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PERFORMANCE EVALUATION OF MERCERIZED COTTON FABRIC TREATED WITH BENZOPHENONE-2

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ABSTRACT

An explorative study was conducted to determine the optimal application procedure of Benzophenone-2 on mercerized cotton fabric as an UV filter. Its effect on fabric's appearance, physical properties, mechanical properties and comfort properties were also studied. To achieve optimum protection, influence of pre-treatment pH, treatment time, treatment temperature and treatment concentration were analysed. The analysis was done on the basis of Ultraviolet protection factor (UPF). The results showed that the pH of 10, temperature of 75^{0}C, time duration of 35 minutes and concentration of 2% were optimal for cotton fabric's treatment with Benzophenone-2. Effect of consecutive launderings on UV treated fabric was also studied. The treatment caused slight yellowing of the cotton fabric. UV absorber treated samples were subsequently tested for any significant change in physical, comfort or mechanical properties of the treated fabric. At 5% level of significance data obtained for physical properties showed no significant changes except for increased fabric weight and decreased crease recovery angle. The results of mechanical properties were found to be non significant at 5 % level of significance for tensile strength but significant increase in percent elongation was observed in warp direction. SEM and FTIR analysis of control and treated fabric was also considered.

KEYWORDS: *Air Permeability, Benzophenone-2, UV Absorber, UV Protection, UPF, Whiteness Index*

Article History

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INTRODUCTION

Fundamentally sun provides all the energy vital to nurture and maintain life. In absence of sun, the life would be impossible on the earth. Sun gives sunlight in the form of electromagnetic energy. Electromagnetic radiation is a wave phenomenon commonly known as light. There are three sorts of ultraviolet rays: UV-A, UV-B and UV-C. The longer wavelength UV rays spanning from 320 nm to 400 nm (UV-A) instigate melanin change in the skin, prompting to accelerated pigmentation. This change sets in within a time of a couple of hours, yet it is just an exceptionally insignificant and of brief span. However, it enters profoundly into the dermis or genuine skin, prompting to untimely maturing and loss of flexibility joined by skin wrinkling and lines. A shorter wavelength of higher energy UV rays (UV-B) which spans from 290 nm-320 nm, infiltrate to a depth of a few millimeters into the skin. It brings a generally stable pigmentation on skin's external layer. This can prompt to intense responses and deteriorations, for example, sunburn or reddening of skin (erythema). The ultraviolet rays known as UV-C has most limited wavelength (100 nm-290 nm), which harm human skin, but they don't reach on the surface of earth as they are sieved through by the ozone layer (Das, 2010).

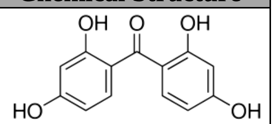
There is a well established relationship between skin cancer and ultraviolet radiation. There are many components resulting in commencement of melanoma and non-melanoma skin malignancies, but overexposure to UV rays has distinctly been recognized as a central element. Solar emission has a perpetual energy spectrum over wavelength ranging from 0.7 nm to 3000 nm. The effective spectrum of the solar radiation reaching on the surface of earth spans from 280 nm to 3000 nm. The UV spectrum lies between the wavelengths of 290 nm to 400 nm. Out of the total incident sunlight reaching on earth’s surface, UV radiation constitutes to 5%, visible light 50% and IR radiation 45%. Despite the fact that, its extent is very low, it has the highest quantum energy contrasted with different radiations. This vitality of UV radiation is the result of organic particle's bond energy; consequently, it has gigantic detrimental impact on human skin (Reinert *et al*, 1997).

