



Synthesis, characterization and application of reactive UV absorbers for enhancing UV protective properties of cotton fabric



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TWO colourless reactive UV absorbers **1** and **2** were synthesized by the condensation reaction of both 2,4,6-trichlorotriazine and sulfanilic acid with 4-aminobenzophenone and ethyl 4-aminobenzoate, respectively. The structures of UV absorbers **1** and **2** were confirmed by IR, NMR (¹H and ¹³C), and elemental analysis. The synthesized reactive UV absorbers were applied to cotton fabric using pad-dry method in comparison with both exhaustion and microwave methods. The results showed that excellent ultraviolet protection factor (UPF) values were achieved on sodium hydroxide-treated cotton fabric using pad-dry method. The optimal conditions using pad-dry method, such as drying temperature, drying time and UV absorber concentration were discussed. It was found that the highest values of UPF and whiteness using reactive UV absorbers **1** and **2** especially after washing can be obtained in the conditions of 80 °C drying temperature, 3 hours drying time and 15 g/l UV absorber concentration. The new reactive UV absorber **1** achieved excellent UPF value (58.2) compared to UV absorber **2** which exhibited poor UPF value (10.5) at the same conditions.

Keywords: Reactive UV absorbers, UPF, Whiteness, Pad-dry method, Sodium hydroxide-treated cotton fabric.

Introduction

Long-term exposure to ultraviolet radiation (UVR) is known to be detrimental to the skin [1-3]. Textiles can be a useful UV barrier and provide great UV protection [4, 5]. Nowadays, considerable attention has been paid to textiles designed for UV protective clothing [6, 7]. Uncoloured and light bleached cotton fabric is the first choice of customers for summer clothing. Such cotton fabric yields very poor UPF values [8]. UV absorber treatments absorb UVR and block its transmission through the fabric to the skin. Hence, the UV protection properties of textiles are significantly enhanced [9]. Therefore, efficient UV cutting materials or UV absorbers development is currently being considered [10, 11]. There are three requirements for an efficient UV absorber [12]: which are the strong absorption in the UV region of the solar spectrum especially for UVB and UVA rays [13], rapid conversion of

UV energy into heat energy without the fabric degradation [14, 15], and ease of its application on the fabric without adding colour to it [14, 15]. Non-reactive UV absorbers include benzophenone, benzotriazole, hindered amine, cyanoacrylate, triazine, oxanilide related compounds and metal oxide nanomaterials have been applied on cotton fabrics [10, 16-26]. The most commonly used UV absorbers of the above are benzophenone, benzotriazole and their derivatives [26]. On the other side, the non-reactive UV absorbers have no reactive group, as a result, they have low affinity and poor wash fastness when applied to cotton fabrics [26-28]. Therefore, UV absorbers comprising reactive groups i.e., reactive UV absorbers are more interesting for cotton fibers [29]. Such UV absorbers could form covalent bonds with the cotton fiber due to the fixation process and then would be able to maintain the protective properties of the modified fabric for a

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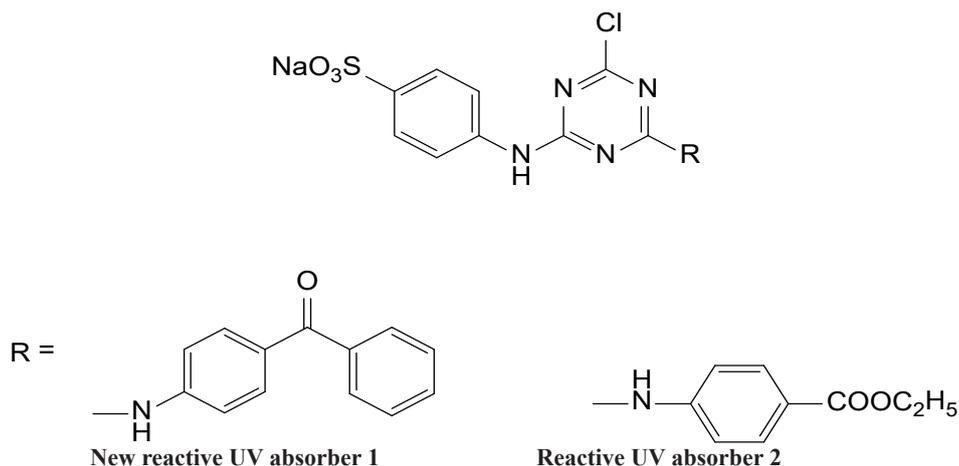
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longer period of time [30]. Where, the introduction of two or more reactive systems to a UV absorber molecule provides it with high fixation efficiency [29]. Interesting representatives of these reactive UV absorbers are those synthesized by the reaction of cyanuric chloride with aromatic amines containing UV absorbing moieties [31]. In the present work, it was anticipated that, synthesis of reactive UV absorbers containing benzophenone and ethylbenzoate moieties

when applied on cotton fabric (using different application methods) would exhibit increasing UV protective properties in comparison with untreated cotton fabric. UPF and whiteness index values of the treated cotton fabric using the synthesized reactive UV absorbers before and after washing were measured.

