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Simultaneous dyeing and anti-bacterial finishing of textile by sol-gel technique

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ABSTRACT

Using the sol–gel process, functional silica coatings can be deposited on textile materials and can lead to new textile properties and applications. In the present study combined dyeing and antibacterial finish was applied on same fabrics. Cotton and polyester/cotton blend were dyed with cheap basic dyes using sol-gel method by adding glycidoxypropyltrimethoxysilane as cross-linking agent. Cetyltriammonium bromide was incorporated to give antibacterial textiles in these coatings. A large reduction in the bacterial count of StaphylococcusAureus was observed.Thus simultaneous dyeing with cheap basic dyes and anti-bacterial finishing is possible by this technique. In addition to antibacterial textiles we also get softening of fabric. However the technique adversely affects the mechanical properties of the fabric.

Keywords: *Antibacterial Textiles, Basic Dyes, Nanotechnology, Silanes, Sol-gel coating*

INTRODUCTION

Sol-gel process has history of more than 100 years as one of the modification and preparation methods of materials since it was invented. Due to the resulting properties of high chemical homogeneity, purity, lower processing temperature, easy shaping and modification[1]. Sol-gel process has widely been applied in areas such as ceramics, pharmacy, orthopedics, and it has been applied in functional finishing of textiles in the last decade.

According to its purpose, silica gel can be divided into two categories. First, the functional gels making the treated textiles obtain desired functions, such as anti-abrasion[2], wrinkle resistance[3], water or oil repellence[4,5,6], antistatic [7], UV shielding [8,9], etc. Second the gels used as carriers to immobilize and/or control functional ingredients, such as improving color fastness of dyed textiles [10,11], preparing bioactive textiles [12], flavor-releasing textiles [13]etc.

Using this process functional silica coatings can be deposited on textile materials and can lead to new textile properties and application [11, 6]. Also, dyes can be embedded into sol–gel coatings and deposited on several types of materials leading to new possible applications, e.g. optical devices or sensors [14, 15].

Literature data reveal that compounds with organic functional groups help in permanent incorporation of antimicrobial components in organic polymers. Organic compounds such as halamines, cationic dyes and polymers, quaternary ammonium compounds, diphenyl ether derivatives help in such incorporation whereas the permanent incorporation of hard metals such as metallic silver or copper into organic polymers has been difficult to achieve. The organic agents do not chemically bond to the textile fibres and the antimicrobial activity is due to the persistent release from the textile to the surrounding. This is because of the non-uniform dispersion of the non-metallic agents into the polymer host[16-27].

In this research work, attempt has been made to incorporate basic dyes and antimicrobial compound in the sol-gel coating of cotton and polyester cotton blend.

MATERIALS AND METHODS

Ready for dyeing plain weave cotton (110 g/m^2, 17picks/cm, 12ends/cm) woven fabric was obtained from Tata Mill, Mumbai. Plain weave polyester-cotton (P/C) (160 g/m^2) fabric was obtained from Piyush Syndicate, Mumbai. Tetraethylorthosilicate (TEOS) was obtained from Evonik Degussa Pvt. Ltd. Mumbai. Glycidoxypropyltrimethoxysilane (GPTMS) was obtained from Dow Corning India Pvt. Ltd. Mumbai. Cetyltriammoniumbromide (CTAB) and Sodium Lauryl Sulphate was obtained from S.D. Fine Pvt. Ltd. Mumbai. Ethanol (98% pure) and Hydrochloride acid (HCl) used was of laboratory reagent grade. Coracryl Violet C3R was obtained from Colourtex Pvt. Ltd. Surat, Gujarat.

2.1 Preparation of sols:

Sols were prepared by taking 50ml ethanol, 34.20ml TEOS, 3.8ml GPTMS and 12ml 0.01N HCl in a beaker to make total volume as 100ml. The mixture was stirred with magnetic stirrer for 24hours at room temperature by covering the beaker with polyethylene sheet. Into these silica sols, CTAB with different concentrations and 10 gplCoracryl Violet C3R dye were added prior to padding.

