to be obtain a good pilling value. However with increasing amount of PVIM application, values are better and nearly no pill was seen on the fabric surface.

Table 6. Pilling results of fabric K-1 treated with different concentrations of PVIM

Article	PVIM amount (gram/liter)	K-1 Pilling degree
A1	0	2-3
A2	10	3
A3	20	3-3.5
A4	30	3-3.5
A5	40	4
A6	50	4-5

For K-1 fabric, high concentrations of polymer solution had to be used. Because of the structure and low pilling grade of the knitted fabric, 50 g/l PVIM was applied to obtain a good pilling value. As can be seen, after treatments nearly 1.5-2 pilling grades of improvement were achieved. Also, for all fabrics, tendency of pilling decreased with the increasing application quantity of the polymer solution. In addition, surface images of PVIM treated and untreated fabrics after the pilling test were compared to prove that PVIM improves pilling values. Photographs showing the surface images of the fabrics are provided in Figure: 7-10. Lots of pills are observed on the surface of untreated fabrics while there are almost no pills on the surface of PVIM treated fabrics.

3.7 Effect of Polymer on Hydrophilicity of the Fabrics

Fabrics treated with PVIM were compared to untreated ones in terms of hydrophilicity. It is clear that PVIM has a positive contribution to hydrophilicity of the fabrics. Results are provided in the tables below.

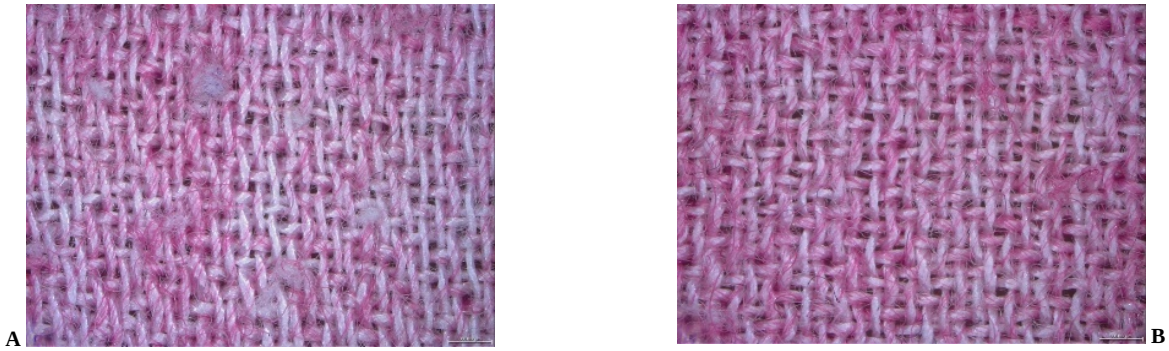

Figure 7. Surface images of W-1 A) Untreated B) 50 g/l PVIM treated

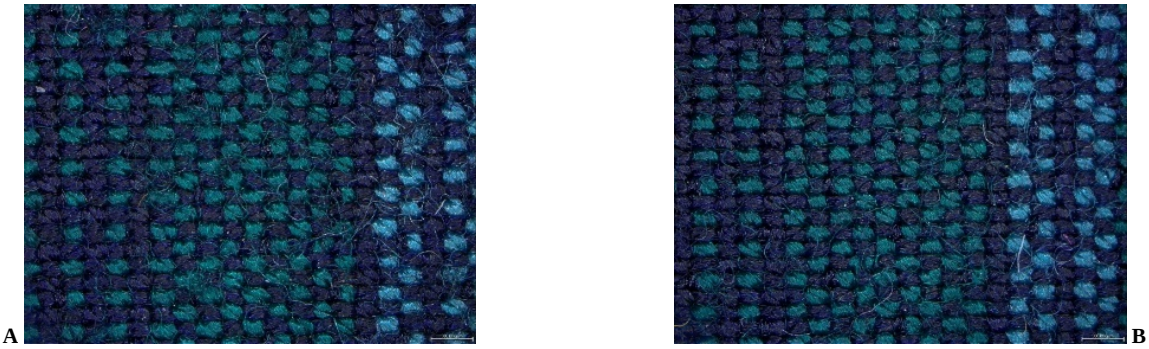

Figure 8. Surface images of W-2 A) Untreated B) 50 g/l PVIM treated

Figure 9. Surface images of W-3 A) Untreated B) 50 g/l PVIM treated

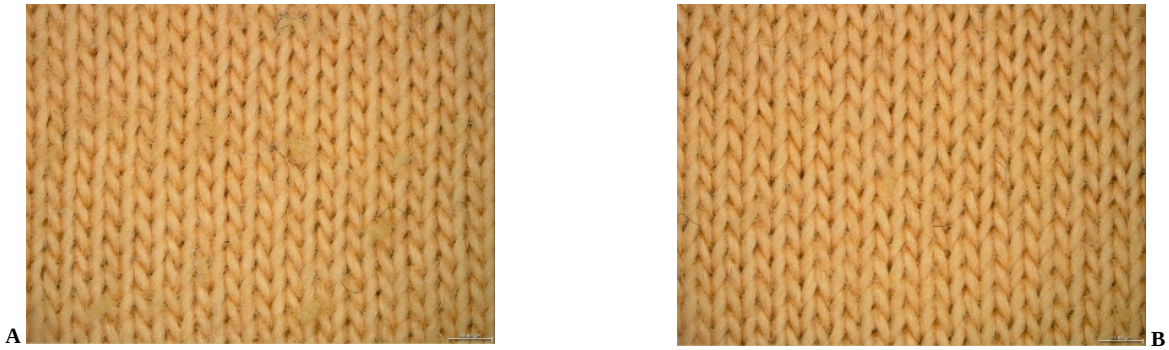

Figure 10. Surface images of W-4 A) Untreated B) 50 g/l PVIM treated

As can be seen from Table 7, hydrophilicity value of untreated W-1 fabric is 1 second. Hence, it absorbs water immediately. When PVIM applies to the fabrics, it is clearly shown that PVIM does not effect the hydrophilicity