Textile materials can be treated with UV absorbers to obtain better security against destructive UV rays. Certain colourless organic or inorganic compounds with strong absorption in the UV scope of 290 nm – 360 nm are known as UV absorbers (Mallik and Arora, 2003). A UV absorber is a molecule which absorbs ultra violet light efficiently and converts the energy into relatively harmless thermal energy (Holme, 2003).UV absorbers function by preferentially absorbing harmful UV radiation and dissipating it as thermal energy. They act primarily by dissipating the absorbed light energy from UV rays as heat by reversible intermolecular proton transfer. UV absorbers should absorb UV light in the 290—400 nm range but also be transparent to other radiation. i.e. be colorless in the visible spectrum. Benzophenones and benzotriazoles are common UV absorbers (Laurence, 2013).UV absorber could he easily dissipated through the substrate. The amount of heat released from this UV absorption mechanistic process is considered very small compared to the total heat load on the samples or polymers, and therefore does not contribute to the thermal degradation of polymers (Durairaj, 2005).

MATERIALS

The materials used to treat the fabric in the study included mercerized cotton fabric, Benzophenone-2, and various chemicals of laboratory grade. The mercerized cotton fabric used for this study was purchased from Ludhiana. Preliminary data of the fabric was collected using standard test methods. To conform that the fabric procured for study is pure cotton, microscopic, burning and chemical tests were conducted. To study the effects of UV absorbers on mercerized cotton fabric, Benzophenone-2 was selected. Details of Benzophenone-2 is given in table 1. Benzophenone-2 was obtained from *Sigma-Aldrich co. LLC, India.*

Table 1: UV Absorber Used in the Study

UV Absorber	Alternate Name	Chemical Formula	Chemical Structure
2,2’,4-4’-tetrahydroxy-benzophenone (97%)	Benzophenone-2	$[(HO)_2C_6H_3]2CO$	

METHODS

UV treatment conditions were optimised on the basis of ultraviolet protection factor (UPF). Treatment parameters optimized for UV absorber included Na_2CO_3 pre-treatment pH, treatment temperature, treatment time and UV absorber concentration. The pH, time, temperature and concentration at which the UV absorbers offered highest UV protection were considered for final treatment.

Recipe for UV Treatment

UV absorber	:	x % (owf)
Liquor ratio	:	1:30
Dyeing pH	:	9-11
Temperature	:	60-75^{0}C
Time	:	25-40 minutes

Optimization of pH

Cotton fabric samples were first treated with sodium carbonate aqueous solution of different pH at 40^{0}C for 30 minutes using 1:30 MLR ratio. Excess sodium carbonate solution was squeezed out of the fabric and was shade dried prior to UV absorber treatment. Water/ethanol mixture (9/1, v/v) was used for the treatment of cotton fabric with UV absorbers. UV absorber was first dissolved in ethanol. Dissolved UV absorbers were then introduced in the beakers containing water. For the optimization purpose 1:30 material-liquor ratio was used. Cotton samples were treated with both UV absorbers (1% on the weight of fabric) in two different beakers using exhaust method. Samples were treated at 75^{0}C for 30 minutes. Samples were stirred continuously in order to achieve a uniform deposition of UV absorbers on the fabric. After 30 minutes, samples were taken out of bath and were washed with water repeatedly in order to remove any excess UV absorber on the fabric surface. Samples were dried in shade afterwards. The pH value at which the treatment gave the highest UPF was considered optimum pH value.

Optimization of Temperature

For determining the optimum temperature, the fabric was first treated with sodium carbonate aqueous solution of optimum pH. Subsequently, UV absorbers were mixed in water/ethanol (9/1, v/v) solution and cotton fabric was treated at four different temperatures of 60^{0}C, 65^{0}C, 70^{0}C and 75^{0}C for 30 minutes. Exhaust method of treatment was used maintaining 1:30 material-liquor ratio. The treatment was followed by thorough washing and shade drying of treated samples. The temperature which gave the best results was selected for further experiment.

Optimization of Time

Mercerized cotton fabric samples were treated with sodium carbonate solution of optimized pH and were treated with UV absorbers using water/ethanol (9/1, v/v) solution at optimum temperature for 25, 30, 35 and 40 minutes separately. For the treatment exhaust method was used with material-liquor ratio of 1:30. Samples were stirred continuously during treatment to achieve an even deposition of UV absorbers on the fabric. Samples were shade dried after thorough washing. The time at which best results were obtained for UPF was selected for the further optimization process.

