DETERIORATION OF TENTAGE IN INDIA—RESISTANCE TO DETERIORATION OF VARIOUSLY DYED/TREATED TENTAGE FABRIC

by

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ABSTRACT

A large number of treatments for protecting the tentage fabric from weathering (i.e. actinic and microbial) degradation have been investigated by outdoor exposure trials. Most of the fabrics were dyed vat, with or without mineral khaki ground and simultaneously exposed to weathering at Kanpur and Bombay representing 'hot-dry' and 'hot-humid' climates. Mineral khaki treatment by itself or as basic treatment on the fabric accords high resistance to weathering. Copper naphthenate by itself or in conjunction with vat dyes impairs resistance. Hydrated manganese oxide has been found to confer outstanding protection to cotton fabric when the exposure is confined to sunlight only.

Introduction

In the earlier experiments[1,2] reported from this laboratory, it was shown that the Indian tentage fabric which was dyed mineral khaki or which had mineral khaki ground on which dyeing with vat colours had been carried out, was more resistant to weathering than those which had been dyed direct with sulphur or vat colours. At the time these experiments were carried out, the approved camouflage shade for tentage was scamic 207 olive green. The experimental fabrics had, therefore been dyed to this shade.

Since the above results were reported, the camouflage shade for tentage has been altered to scamic 314 (olive green) obtained by using a different concentration of the same dye. In the revised context it was necessary to confirm the value of mineral khaki ground in confering resistance to weathering. Since it was apprehended that adequate capacity for basic treatment with mineral khaki may not be always available, it became also necessary to investigate whether the use of copper naphthenate, a rotproofing agent, in conjunction with all vat dyeing will make up for the poor microbial resistance of plain vat dyed fabric.

A further aspect which merited investigation was the practical value of hydrated manganese oxide in protecting the tentage fabric from actinic degradation. It was necessary to ascertain how far the above material can augment the overall weathering resistance of fabrics dyed vat, with or without mineral khaki ground.

It was also of considerable practical interest to ascertain whether the omission of chromium in mineral khaki treatment materially lowered the resistance conferred by this treatment against weathering and if it did, whether an increase in the normal proportion of the iron component could counteract the effect.

The exposure trials described in the present paper deal with the study of the above aspects. The exposures of suitably treated/dyed fabrics were carried out simultaneously at Kanpur and Bombay representing hot-dry' and 'hot-wet' types of climates respectively.

Experimental

Materials—Cotton dosuti which is the basic material of Indian Pattern Tentage was used as the experimental fabric. The fabric was scoured by kier boiling.

Treatment/Dyeing processes

(*a*) *Mineral khaki* (MK)—The mineral khaki liquor was prepared by reducing a 30 per cent solution of sodium dichromate in sulphuric acid medium with coarse sugar (Jaggery). After the reduction was complete, the density of the solution was adjusted to 24° Be by dilution with water. 12 per cent by volume of ferrous sulphate solution (20°Be) was then added. The fabric was padded in the mineral khaki liqu r using a double three bowl padding mangle. The take up of the liquor was 80 per cent. The padded fabric was dried in a hot air chamber at 82·2°C for about fifteen minutes. The dried fabric was then developed at boil in an alkali solution containing 6·8 per cent caustic soda 1·6 per cent soda ash and 0·5 per cent sodium dichromate on a jigger giving 8 ends. The fabric was then washed, soaped, washed again and finally dried.

(*b*) *Mineral Khaki top Dyed Vat to Olive Green Shade*—Fabric treated with mineral khaki was subsequently dyed to olive green shade using the vat colours Cibanone Olive Green A (1·0 per cent) and Cibanone Blue 2R (0·2 per cent).

(*c*) *All Vat to Olive Green Shade 314*—The basic fabric was dyed to olive green shade using the vat dye, Cibanone Olive Green A (1·5 per cent). On a jigger with M/L ratio of 1:5. The temperature of the liquor was maintained at 120°F and the material worked for 8 ends. The excess liquor was removed in a padding mangle. The dyed fabric was aired, rinsed, soaped, washed well and dried in the atmosphere.

(*d*) *Copper Naphthenate in Ammonia*—Requisite weight of copper naphthenate paste (7·3 per cent Cu) was dispersed in dilute ammonia (sp. gr. o. 966) to give a copper content of 0·7 per cent in the liquor. The fabric was given two ends in the dispersion on a jigger maintaining an M/L ratio of 1:10 and the take up of the liquor was 80 per cent. The fabric was dried in the atmosphere.

(*e*) *Dyed vat and treated with copper naphthenate*—Fabric dyed to olive green shade, using vat dyes Cibanone Olive Green A(1·3 per cent) and Cibanone Blue, 2R (0·2 per cent), was treated with copper naphthenate dispersion as described in (*d*) above.

(*f*) *Hydrated manganese oxide*—The fabric was padded in manganese sulphate solution (0·6 per cent metal content) using a double three bowl padding mangle, the take up being 80 per cent. The padded fabric was dried at 71°C on a stenter. The dried fabric was then given 8 ends in a 2 per cent caustic soda solution on a jigger the amount of alkali used being sufficient for complete precipitation of the metal. The fabric was washed free of alkali and dried in the atmosphere.

(*g*) *Mineral khaki treatment followed by hydrated manganese oxide*—The fabric was treated as described in (*a*) and subsequently treated as in (*f*) above.

(h) *Mineral khaki treatment followed by hydrated manganese oxide and finally top dyed vat to olive green shade*—The fabric was treated as in (g) and top dyed vat as in (b) above.

(i) *Hydrated manganese oxide treatment, top dyed vat and finally treated with copper naphthenate*—The fabric was treated as in (g) and top dyed vat as in (b) above.

(j) *Hydrated manganese oxide treatment, top dyed vat and finally treated with copper naphthenate*—The fabric was treated as in (i) and finally treated as in (d) above.

(k) *Mineral khaki without chromium*—The fabric was treated as in (a) using ferrous sulphate solution alone in the place of mineral khaki liquor.

(l) *Mineral khaki without chromium top dyed vat to olive green shade*—The fabric was treated as in (k) and then dyed to olive green shade with vat dyes, Cibanone Olive Green A (2·0 per cent), Cibanone Blue 2R (0·2 per cent).