The general structure of the synthesized reactive UV absorbers:



Experimental

Materials and Equipment

Mill-scoured and bleached cotton fabric, 125 g/m² (fabric density: number of warp yarns = 78 per inch, number of weft yarns = 66 per inch) was obtained from Misr Company for spinning and weaving El-Mahala El-Kobra, Egypt. Before any treatments, the fabric was treated with a solution containing 3 g/l non-ionic detergent (Hostopal CV, Hoechst) and 5 g/l sodium carbonate at liquor ratio 1:50 at boiling for 4 h, thoroughly washed in water and dried at room temperature.

Cyanuric chloride, 4-aminobenzophenone and ethyl 4-aminobenzoate were obtained from Aldrich. Sulfanilic acid was obtained from Acros. All other chemicals and solvents used in this study were of laboratory reagent grade. Thin-layer chromatography (TLC) was performed by a Kieselgel 60 F254 (Merck, Darmstadt, Germany), to follow the reaction progress and the purity of the synthesised UV absorbers.

Methods

Synthesis of new reactive UV absorber 1

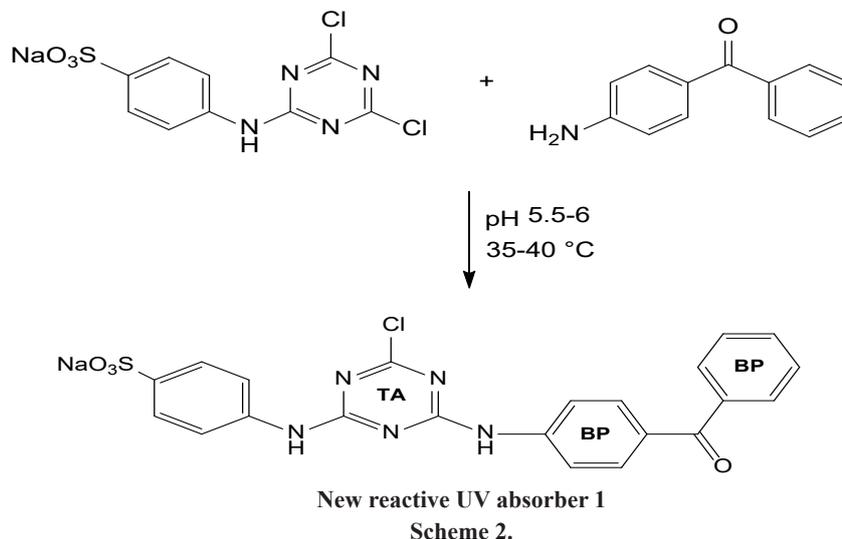
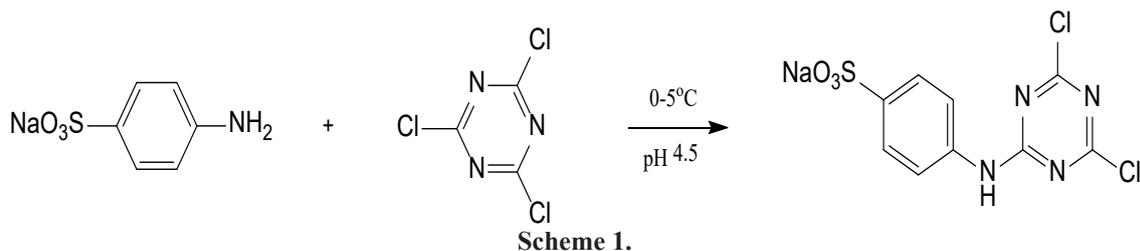
The synthesis of the new reactive UV absorber 1 was carried out in two steps as follows:

The first step: the first condensation reaction for cyanuric chloride with sulfanilic acid (Scheme 1):

The first condensation reaction for cyanuric chloride with sulfanilic acid was carried out by dissolving cyanuric chloride (1.94 g; 0.01 mol; 95%) in acetone. The reaction mixture was poured on ice and sulfanilic acid solution (2.037 g; 0.01 mol; 80%) was added over 30 min. The mixture was stirred at 0–5 °C for 4 h while controlling the pH at 4.5 using 2 M aqueous solution of sodium carbonate and the progress of the reaction was monitored by TLC.

The second step: a second condensation with 4-aminobenzophenone (Scheme 2):

The reaction mixture was further supplied with 4-aminobenzophenone (2.012 g; 0.01 mol; 98%) dissolved in the least amount of acetone over 30 min. and stirred at 35–40 °C, pH 5.5–6 for 5 h. The reaction progress was followed by TLC and the precipitated UV absorber was filtered off and dried in a vacuum oven at 30 °C.



Sodium-4-((4-((4-benzoylphenyl)amino)-6-chloro-1,3,5-triazin-2-yl)amino)benzenesulfonate

The structure of the new reactive UV absorber **1** was confirmed from the following results:

IR, (ν/cm^{-1}): 3418.21, 3287.07 ($-\text{NH}$), 1639.2 ($\text{C}=\text{O}$), 1173.47, 1036.55 ($-\text{SO}_3^-$); $^1\text{H-NMR}$ (400 MHz, $\text{DMSO-}d_6$): δ ppm 7.58–7.85 (13H, m, aromatic-H), 10.45, 10.64 (1H each, br s each, 2 x $-\text{NH}$); $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$): δ ppm 119.86, 120.46, 126.50, 129.06, 129.80, 129.88, 131.30, 132.80, 137.89, 138.81, 143.17, 143.96, 162.66, 164.26, 168.86 (Ar-C), 195.0 (BP-C=O); TA = Triazin; BP = Benzophenone.

