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Efficacy of Enzymes and other Stain Removal Techniques in Museum Textiles

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Abstract: *Textiles and their products are susceptible to a variety of stains due to varied use and environment. Primary reason for the vulnerability of textiles is their organic origin, which renders them fit as food for microorganisms. The problem of pest infestation multiplies in case of museum textiles as most part of it is kept in storage for long periods of time. Even mild pest activity on surface of fabrics can result in tough stains originating from pest saliva, excretion and by-products of fiber breakdown. Termed as 'second-skin' by scholars, textiles evidently witness every single activity that becomes part of our life. Thus, in addition to the common stains like food, cosmetics etc, not so common stains like blood also find place on these organic surfaces. Most of the decorative textiles are either made in decorative weaves or a combination of base fabric with embellishments which many a times happen to be metal in various forms. These in turn give way to the persistent problem of fabric staining and damage due to rust. This research paper would primarily focus on the removal of three stains, i.e., rust, mould and blood and comparison of procedures for stain removal by traditional indigenous techniques, contemporary laboratory procedures and enzymes*

Keywords: *Textile Conservation, Museum Textiles, Stain Removal, Enzyme treatment, Wet Cleaning, Rust, Mould, Blood stains*

I. INTRODUCTION

Stain management is probably one of the most important elements of textile conservation as it impacts both visual and functional aspects of the fabric. Stain removal has essentially remained a spot cleaning technique both in commercial, domestic as well as conservation spaces. The parameters of stain removal in case of conservation are very different from regular domestic or commercial cleaning. In customary cleaning, restoring the original look of the textile is of utmost importance. Thus, the expert has liberty of going a little further, using stronger treatment. However, in case of conservation, spots are removed for varied objectives [1]. At times a spot itself is identified as part of the object's history, thus spot cleaning is done only to remove organic matter present on the stain which would otherwise enhance rate of deterioration of the textile. The resultant colouring from the stain is often conserved and left to narrate the story of the textile and its encounter with the stain. In case the stain is an unwanted, accidental phenomenon, hindering with the look and tale of the object, it will be removed or concealed only to the extent it does not harm the strength and colour of the fabric. [2]. Conservation treatments aim at localised or non-aqueous treatments of fabrics as water can cause lot of damage to the fragile and partially known nature of the textile. If dye on the fabric is not fast enough, aqueous immersion can cause dye bleeding and thus destroying the artefact. Apart from that, fragile artefacts might simply disintegrate by weight of water absorbed by the fibers itself. Also, water might activate a host of other susceptibilities in the fabric, lying dormant in the dry environment. For similar reasons, use of surfactants and chemical bleaches are not a plausible option in case of museum textiles. Blood, rust and mould are three complex stains encountered by restorers and conservators for varied reasons. Blood stains are complicated because of its proteinic character. Use of water at slightly higher temperature would coagulate the proteins and permanently fix it over the fabric. Making matters worse, oxidation of the iron component of blood, makes old stains turn brown and further binds the protein with the fabric. Just like blood, rust is another sensitive stain on fabric for various reasons. As mentioned earlier, rust is formed by oxidation of iron and its derivatives. What makes matters worse in case of textiles is that if the oxidation process is happening for a long duration of time, the fabric in immediate contact of the rusted accessory gets involved in the process of oxidation. Thus, fibers around the affected area get partially or completely eaten up, thus weakening the fabric and starting its disintegration. Unlike blood and rust, fungal stains like mould and mildew pose a completely different set of challenges to the conservators. Precursors of these types of stains could be many, as museum textiles need to be kept in dark storage for most part of their life. Although visible part of mould is the velvety superficial layer on the fabric, challenge for the conservator is to permanently deactivate or remove the stain causing microorganism from the structure of the textile. The present research was designed to compare different techniques of stain reduction in terms of both strength reduction and stain reduction achieved.

It should be noted that primary property of enzymes that makes it a safe bet is its specificity. If used carefully, an enzymatic reagent would not impact the base fabric like bleaches and other detergents. Similarly, indigenous methods like *Reetha* cleaning might provide viable options for museum textiles as they are expected to be less harsh on the base fabric. Novel cleaning techniques like ultrasonic cleaning can also open new prospects for sensitive substrates like museum textiles.

II. LITERATURE REVIEW

Published references about removal of stains using laboratory reagents can be found as early as 1921, when Elledge and Wakefield published a detailed description about use of various acids, alkalis etc. for stain removal from fabrics [3]. Singh et. al. (1992), described how certain chemicals together with Japanese tissue paper can be effectively used for removing stains from *Thangka* paintings [4]. Prasad (1982) explained that *Reetha* solution is slightly acidic in nature and hence the most delicate natural fabrics can be washed without destroying the fibres of the fabric as well as colour of the fabric [5]. The potential of ultrasonic cleaning in conservation has been recognized for some time. Barton et. al. (1986), reported that archaeological conservation in Europe has resorted to this type of cleaning in dealing with waterlogged wood, textiles and leather artefacts [6]. The principle of ultrasonic cleaning is the generation of mechanical impulses through a liquid at high frequencies. These impulses create minute bubbles of vacuum which implode against the immersed object, creating shocks which clean its surface [7]. Literature about use of enzymes is available from as early as 1986. In one research paper Chapman discussed the role of Amylase in effectively removing starch (used for mounting the fabrics) from the back of the fabric [8]. While researching the impact of Cellulase enzyme in combination with scouring chemicals, Csiszar (1998) observed that consecutive Cellulase treatment and alkaline scouring produced bleaching effect on cotton fabric [9]. Also findings of a study conducted by *Diller et. al* (1998) emphasised the use of Glucose oxidase as an effective bleaching agent when used in combination with Cellulase or protease enzymes [11].