Optimization of Concentration

Concentration at which optimum UV protection will be achieved was determined by treating the samples at four different UV absorber concentrations i.e. 1%, 2%, 3%, and 4%. For optimizing the concentration of UV absorber, samples were treated using water/ethanol (9/1, v/v) solution containing UV absorber. The fabric samples were first treated with sodium carbonate solution of optimum pH. UV absorber treatment was given using exhaust method at optimized temperature for optimized time duration using 1:30 material-liquor ratio. The concentration which recorded highest UPF was selected for the final treatment.

Scanning Electron Microcopy (SEM)

Scanning electron microscope was used to obtain the SEM micrographs of untreated and UV treated-dyed cotton samples. Scanning electron microscope was used to produce high resolution images of the cotton fabric surface. SEM imaging generates a three dimensional appearance of samples facilitating useful judgment of the surface structure.

Fourier-Transform Infrared Spectroscopy (FTIR Analysis)

The untreated and UV treated-dyed cotton fabric samples were examined in a double beam FTIR spectrophotometer using ATR (Attenuated Total Reflectance) attachment. The IR transmission spectra were recorded at wave numbers from 700-4000 cm^{-1}.

RESULTS AND DISCUSSIONS

Optimization of pH

Data of pH optimisation is demonstrated in the table 2. The incorporation of Na_2CO_3 in the pre-treatment solution showed an increase of UV absorber absorption in treatment bath. But this positive influence was only limited to pH 10. Once the pH was raised to 11, a decrease in UV-A and UV-B blocking was observed. Highest UPF (30.00) was imparted to the cotton fabric at pH 10. Hence, pH of 10 was selected as optimum pH for further optimization of cotton fabric.

Table 2: Optimization of pH for Benzophenone-2

pH	UPF	Transmission %		Blocking %	
		UV-A	UV-B	UV-A	UV-B
9	25.10	3.33	4.05	96.67	95.95
10	**30.00**	**2.87**	**3.45**	**97.13**	**96.55**
11	27.00	3.02	3.75	96.98	96.25

Optimization of Temperature for Benzophenone-2

Temperature for the treatment was optimised using optimum pH. Data for the temperature optimization is exhibited in the table 3. It was observed from the table that optimum results were obtained at the temperature of 75^0C. Out of the chosen temperatures, the temperature of 75^0C provided highest UV-A and UV-B blocking (96.94 % and 96.84 % respectively). Since, 75^0C was the highest temperature chosen for the optimization purpose, the temperature of 80^0C was additionally examined. After examination of the results, it was observed that the temperature of 80^0C did not influence the cotton fabric positively. Higher temperature also resulted in the uneven uptake of UV absorber. Due to the fact that lowest UV transmission (3.06 %) was observed at 75^0C, it was selected for the further optimization of UV absorber.

Table 3: Optimization of Temperature for Benzophenone-2

Temperature (^{0}C)	UPF	Transmission %		Blocking %	
		UV-A	UV-B	UV-A	UV-B
60	15.50	5.54	6.27	94.46	93.73
65	16.30	5.12	5.90	94.88	94.10
70	25.50	3.76	3.85	96.24	96.15
75	**31.40**	**3.06**	**3.16**	**96.94**	**96.84**
80	17.60	4.68	5.50	95.32	94.50

Optimization of Time Duration for Benzophenone-2

Once the optimum temperature was determined, optimization of time duration was contemplated. Data for the time duration optimization is revealed in table 4. Out of the four time durations chosen for the optimization, treatment for 35 minutes provided the highest UPF (32.10) value. When the treatment time was increased to 40 minutes, a negative effect was observed on UV-A and UV-B blocking of the treated cotton fabric. Thus, 35 minutes was selected as ideal time duration for treatment with Benzophenone-2.

