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Spraying Colloidal Nano TiO2 and Cross-linkable Polysiloxane onto Acrylic Carpet for Self-cleaning

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ABSTRACT

In this study, self-cleaning properties are introduced to acrylic carpet through a simple method of spraying nano titanium dioxide (NTO) onto the carpet surface. A cross-linkable polysiloxane (PS) softener is also applied to enhance the washing durability, self-cleaning properties and handle softness. The self-cleaning properties of the acrylic carpet are investigated by using Solophenyl Red GBL as a synthetic dye and black mulberry as a natural dye to stain the carpet. The PS and NTO concentrations and the type of ultra violet (UV) irradiation are designed based on the statistical approach of a central composite design (CCD). UV irradiation is used under the form of daylight for 16 h, UV-A (400 W) for 1 h and UV-C (20 W) for 3 h. The photocatalytic decolorization values (ΔE^*) of the treated carpet are measured and the optimized models are presented. The PS is capable of stabilizing the NTO particles on the surface of the acrylic carpet and helps to increase the washing durability. Also, the NTO particles can effectively photodegrade both the Solophenyl Red GBL and black mulberry dyes which act as the two coloring stains.

***Keywords*:** Acrylic carpet, Central Composite Design, NTO, Polysiloxane, Self-cleaning, Ultra Violet

1. Introduction

One of the developing usages of acrylic fiber is in carpet production. This could be due to the costly production of woolen carpets and also the lack of elasticity in other fibers, such as those used in polypropylene carpets. In order to improve carpet properties, several studies have been presented, including the reduction of static electrical charges (Kessler & Fisher, 1997; Jassal et al., 2006), recycling of carpet waste (Lee et al., 2006), decrease in pollutants and sensitivity as a result of the applied charge and aromatic powders in carpet preparation which are able to effectively transfer the static charges (Jerrim et al., 2001). In considering the effects of the application of nano particles to create multi-functional characteristics in garment formulation (Pant et al., 2011), there is a great tendency to universalize the use of nano particles in carpets. Nano particles are able to demonstrate remarkable properties, such as the removal of polluted gases (Dong et al., 2007), improvement in the thermal properties (Cai et al., 2011), creation of anti-microbial characteristics with compounds such as nano titanium dioxide (NTO; Dastjerdi & Montazer, 2010; Edwards & Goheen, 2006), zinc oxide (ZnO; Sivakumar et al., 2010) and silver (Ag; Wei et al., 2009) nano particles, N-halamine precursor (Kou et al., 2006; Parthiban, 2012), UV-protection (Gouda & Keshk, 2010), removal of colored pollutants (Lu et al., 2010), photodegradation of synthetic dyes (Li et al., 2007), self-cleaning properties (Gupta et al., 2007), and cross-linking of cellulose (Nazari et al., 2009).

The central composite design method and correlation analysis have been used in recent studies to optimize variables. Among them are the optimizing of wool dyeing conditions with

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madder and liposome (Montazer et al., 2007), photodegradation of the azo dye Reactive Black 5 (RB5), and Reactive Red 120 (RR120) in the NTO/UV system, the extraction process of Poria cocos, and Longon polysaccharides, electrochemical treatment of textile dye wastewater, production of protease, and predicting of stiffness (Tuigong & Xin, 2005).

The extensive use of polymeric agents of cross-linkable PS for the stabilizing of NTO and the preparation of anti-microbial polyester without a decrease in other desired properties, such as strength and comfort (Dastjerdi et al., 2009), addressed Ag/TiO_2 particle durability in order to improve properties such as UV-protection, stain photodegradability, and self-cleaning (Dastjerdi et al., 2010).

In this study, the self-cleaning properties of treated acrylic carpet with NTO photocatalyst particles, cross-linkable PS as a stabilizing compound under different irradiation conditions based on the statistical method of central composite design, are investigated. The influence of NTO photocatalyst on the photodegradation of Solophenyl Red GBL as a synthetic dye and black mulberry as a natural dye is quantified. The discoloration values (ΔE^*) of the irradiated samples under daylight for 16 h, UV-A (400 W) for 1 h and UV-C (20 W) for 3 h are reported, and the optimized models are presented.