2.2 Application of Sol on fabrics:

The fabrics were padded through these sols containing the dye and CTAB using 2 dip 2 nip method with 70% expression for cotton and 60% for P/C. The samples were then dried in air. It is then cured in oven at 120^0Cfor 1 hour. It was then soaped at 40^0C for 2 hours by 10gpl Sodium Lauryl Sulphate at neutral pH. The sample was then washed with water and air dried.

2.3 Color value by reflectance method:

The samples were evaluated for color depth in terms of Kubelka Munk function (K/S) using Computer Color Matching System (Spectra Scan 5100+) of Premier Colors can Instruments Pvt. Ltd. Mumbai.

Kubelka Munk (K/S) function is given by:

$$\frac{K}{S} = \frac{(1 - R)^2}{2R} \qquad (1)$$

Where,
"R" is the reflectance at complete opacity,
"K" is the absorption coefficient,
"S" is the scattering coefficient.

2.4 Fastness testing of dyed fabric:

For washing fastness, light fastness and rubbing fastness, ISO 105 C10, ISO 105-A02 and ISO 105 X-12 were used respectively.

2.5 Antibacterial activity:

The antibacterial efficacy of a compound will vary as per its presence in solution or on the textile substrate. Quantitative assessment of antibacterial activity exhibited on fabrics was carried out by AATCC Test 100-2004 (AATCC 2007) and the colony-forming units (CFUs) were enumerated using Lapiz Coloney Counter (Medica Instrument Mfg.Co., Mumbai, India). The fabrics were introduced in the 100 ml nutrient broth inoculated with the *Staphylococcus aureus (S. aureus)* microbe and incubated at 37°C for overnight (24hours). Microbial inhibition was determined by the reduction in number of bacterial colonies formed with respect to the untreated control sample using following equations:

$$R = \frac{B-A}{B} \times 100 \qquad (2)$$

Where
R = percent reduction in bacteria
A = CFU for test specimen swatches in the jar incubated for 24h contact period,
B = CFU for test specimen swatches in the jar immediately after inoculation (at “0” contact time).

2.6 Wash Durability of Finish

The Antibacterial activity of the finished samples was evaluated after being subjected to 10 wash cycles. The finished fabrics were washed using standard detergent (3% owf) at 40 °C in a Rota dyer.

2.7 Scanning electron microscopy:
Surface morphology of the fabrics was characterized using SEM of JEOL JSM 6380LA, JEOL ltd. Japan.

2.8 Mechanical Properties of the fabric:
For tensile strength and elongation, ASTM D 5034-95 test method was used whereas for tearing strength and stiffness, ASTM D1424-96R04 and ASTM D1388-08(2012) test methods were used respectively.