Table 4: Optimization of Time Duration for Benzophenone-2

Time (Min)	UPF	Transmission %		Blocking %	
		UV-A	UV-B	UV-A	UV-B
25	15.90	5.23	6.07	94.77	93.93
30	18.80	4.36	5.15	95.64	94.85
35	**32.10**	**3.08**	**3.11**	**96.92**	**96.89**
40	30.30	3.12	3.28	96.88	96.72

Optimization of Concentration for Benzophenone-2

After optimization of treatment time duration, optimum concentration of UV absorber was determined. Data for the UV absorber's concentration is reported in the table 5. After scrutinizing the recorded data it was witnessed that UPF accelerated with increased UV absorber dosage. However, after 2% concentration of UV absorber there was a decrease in UPF at 3% and 4% UV absorber concentration. Considering the fact that, higher concentrations resulted in lower UV-A and UV-B Blocking, concentration of 2 % was chosen for the application of UV absorber to the cotton fabric.

Table 5: Optimization of Concentration for Benzophenone-2

Concentration (%)	UPF	Transmission %		Blocking %	
		UV-A	UV-B	UV-A	UV-B
1	32.1	3.08	3.11	96.92	96.89
2	**36.3**	**2.64**	**2.78**	**97.34**	**97.22**
3	29.1	2.75	3.55	97.25	96.45
4	25.9	3.08	3.94	96.92	96.06

Whiteness Index

Recorded values for whiteness index of Untreated and UV treated samples are given in table 6. The experimental data showed that average whiteness index for untreated cotton fabric was 68.99. After the UV absorber treatment a sight yellowing of the treated samples were observed.

Table 6: Whiteness Index of Undyed-Untreated and UV Absorber Treated Fabrics

Treatment	Whiteness Index
Control	68.99
Benzophenone-2	65.31

Benzophenone-2 treated samples showed a whiteness index of 65.31. The data clearly indicated that there was a slight yellowing for both the UV absorbers

Laundering of the UV Treated-Dyed Samples

The treated fabrics would have to undergo laundering as they would be used for the coloured apparels and life style products, so all the UV treated and UV treated-dyed fabrics were subjected to various wash cycles using standard

AATCC home laundry test conditions. The UV treated-dyed samples were laundered at 30^0 C for 16 min using commonly available washing powder in laundrometer. For samples treated with Benzophenone-2, the recorded data unmistakably demonstrated a decrease in UPF from 18.73%, 28.37%, and 36.34% for 5, 10 and 15 wash cycles respectively.

Table 7: Laundering of the UV Treated-Dyed Samples

UV Absorber/ Dye	Before Laundry (UPF)	After Laundry (UPF)		
		5 Wash Cycles	10 Wash Cycles	15 Wash Cycles
Benzophenone-2	36.30	29.50	26.00	23.00

Consequently, when wash cycles were increased to 15 it was found that the durability is not up to the expected level even if protection level was good. The lower UPF indicated low bond formation between fabric and Benzophenone-2. It can be distinguished from the data that UPF was affected negatively by repeated washing even though they sustained good to adequate effectiveness against UV-A and UV-B.

Measurement of Physical and Mechanical Properties

Fabric Weight: It is apparent from the table that the weight of the control fabric was 130.60 g/m^2, which increased significantly (at 5% level of significance) after treatment with benzophenone-2 (131.28 g/m^2).

Fabric Thickness: It can be attributed from table that the thickness for control fabric was 0262 mm. It was witnessed that the thickness of fabric showed no significant difference (at 5% level of significance) after dyeing with Benzophenone-2 (0.264 mm).

Table 8: Fabric Properties of UV Treated Samples

Physical Properties		Control Sample	UV Treated Samples	CD
Fabric weight (g/m^2)		130.60	131.28	0.21
Fabric thickness (mm)		0.262	0.264	NS
Cover factor	Warp	10.33	10.45	NS
	Weft	10.36	10.47	NS
Bending length (cm)	Warp	2.04	2.13	NS
	Weft	2.15	2.25	NS
Flexural rigidity (mg/cm)	Warp	101.38	116.52	NS
	Weft	118.71	136.09	NS
Overall flexural rigidity (mg/cm)		109.74	125.83	NS
Drape coefficient (%)		78.51	79.21	2.4
Crease recovery (degree)	Warp	118.67	116.00	2.25
	Weft	117.67	114.00	2.12
Tensile strength(kg/sq.cm)	Warp	48.50	48.48	NS
	Weft	48.60	48.56	NS
Elongation (%)	Warp	6.30	6.39	0.02
	Weft	6.26	6.26	NS
Air permeability (cc/sec/cm^2)		13.46	12.50	NS