(m) *Mineral khaki without chromium but with excess iron*—The fabric was treated as in (k) but the padding liquor contained 50 per cent more of iron salt.

(n) *Mineral khaki without chromium but with excess iron and finally top dyed vat to olive green shade*—The fabric was treated as in (m) and then top dyed vat to olive green shade as in (i) above.

Plan of exposure

(a) *Type, localities and duration of exposure*—There were two different types of exposures. In one, the fabric was exposed continuously outside to the weathering conditions while in the other, it was exposed only during hours of sunshine. The exposures were conducted at Kanpur (latitude 26°26′N, longitude 80°22′E, altitude 416 ft. above MSL) and Bombay (latitude 18°54′N, longitude 72°49′E, altitude 37 ft. above MSL) representing the 'hot-dry' and 'hot-wet' types of climates respectively. At Kanpur, both the types of exposures referred to above were carried out while at Bombay the weathering exposure alone was carried out. The weathering exposure at both the localities lasted over a period of eighteen months. The actinic exposure at Kanpur was spread over 2500 hours of sunshine. Both the exposures were started simultaneously.

(b) *Details of exposure*

(i) *Weathering exposures at Kanpur and Bombay*—The treated dyed fabrics along with the untreated control dosuti were mounted on wooden frames, one yard square. There were four such frames for each fabric at each exposure site. The wooden frames were supported on steel racks four feet above ground and were exposed inclined at an angle of 45° facing south.

(ii) *Actinic exposure at Kanpur*—Same length of fabric as in the case of weathering exposures were put up in a similar manner but only during hours of sunshine. The frames were removed indoors during the night and also during cloudy weather.

(c) *Sampling procedure*—10 replicates ($10\frac{1}{2}'' \times 2\frac{1}{2}''$ each) were drawn at the time of each sampling according to a randomised plan. The samples were drawn initially and bimonthly thereafter in the weathering exposure. In the actinic exposure, the samples were drawn initially and thereafter every 250 hours of sunshine until 1500 hours were reached. After this period, the sampling was carried out every 500 hours.

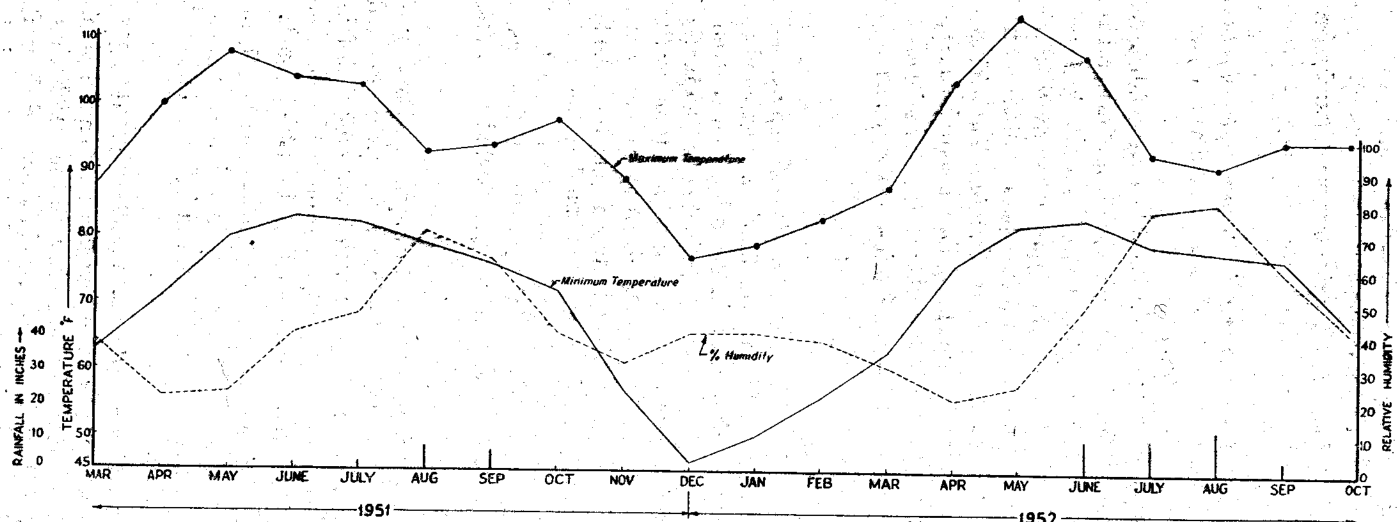
Meteorological Data

The average monthly values for maximum and minimum temperatures, relative humidity and total monthly rainfall during the period of exposure are shown in the Meteorological charts I and II.

METEOROLOGICAL CHART I

KANPUR

(LATITUDE 26° 26'N; LONGITUDE 80° 22'E, ALTITUDE 418 Ft. ABOVE M.S.L.)