The new reactive UV absorber **1** ($\text{C}_{22}\text{H}_{15}\text{ClN}_5\text{NaO}_4\text{S}$) purity was determined by elemental analysis, and gave the following results:

Calculated (%): C, 52.44; H, 3.00; N, 13.90; S, 6.36
Found (%): C, 52.42; H, 2.99; N, 13.88; S, 6.34

Synthesis of reactive UV absorber 2:

The synthesis of the reactive UV absorber **2** was carried out using a method typically described for new reactive UV absorber **1** except in using ethyl 4-aminobenzoate (1.685 g; 0.01 mol; 98%) instead of using 4-aminobenzophenone at the second condensation step (Scheme 3).

Sodium-4-((4-chloro-6-((4-ethoxycarbonyl)phenyl)amino)-1,3,5-triazin-2-yl)amino)benzenesulfonate

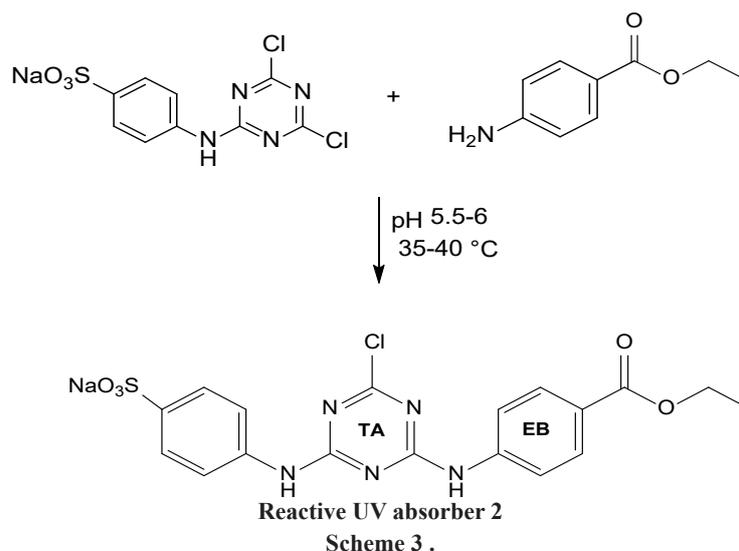
The structure of the reactive UV absorber **2** was confirmed from the following results:

IR, (ν/cm^{-1}): 3415.31, 3284.18 ($-\text{NH}$), 2966.95 (aliphatic CH), 1680.66 ($\text{C}=\text{O}$), 1175.4, 1028.84 ($-\text{SO}_3^-$); $^1\text{H-NMR}$ (400 MHz, $\text{DMSO-}d_6$): δ ppm 1.33 (3H, t, $J=7$ Hz, $-\text{CH}_3$), 4.31 (2H, q, $J=7$ Hz, $-\text{CH}_2$), 7.58–7.95 (8H, m, aromatic-H), 10.45, 10.58 (1H each, br s each, 2 x $-\text{NH}$); $^{13}\text{C-NMR}$ (100 MHz, $\text{DMSO-}d_6$): δ ppm 14.7 ($\text{CH}_3\text{CH}_2\text{-O-CO-}$), 60.9 ($\text{CH}_3\text{CH}_2\text{-O-CO-}$), 120.5, 124.9, 126.5, 130.4, 138.8, 143.4, 144.02, 152.14, 162.8, 164.3, 165.8 (Ar-C), 168.8 (EB-C=O); TA = Triazin; EB = Ethylbenzoate.

The UV absorber **2** ($\text{C}_{18}\text{H}_{15}\text{ClN}_5\text{NaO}_5\text{S}$) purity was determined by elemental analysis, and gave the following results:

Calculated (%): C, 45.82; H, 3.20; N, 14.84; S, 6.80

Found (%): C, 45.80; H, 3.18; N, 14.83; S, 6.78



Application procedures

Exhaustion method

Both reactive UV absorbers **1** and **2** at a liquor ratio 40:1 at 2% shade were applied on cotton fabric using the exhaustion method, similarly as in the case of monochlorotriazine (MCT) reactive dyes. The application process was started at 40 °C, for 30 min, during this period 80 g/l sodium sulphate was added in two portions at an interval of 15 min. Then 15 g/l sodium carbonate was added portionwise while the temperature was raised to 80 °C over 30 min. After that time, the application was continued at this fixation temperature for 1 hour. After application process was completed, all the samples were rinsed with water and air dried.

Microwave method:

Microwave-assisted application of reactive UV absorbers **1** and **2** was carried out in a Milestone Microwave Labstation, with a maximum output power of 1200 W, operating at 2450 MHz. The reactive UV absorbers solutions were applied to cotton fabric, similarly as in the case of MCT reactive dyes in a 250 ml round flask, fitted with condenser, was placed in the microwave oven at 40 °C; the energy level of the microwave oven was adjusted to 380 W and the temperature was raised to 80 °C \pm 2 °C in 4 min. The dyebath was then kept at this temperature for 1 hour. The treated cotton fabric was finally taken out of the microwave oven, rinsed and air dried.