2. Material and Methods

2.1 Material

Carpet with 10.5 Nm, 12 mm high acrylic fibers in piles was used. The polysiloxane (PS) CT 208 E emulsion was supplied by Wacker (Finland). Sodium carbonate was provided by Merck Co. (Germany), and non-ionic detergent (Rucogen, DEN) was composed of a fatty alcohol ethoxylate from Rudolf Chemie Co. (Tehran, Iran). The NTO photocatalyst particles with a special surface (BET) of 50±15 m/g and the average value of the particles and a particle size of 21 nm, namely, Degussa p-25 from Evonik Co., Germany, were applied. Also, a direct dye, namely, Solophenyl Red GBL (Fig. 1) from Ciba Co. (Switzerland) and black mulberry natural dye (Fig. 2) from Iran were used.

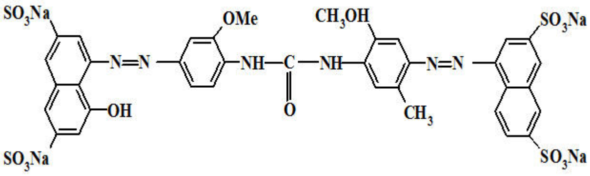


Fig. 1. Chemical structure of Solophenyl Red GBL dye

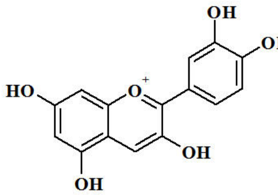


Fig. 2. Chemical structure of black mulberry dye

2.2 Instrument

An ultrasonic bath, Eurosonic 4D (350 W, 220 V, Vicenza, Italy), with a frequency of 50-60 Hz was used. A thermal oven (LO.141) from Fan Chemical Co. (Iran) was used. UV irradiations, which include daylight (Yazd City, Iran, for 16 h, on October 27-29, 2010), UV-A irradiation (400 W) from Philips (Belgium) and UV-C irradiation (20 W) from Sylvania (Belgium), were used. A reflectance spectrophotometer system (color-guide sphere, D/10° spin, Gardco, Germany) with a D65 light source was used to determine the ΔE^*. Scanning electron microscope (SEM) observations on specimens of treated samples was carried out by using a JXA-840 from JEOL Co. (Japan).

2.3 Method

The swatches of carpets (19 cm × 3 cm) were washed at 60°C for 30 min with 0.5 g/L sodium carbonate (Na_2CO_3) and 2 g/L non-ionic detergent (Rucogen DEN), and dried in ambient conditions. A stabilized suspension was obtained through various concentrations of NTO particles with the required volume of distilled water in an ultrasonic bath for 15 min. The acrylic carpet samples were treated by using a spray technique with various concentrations of NTO and then dried in ambient conditions. The post-treatment was performed via spraying a solution of PS followed by curing at 165°C for 90 s. In order to remove the unfixed particles, the treated carpets were washed at 70°C for 30 min with 1 g/L Na_2CO_3 and 1 g/L non-ionic detergent (Rucogen DEN). The treated carpets were rinsed several times and then dried at room temperature. Photodegradation evaluations of the natural and synthetic dyes were conducted through discoloration after exposure to daylight, UV-A (400 W) and UV-C (20 W) for 16, 1, and 3 h, respectively. The complete process of the carpet treatment is shown in Fig 3.

The treated carpets were placed on a flat surface and then one drop of mulberry and Solophenyl Red GBL (1 g/L) was vertically dripped onto the surface of the carpet by using a burette (50 mL), positioned 1 cm above the carpet. A color stain on the carpet surfaces was created with an approximate diameter of 1.30 cm. The dye stained carpets were then dried at room temperature. Virtually, the amounts of ΔE^* for the blank and the treated carpets were measured.