KEY

——— MEAN MAXIMUM TEMPERATURE F°

——— MEAN MAXIMUM TEMPERATURE F°

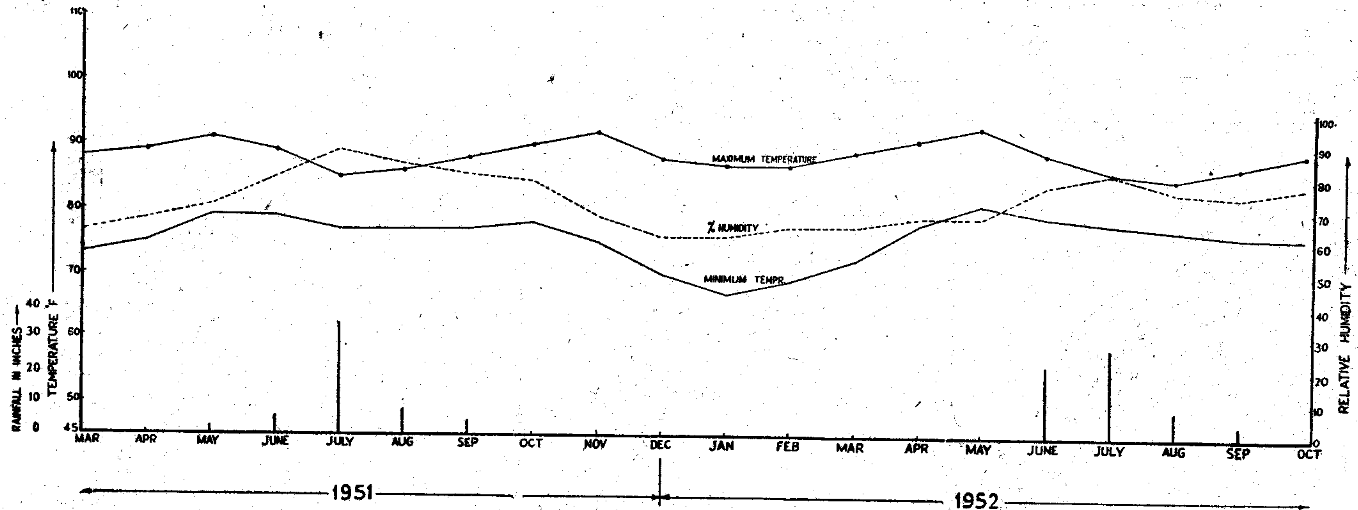
- - - - RELATIVE HUMIDITY

| RAINFALL IN INCHES

METEOROLOGICAL CHART II

BOMBAY

(LATITUDE 18° 54′ N, LONGITUDE 72° 49′ E. ALTITUDE 37 FT. ABOVE M.S.L.)



KEY

——— MEAN MAXIMUM TEMPERATURE F°

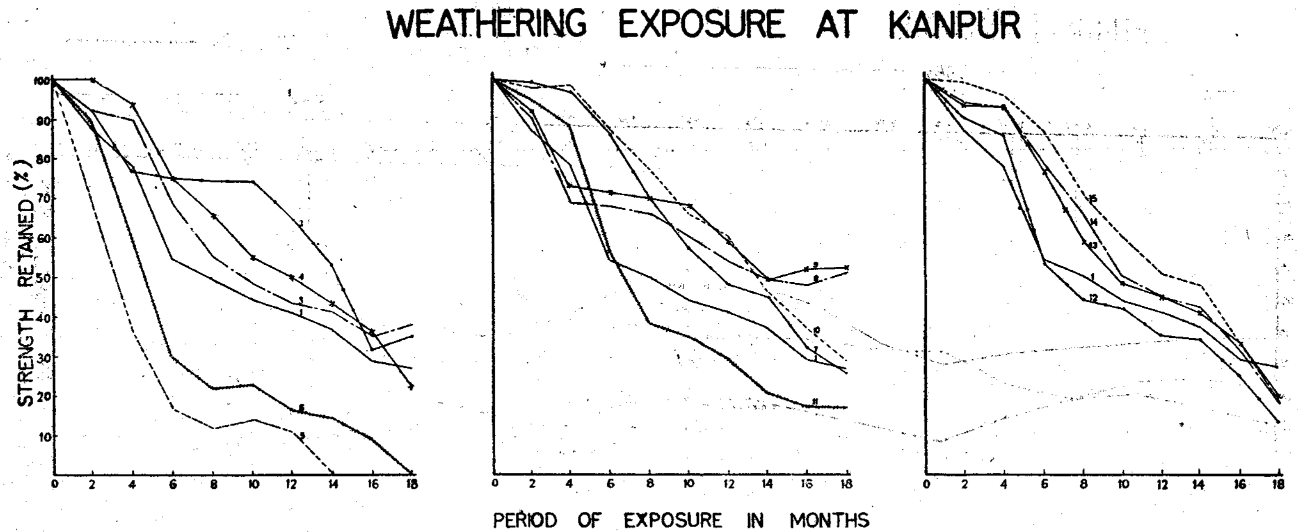
——— MEAN MAXIMUM TEMPERATURE F°

- - - - - - RELATIVE HUMIDITY

| RAINFALL IN INCHES

Test Methods and Results

(*a*) *Breaking strength*—Samples were conditioned at 21°C and 65—70 per cent R. H. for 48 hours and were broken warpwise ($2'' \times 6\frac{5}{8}''$) between grips in a Good Brand cloth testing machine, in which the test piece was stretched at a constant rate of 18 inches per minute. The results obtained in the weathering and actinic exposures are represented graphically in Figs 1, 2 and 3.



KEY

1 UNTREATED [CONTROL]
2 MINERAL KHAKI (MK)
3 MK + VAT
4 ALL VAT
5 COPPER NAPHTHENATE IN AMMONIA
6 VAT + COPPER NAPHTHENATE
7 HYDRATED MANGANESE OXIDE [Mn $(OH)_2/MnO$]
8 MK + Mn $(OH)_2/MnO$
9 MK + Mn $(OH)_2/MnO$ + VAT
10 Mn $(OH)_2/MnO$ + VAT
11 Mn $(OH)_2/MnO$+VAT+COPPER NAPHTHENATE
12 MK WITHOUT CHROMIUM
13 MK WITHOUT CHROMIUM + VAT
14 MK WITHOUT CHROMIUM BUT WITH EXCESS IRON
15 MK WITHOUT CHROMIUM BUT WITH EXCESS IRON + VAT