Pad-dry method using NaOH-treated cotton fabric

Cotton fabric was firstly pretreated in 15% NaOH solution at 50 °C for 30 min. and dried at

80 °C for 3 min., and then the cotton fabric was washed with water and dried at 80 °C for 3 min. The NaOH-treated cotton fabric was then treated with the synthesized reactive UV absorbers **1** and **2** using pad-dry method as follow:

NaOH-treated cotton fabric (1 g) was immersed in a beaker containing the synthesized reactive UV absorbers **1** and **2** at various concentrations (5, 10 and 15g/l) at 40 °C for 30 min, then padded and finally dried in oven at various temperatures (80, 100 and 120 °C) at various drying times (1.5, 3 and 5 hours). After that, the treated cotton fabric was washed using 2 g/l nonionic detergent. Treated cotton fabric before and after different washing cycles were characterized for their UV protection properties and their whiteness index values.

Measurements

General

Infrared (IR) spectra were recorded on a Jasco FT/IR 4700. The transmittance between 400 and 4000 cm^{-1} was recorded. The NMR spectra were recorded on a Bruker High performance Digital FT-NMR Spectrometer Advance III: 400 MHz for ^1H NMR and 100 MHz for ^{13}C NMR; the chemical shift values are expressed in δ ppm and the J values are given in Hz for ^1H NMR. The UPF values were measured on JASCO V-750 UV-Visible spectrophotometer (United States). The Hunter Whiteness Index (WI) E 313 was measured on untreated and treated fabrics after washing using a Datacolor International SF 600 Plus spectrophotometer (Datacolour, Switzerland)

under illuminant D 65, 10° standard observer.

UPF of the treated cotton samples was evaluated using AS/ NZS 4399:1996 test method by cutting each specimen at least 5 x 5 cm place the specimen flush against the sample transmission port opening in the sphere. The UVR transmittance of fabric was measured over the UV spectrum (290–400 nm) with 5 nm intervals. The UPF was calculated by Equation (1) [32, 33].

$$UPF_i = \frac{\sum_{\lambda=290nm}^{400nm} E_{\lambda} \times S_{\lambda} \times \Delta\lambda}{\sum_{\lambda=290nm}^{400nm} E_{\lambda} \times S_{\lambda} \times T_{\lambda} \times \Delta\lambda} \quad (1)$$

Where E_{λ} is relative erythemal spectral effectiveness, S_{λ} is solar spectral irradiance in $W.m^{-2}.nm^{-1}$, T_{λ} is spectral transmittance of the fabric, λ is wavelength in nm and $\Delta\lambda$ is bandwidth in nm.

Results and Discussion

Characterization of the reactive UV absorbers 1 and 2

The successful synthesis of the two reactive UV absorbers **1** and **2** was proved by recording their spectral data IR and NMR (1H and ^{13}C) and elemental analysis. The IR spectra of the two reactive UV absorbers **1** and **2** showed two bands at 3418.21, 3287.07 (for UV absorber **1**) and 3415.31, 3284.18 (for UV absorber **2**) characteristic for the two (–NH) groups in each one. The characteristic band of (C=O) group appeared at 1639.2 for UV absorber **1** and at 1680.66 for UV absorber **2**. The two characteristic bands of the (–SO₃[–]) group appeared at 1173.47, 1036.55 cm^{-1} for UV absorber **1** and at 1175.4, 1028.84 cm^{-1} for UV absorber **2**. This is in addition to a strong band at 2966.95 cm^{-1} indicating the presence of the aliphatic (–CH) group in UV absorber **2** only. The 1H -NMR spectra showed the aromatic-Hs as a multiplet signal at \approx 7.5–8.0 ppm in both reactive UV absorbers **1** and **2**. Moreover, the aliphatic-category of protons was explained for the –OCH₂CH₃ group as A₂X₃– coupling system at ppm 4.3 (q, –CH₂–) and 1.3 (t, –CH₃) only in case of reactive UV absorber **2**. Further confirmation for the successful synthesis of reactive UV absorbers **1** and **2** was concluded from the ^{13}C -NMR spectra that showed the most downfield signal of carbonyl-benzophenone at 195 ppm in case of reactive UV absorber **1**. In case of reactive UV absorber **2** the ethyl ester was confirmed by its three ^{13}C -resonances at about 14.7, 60.9 and 168.8 ppm for ethyl and carbonyl groups.

Effect of application methods

Our study included a comparison between the exhaustion, microwave and pad-dry application methods of the two reactive UV absorbers **1** and **2** applied to cotton fabric.

Figure 1 summarize the results of UPF values of the two reactive UV absorbers **1** and **2** which were applied on cotton fabric using exhaustion, microwave and pad-dry application methods. The two reactive UV absorbers were applied on cotton fabric using pad-dry method at 5g/l UV absorber concentration, for 3 hours drying time and at 80 °C drying temperature. The results showed that the treated cotton samples using pad-dry method revealed the best UPF values compared with exhaustion and microwave methods. This could be attributed to that the cotton fabric are firstly pretreated in 15% NaOH at 50 °C for 30 min and dried at 80 °C for 3 min, which has the most significant effect on swelling of cotton fabric. This leads to maximize the uptake and fixation reaction completion of the reactive UV absorbers on NaOH-treated cotton fabric *via* MCT reactive group. This results in achieving higher UPF values for the treated cotton fabric. Also it is clear that; the new reactive UV absorber **1** has higher UPF values on the treated cotton fabric compared with those achieved using reactive UV absorber **2** in the all studied application methods. The pad-dry method is considered to be the best application method, so we have to study its parameters in details. The effects of reactive UV absorber concentrations, drying temperature and drying time using the pad-dry method on the UPF and the whiteness of the treated cotton fabric by the two reactive UV absorbers were measured.