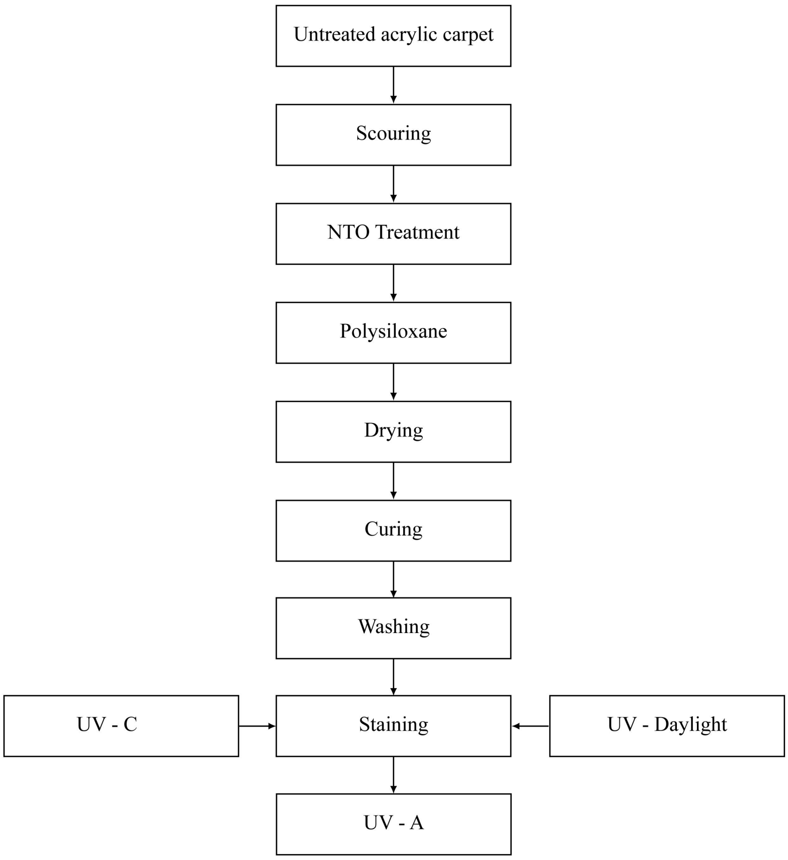


Fig. 3. The processing treatment of the acrylic carpet

2.4 Experimental Design

The central composite design was used as part of the experimental plan with three variables. These variables included the amounts of PS, NTO and different UV conditions. The ranges of these variables are: PS (0.00-4.00%), NTO (0.00-2.00%) and UV irradiation conditions (daylight, UV-400 W, and UV-20 W). Details of the design for the acrylic samples with PS in the presence of NTO are presented in Table 1 (Runs 1-13). Also, the influence of the variables on the results Y (ΔE^*) was adjusted by using the following second order polynominal function:

$$Y = b_0 + \sum b_i X_i + \sum b_{ij} X_i X_j + \sum c_i X_i^2 \quad i \geq j \quad i,j = 1,2,3 \qquad (1)$$

In this equation, b_0 is an independent term according to the mean value of the experimental plan, b_i are the regression coefficients that explain the influence of the variables in their linear form, b_{ij} are the regression coefficients of the interaction terms between variables, and c_i are the coefficients in the quadratic form of the variables.

3. Results and Discussion

3.1 Self-Cleaning Properties

The total color differences (ΔE^*) of the Solophenyl Red GBL and black mulberry stains on the carpets treated with PS and NTO under various UV irradiations are shown in Table 1, which indicate that an increase in the PS concentration means that the $\Delta E^*$of the Solophenyl Red GBL stain increases; whereas a PS above 3.00% does not have any noticeable effect on the efficiency of the NTO particles. This may be due to the thickness of the polymer layer on the NTO particles which prevents UV irradiation from affecting the particles and then results in a decrease of the photocatalyst efficiency. Also, the increasing of the NTO content in the finishing bath to 1.50% increases the ΔE^* of the Solophenyl Red GBL stained carpet. Above this NTO content, the self-cleaning effect is reduced. This could be attributed to the NTO particle agglomeration on the carpet or in the finishing bath. The agglomeration of NTO particles decreases the photocatalyst efficiency of the NTO. Also, the initial increase in the PS and NTO contents increases the ΔE^* of the black mulberry stained carpets, whereas above 3.00% PS and 1.50% NTO, the ΔE^* of the black mulberry stained carpets is decreased, similar to the Solophenyl Red GBL stained carpets.

Meanwhile, with the same content of PS and NTO under UV irradiation, higher photodegradation was observed on the black mulberry stained carpets as compared with the Solophenyl Red GBL stained carpets. This can be due to the lower stability of the black mulberry chromophores as opposed to the Solophenyl Red GBL dye against the NTO photocatalyst particles.