RESULTS AND DISCUSSION

3.1 Dyeing property of the fabrics

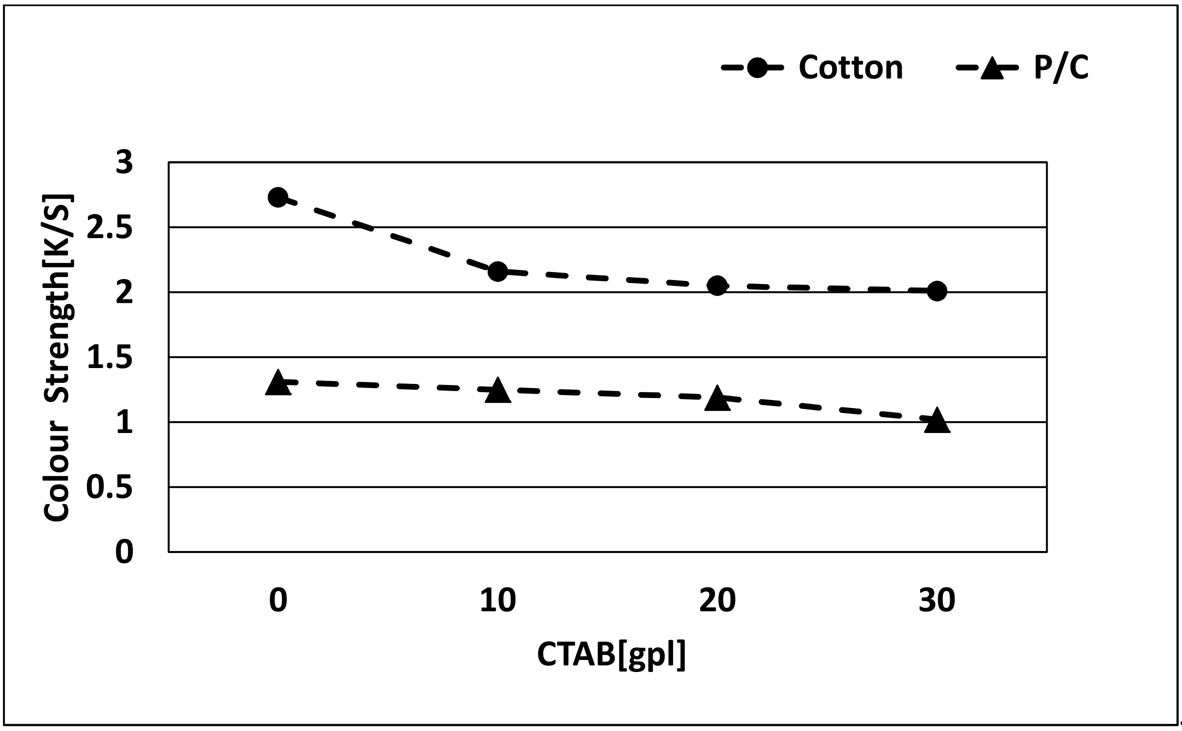

Fig 1

Figure 1shows the plot of Color strength of the fabric samples dyed by Coracryl Violet C 3R for Cotton and P/C respectively. It can be seen that addition of CTAB results in marginal decrease in the color strength. This behavior is because there is a competition between the dye and CTAB, which are both cationic in nature,to bond to the negatively charged silane film. This results in formation of some bonds between silane and CTAB which instead could have been formed between the dye and silane.

Table 1: Fastness properties for dyed and CTAB finished Cotton and P/C fabric with different concentration of CTAB

Concentration of CTAB (gpl)	Cotton					P/C				
	Rubbing Fastness		Wash Fastness		Light Fastness	Rubbing Fastness		Wash Fastness		Light Fastness
	Dry	Wet	Staining	Colour Change		Dry	Wet	Staining	Colour Change	
0 (Control)	4-5	4	4-5	2	3-4	2-3	2	3	3	3-4
10	2	2-3	2-3	2-3	2-3	2	2-3	2-3	2-3	2-3
20	2	2-3	2	2-3	2-3	2	2-3	2	2-3	2-3

Table 1 shows the fastness data of cotton and P/C blend samples. We can observe that all types of fastness get impaired when CTAB is added into the bath due to the reduction in number of ionic bonding points between dye and silane owing to the presence of CTAB. Moreover sol-gel being essentially a coating, the rubbing fastness is not expected to be of excellent quality.

However, one of the most important factors that influence fastness is the electrical net charge of the dye. It is known that silica sol coatings have an isoelectric point around pH 4, so at neutral pH they are negatively charged. For this reason positively charged dyes can interact with the surrounding silica matrix. Therefore their fastness is better than that of uncharged or negatively charged dyestuffs. Hence basic dyes are more suitable for incorporating into silica sols.

