



TEKSTİL VE MÜHENDİS
(Journal of Textiles and Engineer)

<http://www.tekstilvemuhendis.org.tr>



Azo Boyarmaddeler ile Boyanan Tekstil Malzemelerinin Haslık Özelliklerinin İncelenmesi

An Investigation of Fastness Properties of Textile Materials Colored with Azo Dyes

Emin KARAPINAR¹, İlker AKSU²

¹ Department of Chemistry, Faculty of Arts and Sciences, Pamukkale University, 20070, Denizli, Turkey

² Deniz Textile Factory, Organized Industrial District, 20017, Denizli, Turkey

Online Erişime Açıldığı Tarih (Available online): 30 Haziran 2013 (30 June 2013)

Bu makaleye atıf yapmak için (To cite this article):

Emin KARAPINAR, İlker AKSU (2013): An Investigation of Fastness Properties of Textile Materials Colored with Azo Dyes, Tekstil ve Mühendis, 20: 90, 17-24.

For online version of the article: <http://dx.doi.org/10.7216/130075992013209002>



AN INVESTIGATION OF FASTNESS PROPERTIES OF TEXTILE MATERIALS COLORED WITH AZO DYES

Emin KARAPINAR^{1*}
İlker AKSU²

¹Department of Chemistry, Faculty of Arts and Sciences, Pamukkale University, 20070, Denizli, Turkey,
²Deniz Textile Factory, Organized Industrial District, 20017, Denizli, Turkey,

Gönderilme Tarihi / Received: 26.02.2013

Kabul Tarihi / Accepted: 16.05.2013

ABSTRACT: In this study, a series of disazo-3-methyl-1H-pyrazole-5-on derivatives (5a-e), which can also be found in previous studies, has been re-synthesized. Their structures were verified with FT-IR results and the melting points. Preliminary dyeing experiments with synthesized dyes had been applied to multifiber fabric. All dyeing experiments to Nylon 6,6 (PA6.6) and Polyester (PES) fabric were made in the laboratory and dyeing properties of textile materials used were investigated. In the end, it was found that the synthesized dyes could be applied to fabric, which is made of PA6.6 and PES. Fastnesses were examined according to ISO standards. The following were the applied fastnesses: water, perspiration, washing at 40°C and 60°C, in pool water with 20 ppm, and wet/dry rubbing. All results were compared with closest commercial dyes (T1 and T2). As the results of the fastness tests have been investigated, it can be summarized that staining values of synthesized 5a, 5b, 5c, 5d and 5e dyes, especially for nylon, acetate and PES fibers, are better than T1 and T2, whereas wet and dry rubbing values are worse being between 0.5 and 3s. Chlorinated water fastness and color changes of all dyes are equal to each other.

Keywords: Fastness, azo dyes, grey scale, HT dyeing, dyeing properties

AZO BOYARMADDELER İLE BOYANAN TEKSTİL MALZEMELERİNİN HASLIK ÖZELLİKLERİNİN İNCELENMESİ

ÖZET: Bu çalışmada, literatürde bulunan bir seri disazo-3-metil-1H-pirazol-5-on türevleri (5a-e) yeniden sentezlenmiştir. Yapıları FT-IR ve erime noktası ile doğrulanmıştır. Sentezlenen boyarmaddeler ile multifibre kumaş üzerine boyama ön denemeleri yapılmıştır. Boyarmaddelerin naylon 6,6 ve poliesteri boyayabildiği görülmüştür. Naylon 6,6 ve poliester kumaş üzerine laboratuvar ortamında boyamalar yapılmış ve bu tekstil malzemelerini boyama özellikleri incelenmiştir. ISO standartlarına göre, suya karşı, asidik ve bazik tere karşı, 40 °C ve 60 °C yıkamaya karşı, 20 ppm klorlu suya karşı, yağ ve kuru sürtmeye karşı haslıkları incelenmiştir. Bulunan haslık sonuçları bunlara yakın renk veren ticari boyalar (T1 ve T2) ile kıyaslanmıştır. Yapılan haslık testlerinin sonuçları incelendiğinde, sentezlenen 5a, 5b, 5c, 5d ve 5e boyarmaddelerinin özellikle naylon, asetat ve PES elyaflarını kirletme değerleri T1 ve T2' ye nazaran daha iyi, buna karşın yağ ve kuru sürtme değerlerinin yarım ile 3 puan arası kötü olduğu şeklinde özetlenebilir. Tüm boyaların klorlu su haslıkları ve renk değişimleri birbirine denktir.

Anahtar Kelimeler: Haslık, azo boyarmadde, gri skala, HT boyama.

