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Low Temperature Dyeing of Plasma Treated Luxury Fibres. Part I: Results for Mohair (Angora Goat)

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*Department of Textile Engineering, Ege University, Bornova, Izmir. Turkey Abstract

This study focused on the usage of plasma treatment for the modification of fibre surfaces to achieve the dyeing of mohair fibres at lower temperatures without causing a decrease in dye-exhaustion. The study was carried out using different gases under various powers and times. The effect was assessed in terms of colour, and the test samples were also evaluated using scanning electron microscopy (SEM). The optimum conditions of plasma treatment for improving mohair fibre dyeability are treatments carried out using Ar gas at 140 W for 60". According to the results of the experiments, it can be concluded that plasma treated mohair fibres can be dyed at lower temperatures (90 °C) and for shorter times (1 h instead of 1.5 h) with reactive dye without causing any decrease in colour yield. Dyeing kinetics were also researched in the study, and it was demonstrated that the rate constant and standard affinity of the plasma treated sample increased.

Key words: *Mohair, plasma, low temperature dyeing, SEM, dyeing kinetic.*

Introduction

Mohair fibres are found in limited regions throughout the world and are produced in small quantities. These factors combine to make the finished products very expensive, and hence mohair fibres are termed "Luxury Fibres" [1].In the domain of colour and luster, Mohair fibres have superiority when compared to wool fibres [1 - 3]. Mohair is famous for its strength, durability and shine. It is stronger and warmer than wool and is not subject to shrinking or wrinkling [4]. Mohair can be used in many items: accessories for hats, scarves, lounging boots, slippers, throws, blankets, carpeting and rugs, wigs, paint rollers, ink transfer pads, and children's toys [5]. As these fibres are also protein based, their dyeing characteristics are similar to those of wool. However, there are some differences between them. It is well known that mohair tends to lose its luster when dyed for prolonged periods at a boiling temperature, and to preserve its luster, it is generally necessary to use short dyeing cycles or low dyeing temperatures [6].

In recent years, many attempts have been made to improve various aspects of dyeing, and new technologies have been, and are still being, developed to reduce fibre damage, decrease energy consumption and increase productivity. Conventional processes, such as chlorination, do not comply with environmental legislation due to the adsorbable organo halogens (AOX) that are generated during processing [7]. Alternative surface modifications for improving wool dyeability are therefore being explored, one of which is plasma treatment. Plasma technology

is an important alternative to wet treatments because there is no water usage: Treatment is carried out in a gas phase; a short treatment time is enough; it does not cause industrial waste, and it saves energy [8].

The plasma treatments of wool [9 - 16] have already been investigated. For the reason of having a similar structure to that of mohair fibre, some of the studies carried out on wool will be summarised.

Kan *et al.* studied the influence of the nature of gas (oxygen, nitrogen, and a 25% hydrogen/75% nitrogen gas mixture) in plasma treatments on the fibre-to-fibre friction, feltability, fabric shrinkage, surface structure, dyeability, alkali solubility and surface chemical composition properties of wool substrates. After low temperature plasma (LTP) treatment, those properties of the LTP-treated substrates changed depending on the nature of the plasma gas used [9].

Kan et al. have also researched the surface characteristics of wool fibres treated with LTP using different gases, namely, oxygen, nitrogen and a gas mixture (25% hydrogen/75% nitrogen). The investigations showed that the chemical composition of a wool fibre surface varied differently with the different plasma gases used. The surface chemical composition of the different LTP-treated wool fibres was evaluated with different characterisation methods, namely FTIR-ATR, XPS and the saturated adsorption value [10]. Sun and Stylios investigated the effects of LTP on the pre-treatment and dyeing processes of cotton and wool. The contact angles, wicking properties, scourability and dyeability of wool and cotton fabrics were affected by low-temperature plasma treatments. After treatment, the wool and cotton fabric specimens showed increased hydrophilicity and improved scouring as well as dyeing processing by nearly 50% [11].