Cover Factor: Cover factor for control fabric was 10.33 and 10.36 in warp and weft direction respectively. For cover factor in warp as well as weft direction there was a no significant difference (at 5 % level of significance).

Bending Length: The mean bending length of control fabric in warp direction and weft direction was recorded to be 2.04 cm and 2.15 cm respectively. For the warp direction as well as weft direction, UV treatment showed no significant difference in bending length at 5% level of significance.

Flexural Rigidity: Observed flexural rigidity for control fabric was 101.38 mg/cm and 118.71 mg/cm in warp and weft direction correspondingly. An overall flexural rigidity of 109.74 mg/cm was recorded. No significant difference in flexural rigidity was observed at 5% significance level.

Drape Coefficient: drape coefficient of the control fabric was 78.51%. Interpreting from the recorded values it was witnessed that drapability decreased slightly after treatment with Benzophenone-2 but it was found non significant at 5% level of significance.

Crease Recovery: The crease recovery angles of control fabric samples were. The observed crease recovery angle in warp direction for control fabric was 118.67 and for the weft direction recorded crease recovery was 117.67. Both Crease recovery values showed significant decrease in crease recovery angle at 5% level of significance.

Tensile Strength: The tensile strength in warp and weft direction for control fabric was 48.50 kg/sq.cm and 48.60 kg/sq.cm respectively. There was no significant difference in tensile strength for the warp and weft direction at 5% level of significance.

Elongation: Elongation of control fabric was witnessed to be 6.30 % and 6.26 % in warp and weft direction respectively. It was observed that elongation in both dyed samples has occurred in warp and weft direction. A significant increase in % elongation was observed in warp direction at 5% level of significance in warp direction. For weft direction, no significant difference was observed at 5% level of significance.

Air Permeability: it can be observed that the air permeability of control fabric was 13.46 cc/sec/cm^2. After UV treatment, no significant difference in air permeability was found at 5% level of significance.

Scanning Electron Microcopy (SEM)

SEM indicated that the Benzophenone-2 was present in the spaces of the fibre assembly of fabric as shown in Figure 2. After the treatment with Benzophenone-2, chemical compound can be observed on the surface of the fibre in the form of flakes and small clusters.

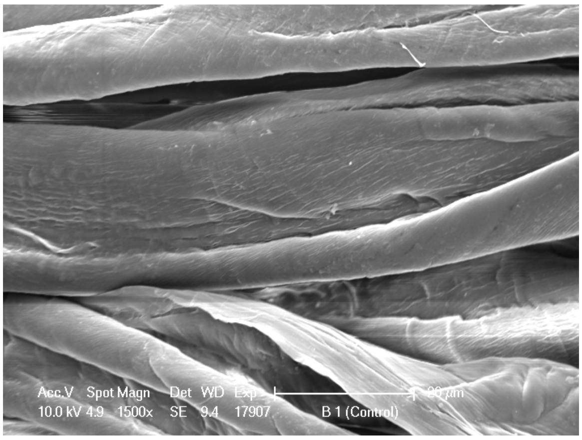


Figure 1: Scanning Electron Photomicrograph of Untreated Cotton Fabric at 20 µm

Figure 2: Scanning Electron Photomicrograph of Benzophenone-2 Treated Cotton Fabric at 20 µm

FTIR Study of Benzophenone-2 Treated Fabric

The FTIR frequencies of both untreated and UV treated cotton fabrics are illustrated in the figure 3. It can be observed from the table 9 that after the treatment of the cotton with Benzophenone-2, both untreated and UV treated samples showed similar frequencies. The only two exceptions was disappearance of the C-H bending in the frequency range of ~850 cm^{-1} and appearance of a new peak at 1649 cm^{-1} belonging to C=C bending.