* Sorumlu Yazar/Corresponding Author: ekarapinar@pau.edu.tr

DOI: 10.7216/13007592013209002, www.tekstilvemuhendis.org.tr

1. INTRODUCTION

As a result of flood disasters throughout world and especially in the Asian continent in 2010, cotton prices have been tripled by not coming across in the past, by wasting cotton harvest in Pakistan, which is one of the most important cotton producers in the world, and other reasons. This situation has troubled every sector from producer to consumer.

Under these circumstances, it can be said that synthetic fibers and synthetic dyes are suitable to develop and spread more in the long term.

In recent years, because of being a large portion of the world's fiber production of polyester fibers, synthesis of disperse azo dyes has become important. Most dyes, synthesized for this purpose, contain carboxylic diazo and coupling components. With the synthesis of azo dyes, they give dull color as a disadvantage and more brilliant colors are acquired by starting to use heterocyclic compounds [1].

In recent years, it has been focused on heterocyclic diazo and coupling components in the synthesis studies. Sabnis and his friends notes that dyes, which were obtained from a derivative of 2-aminotiyofenin with four different heterocyclic coupling compounds like enol, had given good results on polyester fiber [2]. In addition, intensive studies can be seen about synthesis of monoazo dyes, a derivative of pyrazole in literature and its properties of dyeing [3,4]. 5-aminopyrazoles can be used as starting materials for the synthesis of many polisubstitue compounds [5,6].

4- Arilazo-5-aminopyrazoles can be obtained easily from the reaction of arildiazonyum salts with active methylene compounds containing nitrile group and benefiting from clamping ring closure reaction of hydrazine derivatives of these compounds [7,8]. Since 5-aminopyrozoles are heteroaromatic amines, they can

clamp together with active methylene compounds by diazo again.

In accordance with the studies in the literature, it is found that heterocyclic monoazo dyes show good fastness properties. But these dyes are monoazo dyes. Disazo dyes containing more than one heterocyclic component in the literature are limited.

In this study, synthesis of disazo dyes containing more than one heterocyclic component is investigated and the possibility for the fabrics dyed with these dyes becoming a commercial product in the market is also examined according to washing, water, chlorinated water, perspiration and rubbing fastness called consumer satisfaction index.

Color fastness is the most important characteristic of dyed material. However, fastness of a dye is not an absolute property. It shows difference depending on both a certain factor like light, water, transpiration and also dye applied material. For instance, as fastness of a dye can be low on cotton but high on Orlon, it can be high against light but low against another factor (water, chlorine etc.) [9].

Fastness is defined as resistance power of color of a textile material against many factors during production and usage.

2. MATERIAL and METHOD

In this study, 100% PES plain knitted and 100% Nylon 6,6 plain knitted fabrics were dyed. Dyeing Graph is shown in Figure 1.

2.1 Color Fastness to Chlorinated Water

This standard (ISO 105 E03) embraces the method of determination of color fastness of dyed and/or printed textile materials against different concentrations of active chlorine used to disinfect swimming pools [10].

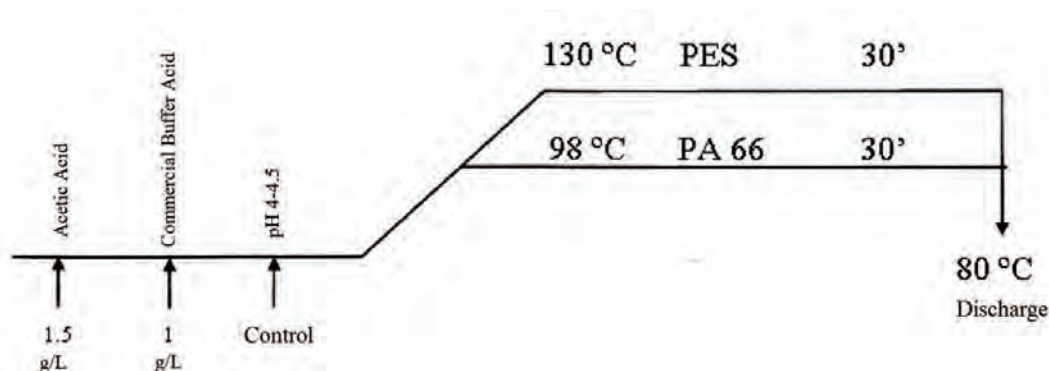


Figure 1. Dyeing Graph

2.2 Color Fastness to Rubbing

This standard (ISO 105 X12) embraces the method of determination of resistance to flow and rubbing against color of all kinds of textile materials especially floor covering materials and other pile fabrics and their products. A dry rubbing cloth and a wet rubbing cloth are rubbed to pieces of textile test materials. Color flows to rubbing pieces are evaluated with grey-scale [11].