Effect of reactive UV absorber concentrations in the pad-dry method

The extent of the UPF values of NaOH-treated cotton with different concentrations of reactive UV absorbers **1** and **2** (5, 10 and 15g/l) was investigated at 3 hours drying time and 80 °C drying temperature. The results are shown in Figure 2. From this Figure, it is clear that the highest UPF values were secured using the new reactive UV absorber **1** over the range of UV absorber concentrations studied. Also the results for the reactive UV absorbers studied indicate that the UPF values of the NaOH-treated cotton fabric increases as the reactive UV absorber concentration increases. This emphasizes that the fabric reactive UV absorber content plays an important role in increasing the UV protective properties of the NaOH-treated cotton fabric.

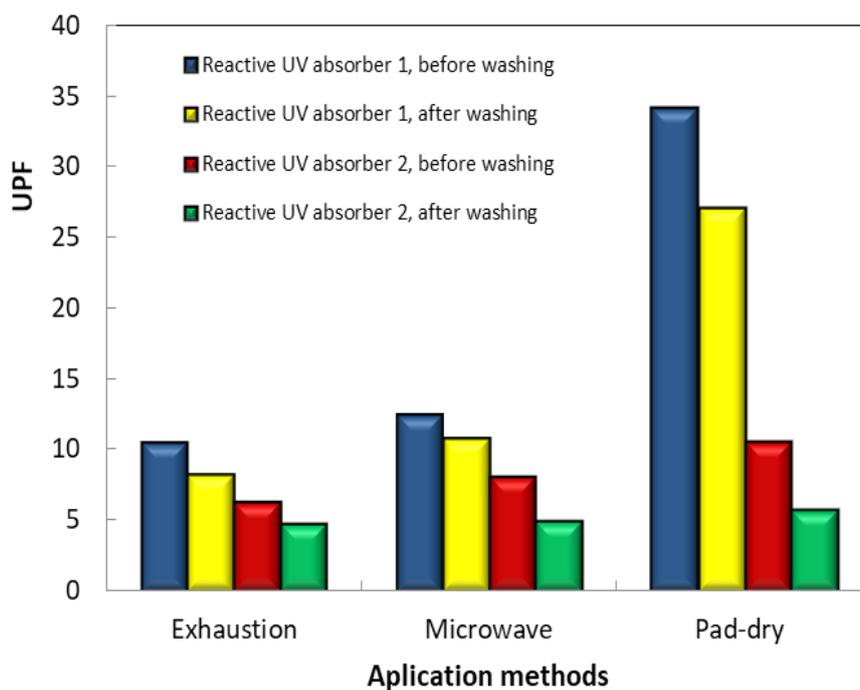


Fig. 1. UPF values of cotton fabric treated with reactive UV absorbers 1 and 2, using different application methods.

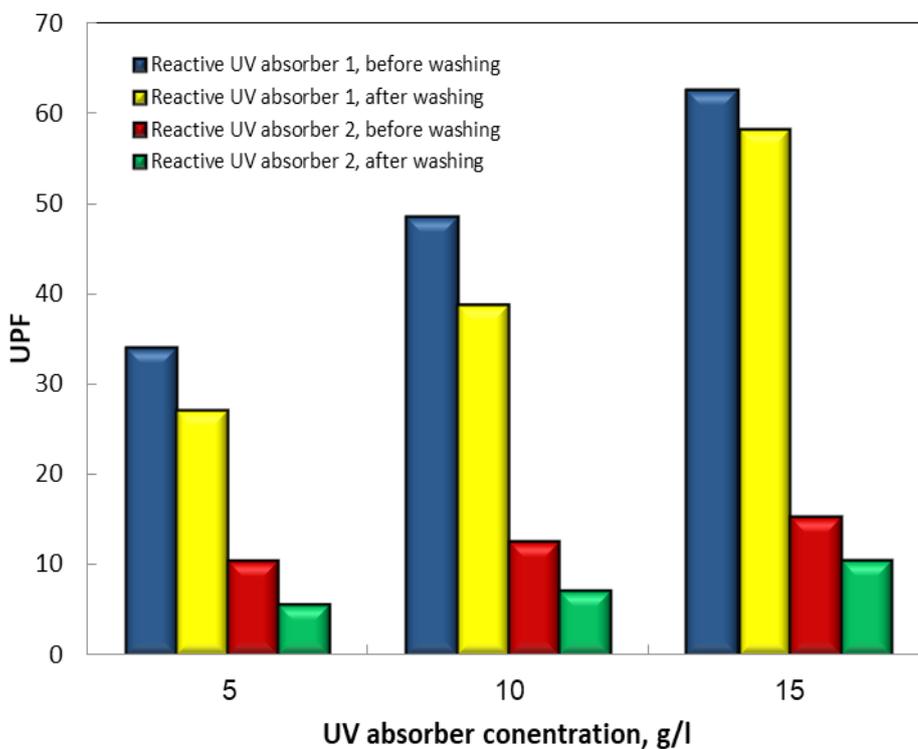


Fig. 2. Effect of the reactive UV absorber concentrations on UPF values of NaOH-treated cotton fabric using the pad-dye method at 80 °C drying temperature, 3 hr. drying time.