Biniaś *et al.* investigated the effect of low temperature plasma on selected properties (surface characteristic, water absorption capacity, capillarity, dyeability) of wool fibres. The selected properties of wool textiles were changed by the influence of low-temperature plasma on the fibre's surface layers, acting only on a very small thickness. The level of changes was limited by the parameters of the low-temperature plasma. The lowering of the dyeing temperature was achieved [12].

Jocic *et al.* investigated the influence of low-temperature plasma and biopolymer chitosan treatments on wool dyeability. Wool knitted fabrics were treated and characterised by whiteness and shrink-resistance measurements. The surface modification was assessed by contact-angle measurements of fibres of human hair. It was stated that after plasma treatment the whiteness degree and hydrophility of the fibres increased, and fibre dyeability was improved [13].

Sun and Stylios determined the mechanical and surface properties and handle of wool and cotton fabrics treated with LTP. This investigation showed that the mechanical properties of wool and cotton changed remarkably after oxygen plasma treatment. There were no significant differences observed between plasma treat-

ed and un-treated fabrics after scouring and dyeing [14].

Mori and Inagaki studied the dyeing properties of Argon (Ar)-plasma treated wool using the six classes of dyestuffs, i.e., acid, acid metal complex, acid mordant, reactive, basic and disperse dyes. Ar-plasma treatment greatly improved the colour yield and levelness, together with a decrease in tippy dyeing. The conditions of the plasma treatment enhanced not only the colour yield but also the anti-felting performance. The relationship between the improvement of dyeing properties by the plasma treatment and the chemical structure of the dyes was also examined. In the case of acid dyes, the effect of plasma treatment on the colour yield was more significant for milling type dyes of large molecular weight than for leveling type dye of low molecular weight. Furthermore, the hot water and rubbing fastness were improved by Arplasma treatment [15].

Demir et al. treated knitted wool fabrics with atmospheric argon plasma, enzyme (protease), chitosan, and a combination of these processes. The fabrics treated were evaluated in terms of their dveability, colour fastness and shrinkage properties, as well as bursting strength. Their surface morphology was characterised by SEM images. In order to show the fictionalisation of a wool surface after plasma treatment, XPS analysis was done. According to the results of the experiment, it was stated that atmospheric plasma has an etching effect and increases the functionality of a wool surface, which is evident from the SEM and XPS analyses [16].

This study focused on the usage of plasma treatments for the modification of fibre surfaces to achieve the dyeing of mohair fibres at lower temperatures without causing any decrease in dye-exhaustion. The study was carried out using different gases under various powers and times. The effect was assessed in terms of colour, and test samples were also evaluated using scanning electron microscopy (SEM).

Material and method

Materials

Mohair fibre of $31.47 \mu m$ mean fibre diameter, used in all the experiments, was supplied by IPLIKSAN A.Ş. / Turkey. The dyes used were Telon Blue BRL Micro (C.I. Acid Blue 324) (acid leveling

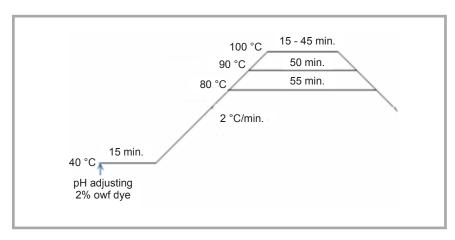


Figure 1. Dye profile of mohair dyed with different dyes at 80 °C, 90 °C or 100 °C.

dye), Telon Blue M-RLW (C.I. Acid Blue 204) (acid milling dye), Isolan Dark Blue 2S-GL (C.I. Acid Blue 193) (1:2 metal complex dye), and Realan Blue RC (reactive dye), which were kindly supplied by Dystar. All the experiments were carried out using soft mill water.

Plasma treatment

For plasma treatment, a dielectric barrier discharge (DBD) atmospheric plasma device was used. The samples were placed between the electrodes, the distance between which being 0.2 cm. Air, nitrogen and argon were used as the processing gases with a power of 70 and 140 watts, over different time intervals, namely 30, 60 and 120 seconds. It was not possible to pass fibres through the electrodes. For this reason, fibres were sewn on woven cotton fabrics, and passed through the electrodes.