2.3 Color Fastness to Water

This standard (ISO 105 E01) embraces the method of determination of color fastness dyed and/or printed textile materials against water. One or two features specified escort pieces in contact with test samples are immersed in water and filtrated and placed between two plates of the device under a specific pressure. Sample and escort cloth/clothes are dried. Discoloration of the test sample and flow to escort cloth are evaluated by comparing grey-scale [12].

2.4 Color Fastness to Washing

This standard (ISO 105 C06) embraces the method of determination of color fastness dyed and/or printed textile materials accordingly A1S (40 °C) and C2S (60 °C). It is a process of washing, rinsing, wringing and drying of a textile sample in contact multifiber fabric.

In order to get the results representing the normal operation in a short time, samples are kept at the appropriate temperature, alkalinity, bleaching operation and abrasive operation. Abrasive operation is done by using appropriate quantity of steel balls and low bath ratio.

The value of the color fastness of the sample with the value of color fastness by straining of multifiber is determined by grey-scales [13].

2.5 Color Fastness to Perspiration

This standard (ISO 105 E04) embraces the method of determination of color fastness of all kinds of textile products against human perspiration. The test samples, which were placed between the escort clothes, are

treated with two different solutions containing histidine and a specific pressure is applied by experiment device. The experiment samples and the escort clothes are dried by separating from each other. Color discoloration and color flow to escort cloth in each sample are evaluated to be compared with grey-scale [14].

3. EXPERIMENTAL SECTION

3.1 Chemicals Used in Experiments

The chemicals, which were used in the synthesis step, were provided from Aldrich, Merck, Acros and Sigma companies. Because they are pure enough, they were used without purification. And the chemicals that were used in dyeing and fastness tests were obtained from "Deniz Textiles".

Cotton rubbing cloth, which is suitable for Multifiber DW-ISO 105 F10 is suitable for ISO 105 F09 and the formulas of dyes (Figure 2) used as reference are T1: Setapers Yellow CE-5G (C₁₄H₁₀Cl₂N₄O₂), T2: Setapers Yellow Brown CE-RN (C₁₉H₁₈ClN₅O₄).

3.2 Devices Used in Experiments

FT-IR, Perkin Elmer spectrum, BX spectrophotometer and Electro thermal 9100 and melting point apparatus with a magnetic stirrer and ice machine (Scotsman AF-80) device that was used in order to correct structures of compounds obtained are utilized from laboratory facilities of the Pamukkale University, Department of Chemistry, Faculty of Science and Literature. Also friction device (Crock meter-James H. Heal), grey scale for color staining (James H. Heal), grey scale for color change (ADD), precision balance (Sartorius), oven (James H. Heal), (37°C ±2°C) and 5 kg weight (12.5 kPa), Gyro washing machine (James H. Heal), stainless steel balls about 0.6 cm in diameter, multifiber is appropriate for DW-ISO 105 F10, perspirometer apparatus (James H. Heal), acrylic plates in sizes 60mm x 115mm x 1.5mm, devices, plates, and tools are utilized from Deniz Textile laboratory facilities.