FIG. 1. PERCENTAGE STRENGTH RETAINED

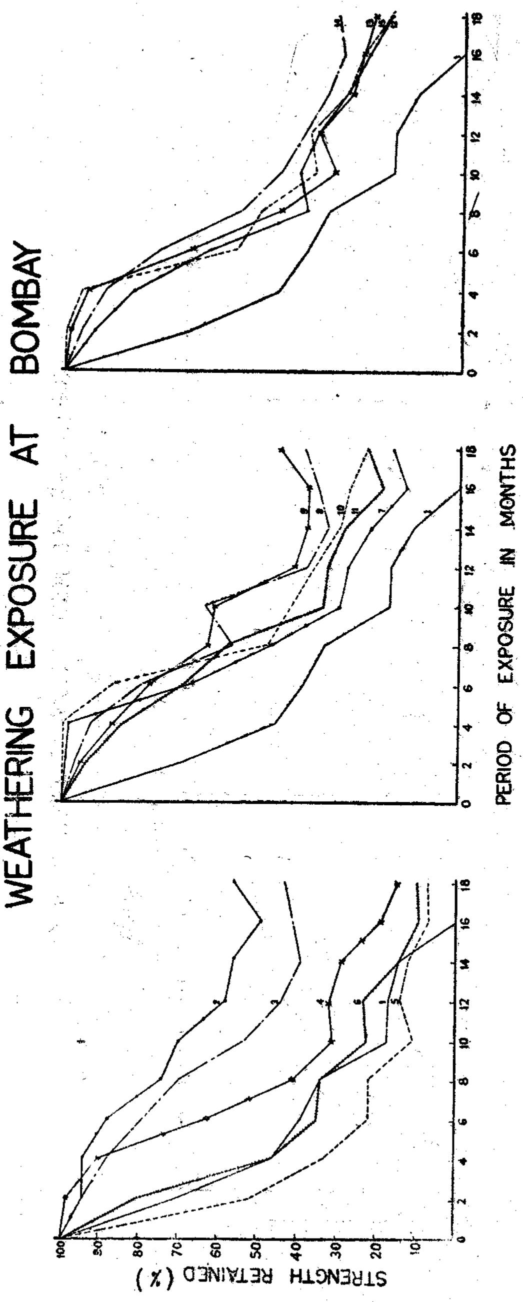


FIG. 2. PERCENTAGE STRENGTH RETAINED

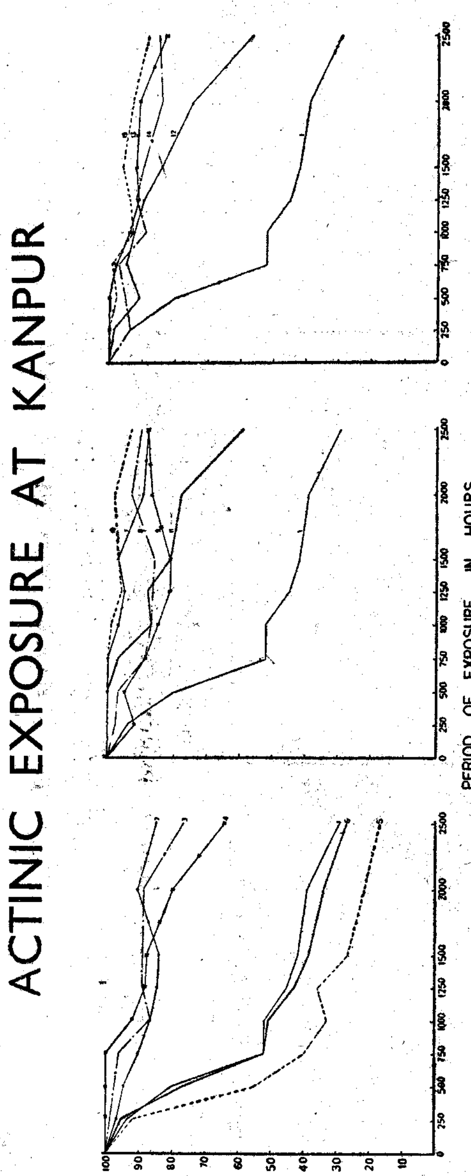


FIG. 3. PERCENTAGE STRENGTH RETAINED

(*b*) *Copper number*—The copper number of the samples was determined by the method of Clibbens and Geake[4]. The results are given in Fig 4.