III. METHODOLOGY

Stained samples were simulated in plain weave, undyed cotton fabric of medium thread count. Stains selected for the purpose were blood, mould and rust.

- 1) *Rust*: For the purpose of simulation, all iron pins were placed on a moist fabric and the same was left wrapped for a period of seven days for assisting the rust stain.
- 2) *Mould*: AATCC test method-30-2004, (Antifungal Activity, Assessment on Textile Materials: Mildew and Rot resistance of Textile Materials) was used to develop mould on the test fabrics. Cotton cloth was exposed in the soil bed for seven days during the test period to verify fungal activity. Specimens were buried horizontally on 10.0+/- 1.0 cm (3.9+/-0.4 in) of soil, spaced at least 2.5 cm (1.0 in) apart and then cover with 2.5 +/- 0.5 cm (1.0+/- 0.2 in) of test soil.
- 3) *Blood*: AATCC test method 104-1999 (Colourfastness to water spotting) was referred for placing stain on the test fabrics as the pressure and amount of stain placed on the fabric had to be uniform. With tip of pipette in contact with fabric, blood was run onto the specimen (0.15ml) at room temperature. It was worked with rounded glass rod to assist penetration. Since the time duration for mould simulation was 7 days, blood stains were also cured for seven days before any removal treatment was performed. Blood was obtained from a pathological laboratory. The blood sample was provided from left-over blood samples after the required testing had been completed by the laboratory.

A. Removal of Stains

Samples were cleaned with different wet treatments (water treatment, reagent spotting, dry-cleaning, ultrasonic cleaning, enzyme treatment and indigenous treatment).

- 1) *Water treatment*: the stains were dipped in distilled water for a period of 10mins, to assist penetration and loosening of stain. Further soft paint brush was used to remove the superficial layer of stain. No mechanical agitation was applied to the fabric, keeping into consideration that the procedure is being tested for potentially weak fabrics. Samples were flat dried thereafter.
- 2) *Home Laundry*: Home laundry techniques are probably the oldest and simplest means of sanitizing fabrics. Primary merit of this method is that worker gets to closely interact with fabric at every stage of treatment. This ensures possibility of simultaneous improvisation, while fabric is still under treatment. For the purpose of this study AATCC test method 61-2007 was followed.
- 3) *Method*: Test no 1A- was used. Laundering machine was adjusted to maintain the designated bath temperature of 40+/-2⁰C. The wash liquor was prepared with total liquor volume of 200ml and detergent concentration at 0.37%. Test was run in lever lock stainless steel canisters of size 75X125 mm with 10 steel balls in each canister. The laundering machine was run for 45mins after which each test specimen was rinsed in a separate beaker. Each specimen was rinsed three times in distilled water at 40+/- 2⁰C with occasional stirring and hand squeezing. To remove excess water, flat specimens were pressed between folds of blotting paper. Thereafter, specimens were air-dried, placed flat on a blotting paper. A commercial detergent was used for cotton fabrics whereas a neutral soap was used as 'non-ionic' detergent for wool and silk. This was used as commonly available market reagents are being used by most conservation laboratories in India.

4) **Laboratory reagent spotting:** Stains were first treated with water as in the first process and further treated with advised laboratory reagents, commonly used in conservation laboratories. Following reagents were used for respective stains as per data collected in the first phase of study:

- a) **Rust:** Oxalic acid (2-3%), (ph-8.3-10) after treatment with lime water
- b) **Blood:** Thioglycolic acid (> 1% solution)
- c) **Mould:** Denatured spirit

After treatment, all samples were washed with distilled water and then dried before placed for evaluation.

5) **Enzyme treatment:** as evident from the literature review on enzymes, there is no specific enzyme proposed for the nature of stains selected in the research. However, published research has repeatedly emphasized the substitution of chemical bleaches by Glucose Oxidase for the purpose of enhancing whiteness of fabrics without considerable strength loss (11). Thus Glucose Oxidase was used as enzymatic bleaching agent for stain removal. The procedure used was as follows:

Glucose Oxidase (SRL brand -074040-Glucose Oxidase (GOD) extra pure)

- a) **Step I:** Glucose Oxidase-25units/ml (0.04mg/ml) Liquor ratio-1:20 / D glucose – 10g/l / pH-5 (acetate buffer) / Temp-35° C / Time – 60mins
- b) **Step II:** pH-10/Magnesium Sulphate – 2g/l/Non-ionic surfactant (triton *100)/Temp-85° C Time– 90mins [12], [13]

6) **Dry cleaning:** The stained samples were provided with PERC treatment as advised in AATCC test method 158-1995.