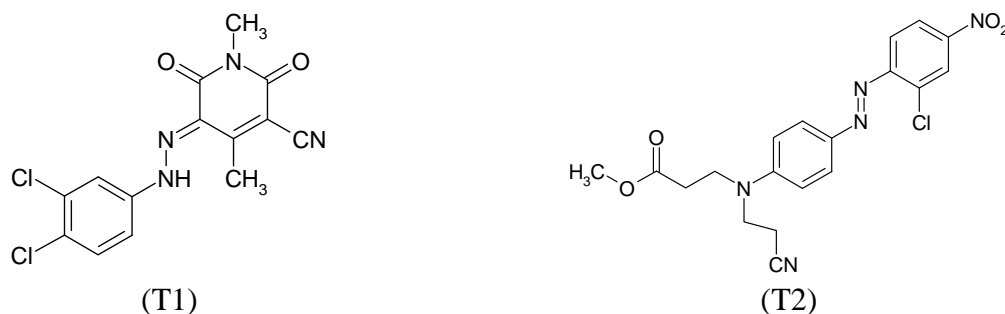


Figure 2. Formulas of Commercial dyes

3.3 Disazo Dyes Synthesis, Application and Fastness Tests

Obtained compounds of 2-arylhydrazon-3-ketiminokrotononitril (1a-1e) and 5-amino-4-arylazo-3-metil-1H-pirazol (2a-2e) are synthesized according to procedure in the literature [7,15] and obtained compounds 4-(3'-metil-4'-fenilazo-1'H-pirazol-5'-ilazo)-3-metil-1H-pirazol-5-on (5a), 4-(3'-metil-4'-(4"-nitrofenilazo)-1'H-pirazol-5'-ilazo)-3-metil-1H-pirazol-5-on (5b), 4-(3'-metil-4'-(4"-metoksifenilazo)-1'H-pirazol-5'-ilazo)-3-metil-1H-pirazol-5-on (5c), 4-(3'-metil-4'-(4"-klorfenilazo)-1'H-pirazol-5'-ilazo)-3-metil-1H-pirazol-5-on (5d) and 4-(3'-metil-4'-(4"-metilfenilazo)-1'H-pirazol-5'-ilazo)-3-metil-1H-pirazol-5-on are synthesized according to procedure in the literature and schematics of synthesis is given in Figure 3 [15].

(5a) compound is orange-yellow, mp: gray>300 °C, (5b) compound is yellow, mp: gray>350 °C, (5c) compound is red, mp: gray>315-316 °C, (5d) compound is light brown, mp: gray>360 °C and (5e) compound is orange-yellow, mp: 317-318 °C.

3.3.1 Dyeing of PA6.6 and PES with 5a-5e, T1 and T2 dyes

Since synthesized dye is in solid soil form; firstly they are powdered in a clean glass beaker with a glass rod. This pulverized dye and T1 and T2 dyes were weighed

in a sensitive balance as 0.1 gram amounts in order to dye 1% of 10 gram fabric. These dyes used for dyeing were tried to bring a homogeneous solution with water. The water solubility of the dyes is poor. These dyes used in dyeing at 80 °C were processed to make a homogeneous solution in water. Dye solutions were taken in dyeing tubes and their pH levels were adjusted to be about 4-4,5 by adding 1.5 g/L of acetic acid and 1 g/L of commercial buffer acid. Any commercial dispersing agent was not used. Then these dye solutions were completed to 100 mL with water. Fabric of polyester and polyamide 6.6 obtained from Deniz Textile were weighed to be 10 grams in sensitive balance and put in adjusted dye solutions. Immediately, tubes were closed and shaken by hand to spread dyes regularly. After that, dyeing processes were started with the Termal laboratory dyeing machines. The dyeing started with cold heated to 130°C with 1.5 °C/min increase interval and PA was heated to 98°C with 1.5°C/min intervals at the same time. This process carried on for 30 minutes. At the end of the time, the processes in each machine were cooled to 80°C and tubes were removed from the machines. After fabrics were rinsed under tap water for about 5 minutes, they were washed twice with commercial soap 1g/L about 10 minutes at 95°C and the washing process was completed with cold rinsing. Finally they were dried in the oven at 120°C.