Table 9: IR Absorptions for Control and Benzophenone-2 Treated Cotton Fabric

Control Sample			Benzophenon-2 Treated Sample		
Sr. No.	**Frequency (cm^{-1})**	**Vibrational Assignments**	**Sr. No.**	**Frequency (cm^{-1})**	**Vibrational Assignments**
1.	850.61	C-H bend	1.	983.70	C-H (out-of-plane) bend
2.	983.70	C-H (out-of-plane) bend	2.	1028.06	C-O-C stretch
3.	1029.99	C-O-C stretch	3.	1053.13	C-O-C stretch
4.	1053.13	C-O-C stretch	4.	1103.28	C-O-C stretch
5.	1109.07	C-O-C stretch	5.	1161.15	C-O-C stretch
6.	1161.15	C-O-C stretch	6.	1201.65	C-OH stretch
7.	1203.58	C-OH stretch	7.	1317.38	CH2 wagging
8.	1317.38	CH2 wagging	8.	1423.47	CH_2- bending
9.	1423.47	CH_2- bend	9.	1535.34	C=C bending
10.	1517.98	C=C bend	10.	1649.14	C=C bending
11.	2316.51	Carboxylic Acid -OH Stretch	11.	2314.58	Carboxylic Acid O-H stretch
12.	2895.15	Carboxylic Acid -OH Stretch	12.	2881.65	Carboxylic Acid O-H Stretch
13.	3334.92	water -OH Stretch	13.	3309.85	water -OH stretch
14.	3728.40	water -OH Stretch	14.	3745.76	water -OH stretch

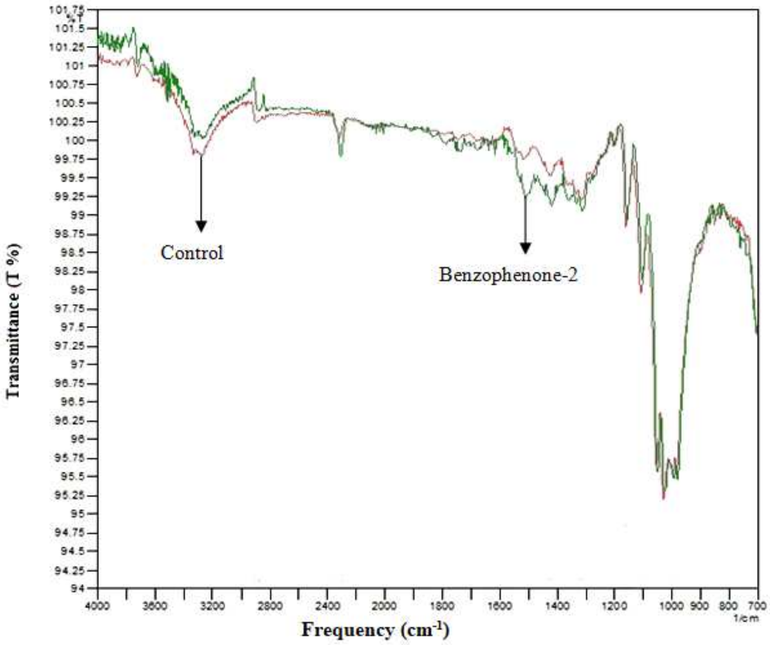


Figure 3: FTIR of Untreated and Benzophenone-2 Treated Cotton Fabric

CONCLUSIONS

To achieve optimum UPF, the application of Benzophenone-2 should be done at a pH of 10 applying temperature of 75^{0}C for time duration of 35 minutes using concentration of 2%. At 5% level of significance, data obtained for physical properties showed no significant changes except for increased fabric weight and decreased crease recovery angle. The results of mechanical properties were found to be non significant at 5 % level of significance for tensile strength. A significant increase in percent elongation was observed in warp direction after the treatment.

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