Three methods of UV irradiation, including by daylight, and at 400 W (UV-A) and 20 W (UV-C), were used for 16, 1, and 3 h of irradiation respectively. The order of increase in the ΔE^* for the three methods was: UV-C<UV-A<Daylight. Daylight irradiation for 16 h has the highest efficiency among all three choices. The results demonstrated that all three irradiation methods produce various chemical active species on the NTO surfaces which are then transferred to the black mulberry and Solophenyl Red GBL stains, and photodegrade the color stains. The formation of a colorless network structure as a result of the action of the silicone can act as a protective layer for the fibers in the carpet. This technique includes the use of NTO particles with a PS cross-linkable agent in two separate stages.

This finishing material can be applied by using common techniques. The PS treatment can create a polymeric layer on the fiber surface. This layer is stable against light, temperature, and chemical and microbial attacks. In this study, a remarkable control of photocatalyst material release speed from the coating layer is obtained. As a result, the acrylic carpets treated with NTO and the PS cross-linkable agent are able to effectively photodegrade the black mulberry and Solophenyl Red GBL stains.

Table 1. Central composite design for color difference (ΔE^*) of the carpets with PS and NTO under different UV irradiations

Run			Black	Mulberry		Solo- phenyl	Red	GBL
	A: NTO (%)	B: PS (%)	ΔE^*-400 W	ΔE^*-20 W	ΔE^*-Day light	ΔE^*-400 W	ΔE^*-20 W	ΔE^*-Day light
Blank	0.00	0.00	0.69	0.55	0.74	0.41	0.38	0.49
1	1.00	4.00	19.14	18.67	19.16	15.75	14.10	18.11
2	2.00	2.00	17.03	16.26	19.57	16.14	13.41	16.78
3	1.71	3.41	17.28	17.09	17.94	18.54	16.09	18.62
4	1.00	0.00	2.76	1.19	4.09	3.22	1.66	4.85
5	1.00	2.00	17.04	14.31	19.31	17.49	12.39	20.28
6	1.00	2.00	16.78	15.34	18.65	17.22	13.55	19.48
7	0.29	3.41	16.99	16.60	17.83	16.68	10.25	16.97
8	0.29	0.59	13.42	11.92	13.62	11.02	8.60	13.17
9	1.00	2.00	16.72	16.25	18.19	16.76	14.65	19.07
10	1.71	0.59	14.65	14.45	15.50	12.73	10.65	15.42
11	1.00	2.00	16.25	16.78	18.17	16.59	14.81	18.59
12	1.00	2.00	16.21	14.31	17.88	16.49	12.39	18.44
13	0.00	2.00	4.19	2.41	5.82	8.99	5.08	12.58

3.2 Statistical Analysis

To photodegrade the Solophenyl Red GBL synthetic dye, an analysis of variance (ANOVA) was carried out, and shown in Table 2. According to the ANOVA results, the fitted models of self-cleaning (ΔE^*) by using Design-Expert software are given in Eqs. 2–4, respectively.

$$(\Delta E^*)^{2.06}_{\text{UV-Sun}} = -32.28459 + 283.42853 \times NTO + 200.17493 \times PS - 103.91804 \times NTO^2 - 34.33599 \times PS^2 \quad (2)$$

$$(\Delta E^*)^{2.06}_{\text{UV-400 W}} = -101.59998 + 283.42853 \times NTO + 200.17493 \times PS - 103.91804 \times NTO^2 - 34.33599 \times PS^2 \quad (3)$$

$$(\Delta E^*)^{2.06}_{\text{UV-20 W}} = -199.43325 + 283.42853 \times NTO + 200.17493 \times PS - 103.91804 \times NTO^2 - 34.33599 \times PS^2 \quad (4)$$

To photodegrade the black mulberry stains, an analysis of variance (ANOVA) was carried out and shown in Table 3. According to the experimental design, the result is analyzed and approximate functions of self-cleaning for all of the UV irradiation were obtained by Eq. 5.

$$\Delta E^* = +0.56752 + 10.99812 \times NTO + 6.29968 \times PS - 3.62167 \times NTO^2 - 0.91667 \times PS^2 \quad (5)$$

Figs. 4a-b show the surface response of the model for the treated carpets. By using the Design-Expert software, the optimum condition (ΔE^*=19.44) with a desirability of 0.975 is found with the Solophenyl Red GBL stain (Fig. 4a) which contains 3.06% PS and 1.41% NTO and subjected to daylight irradiation. Besides that, Fig. 4b shows the optimum design point (ΔE^*=19.57) with a desirability of 0.975 for the black mulberry stain which has a PS concentration of 3.06% and NTO concentration of 1.41% , and subjected to daylight irradiation.