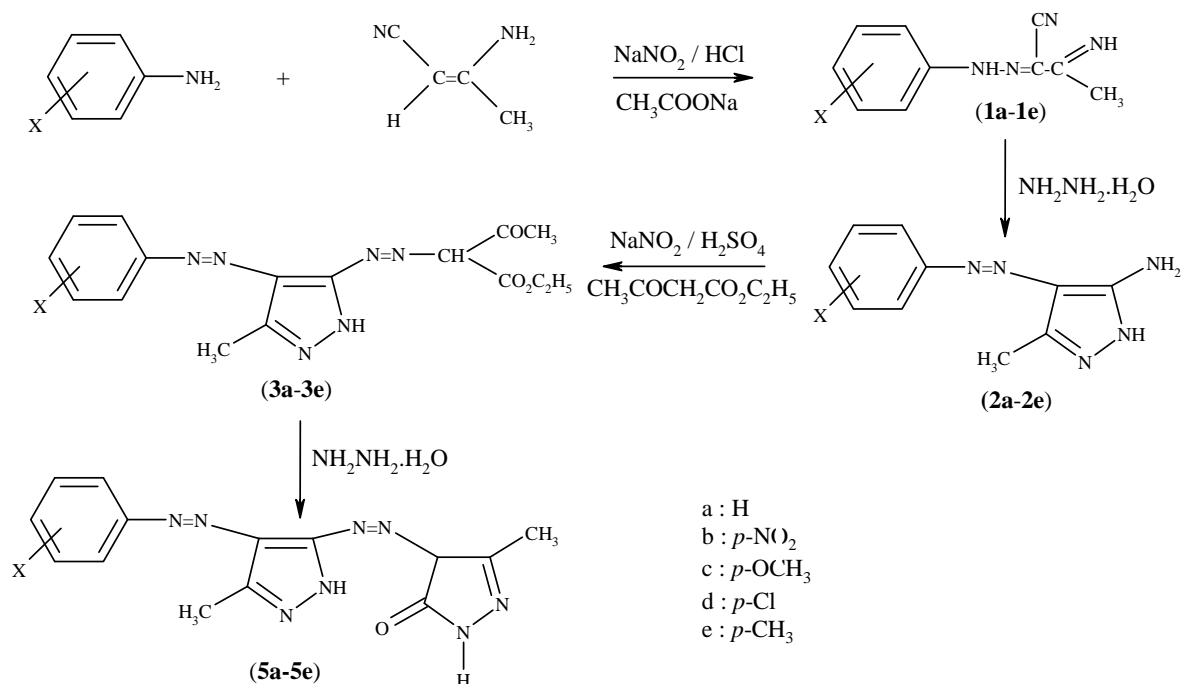


Figure 3. Schema of the synthesis of the compounds obtained.

3.3.2 Color fastness to washing test of 5a-5e, T1 and T2.

PES and PA6.6 fabrics, dyed with (5a-5e), T1 and T2, were cut in sizes of 10x4 cm and sewn to the multifiber fabrics with the short and front side of the test samples. United test samples were treated to 40°C (ISO 105 C06 A1S) and 60°C (ISO 105C06 C2S) washing fastness tests according to the parameters (A1S; Flotte Volume: 150 mL, Detergent: 4 g/L, time: 30 minute, Number of steel Ball: 10; C2S; Flotte Volume: 50 mL, Detergent: 4 g/L, Sodium Carbonate: 1 g/L, Sodium Perborate: 1 g/L, time: 30 minute, Number of steel Ball: 25, pH: 10.5±0.1) in the Gyrowash washing machine. Specific amounts of washing solutions were added to test tubes. Flotte was adjusted to the specific temperature and test samples were placed into the tubes. After tubes were closed, the device was run for the indicated time and temperature according to the parameters.

After washing, two rinsing operations were applied with 1 minute repeats in a 100 mL, 40°C of water to united test sample for every test. As a result of the rinsing operation, excess water on united test samples were received. Escort fabric and samples were dried in oven by not contacting each other with maximum 60°C temperature. After drying, color change and staining degree of samples for escort fabrics were evaluated with grey scale under D65 light (standard sun light).

3.3.3 Color fastness to chlorinated water test of 5a-5e, T1 and T2.

Test samples of PES and PA6.6 fabrics, dyed with (5a-5e), T1 and T2, in sizes 10x4 cm were prepared. Solutions containing 20 ppm active chlorine, with flotte ratio of 1/100, were taken to tubes of Gyrowash machine. After their covers were closed, test samples in test solution were shaken for 1 hour in a dark environment at 27±2 °C. At the end of the time, test samples received from solution were squeezed and dried by hanging with room temperature and less light. Dried test samples were compared with original fabric under D 65 light (standard sun light). The color difference degree between test samples and pure samples were determined by grey scale.

3.3.4 Color fastness to rubbing test of 5a-5e, T1 and T2.

PES and PA6.6 fabrics, dyed with (5a-5e), T1 and T2, in sizes 5x14 cm were taken as test samples. Bleached, unpolished and cottony fabric in sizes 5x5 cm were used as rubbing fabric.

Samples were placed in fixing part of the rubbing fastness test device and immobilized. Rubbing fabric was placed in rubbing finger of the rubbing fastness test device by not spattering, then it was immobilized with the ring. After rubbing fabric was placed, the upper arm was put on fabric and it was moved back and forth 10 times in ten seconds. The same process was done with rubbing fabric soaked by %100 AF. (For AF = %100, five drops of pure water were dropped on rubbing fabric with plastic dropper). The result of wet rubbing test was evaluated after drying by not spattering at room temperature. After the drying process, fibers that remained mechanically on the rubbing fabrics were cleaned with a sticky tape. The evaluation was done after that. The staining degrees of rubbing fabrics were evaluated with appropriate grey scale according to wet and dry rubbing test results.

3.3.5 Color fastness to water test of 5a-5e, T1 and T2.

PES and PA6.6 fabrics, dyed with (5a-5e), T1 and T2, were cut in sizes of 10x4 cm and then sewn to the multifiber fabrics in the the same size with the short and front side of the test samples. United test samples were left for 30 minutes in 100 mL pure water. Afterwards united samples were put between glass plates under the pressure of 12.5 kPa. They were kept 4 hours in the oven heated before as the temperature is 37±2 °C. Test samples between the glass plates, which were sewn only one side and not connected at other sides, were dried in open air at 60°C maximum. After the drying process, color changes of the test samples and amount of staining for the escort fibers were evaluated with grey scale under D65 light (standard sun light).