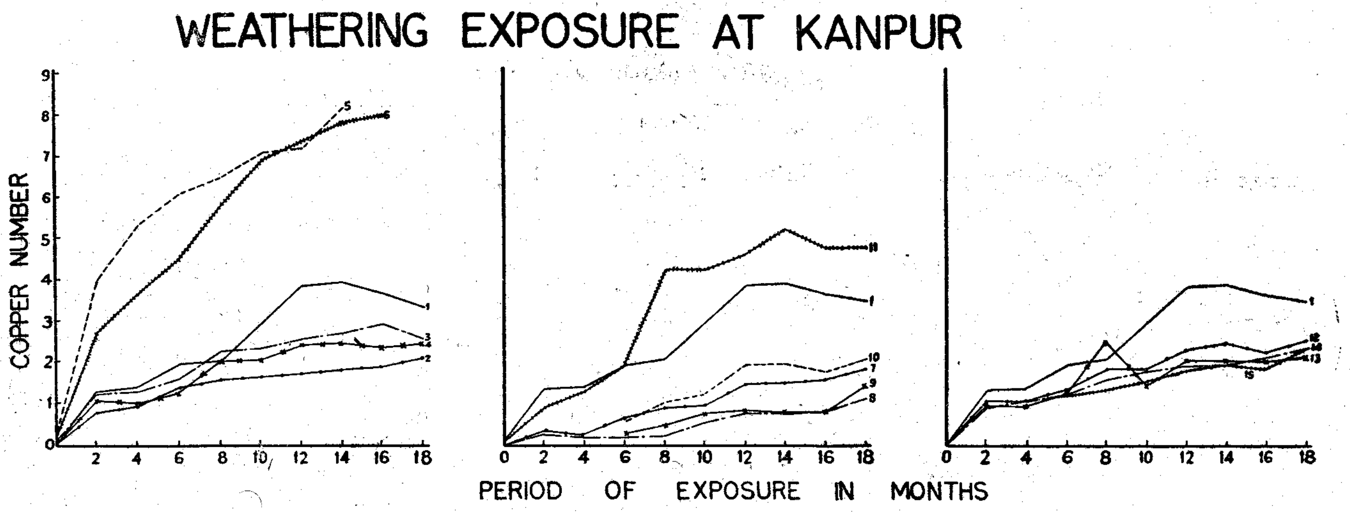


FIG. 4. COPPER NUMBER

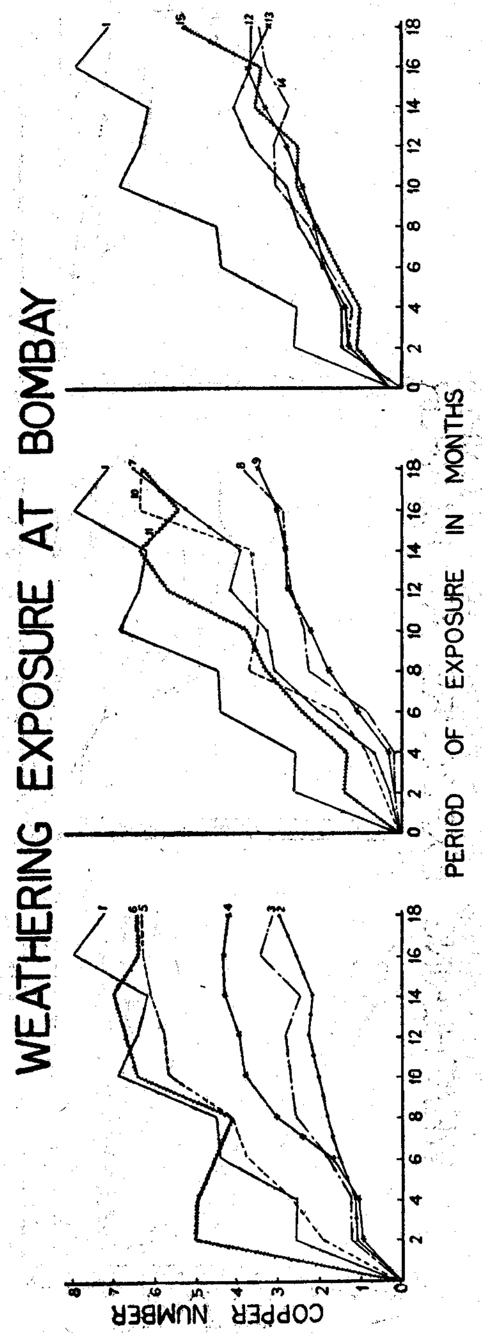


FIG. 4. COPPER NUMBER

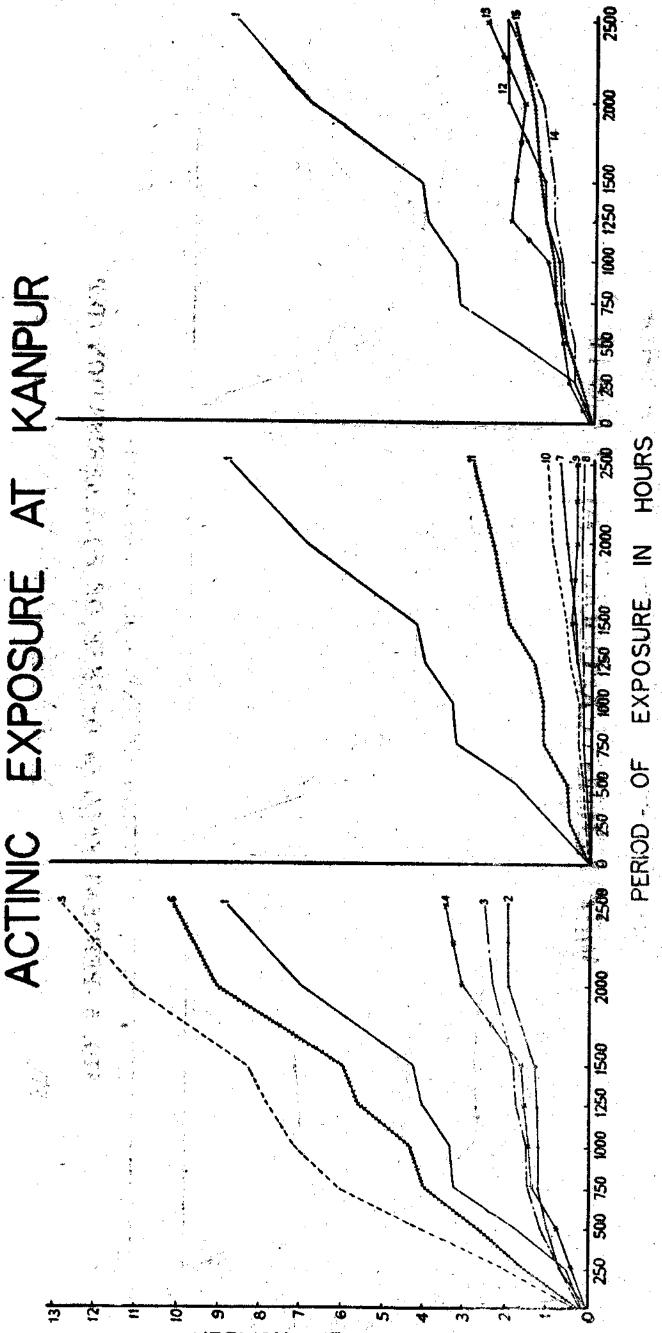


FIG. 4. COPPER NUMBER

(c) *Degree of Polymerisation*—This was carried out through measurement of the intrinsic viscosity o the corresponding cellulose nitrate in acetone solution[5]. The values for intrinsic viscosity were converted into degree of polymerisation by using the mathematical function[6,7,8] relating intrinsic viscosity $[\xi]$ with molecular weight M of cellulose nitrate $[\xi] = km^{a}$. The values of the constants K and α were $3 \cdot 8$ and $0 \cdot 93$ respectively. The results are represented in Fig 5.