Fibre dyeability

To determine the effect of plasma treatment on the dye exhaustion of mohair fibre, the treated samples were dyed with milling acid dye (Telon Blue M-RLW) to a depth of 2% at 80 °C (see *Figure 2*). Color yield (K/S) values of the dyed samples were measured with a Minolta 3600d spectral photometer (D65/10°). For optimisation of the plasma treatment statistically, a General Linear Model was used.

Alkali solubility

After determining optimum plasma treatment conditions for increasing fibre dyeability, alkali solubility values of untreated fibres and those plasma treated at optimum conditions were realised in order to determine if significant damage occurs during plasma treatment or not. The IWTO-4-60 standard test method

was used. The value was calculated as a percentage of the original mass, according to the equation given below:

Alkali solubility in % =
$$= (M_1 - M_2)/M_1 \times 100$$

where: M₁- mass of oven-dry sample before sodium hydroxide treatment, and M₂- mass of oven-dry sample after sodium hydroxide treatment.

Scanning Electron Microscopy (SEM)

The surface modification was evaluated by taking photographs of the fibres with a Phillips XL-30S FEG scanning electron microscope (SEM).

Fibre dyeability of optimised plasma treated mohair using different classes of dyes

After determining the optimum plasma treatment conditions for increasing fibre dyeability, the dyeing properties of the plasma treated mohair fibres were tested with various dye classes for different temperatures and times. Dveing procedures were carried out at a 1:30 liquor ratio and dyeing depth of 2%. The dyeing graph is given in Figure 1. The pH of dyeing chosen for leveling acid dye (Telon Blue BRL Micro) was 3 (with HCOOH), for milling acid dye (Telon Blue M-RLW) and 1:2 metal complex dye (Isolan Dark Blue 2S-GL) - 6 (with CH₃COOH), and for reactive dye (Realan Blue RC) - 5 (with CH₃COOH). In order to prohibit the affecting dyeing properties of fibres, the usage of auxiliaries (equalising agent etc.) or salts was avoided. As can be seen from *Figure 1*, the dyeings were carried out at three different temperatures (100, 90 and 80 °C), where the total dveing time was constant (90 min.). Furthermore, at 100 °C the

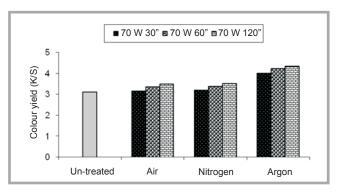


Figure 2. Effect of gas type and time of plasma treatments carried out at 70 W on the colour yield (K/S at 630 nm) of mohair dyed with 2% owf Telon Blue M-RLW.

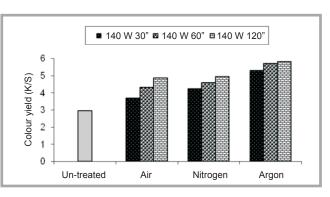


Figure 3. Effect of gas type and time of plasma treatments carried out at 140 W on the colour yield (K/S at 630 nm) of mohair dyed with 2% owf Telon Blue M-RLW.

dyeing time chosen was 15 and 45 min. After dyeing, the liquor was cooled down to 60 °C at a rate of 2 °C/min. and next the fibres taken out. Then the dyed samples were rinsed with cold (5 min.) -warm (at 50 °C 5 min.) - cold (5 min.) water, respectively, and dried. All experiments were carried out in a Thermal HT type dyeing machine.

Colour yield (K/S) and CIE L*a*b* colour space values of the dyed samples were measured with a Minolta 3600d reflectance spectrophotometer, with the illumination/observer conditions set at D65/10°. Light fastness tests according

to the ISO 105 B02 standard and washing fastness tests according to the ISO 105 C06 standard were also carried out.

Results and discussion

Optimisation of the plasma treatment

Effects of gas type, power and time on the plasma treatment efficiency of mohair fibre

Results of the experiments carried out to optimise the parameters of plasma treatment are given in *Figures 2 - 3*, and also statistical evaluation results can be seen in *Table 1*.