3.2 Antibacterial characteristics of the fabric

The sol-gel coated samples were evaluted quantitaively using AATCC Test 100-2004 (AATCC 2007) for the antimicrobial test and the results for percent reduction of colonies against *S.aureus* are shown in **Figures 2 &3 and Tables 2 & 3.**

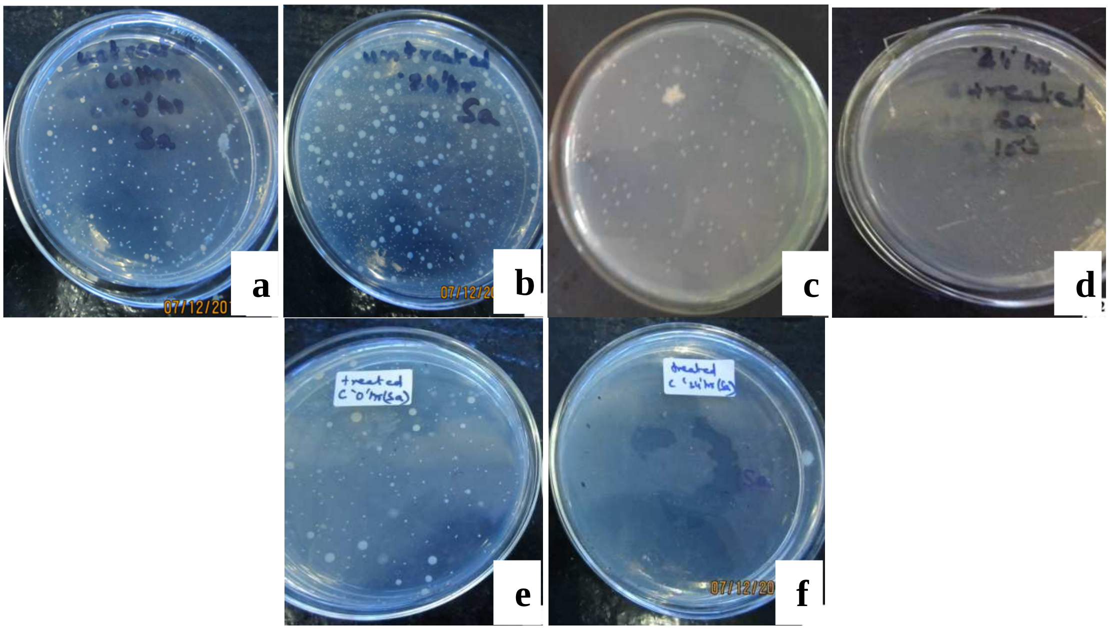

Fig 2

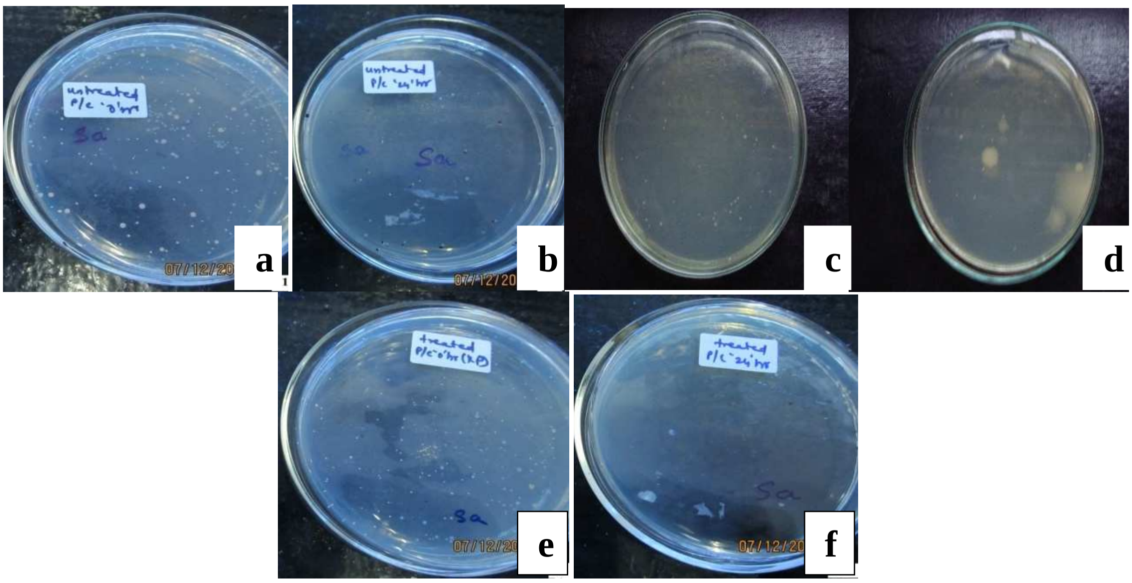

Fig 3

Table 2: Antibacterial activity for dyed and CTAB finished Cotton with CTAB for different concentration of CTAB against *S.Aureus*

Concentration of CTAB (gpl)	Before Wash			After 10 washes		
	Number of Colonies after		Reduction in colonies (%)	Number of Colonies after		Reduction in colonies (%)
	0 hrs	24 hrs		0 hrs	24 hrs	
0 (Control)	$1.53x10^5$	$13.3x10^5$	Nil	$1.73x10^5$	$15.3x10^5$	Nil
10	$2.00x10^5$	$0.326x10^5$	83.7	$3.52x10^5$	$0.99x10^5$	71.88
20	$1.63x10^5$	$0.075x10^5$	95.3	$2.09x10^5$	$0.42x10^5$	78.46

Table 3: Antibacterial activity for dyed and CTAB finished P/C with CTAB for different concentration of CTAB against *S.Aureus*

Concentration of CTAB (gpl)	Before Wash			After 10 washes		
	Number of Colonies after		Reduction in colonies (%)	Number of Colonies after		Reduction in colonies (%)
	0 hrs	24 hrs		0 hrs	24 hrs	
0 (Control)	$0.30x10^5$	$1.84x10^5$	Nil	$0.92x10^5$	$3.34x10^5$	Nil
10	$2.73\ x10^5$	$0.28\ x10^5$	89.74	$3.67x10^5$	$0.92x10^5$	74.93
20	$2.20x10^5$	$0.024x10^5$	98.80	$3.98x10^5$	$0.72x10^5$	81.91