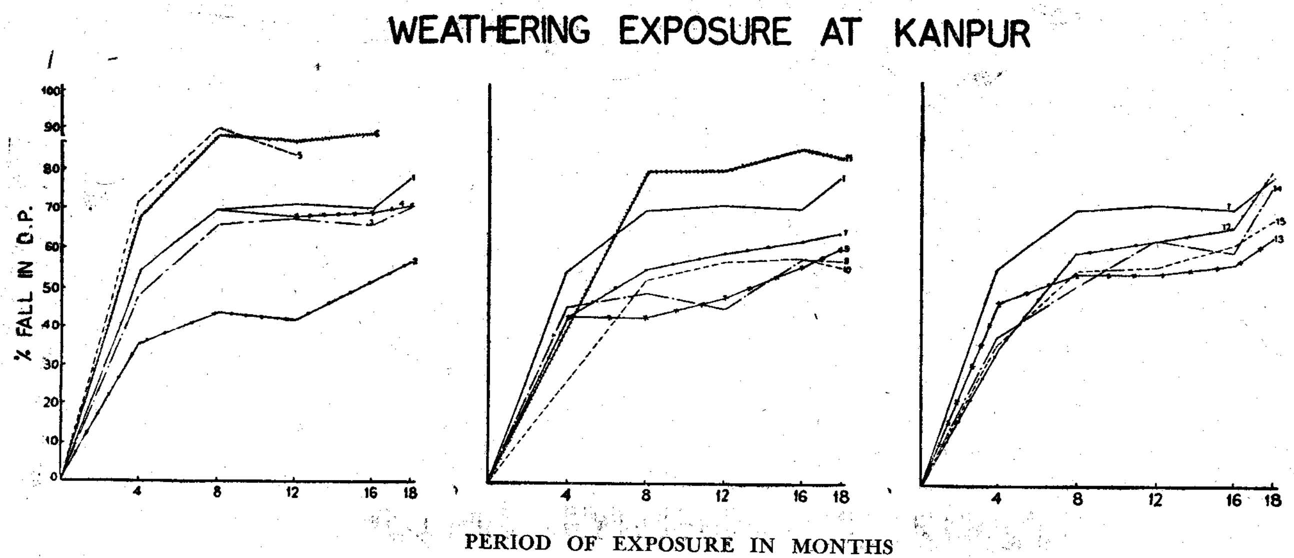


FIG. 5. PERCENT FALL IN DEGREE OF POLYMERISATION (D.P.)

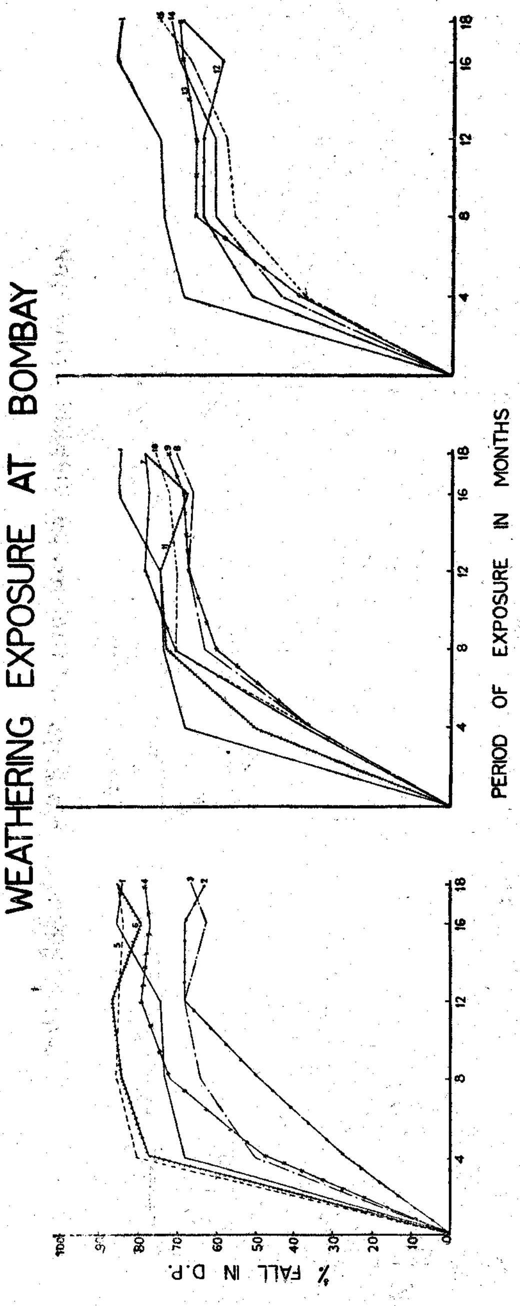


FIG. 5. PERCENT FALL IN DEGREE OF POLYMERISATION (D.P.)

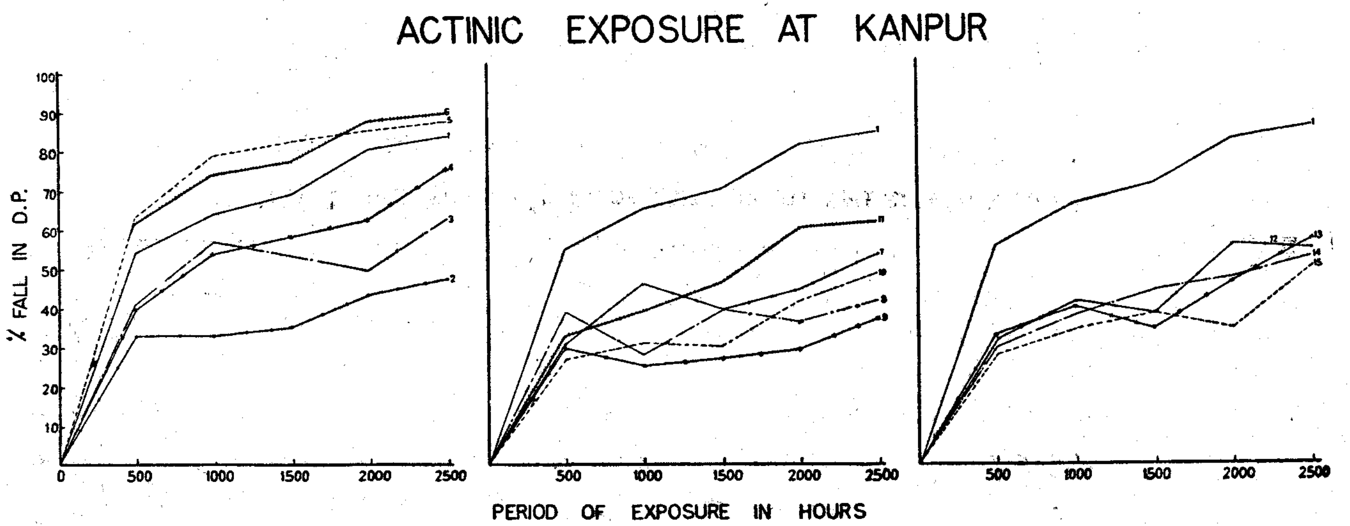


FIG. 5. PERCENT FALL IN DEGREE OF POLYMERISATION (D.P.)