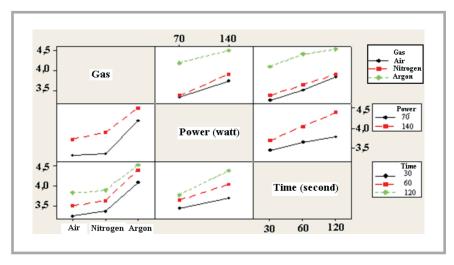


Figure 4. Interactions related to the colour yield (K/S at 630 nm) values of mohair fibres plasma treated under various conditions and dyed with 2% owf Telon Blue M-RLW.

Table 1. Variance analysis results of dyeings carried out after plasma treatments in various conditions.

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Gas	2	2,36274	2,36274	1,18137	79,02	0,000
Power	1	0,77709	0,77709	0,77709	51,98	0,000
Time	2	0,78714	0,78714	0,39357	26,33	0,000
Error	12	0,17940	0,17940	0,01495		
Total	17	4,10638				
S = 0,122270 R-Sq = 95,63% R-Sq(adj) = 93,81%						

According to the statistical evaluations, it can be stated that there are statistically important differences (p > 0.05) with respect to gas type, power and time. All results can be seen in *Figure 4* to compare.

When figures are examined, it can be understood that colour yields obtained in dyeing increase with an increase in the power and time of plasma treatment. Comparing the gases, it can be said that the best results are obtained with Ar gas.

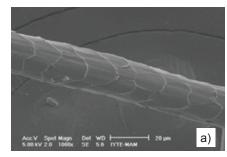
For Argon gas, the results obtained at 140 watts are better. As for the effect of the time, it can be said that, although by increasing the time from 30 sec. to 60 sec. the colour yield of dyeing increases significantly, by increasing it from 60 sec. to 120 sec. the increase in colour yield is not essential. For this reason it can be concluded that the optimum conditions of plasma treatment for improving mohair fibre dyeability are treatments carried out using Ar gas at 140 W for 60".

The increase in colour yield for plasma treated fibres can be attributed to the oxidation effect brought about by the plasma treatment of the fibre surface, including the creation of polar groups by oxidation and removing surface lipids. The formation of hydrophilic groups in the hydrocarbon chains of the lipid layer and/or its elimination makes the fibre surface more hydrophilic. Plasma pretreatment more or less removes the surface barrier present on the fibre surface, meaning that dyes can enter the treated fibres more easily in comparison with un-treated fibre [13]. Furthermore, although the wool fibre itself contained amino groups (-NH₂), further introduction of amino groups by plasma treatment may have enhanced the absorption of anionic dye during the dyeing process [10].

Evaluation of optimised plasma treated mohair fibre

After determining the optimum conditions of plasma treatment as Argon gas, 140 W and 60", the alkali solubility of the plasma treated fibre was tested at optimum conditions and compared with the un-treated sample. While un-treated mohair fibre has about 9.6% solubility, plasma treated mohair fibre has about 12.5%; however, by taking into consideration that the alkali solubility values are between 9 and 15% for undamaged wool [17], it can be concluded that fibre damage is not excessive. The increase in the alkali solubility of the plasma treated fibres may have been due to abrasion of the fibre surface after the plasma treatment, which causes holes on the fibre surface, forming a pathway for the penetration of caustic species into the fibre during the alkali solubility test [9].

In order to observe the modification that occurred on the fibre surface, SEM photographs of the un-treated and plasma treated mohair fibres were taken. It was observed that the cuticle layer of mohair fibre was damaged by plasma treatment (*Figure 5*). As is generally known, the cuticle layer shows a barrier effect for dye diffusion. The partial decomposition of this layer after plasma treatment



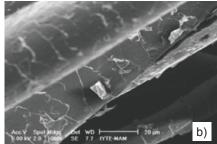


Figure 5. SEM photos of the plasma treated and un-treated mohair fibres; a) un-treated, b) plasma treated (Argon, 140 W, 60").

clearly explains the increase in fibre dyeability.