The results indicate that cotton fabric showed colony reduction and it increased with increase in concentration of CTAB in the sols, indicating progressive increase in antibacterial activity. We can observe similar trend for P/C samples as well. This is on expected lines as CTAB is a known antibacterial compound. Just like basic dyes the positively charged CTAB gets entrapped by way of ionic bonding with the negatively charged silane film and thus it exhibits antibacterial property.The presence of coating can be confirmed by the SEM photographs of the samples as shown in **Figures 4 and 5**.

The antibacterial activity was found to decrease after subjecting the fabric samples for 10 washes. This is because of leaching of sol-gel coating due to the action of soap on the fabric.

Table 4: Mechanical properties of dyed and CTAB finished Cotton fabric for different concentration of CTAB

Concentration of CTAB (gpl)	Tensile Strength (Kgf)		Elongation (%)		Tearing Strength(Kgf)		Bending Length (cm)
	Warp	Weft	Warp	Weft	Warp	Weft	
Untreated	63.9	33.36	11.46	17.07	1.22	1.41	2.81
0 (Control)	57.7	30.55	14.21	19.23	0.64	0.69	2.71
10	56	29.04	15.47	19.87	0.64	0.67	2.64
20	53	24.63	16.10	20.04	0.62	0.65	2.57

Table 5: Mechanical properties of dyed and CTAB finished P/C fabric for different concentration of CTAB

Concentration of CTAB (gpl)	Tensile Strength (Kgf)		Elongation (%)		Bending Length (cm)
	Warp	Weft	Warp	Weft	
Untreated	111.7	63.3	59.7	32.5	1.53
0 (Control)	117.8	52.8	52.1	32.6	1.23
10	116.8	51.5	54.5	35.2	1.21
20	115.8	50.43	55.7	36.1	1.19