Effect of drying temperature in the pad-dry method

After determining the optimum UV absorber concentration, the study was extended to investigate the effect of the drying temperature. The drying temperature was varied to determine the optimum drying temperature. Figure 3 illustrates the UPF values of reactive UV absorber 1 and 2 on NaOH-treated cotton fabric at different drying temperatures (80, 100 and 120 °C). It is noticed that the new reactive UV absorber 1 has higher UPF values compared with reactive UV absorber 2 at all drying temperatures. The results also show that, the best results for both reactive UV absorber 1 and 2 were achieved at 80 °C drying temperature. On the other hand, the hydrolysis of MCT of reactive UV absorbers 1 and 2 tended to increase above 80 °C. Thus the optimum drying temperature was 80 °C.

Effect of drying time in the pad-dry method

The influence of drying time (1.5, 3 and 5 hours) on UPF values was also examined. The results are shown in Figure 4. From this Figure, it is clear that; reactive UV absorber 1 has higher UPF values compared with reactive UV absorber 2 at all drying times. The results also show that, higher UPF values were achieved at 3 hours drying time for both reactive UV absorbers. On the other hand, the UPF decreased at 5 hours drying time. This could be attributed to that the MCT reactive group is easy to be hydrolyzed at longer drying time. The hydrolysis of MCT reactive groups in turn leads to decrease the anti-UV protection of both the UV absorbers. Therefore, it could be concluded that the optimum drying time is 3 hours.

The UV protective properties of both UV absorbers 1 and 2 at their optimum conditions

In continuation to the results mentioned in Figure 1, it is clear from Figure 5 that the UPF value of both reactive UV absorbers 1 and 2 on NaOH-treated cotton fabric using pad-dry method at the optimum conditions are definitely more enhanced than those using both exhaustion and microwave methods. The optimum UPF values of both UV absorbers 1 and 2 were achieved at 15 g/l reactive UV absorber concentration and 80 °C drying temperature at 3 hours drying time. The new UV absorber 1 achieved excellent (UPF = 58.2) UV protection properties, while the UV absorber 2 achieved poor (UPF = 10.5) UV protection properties after washing.

Effect of concentration of reactive UV absorbers on the whiteness of cotton fabric

The whiteness was measured for four times at different portions of the fabric surface. The final Hunter whiteness was the average of the four values [26]. The whiteness index values of untreated and treated cotton fabric with reactive UV absorbers 1 and 2 using pad-dry method after washing (at optimum conditions) are given in Figure 6. From this Figure, it is clear that; NaOH-treated cotton fabric using the new reactive UV absorber 1 have high whiteness values at all concentrations compared with those using reactive UV absorber 2. The whiteness values of the NaOH-treated cotton fabric treated using new reactive UV absorber 1 after washing decreased on average less than 1 degree (15 g/l) and less than 3 degrees (5 g/l) compared with untreated cotton fabric. However, the whiteness values of NaOH-treated cotton fabric using reactive UV absorber 2 decreased on average less than 3 degrees (15 g/l) and less than 10 degrees (5 g/l) compared with untreated cotton fabric. Therefore, the effect of new reactive UV absorber 1 on the whiteness of cotton fabric is not obvious compared with reactive UV absorber 2.

Conclusion

Two colourless reactive UV absorbers 1 and 2 were conveniently synthesized and characterized using elemental analysis and spectral data IR and NMR (¹H and ¹³C). Pretreatment of cotton fabric with sodium hydroxide enhances the uptake and fixation of the synthesized reactive UV absorbers 1 and 2 using pad-dry method. Meanwhile the synthesized reactive UV absorbers 1 and 2 have MCT reactive group to react with cotton fabric by covalent bonds to prevent UVR. The best conditions for the application of the synthesized reactive UV absorbers 1 and 2 on sodium hydroxide-treated cotton fabric are drying temperature (80 °C), drying time (3 hours) and UV absorber concentration (15 g/l) using a pad-dry method. Treatment of sodium hydroxide-treated cotton fabric with the synthesized reactive UV absorbers 1 and 2 exhibited high UPF especially after washing compared with those using both exhaustion and microwave methods. The new reactive UV absorber 1 achieved excellent UPF value (58.2) at the optimum conditions, while the UV absorber 2 obtained poor UPF value (10.5) at the same conditions. The effect of new reactive UV absorber 1 on the whiteness of cotton fabric using pad-dry method is not obvious compared with reactive UV absorber 2.