Effect of the plasma treatment on fibre dyeability with various dye classes

The colour yields of the plasma treated and un-treated fibres dyed at various temperatures and times are given in *Figures* 6 - 9.

From the figures it can be seen that the plasma treated fibres were dyed darker than the un-treated ones, but for leveling acid dye there is no difference between them in the colour yield; and also the dyeing temperature is not important (see *Figure 6*). Leveling acid dyes are small molecular dyes that they do not need

high energy for diffusion, and for this reason they can also be exhausted by fibre at lower temperatures than the boiling point. As a result, it can be suggested that acid dyes of low molecular weight should be preferred for preventing colour yield losses during low temperature dyeings; however, this will not be a good solution when high wet fastness values are desired as these dyes have fairly low wet fastness properties. However, if low temperature dyeing can be achieved with big molecular dyes without causing any decrease in colour yield, it would result in improved dyeing characteristics, such as wet fastness, of the final product.

The increase in colour yield for plasma treated fibres dyed with milling acid, 1:2

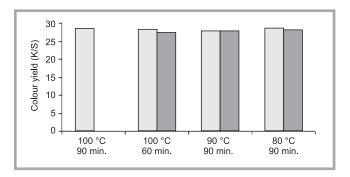


Figure 6. Colour yield results (K/S at 630 nm) for un-treated and plasma treated mohair dyed with 2% owf Telon Blue BRL Micro (acid leveling dye) at different temperatures.

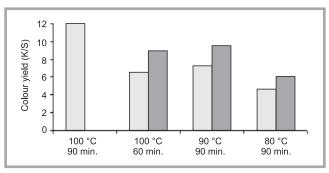


Figure 7. Colour yield results (K/S at 630 nm) of un-treated and plasma treated mohair dyed with 2% owf Telon Blue M-RLW (acid milling dye) at different temperatures.

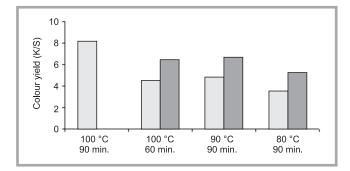


Figure 8. Colour yield results (K/S at 590 nm) of un-treated and plasma treated mohair dyed with 2% owf Isolan Dark Blue 2S-GL (1:2 metal complex dye) at different temperatures

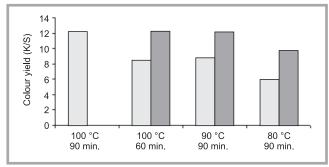


Figure 9. Colour yield results (K/S at 620 nm) of un-treated and plasma treated mohair dyed with 2% owf Realan Blue RC (reactive dye) at different temperatures.

metal complex, and reactive dye can be attributed to the oxidation effect brought about by plasma treatment of the fibre surface, including the creation of polar groups by oxidation and removing surface lipids. The formation of hydrophilic groups in the hydrocarbon chains of the lipid layer and/or its elimination makes the fibre surface more hydrophilic. Plasma pretreatment more or less removes the surface barrier present on the fibre surface, meaning that dyes can enter the treated fibres more easily in comparison with un-treated fibre [13]. Furthermore, although the wool fibre itself contained amino groups (-NH₂), further introduction of amino groups by plasma treatment may have enhanced the absorption of anionic dye during the dyeing process [10].

While the colour yields of plasma treated mohair fibres reactively dyed at 90°C are approximately equal to un-treated fibres dyed at a boiling temperature, the colour yields of plasma treated fibres dyed with milling acid dye or 1:2 metal complex dye at 90 °C are lower than un-treated fibres dyed at a boiling temperature. The reason for these differences is that even if the fibre structure becomes more diffusible to dyes because of the decomposition of the cuticle layer (see Figure 5), for big molecular dyes, such as milling acid and 1:2 metal complex, at lower temperatures (such as 90 °C) it is difficult to reach and diffuse into fibres because of low kinetic energy in the medium. From the figures it can generally be concluded that plasma treated mohair fibres can be dyed at lower temperatures (90 °C) over shorter times (1 h instead of 1.5 h) with reactive dye without causing any decrease in colour yield.