3.3.6 Color fastness to perspiration test of 5a-5e, T1 and T2.

Acidic and basic perspiration solutions were prepared as below.

a) Acidic perspiration solution: 0.5 g L-Histidine mono hydro-chloride monohydrate ($C_6H_9O_2N_3.HCl.H_2O$), 5 g sodium chloride (NaCl), 2.2 g sodium dihydrogen ortho-phosphate dihydrate ($NaH_2PO_4.2H_2O$) were mixed and pH were adjusted to 5.5 by 0.1 N NaOH.

b) Basic perspiration solution: 0.5 g L-Histidine mono hydro-chloride monohydrate ($C_6H_9O_2N_3.HCl.H_2O$), 5 g sodium chloride (NaCl), 2.5 g disodium hydrogen ortho-phosphate dihydrate ($Na_2HPO_4.2H_2O$) were mixed and pH were adjusted to 8.0 by 0.1 N NaOH.

PES and PA6.6 fabrics, dyed with (5a-5e), T1 and T2, were cut in sizes of 10x4 cm and then sewn to the multifiber fabrics in the the same size with the short and front side of the test samples. United test samples, which were wetted in two perspiration solutions —one of them was acidic and the other was basic as FO=1/50—were allowed to stay at room temperature for 30 minutes. During this procedure test samples were mixed frequently. At the end of the duration, test samples were squeezed by two glass rods in order to remove excess

water. United samples were put between glass plates under the pressure of 12.5 kPa. They were allowed to stand for 4 hours in the oven heated before as the temperature is 37+/- 2 oC. Test samples between the glass plates, which were sewn only at one side and not connected at other sides, were dried in open air at 60oC maximum. After drying process, color changes of the test samples and amount of staining for the escort fibers were evaluated with grey scale under D65 light (standard sun light).

Table 1. The fastness values of %1 PA6.6 dyeing with 5a,5c,5d,5e and T1

Applied fastnesses	Trash and color change values of multifibre																			
	Acetate					Cotton					Nylon					PES				
	T1	5a	5c	5d	5e	T1	5a	5c	5d	5e	T1	5a	5c	5d	5e	T1	5a	5c	5d	5e
A1S	4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4	4	4/5	4/5	4	4/5	4/5	4/5	4/5	4/5
C2S	2	3	4/5	4/5	4	4	4	4/5	4/5	4/5	2	2/3	3/4	4/5	3	4/5	4/5	4/5	4/5	4/5
Water fastness	4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
Acidic perspiration F.	4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4	4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
Alkaline perspiration F.	3/4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	3/4	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5

Table 2. The fastness values of %1 PA6.6 dyeing with 5a,5c,5d,5e and T1

Applied fastnesses	Trash and color change values of multifibre														
	Acrylic					Wool					Color change				
	T1	5a	5c	5d	5e	T1	5a	5c	5d	5e	T1	5a	5c	5d	5e
A1S	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
C2S	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
Water fastness	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
Acidic perspiration F.	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
Alkaline perspirationF.	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5

Table 3. The fastness values of %1 PES dyeing with 5a,5c,5d,5e and T1

Applied fastnesses	Trash and color change values of multifibre																			
	Acetate					Cotton					Nylon					PES				
	T1	5a	5c	5d	5e	T1	5a	5c	5d	5e	T1	5a	5c	5d	5e	T1	5a	5c	5d	5e
A1S	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
C2S	4/5	4/5	4	4/5	4/5	4/5	4/5	4	4/5	4/5	4/5	4/5	3	4	3/4	4/5	4/5	4/5	4/5	4/5
Water fastness	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
Acidic perspiration F.	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
Alkaline perspirationF.	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5

Table 4. The fastness values of %1 PES dyeing with 5a,5c,5d,5e and T1

Applied fastnesses	Trash and color change values of multifibre														
	Acrylic					Wool					Color change				
	T1	5a	5c	5d	5e	T1	5a	5c	5d	5e	T1	5a	5c	5d	5e
A1S	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
C2S	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
Water fastness	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
Acidic perspiration F.	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
Alkaline perspirationF.	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5

Table 5. The fastness values of %1 PES and %1 PA6.6 dyeing with 5a,5c,5d,5e, T1 and T2

Applied fastnesses	The fastness values of %1 PA6.6 dyeing										The fastness values of %1 PES dyeing				
	T1	5a	5c	5d	5e	T2	5b	T1	5a	5c	5d	5e	T2	5b	
Wet rubbing fastness	4	3	3/4	2/3	2/3	4/5	3	4/5	4	3/4	4	3/4	4/5	3/4	
Dry rubbing fastness	4/5	1/2	2/3	1/2	2/3	4/5	1/2	4/5	3	1/2	2	2/3	4/5	3	
20 ppm Fastness to chlorinated water	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	

4. RESULTS AND DISCUSSION

FT-IR spectrums of re-synthesized dyes (a series of disazo-3-methyl-1H-pyrazole-5-on derivatives) according to the literature were obtained and their melting points were determined. The results, which are appropriate with Demircali, (2006) have shown accuracy of the synthesis [15].