The reactions for the production of the electrons and holes created on the NTO particles as a result of UV irradiation and consequently the oxidation and reduction reactions in order to photodegrade the Solophenyl Red GBL are shown in Fig. 5. During the photocatalyic reaction process, NTO particles under irradiation (λ<385 nm) form a pair of electrons (e^-) and holes (h^+) on the NTO particles [Eq. 1] which have the ability to transform the water and oxygen available in the environment into superoxide ions and hydroxyl free radicals. Also, the created holes cause the production of radical hydroxyl [Eq. 2], where hydroxyl radicals absorb electrons and are changed into OH^- [Eq. 3]. More likely, the acidic conditions generated by the NTO or produced radicals lead to the breaking up of the azo groups and finally decomposing the Solophenyl Red GBL. The suggested mechanism of the dye photodegradation in the presence of H^+ is illustrated in Eqs. 4, 5, and 6.

Although the photodegradation mechanism of black mulberry has not been experimentally investigated, but a related mechanism reaction by using NTO particles is suggested in Fig. 6. Specified and well-known reactions, which use NTO under UV irradiation, are used [Eqs. 1, 2, and 3]. In the next stage, the hydroxyl anions attack the oxygen of the dye, which has a positive charge [Eq. 4], thus causing the opening of the pyran ring structure and leading to the disintegration of the chromophore group [Eq. 5]. Consequently, this causes the decolorization of the black mulberry.

The reflectance curves of the blank and treated carpets in the UV region (200-400 nm) are shown in Fig. 7. The reflectance spectrum values of the blank sample are more than the other samples; whereas the treated sample with 1.00% NTO and 3.00% PS, and subjected to UV irradiation under daylight has the least amount of reflectance. In other words, the most absorption occurred in the UV region. This confirms the creation of a suitable polymeric layer via a PS polymeric agent for the protection of the NTO particles. In the sample that contained PS, the amounts of reflectance decreased as compared with the blank sample. Therefore, NTO particles are able to absorb wavelengths less than 385 nm and the obtained acrylic carpet has a protective property against UV.

Table 2. ANOVA for quadratic model of surface response of Solophenyl Red GBL synthetic stains

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	5.791E+005	6	96516.96	36.91	<0.0001	Significant
A [NTO]	68570.63	1	68570.63	26.22	<0.0001	
B [PS]	1.895E+005	1	1.895E+005	72.47	<0.0001	
C [UV-irradiation]	1.834E+005	2	91681.73	35.06	<0.0001	
A^2	56342.39	1	56342.39	21.55	<0.0001	
B^2	98417.52	1	98417.52	37.64	<0.0001	
Residual	83675.55	32	2614.86			
Lack of FR	71334.39	20	3566.72	3.47	0.0155	Significant
Pure Error	12341.16	12	1028.43			
Cor Total	6.628E+005	38				

Table 3. ANOVA for quadratic model of surface response of black mulberry natural stains

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	624.54	4	156.14	13.60	<0.0001	Significant
A [NTO]	169.18	1	169.18	14.74	0.0005	
B [PS]	332.77	1	332.77	28.98	<0.0001	
A^2	68.43	1	68.43	5.96	0.0200	
B^2	70.14	1	70.14	6.11	0.0186	
Residual	390.37	34	11.48			
Lack of FR	384.95	22	17.50	38.74	<0.0001	Significant
Pure Error	5.42	12	0.45			
Cor Total	1014.91	38				