values negatively. For all treated fabrics with different concentrations of PVIM, hydrophilicity values are recorded as a 1 second like untreated fabric.

Table 7. Hydrophilicity results of fabric W-1 treated with different concentrations of PVIM

Article	PVIM amount (gram/liter)	W-1 Hydrophilicity value
A1	0	1 s
A2	10	1 s
A3	20	1 s
A4	30	1 s
A5	40	1 s
A6	50	1 s

It is clearly demonstrated from Table 8 that PVIM contributes hydrophilicity of the W-2 fabric. When hydrophilicity value of untreated fabric is 4 minute; hydrophilicity value of fabric treated with 10 g/l PVIM: 2 minute.

Table 8. Hydrophilicity results of fabric W-2 treated with different concentrations of PVIM

Article	PVIM amount (gram/liter)	W-2 Hydrophilicity value
A1	0	4 min
A2	10	2 min
A3	20	1,5 min
A4	30	1 min
A5	40	45 s
A6	50	8 s

Hydrophilicity of W-3 untreated fabric was not good enough and was measured as 65 s (Table 9). PVIM contributes hydrophilicity of these fabrics like W-2 fabrics 10 g/l PVIM application increased hydrophilicity to 45 s. With increasing amount of PVIM application, values were better and hydrophilicity value of fabric treated with 50 g/l PVIM was measured as 12 s.

Table 9. Hydrophilicity results of fabric W-3 treated with different concentrations of PVIM

Article	PVIM amount (gram/liter)	W-3 Hydrophilicity value
A1	0	65 s
A2	10	45 s
A3	20	40 s
A4	30	35 s
A5	40	25 s
A6	50	12 s

Like W-1 fabric, untreated K-1 fabric can absorbs water immediately. Its hydrophilicity value was measured as a 1 s. When different amounts of PVIM was applied to the fabric, hydrophilicity did not affected and remained same. To conclude, PVIM helps increasing the hydrophilicity of the fabrics.

Table 10. Hydrophilicity results of fabric K-1 treated with different concentrations of PVIM

Article	PVIM amount (gram/liter)	K-1 Hydrophilicity value
A1	0	1 s
A2	10	1 s
A3	20	1 s
A4	30	1 s
A5	40	1 s
A6	50	1 s

3.8 Effect of Polymer on the Whiteness Values of the Fabrics

To investigate the yellowing effect of PVIM, white fabric was treated with 50 g/l PVIM and was exposed to 170°C for 1 minute in a Mathis-stenter frame. The whiteness value of this fabric was measured using the Datacolor 600™ device. The value obtained was 155 Berger. The whiteness value of the untreated fabric was also measured and recorded as 154 Berger. When the results obtained were compared, it was clearly seen that there was no significant difference, so PVIM does not effect the whiteness of the fabrics.

3.9 Hand of the Fabrics

A hand value of 5 out of 5 means very soft hand, a value of 1 means very hard hand. While the hand of untreated fabrics can be defined as 2-3.5, the hand of fabrics treated with PVIM can be defined as 3-4. This is the proof that PVIM does not adversely affect the hand. For some treated fabrics, hand is better than untreated ones. On the other hand, some treated fabrics are not good enough in terms of softness and slippery. For this case, softener can be added in the solution. Polymer and softener combinations were prepared and applied to the fabrics. As a result, hand value of these fabrics were 4-5 and softener did not adversely affect the pilling values of the fabrics. It can be stated that there is no drawback in adding suitable softeners as additives to improve the hand of the applied fabrics.

4. CONCLUSION

- For the aim of decreasing pilling tendency, polyvinylimidazole (PVIM) was synthesized and characterized successfully.
- Pilling values of fabrics treated with functional polymer increased by around 1,5-2 pilling grade. With the increased application amount of PVIM, pilling grades further improve. At 50 g/l PVIM treatment, for all fabrics, pilling grade of 4-5 was obtained, means there is almost no pills on the fabric surface. In fact, improvements at 40 g/l PVIM treatment could be sufficient for all fabrics.
- According to study results, PVIM didn't decrease the hydrophilicity of the fabrics similar to other chemicals

which are used in pilling improvement. On contrary, PVIM improves hydrophilicity of the fabrics.

- It was found that treated PVIM fabrics had same optical brightness value in berger unit as untreated fabrics when they are both exposed to high temperatures during drying process.
- To be included in the product list of Rudolf-Duraner, PVIM is defined as "RUCO-PLAST EPG 18042". It is proved that RUCO-PLAST EPG 18042 can be used as an anti-pilling agent for treated fabrics.

Acknowledgement

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