CIEL*a*b* values of the dyed fibres (untreated and plasma treated) are illustrated in *Table 2*.

If the L* values are examined, it can be seen that the L* values of un-treated fibres are higher except for leveling acid dye (Telon Blue BRL Micro). The L* value is that of lightness-darkness, and an increase in it shows that the colour is getting lighter. Furthermore the L* values for plasma treated fibres dyed at 90 °C with reactive dye are similar to those for un-treated fibres dyed at 100°C, which means that the fibres can be dyed at 90 °C with reactive dye without any decrease in colour yield. From this point of view, the results obtained are parallel with the K/S values. From *Table 1* it can be seen

Table 2. CIEL*a*b* values of un-treated and plasma treated mohair dyed with four different dyes.

Dye	Dyeing Temp Time	Sample	L*	a*	b*
Telon Blue BRL Micro	100 °C - 90 min.	un-treated	21.97	2.76	-30.91
	400.00 .00'.	un-treated	22.82	2.47	-32.06
	100 °C - 60 min.	plasma treated	22.49	2.52	-30.92
	90 °C - 90 min.	un-treated	23.10	1.49	-31.53
	90 °C - 90 mm.	plasma treated	22.33	2.85	-31.40
	80 °C - 90 min.	un-treated	22.40	1.86	-30.81
	60 °C - 90 Min.	plasma treated	22.42	2.78	-31.35
	100 °C - 90 min.	un-treated	40.12	-0.69	-37.45
Telon Blue M-RLW	100 °C - 60 min.	un-treated	46.32	-3.68	-31.86
	100 °C - 60 min.	plasma treated	43.24	-2.35	-33.39
	90 °C - 90 min.	un-treated	45.58	-3.51	-32.16
वृं घ	90 °C - 90 Min.	plasma treated	42.35	-1.10	-36.20
	80 °C - 90 min.	un-treated	50.91	-4.78	-28.66
	60 C - 90 IIIII.	plasma treated	49.06	-4.17	-29.83
	100 °C - 90 min.	un-treated	33.26	-1.20	-14.40
e P	100 °C - 60 min.	un-treated	41.28	-1.92	-13.49
ᄎ ᄀ <u>ᇤ</u>	100 C - 60 111111.	plasma treated	36.39	-1.39	-14.19
n Dark 2S-GL	90 °C - 90 min.	un-treated	40.46	-1.69	-14.26
İsolan Dark Blue 2S-GL	90 0 - 90 11111.	plasma treated	35.74	-1.45	-13.58
<u>8</u>	80 °C - 90 min.	un-treated	44.94	-2.05	-13.03
	00 C - 90 IIIII.	plasma treated	38.94	-1.65	-13.34
	100 °C- 90 min.	un-treated	34.72	-2.05	-33.26
Realan Blue RC	100 °C-60 min.	un-treated	38.96	-3.67	-31.29
	100 C-00 IIIII.	plasma treated	34.63	-1.83	-33.66
an RC	90 °C- 90 min.	un-treated	38.63	-3.02	-32.64
Real	90 C- 90 IIIII.	plasma treated	34.28	-1.56	-32.93
	80 °C- 90 min.	un-treated	47.15	-5.43	-27.10
	00 C- 90 IIIII.	plasma treated	43.27	-4.01	-30.68

that generally the differences in the a* and b* values of the colour obtained for untreated and plasma treated fibers are smaller compared to the differences in the L* values. a* values are higher for the plasma treated samples, which means the colours are redder. The b* values of the plasma treated fibers are generally lower compared to those of untreated ones, which means the colors are bluer.

In *Table 3* washing and light fastness values of the dyed samples are given.

When *Table 3* is examined, it can be seen that there are not any big differences in washing fastness among the un-treated and plasma treated fibres; however, the light fastness of plasma treated fibres is higher except for leveling acid dye (Telon Blue BRL Micro), the reason for which is that plasma treated fibres were dyed darker. Because the dye amount which is damaged by the effect of light is consistent, and if the dyeing shade is higher, the dye percentage damaged will decrease, hence light fastness values will be higher [18].