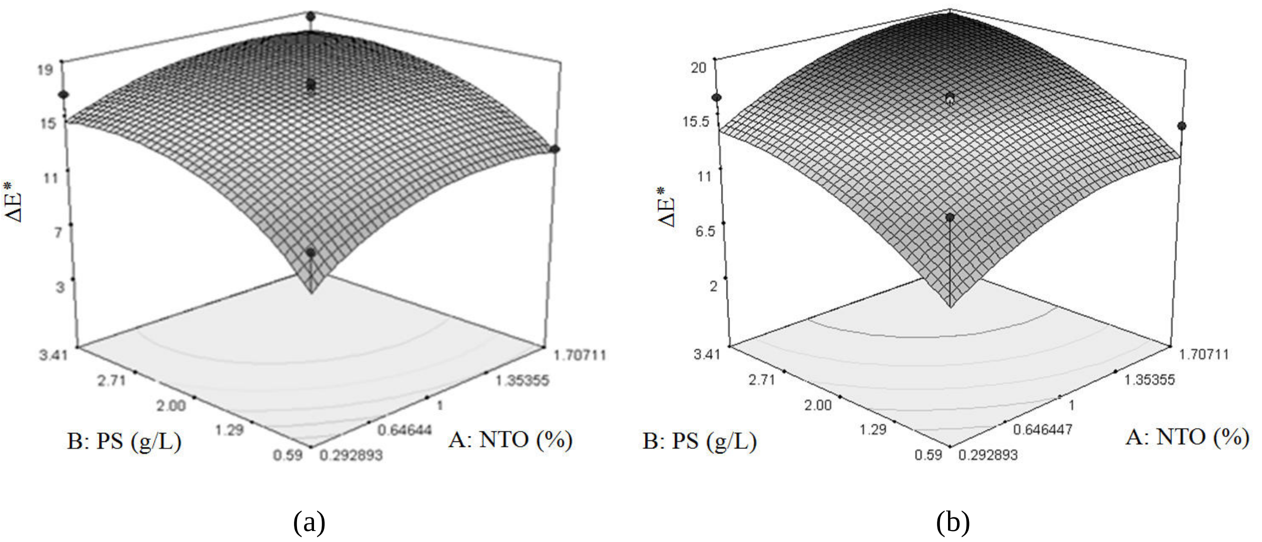
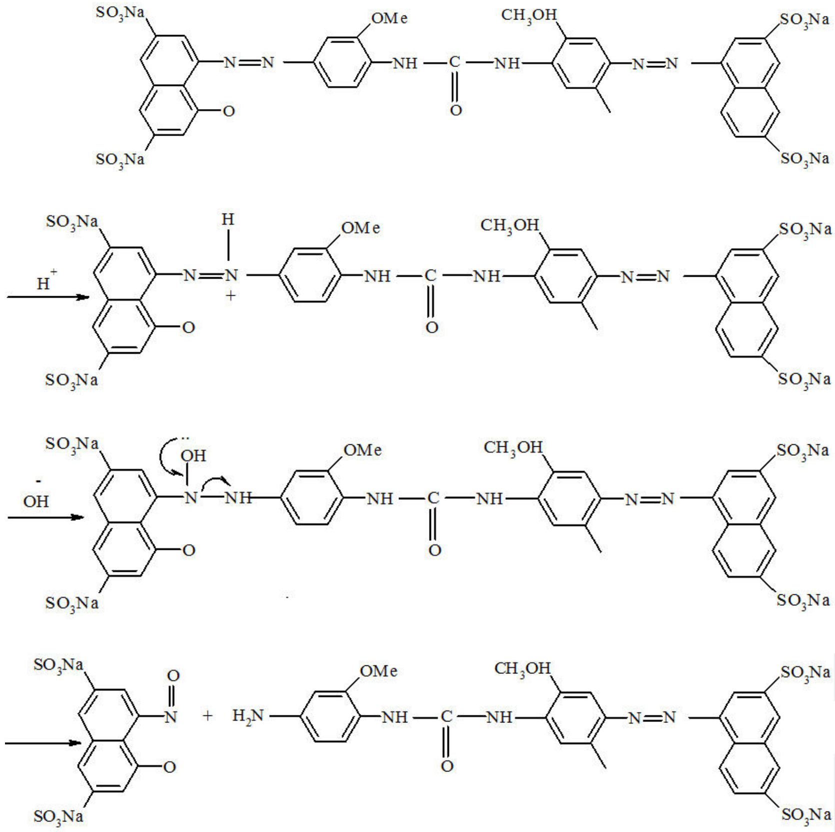


Fig. 4. Design-expert plot for Solophenyl Red GBL stain (a) and black mulberry stain, (b) with NTO and PS, and subjected to daylight irradiation

$$TiO_2 \longrightarrow e^- + H^+ \quad (1)$$

$$H^+ + O \rightleftharpoons H^+ + \dot{O}H \quad (2)$$

$$\dot{O}H + \bar{e} \rightleftharpoons OH^- \quad (3)$$



(4)

(5)