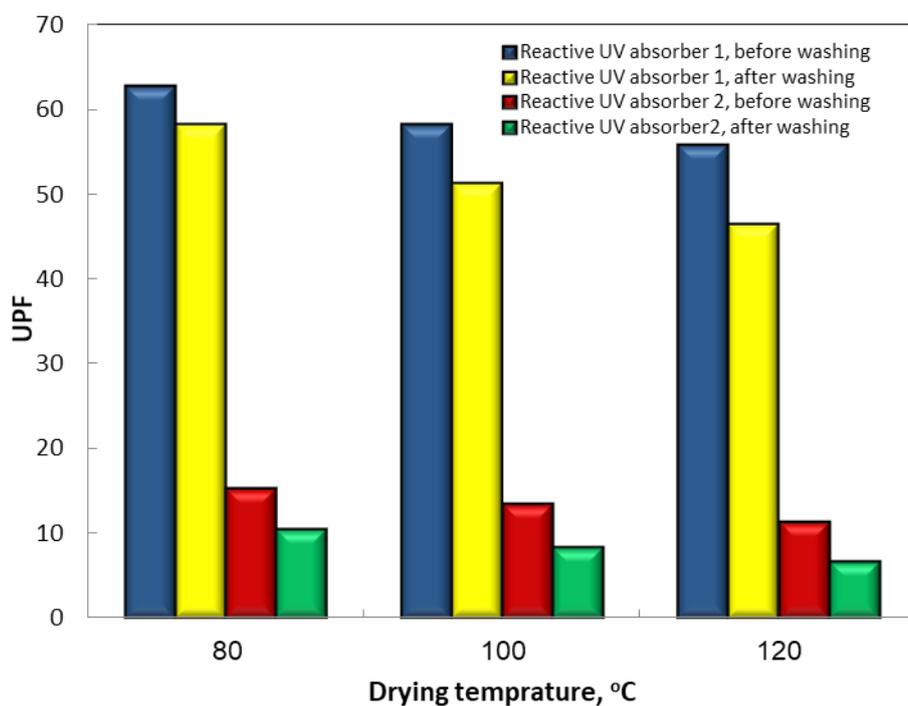


Fig. 3. UPF values of reactive UV absorbers 1 and 2 on NaOH-treated cotton fabric at different drying temperatures at 15 g/l reactive UV absorber concentration and 3 hr. drying time.

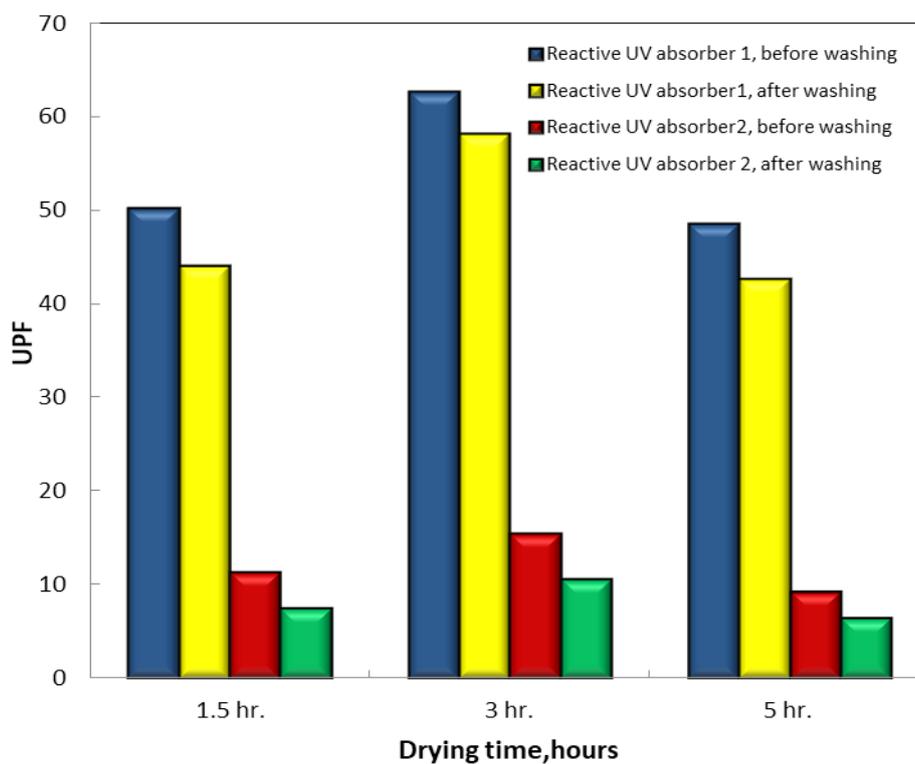


Fig. 4. Effect of drying time on UPF values of reactive UV absorbers 1 and 2 on NaOH-treated cotton fabric at 15 g/l reactive UV absorber concentration and 80 °C drying temperature .