Table 4 and 5shows the results obtained for tensile strength testing of Cotton and P/C fabrics coated with sol-gel containing CTAB. We can note that for untreated Cotton, tensile strength was highest which got reduced when treated with TEOS and GPTMS which after addition of CTAB got further reduced with increase in concentrations of CTABin both warp and weft direction. The acidity of the sol used for coating has caused damage to fibreswhich could be the reason for the decrease of the tensile strength of the fabric coated by the sol with high silica content [28].

For P/C blend there was slight increase in tensile strength but reduced slightly with addition of CTAB.

Percentage of elongation went on increasing for both the fabric samples as the concentration of CTAB was increased. However this change in tensile strength and percentage of elongation was pronounced in case of cotton and marginal for P/C blend.

The tearing strength of cotton samples are also shown in **table 4**. It is seen that the tearing strength got substantially reduced for silane treated samples. These changes in mechanical properties can be attributed to the thin gel film formation on the fibre surface and the adhesion of net-like gel between fibers, which can be obviously observed from SEM in **Figures 4 and 5**.

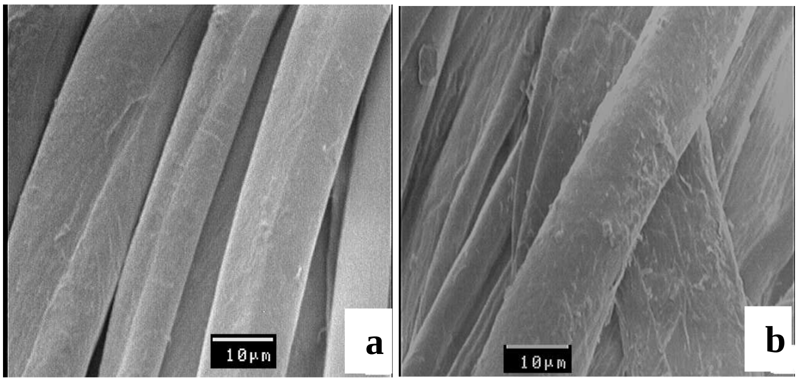

Fig 4

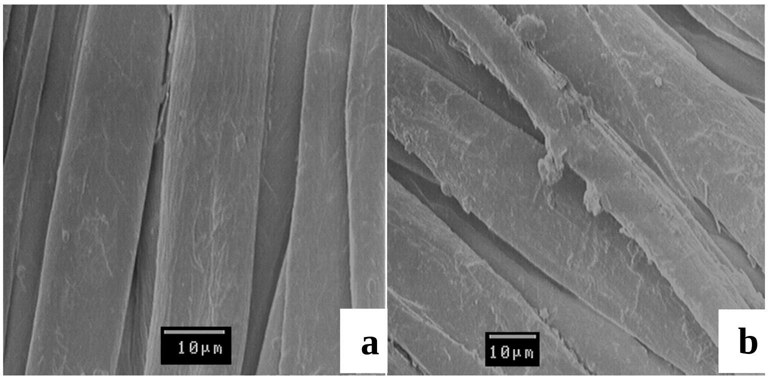

Fig 5

One interesting thing to note is that the fabric becomes softer after this sol-gel coating process containing CTAB. This is because the CTAB, which is long chain quaternary ammonium compound, might be giving cationic softener like effect to the fabric samples. This statement is supported by the reduction in bending length of the fabric.

CONCLUSION

Cotton and P/C blend was dyed with cheap basic dyes using sol-gel method by adding GPTMS as cross-linking agent. CTAB was also addedin the coating to give antibacterial textiles. Simultaneous dyeing and anti-bacterial finishing was made possible by this technique. In addition to antibacterial textiles we also get softening of fabric.However the technique adversely affected the mechanical properties of the fabric.

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