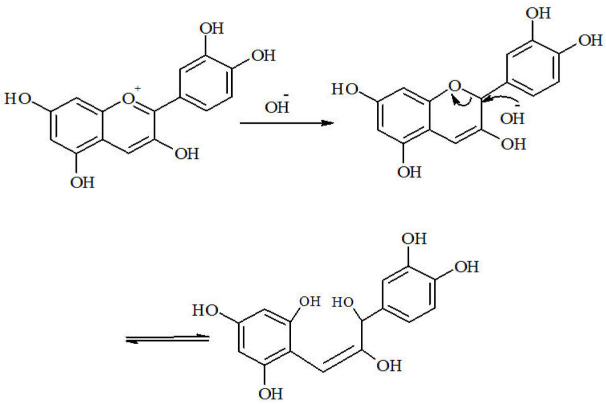
(6)

Fig. 5. Photodegradation mechanism of Solophenyl Red GBL that uses NTO photocatalyst particles

$$TiO_2 \longrightarrow e^- + H^+ \qquad (1)$$

$$H^+ + O \rightleftharpoons H^+ + \dot{O}H \qquad (2)$$

$$\dot{O}H + e^- \rightleftharpoons OH^- \qquad (3)$$



(4)

(5)

Fig. 6. Natural photodegradation mechanism of black mulberry that uses NTO photocatalyst particles

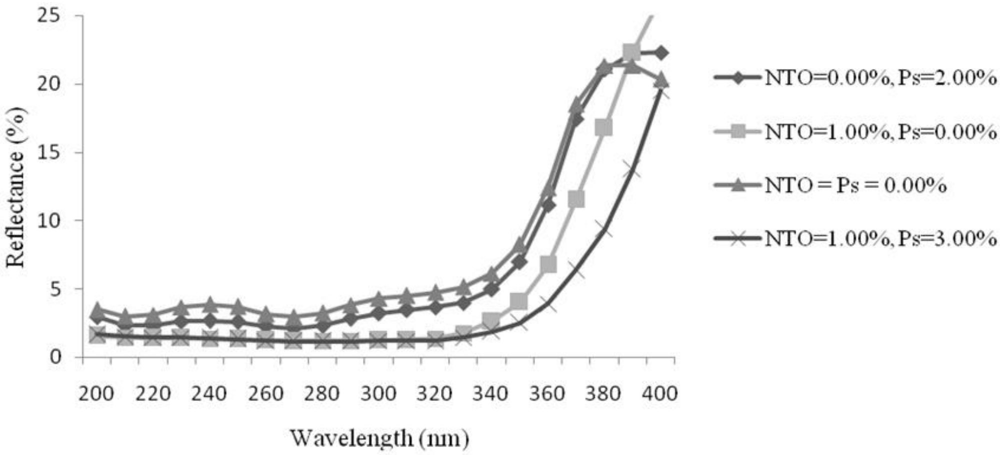
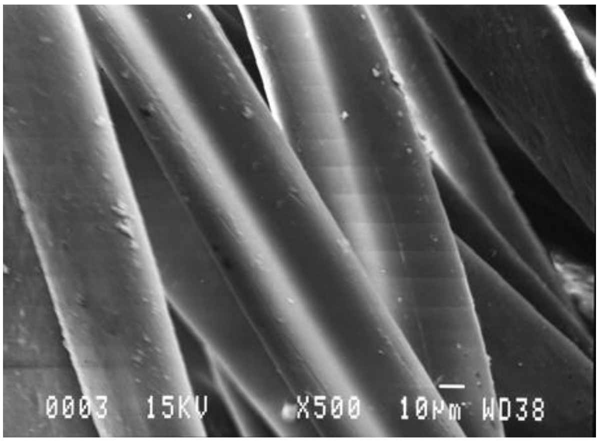


Fig. 7. Reflectance curves of blank and treated acrylic carpets with different concentrations of NTO and PS, and subjected to daylight irradiation