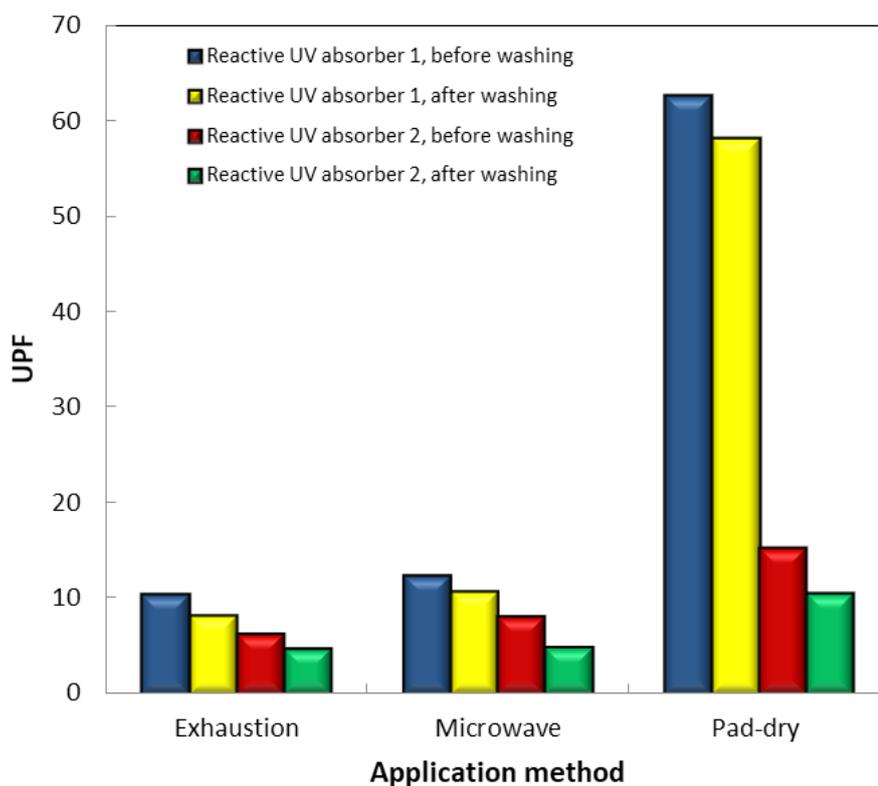


Fig. 5. UPF values of cotton fabric treated with both reactive UV absorbers 1 and 2 using pad-dry (at optimum condition), exhaustion and microwave methods.

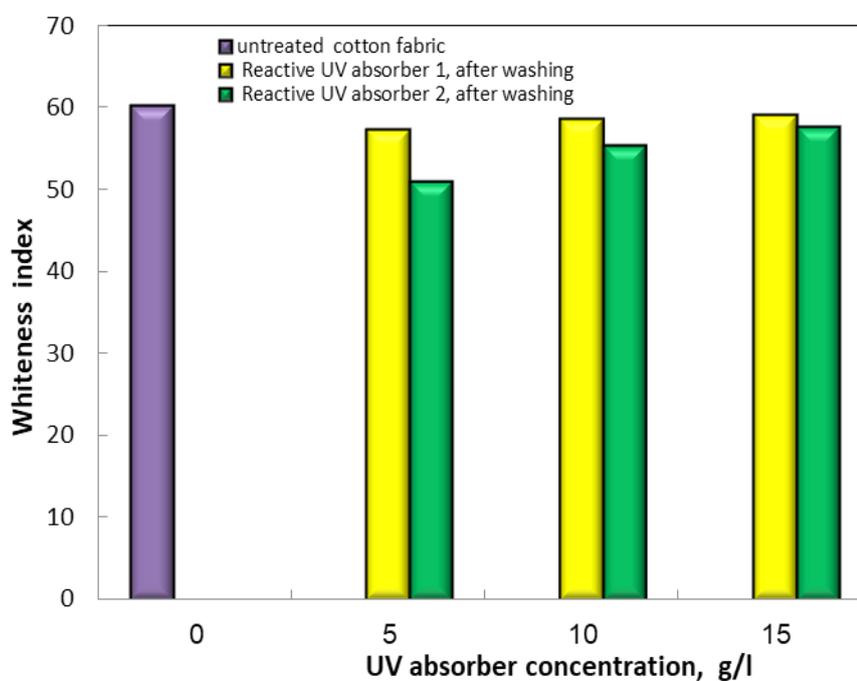


Fig. 6. whiteness index values of untreated and NaOH-treated cotton fabric using reactive UV absorbers 1 and 2 after washing using pad-dry method at optimum conditions .

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تحضير، توصيف وتطبيق مواد ماصة نشطة لتحسين خواص الحماية من الأشعة فوق البنفسجية للأقمشة القطنية

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تم تحضير مادتين نشطتين ماصتين للأشعة فوق البنفسجية **1** و **2** عن طريق تفاعل التكاثف بين 2,4,6- ثلاثى كلوروترايزين وحمض السلفنيليك مع 4- امينوبنزوفينون و اثيل-4- امينوبنزويت على الترتيب. تم توصيف المركبات المحضرة باستخدام الأشعة تحت الحمراء والرنين النووى المغناطيسى (البروتونى والكربونى). تم تطبيق المواد الماصة النشطة على الاقمشة القطنية باستخدام طريقة الغمر- التجفيف ومقارنتها مع طريقة الاستنفاد و طريقة المايكروويف. لقد اظهرت النتائج معامل حماية من الاشعة فوق البنفسجية (UPF) ممتازاً للأقمشة القطنية المعالجة أولياً بهيدروكسيد الصوديوم باستخدام طريقة الغمر- التجفيف لتطبيق المواد الماصة النشطة **1** و **2**. وقد تمت دراسة الظروف المثلى باستخدام طريقة الغمر- التجفيف مثل درجة حرارة التجفيف ووقت التجفيف و تركيز المادة الماصة النشطة. لقد وجدنا ان اعلى قيم لمعامل الحماية (UPF) و البياض باستخدام المواد الماصة النشطة خاصة بعد الغسيل تم الحصول عليها عند ظروف 80 درجة مئوية كحرارة تجفيف و 3 ساعات كوقت تجفيف و 15 جم/لتر كتركيز المادة الماصة النشطة. المادة الماصة النشطة الجديدة **1** حصلت على معامل حماية (UPF) ممتاز بقيمة (58.2) مقارنة بالمادة الماصة النشطة **2** التى حصلت على معامل حماية منخفض بقيمة (10.5) عند نفس الظروف.