Preliminary dyeing experiments with synthesized dyes had been applied to multifiber fabric. All dyeing experiments onto Nylon 6,6 (PA6.6) and Polyester (PES) fabrics were made in the laboratory and dyeing properties of these textile materials were investigated. In the end, it was found that the synthesized dyes could be used in the dyeing of fabrics, which are made of PA6.6 and PES.

Fastness results of PES dyeings of (5a, 5b, 5c, 5d and 5e) are given respectively in Table 1, 2, 5 and results of PA6.6 are given respectively in Table 3, 4, and 5. Change in color was assessed using the standard grey scale, where a rating of 5 is considered as excellent and 1 is poor.

As a result of the dyeing, it was observed that 5a seems yellow T1 and their fastness values were compared with this dye. As to T2, it dyed materials in brown. As a result of the dyeing, 5b dye had given brown color similar to T2 and their fastness values were compared with T2. As a result of the dyeing, 5c, 5d, and 5e dyes had given yellow color similar to T1 and their fastness values were compared with T1.

When staining of the adjacent multifiber values of PA6.6 fabric dyed with T1 and 5a were compared, acetate and PA6.6 fabric values of 5a are better by half or 1 point than T1. Staining of cotton, PES, acrylic and wool are equal to each other, whereas wet and dry rubbing values are worse between 1 and 3 points. When staining of the adjacent multifiber values of PES dyed with T1 and 5a were compared, all staining values are equal to each other whereas wet and dry rubbing values are worse by between half and 1 point. The synthesized dyes have poor ability of dyeing and have less application to the fabric. Since they leave less waste dye into the medium, which means less degree of pollution, has been observed and wash fastness value is better.

When staining of the adjacent multifiber values of PA6.6 dyed with T2 and 5b were compared, acetate, nylon, cotton, PES and acrylic values of 5b are better by

between half and 1 point than T2 while wool staining values are equal to each other, whereas wet and dry rubbing values are worse between 1 and 3 points.

When staining of the adjacent multifiber values of PES dyed with T1 and 5b were compared, all staining values are equal to each other whereas wet and dry rubbing values are worse between 1 and 1.5 points.

When staining of the adjacent multifiber values of PA6.6 dyed with T1 and 5c were compared, all staining values are equal to each other. Acetate, nylon, and cotton values of 5c are better by between half and 2.5 points than T1. PES, acrylic and cotton staining are values equal to each other whereas wet and dry rubbing values are worse between half and 2.5 points. When staining of the adjacent multifiber values of PES dyed with T1 and 5c were compared, nylon staining at washing fastness of 5c at 60°C, are worse by 1.5 points than T1, all other staining values are equal to each other, whereas wet and dry rubbing values are worse between 1 and 3 points.

When staining of the adjacent multifiber values of PA6.6 dyed with T1 and 5d were compared, acetate, nylon and cotton values of 5d are better between half and 2.5 points than T1. Staining of PES, acrylic and wool are equal to each other, whereas wet and dry rubbing values are worse between 1.5 and 3 points. When staining of the adjacent multifiber values of PES dyed with T1 and 5e were compared, all staining values are equal to each other, whereas wet and dry rubbing values are worse between half and 2.5 points.

When staining of the adjacent multifiber values of PA6.6 dyed with T1 and 5e were compared, acetate, nylon and cotton values are better between half and 2 points than T1. Staining of PES, acrylic and wool are equal to each other, whereas wet and dry rubbing values are worse between 1.5 and 2 points. When staining values of the adjacent multifiber of PES dyed with T1 and 5e were compared, all staining values are equal to each other, whereas wet and dry rubbing values are worse between 1 and 2 points.

Chlorinated water fastness values and color changes of all dyes are equal to each other. When dyes synthesized for dyeing process were solved in water and tried to prepare a homogeneous solution, it was observed that 5a and 5b as against T1 and T2 had resolved difficultly, hadn't formed homogeneous solution and other dyes had given close results to T1 and T2.

Light irregularities were observed in all dyeings with synthesized dyes. This result can be interpreted as since resolution of dyes is weak, it caused staining.