Conclusion

The textile industry is searching for innovative production techniques to improve product quality, and society requires new finishing techniques working with respect for the environment. Plasma surface treatments show distinct advantages because they are able to modify the surface properties of inert materials, sometimes with environmentally friendly devices [19]. In this investigation, an alternative ecofriendly pre-treatment is suggested for improving the dyeability of mohair. According to the results of the experiment, it can be concluded that plasma treated mohair fibres can be dved at lower temperatures (90°C) over shorter times (1 h instead of 1.5 h) with reactive dye without causing any decrease in colour yield.

Acknowledgements

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Table 3. Effect of plasma treatment on the washing and light fastness properties of the dyed mohair samples (WO: Wool, PAC: Polyacrylonitrile, PES: Polyester, PA: Polyamide, CO: Cotton, CA: Cellulose Acetate).

Dye	Sample	D T T	Washing Fastness					Light	
		Dyeing Temp Time	wo	PAC	PES	PA	СО	CA	fastness
Telon Blue BRL Micro	un-treated	100 °C - 90 min.	4	4 - 5	4 - 5	2 - 3	3	4	7 - 8
	un-treated	100 °C - 60 min.	4	4 - 5	4 - 5	2 - 3	3	4	7 - 8
	plasma treated	100 °C - 60 min.	4	4 - 5	4 - 5	2	2 - 3	4	7 - 8
	un-treated	90 °C - 90 min.	4	4 - 5	4 - 5	2 - 3	3	4	7 - 8
	plasma treated	90 °C - 90 min.	4	4 - 5	4 - 5	2	2 - 3	4	7 - 8
	un-treated	80 °C - 90 min.	4	4 - 5	4 - 5	2 - 3	3	4	7 - 8
	plasma treated	80 °C - 90 min.	4	4 - 5	4 - 5	2	2 - 3	4	7 - 8
Telon Blue M-RLW	un-treated	100 °C - 90 min.	5	5	5	4	4 - 5	5	7
	un-treated	100 °C - 60 min.	5	5	5	4	4 - 5	5	6 - 7
	plasma treated	100 °C - 60 min.	5	5	5	4	4 - 5	5	7
	un-treated	90 °C - 90 min.	5	5	5	4	4 - 5	5	6 - 7
	plasma treated	90 °C - 90 min.	5	5	5	4	4 - 5	5	7
	un-treated	80 °C - 90 min.	5	5	5	4	4 - 5	5	6
	plasma treated	80 °C - 90 min.	5	5	5	4	4 - 5	5	6 - 7
isolan Dark Blue 2S-GL	un-treated	100 °C - 90 min.	5	5	5	4	4 - 5	5	6 - 7
	un-treated	100 °C - 60 min.	5	5	5	4	4 - 5	5	6
	plasma treated	100 °C - 60 min.	5	5	5	4	4 - 5	5	6 - 7
	un-treated	90 °C - 90 min.	5	5	5	4	4 - 5	5	6
	plasma treated	90 °C - 90 min.	5	5	5	4	4 - 5	5	6 - 7
	un-treated	80 °C - 90 min.	5	5	5	4	4 - 5	5	5 - 6
	plasma treated	80 °C - 90 min.	5	5	5	4	4 - 5	5	6
Realan Blue RC	un-treated	100 °C - 90 min.	5	5	5	4 - 5	4 - 5	5	6
	un-treated	100 °C - 60 min.	5	5	5	4 - 5	4 - 5	5	5 - 6
	plasma treated	100 °C - 60 min.	5	5	5	4 - 5	4 - 5	5	6
	un-treated	90 °C - 90 min.	5	5	5	4 - 5	4 - 5	5	5 - 6
alan	plasma treated	90 °C - 90 min.	5	5	5	4 - 5	4 - 5	5	6
Rea	un-treated	80 °C - 90 min.	5	5	5	4 - 5	4 - 5	5	5
	plasma treated	80 °C - 90 min.	5	5	5	4 - 5	4 - 5	5	5 - 6

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