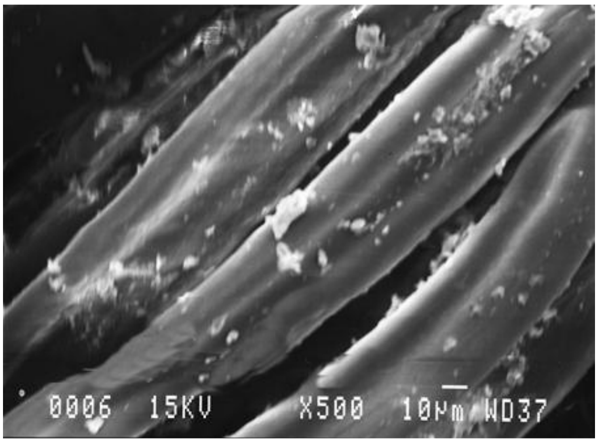
3.3 Scanning Electron Microscope

Figs. 8a-d show the SEM images of (a) blank acrylic fibers, (b) fibers treated with 1.00% NTO, (c) fibers treated with 3.00% PS, and (d) fibers treated with 1.00% NTO and 3.00% PS (magnification =500×).

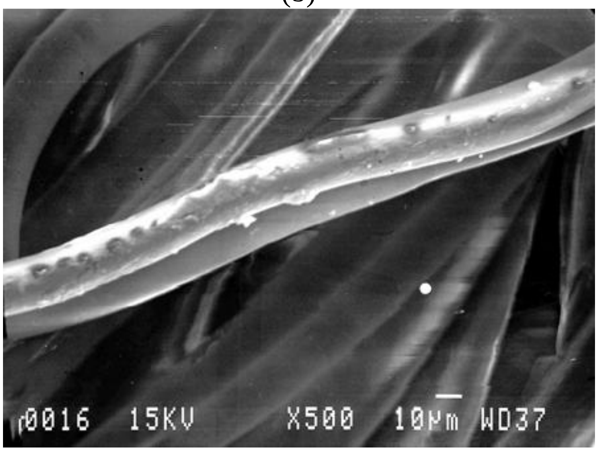
The cross-linked structures are created as colorless and clear layers on the fibers via PS as the polymeric agent and stabilized NTO particles that act as the protective layer. The image of the acrylic fibers treated with 1.00% NTO and 3.00% PS (Fig. 8d) reveals the ability of the NTO particles to remain on the acrylic fibers when PS is used. This causes an increase in the efficiency of properties, such as self-cleaning and protection against UV irradiation.



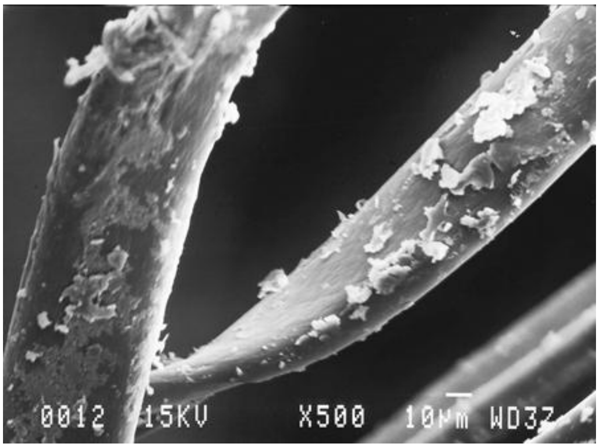
(a)



(b)



(c)



(d)

Fig. 8. SEM images of (a) the blank acrylic fiber, treated with (b) 1.00% NTO, (c) 3.00% PS, and (d) 1.00% NTO and 3.00% PS, (magnification =500×) under UV of daylight irradiation

4. Conclusions

This study has proposed and investigated a new idea to assess optimized permanent self-cleaning on acrylic carpet surfaces. This technique includes the embedding of NTO particles with a cross-linkable PS layer through two-steps of spraying. This functional coating enables remarkable improvements in the performance and durability of functionalized acrylic carpets. Optimum conditions of acrylic carpet production with self-cleaning properties are recommended. Three UV irradiation conditions have been compared for two different dyes, synthetic and natural (Solophenyl Red GBL and black mulberry, respectively), as the coloring stains. Meanwhile, the suggested photodegradation mechanisms for black mulberry and Solophenyl Red GBL dyes are presented. A remarkable increase in the self-cleaning properties of the NTO particles is obtained when used together with PS. However, the discoloration of the black mulberry dye in optimized conditions is better than that of Solophenyl Red GBL. The acrylic carpet treated with NTO and PS cross-linkable polymer is capable of effectively photodegrading the black mulberry and Solophenyl Red GBL stains. Also, the treated acrylic carpets have the desired softness and suitable protection against UV irradiation.

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