(*d*) *Metal contents*—The metal contents of the fabrics were followed up during the weathering exposures and the results are given in Table I.

(*e*) *Isolation of microorganisms*—The samples in the weathering exposures were examined for visible mould growth and discolouration. Portions (1 cm square) taken at random were repeatedly washed with distilled water to remove superficial impurities and then aseptically planted separately on water-agar, Thornton and Waksman's media in Petri dishes and on moist filter paper in flasks. The Petri dishes and flasks were incubated at 30±2°C for 7—10 days and then examined for the presence of organisms. The results of isolation of fungi are given in Table II.

TABLE I

Percent Metal Contents—Weathering Exposures at Kanpur and Bombay

Serial No	Treatments/ Dyeing processes	Metal	Kanpur: Period of exposure in months 0	6	12	18	Bombay: Period of exposure in months 0	6	12	18
1	Mineral khaki (Mk)	Fe	0·77	0·77	0·82	0·83	0·77	0·81	0·79	0·80
		Cr	3·10	3·10	2·90	..	3·10	3·10	3·10	..
2	Mk + Vat	Fe	0·60	0·67	0·68	0·75	0·60	0·69	0·63	..
		Cr	3·0	2·60	2·50	1·20	3·0	2·63	2·60	2·40
3	Copper naphthenate in ammonia.	Cu	0·94	0·017	traces	traces	0·94	0·042	traces	traces
4	Vat+copper naphthenate	Cu	0·88	0·12	0·03	traces	0·88	0·014	,,	,,
5	Hydrated manganese oxide (Mn $(OH)_2$/MnO)	Mn	0·38	0·22	0·06	0·034	0·38	0·13	0·08	traces
6	Mk+Mn $(OH)_2$/MnO	Mn	1·01	0·75	0·50	0·25	1·01	0·38	0·22	0·10
		Fe	0·74	0·74	0·74	0·81	0·74	0·72	0·71	0·69
		Cr	2·97	2·50	2·90	..	2·97	2·03	3·0	..
7	Mk+Mn$(OH)_2$/MnO + Vat	Mn	0·79	0·83	0·40	0·14	0·79	0·38	0·19	0·08
		Fe	0·67	0·68	0·68	0·78	0·67	0·78	0·75	0·77
		Cr	2·87	2·66	2·60	..	2·87	2·80	2·80	..
8	Mn $(OH)_2$/MnO+Vat	Mn	0·39	0·18	0·08	0·029	0·39	0·09	0·07	0·024
9	Mn $(OH)_2$/MnO+Vat + copper naphthenate	Mn	0·37	0·08	0·12	0·003	0·37	0·11	0·13	0·041
		Cu	0·71	0·13	0·13	0·019	0·71	0·014	0·01	0·001
10	Mk without chromium	Fe	0·46	0·55	0·63	0·63	0·46	0·58	0·68	0·60
11	Mk without chromium +Vat	Fe	0·50	0·63	0·61	0·65	0·50	0·57	0·69	0·64
12	Mk without chromium but with excess of iron	Fe	0·80	0·85	0·91	0·92	0·80	0·76	0·84	0·78
13	Mk without chromium but with excess of iron+Vat	Fe	0·71	0·77	0·86	0·92	0·71	0·75	0·83	0·89

TABLE II

Incidence of fungi

	FUNGI	KANPUR									Total for the entire period of exposure	BOMBAY									Total for the entire period of exposure
		May 51	July	Sept.	Nov.	Jan. 52	March	May	July	Sept.		May 51	July	Sept.	Nov.	Jan. 52	March	May	July	Sept.	
1	Actinomyces sp.							1			1										0
2	Acrothecium sp.										0								2		2
3	Alternaria sp.					3	1				4				1						1
4	Aspergillus spp.	2	1	1	5	12	15	11	15	13	75	3		7	7	6	6	16	10	10	65
5	Botrytis sp.					1			1		2									1	1
6	Chaetomium sp.								2	2	4					2			2		4
7	Cladosporium sp.					1			1		2										0
8	Cunninghamella sp.								1		1										0
9	Dematium sp.			12	8	2					22				2						2
10	Dematiaceae (Non-sporulating forms)	1			3	6	4		2	3	19	1		10	12	6	2		2	3	36
11	Fusarium sp.										0								1	1	2
12	Haplaria sp.										0								1		1
13	Helminthosporium sp.					4	3				7				5					4	9
14	Memnoniella sp.				3					1	4				4	1	1		4	4	14
15	Monilia sp.										0						1				1
16	Mucor sp.										0								1		1
17	Paecilomyces sp.				1		1		1		3			1					3	2	6
18	Penicillium spp.	1		1	2	3	6		1	2	16	7		1	3	2	12		2	1	28
19	Phoma sp.					1	2				3										0
20	Pyrenomycetes (Group)			2							2										0
21	Rhizopus sp.					1		1	2	1	5							6			6
22	Rhodotorula sp.					1					1										0
23	Sphaeropsidales sp.					1	1				2										0
24	Trichoderma sp.								12	12	24								2	9	11
25	Tritirachium sp.										0				1						1
26	Torula sp.				1	2					3										0
27	Verticilium sp.			2	4	2	1	2	4	6	21	1		2	2	1	1	7	7	3	24
28	Unidentified isolates			4	9	11	15	5	14	12	70			6	8	19	19	12	14	18	96
	Total	4	1	22	36	51	49	20	56	52	291	12		27	45	37	42	41	51	56	311

N.B.—The figures shown against the various organisms represent the number of isolates of each sampling.

Discussion of Results

Breaking strength

Actinic exposure

(*a*) The following treatments have proved to be best:

(*i*) Hydrated manganese oxide ($Mn(OH)_2/MnO$).

(*ii*) Hydrated manganese oxide+Vat

(*iii*) Mineral khaki without chromium+Vat, and

(*iv*) Mineral khaki without chromium but with excess iron+Vat.

The above four treatments are not significantly different among themselves. The strengths retained by the treated fabrics at the end of 2500 hours of sunshine are very high (83—88%).

(*b*) The omission of chromium from mineral khaki impairs greatly the resistance of the latter but the addition of excess iron *in lieu* or top dyeing with Vat restores the resistance to about the same order.