As the results of the fastness tests have been investigated, it can be summarized that staining values of synthesized 5a, 5b, 5c, 5d and 5e dyes, especially for nylon, acetate and PES fibers, are better than T1 and T2, whereas wet and dry rubbing values are worse between half and 3 points.

It can be thought that since dyeing capabilities of synthesized dyes are weak, result of this situation reflects to fastness tests and finally staining comes out good.

The fact that wet and dry values is worse, is an indicator that there are a lot of dyes on the fabric surface and this situation shows dyeing ability of the dyes is weak and molecule sizes of the dyes are not appropriate.

NOMENCLATURE

FT-IR	:Fourier Transform Infrared	HT	: High Temperature
PES	: Poliester	D65	: Daylight 6500 K
PA	: Poliamid	T1	
ISO	:International Organisation For Standardization	T2	
mp	: Melting point		

ACKNOWLEDGEMENT

Authors thank to the Research Foundation of Pamukkale University for the financial support (BAP, 2010FBE025) and Deniz Textile Factory.

REFERENCES

- Karci, F., Sener, N., Yamac, M., Sener, I., Demircali, A., (2009), "The synthesis, antimicrobial activity and absorption characteristics of some novel heterocyclic disazo dyes", *Dyes and Pigments.*, 80(1): 47-52.
- Sabnis, R.W., Rangnekar, D.W., (1990), "Synthesis of 2-azo-3-cyano-5-carbethoxy thiophene derivatives and their application on polyester fibres", *J. Chem. Tech. Biotechnol.*, 47(1): 39-46.
- Karci, F., (2005), "Synthesis of disazo dyes derived from heterocyclic components", *Coloration Technology.*, 121(5): 275-280.
- Hanna, M.A., Girges, M.M., Fadda, A.A., (1992), "New Dye-stuffs for Polyester Fibres-Synthesis and comparative Tinctorial Behaviour of 3-Alkyl-4-aryl hidrazono-N¹-picolinoyl-2-pyrazolin-5-ones and their isomeric pyrid-3-and-4-yl Analogues". *J. Chem. Tech. Biotechnol.*, 55(1): 9-16.
- Abdel-Latif, F.M., Barsy, M.A., Elbadry, E.A., Hassan, M., (1999), "New routes for novel pyrazolo[3,4-b][1,6]-naphthyridine, pyrazolo [3,4-b] pyridine and pyrazolo [3,4:2,3] pyrido [6,1-a] benzimidazole derivatives", *J. Chem. Res. (S).*, 12: 696-697.
- Elagamey, A.G.A., Mohamed, F., Eltaweel, A.A., (1991), "Nitriles In Heterocyclic Synthesis -Synthesis of Pyrazolo[1,5-a]Pyrimidine, Pyrazolo[1,5-c][1,2,4]Triazine And Pyrazolo[4,3-e][1,2,4]Triazine Derivatives". *J. Prakt. Chem.*, 333(2): 333-338.
- Ho, Y.W., (2005), "Synthesis of some new azo pyrazolo[1,5-a] pyrimidine-thieno[2,3-b]pyridine derivatives and their application as disperse dyes", *Dyes and Pigments.*, 64(3): 223-230.
- Tsai, P.C., Wang, I.J., (2005), "Synthesis and solvatochromic properties of some disazo dyes derived from pyrazolo[1,5-a]pyrimidine derivatives", *Dyes and Pigments.*, 64(3): 259-264.
- Ozcan, Y., (1984), *Textile Fibers and Dyeing Technique*, Istanbul University Publications, No:3176, Istanbul.
- TS 837 EN ISO 105-E03/April, (2006): *Textile- Color Fastness Tests- Volume: E03: Color fastness to chlorinated water (swimming pool water)tests methods*, Ankara.
- TS 717 EN ISO 105-X12/ April, (2006): *Textile- Color Fastness Tests- Volume:X12: Color fastness to rubbing test methods*, Ankara.
- TS 396 EN ISO 105-E01/ April, (2006): *Textile- Color Fastness Tests- Volume:E01: Color fastness to water test methods*, Ankara.
- TS 7584 EN ISO 105-C06/November, (1989): *Color fastness test methods for dyed and/or printed textile products-Colour fastness to domestic and commercial laundering process*, Ankara.
- TS 398 EN ISO 105-E04/ April, (2006): *Textile- Color Fastness Tests- Volume:E04: Color fastness to perspiration test methods*, Ankara.
- Demircali, A., (2006), "Synthesis Of New Disazo Dyes And Investigated Of Their Absorption Properties", M. Sc. Thesis in Chemistry, Pamukkale University, Graduate School of Education Sciences, Denizli.