(*c*) The least satisfactory treatments are given below in the decreasing order of resistance to actinic attack.

(*i*) Untreated control.

(*ii*) Vat+Copper naphthenate.

(*iii*) Copper naphthenate.

(*d*) It will be obvious that while the above results are important, they cannot be viewed in isolation, since under field conditions of exposure of cotton material where actinic degradation takes place, microbiological degradation also occurs to a greater or less extent.

Weathering Exposures

(*a*) *The role of mineral khaki in conferring protection against weathering*—The best treatments at both Kanpur and Bombay are as follows:

(*i*) Mineral khaki+$Mn(OH)_2/MnO$+Vat.

(*ii*) Mineral khaki.

(*iii*) Mineral khaki+$Mn(OH)_2/MnO$, and

(*iv*) Mineral khaki+Vat.

It is clear that mineral khaki by itself or in the presence of vat confers the best resistance at both 'hot-dry' and 'hot-wet' localities. This finding is in agreement with the results reported previously from this establishment[1, 2].

(*b*) *The value of hydrated manganese oxide in conferring protection against weathering*—It has been shown under discussion on actinic exposure that treatment with hydrated manganese oxide ($Mn(OH)_2/MnO$) confers excellent protection. In the weathering exposures at both Kanpur and Bombay the best treatment is the one in which $Mn(OH)_2/MnO$ is present along with mineral khaki and vat. It will be obvious that where tentage fabric is to be dyed comouflage shade 314, it is best to have mineral khaki ground, treated subsequently with $Mn(OH)_2/MnO$ and then dyed to vat. The strength retained by fabrics so processed after 18 months of exposure at both Kanpur and Bombay is about 45—52%. Where vat dyeing is not necessary finally, no special advantage accrues by resorting to treatment with $Mn(OH)_2/MnO$ on mineral khaki ground.

(*c*) *The value of copper naphthenate when used in conjunction with all vat dyeing*—At both Kanpur and Bombay, the incorporation of copper naphthenate in all vat dyeing, far from improving weathering resistance, actually impairs it. The effect is evident even in first four months of exposure. The effect is no doubt due to the fact that the protection conferred by copper naphthenate in countering microbial degradation is far outweighed by the rotproofing agents' capacity to accelerate actinic degradation.

(*d*) *The effect of omission of chromium in mineral khaki on its weathering resistance*—There is very definite evidence that the omission of chromium in mineral khaki reduces the resistance of the latter both at Kanpur and Bombay. The addition of excess iron *in lieu* of chromium increases the weathering resistance slightly at Kanpur but at Bombay the effect is not significantly different. Similar results have been obtained where instead of excess iron, top dyeing with vat has been resorted to.

Copper number

(*a*) *Actinic exposure*—The results shown in Fig 4 reveal *inter alia* that:

(*i*) wherever hydrated manganese oxide ($Mn(OH)_2/MnO$) or mineral khaki is present, the degradation is very low; where both of them are present together, the degradation is least, and

(*ii*) copper naphthenate accelerates actinic degradation (vide treatments 1, 4, 5, 6, 10 and 11 in Fig 4).

(*b*) *Weathering exposure*—The results shown in Fig 4 reveal *inter alia* that

(*i*) considerable actinic degradation takes place at Bombay.

(*ii*) treatments in which mineral khaki is present, suffer the least degradation at both Kanpur and Bombay, and

(*iii*) omission of chromium in mineral khaki increases the extent of degradation of the latter both at Kanpur and Bombay.

Degree of Polymerisation—The values for percentage fall in degree of polymerisation in both actinic and weathering exposures show clearly that:

(*a*) where copper naphthenate is present, the resistance to deterioration is poor,

(*b*) there is a tendency for mineral khaki wherever present, to improve the resistance to deterioration, and

(*c*) omission of chromium from mineral khaki impairs the latter's resistance to deterioration.

Excepting in isolated cases in well defined groups, it is difficult to select outstanding treatments from the data provided in Fig 5. These data do not bear a close relationship to those recorded under breaking strength. This lack of relationship indicates that in exposure trials the breaking strength of a woven fabric such as dosuti is not entirely influenced by the average molecular weight of the cellulose. Under controlled conditions of irradiation of a woven fabric to ultraviolet rays in the laboratory, a parallelism has been found to exist between average molecular weight of the cellulose and breaking strength of fabric[9].

Metal Contents—The results summarised in Table I indicate that while chromium and iron as oxides are generally resistant to leaching effects during weathering, copper as copper naphthenate and manganese as hydroxide/oxide are very rapidly lost, more so at Bombay than at Kanpur. This result is of some importance since the protective action of hydrated manganese oxide is dependent on metal content[10].

Incidence of microorganisms

(*a*) Although the weathering exposures commenced in the middle of March, 1951 the first sign of mould growth appeared during the early stages of monsoon (1951) at both Kanpur and Bombay. This is in conformity with the finding reported in previous exposure trials[11]. Side by side the fungal growth, algal growth was observed at Bombay.

(*b*) During the course of isolation of organisms in the laboratory a large number of bacteria appeared in the media in addition to fungi. They were rather numerous during the earlier part of the month (1951) and disappeared later gradually. This is, no doubt, due to the fact that high moisture requirement for bacterial growth can be met with on exposed fabric, only during monsoon. The bacteria, however, did not occur during the subsequent monsoon (1952) of the exposure period. The non-occurrence of bacteria is to be associated with the intense fungal activity and a parallel instance can be seen in the deterioration of a fabric buried in moist soil[12], where bacteria flora are predominant during early stages, but disappear with the gradual advent of the fungion the fabric.

(*c*) The fungal flora isolated from the exposed fabrics do not show any notable difference in the types of fungi so far as the two localities are concerned. About 20 important types of fungi have been found to occur at both the localities. Amongst these *Aspergillus, Penicillium, Curvularia and Trichoderma* are the predominant genera. Non-sporulating forms, as also certain unidentified types of Dematiaceae have also been found. These fungi were present on fabrics irrespective of treatments/dyeing processes. *Memnoniella echinata* and *Chaetomium* sp. which have not been commonly accorded on exposed fabrics, have been found to occur at both the localities.

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