7) **Ultrasonic Cleaning:** the stained samples were placed in a smaller ultrasonic machine and treated in the presence of ‘surf excel’ as reagent for a period of 5mins. Time period of 5mins was selected as this time period proved to cause minimum strength damage as per the results obtained in the previous section of work. The treatment was performed at room temperature.

8) **Indigenous technique: Reetha** (*Sapindus Mukorossi*) was tried for stain removal as literature review suggested efficiency of this indigenous medium for cleaning of stained and weakened textiles. Thus fresh solution of *reetha* powder in water (10 %) was utilized for the purpose. The samples were dipped in this solution for a period of two hours. After every 30 mins sponge roller was worked on the samples to ensure soft agitation and better penetration of reagent in the stains. The process was carried out at room temperature.

B. Evaluation

Evaluation of treated samples was done based on two criteria, i.e., fabric strength reduction and efficiency of stain removal. Thus, fabrics were individually treated with stain removal procedure and made to undergo tensile testing (Grab Test- ASTM D 5034-09). Efficiency of stain removal was tested using ASTM D 4265-98. Visual examination technique was utilized as samples like mould and rust cannot be produced in a controlled manner. The treated test-swatches and the control sample for each group were displayed on a flat, neutral-coloured (gray) non-glare finished surface under standard daylight. The stained/treated samples were displayed in three groups, i.e., rust, mould and blood. In each set, the stained/treated samples were coded for their treatment from 1-6. Three judges were independently provided with a tabular grading sheet to grade the removal of stains on swatches to a nearest rating (Appendix-IV). Ratings were done in on a nine-point scale (Table-1) from A-E, where rating of A was suggested for a sample displaying no-change in stain, and a rating of E was kept for samples displaying complete removal of stain. Intermediate ratings between A-E were used to grade the removal of stain between these two conditions. Also, samples falling between two categories were rated in between the corresponding categories.

Table 1: Rating Scale used to evaluate Stained samples

RATING	INTERPRETATION
A	STAIN UNCHANGED
A-B	IN BETWEEN A & B
B	STAIN SLIGHTLY REDUCED
B-C	IN BETWEEN A & B
C	STAIN CONSIDERABLY REMOVED
C-D	IN BETWEEN A & B
D	STAIN ALMOST REMOVED
D-E	IN BETWEEN A & B
E	STAIN COMPLETELY REMOVED

After the completion of evaluation exercise, the ratings in the evaluation sheet were summarized on a master evaluation sheet, where the most dominant rating in each category was identified. This set of most dominant ratings was then considered as reference for final inference. Final inference was derived not only based on best stain removal but also taking into consideration the implication of each treatment on the strength of the fabric. Thus, combining the results of stain removal efficiency and reduction of fabric strength, most suitable technique for removal of each stain was identified.

IV. RESULTS & DISCUSSIONS

A. Tensile Strength

It can be seen from Table 2. that washing with distilled water causes negligible strength reduction in the fabric, both in terms of stress and strain. Further, dry-cleaning comes out to be the most feasible option in terms of least strength reduction. Ultrasonic wash 5 minutes also provides equivalent option in terms of lesser strength reduction, closely followed by *reetha* treatment. Also, SD and CV% values indicate the accuracy of the data obtained.

Since, Laboratory reagents have been used as spot cleaning treatments as opposite to other cleaning treatments which are full immersion techniques; there tensile strength was not evaluated by above technique. However, chemical reagents used cannot be expected to cause lesser strength reduction than above mentioned techniques.

Table 2: Effect of Stain Removal Methods on Breaking Load and Extension of Cotton Fabric

	S.No	Control Sample	Water Wash	Home Laundry	Dry-Cleaning	Enzyme Wash	Ultrasonic 5mins	<i>Reetha</i> Treatment
BREAKING LOAD (Warp) (N/m ²)	S-1	7.00	7.4	4.10	5.30	4.20	4.90	4.6
	S-2	8.80	8.1	4.70	5.20	4.70	4.40	5.1
	S-3	8.30	8.2	5.00	5.10	5.30	4.80	4.7
	S-4	8.00	7.9	4.70	5.10	4.90	5.20	5.2
	S-5	7.70	7.5	5.80	5.20	4.70	4.80	4.8
	Average	7.96	7.82	4.86	5.18	4.76	4.82	4.88
	Standard Deviation	0.67	0.36	0.62	0.08	0.40	0.29	0.26
	CV %	8.46	4.56	12.73	1.62	8.35	5.94	5.30
	% Change	-	1.76	38.95	34.92	37.69	39.45	37.94
BREAKING LOAD (Weft) (N/m ²)	S-1	6.90	7.70	4.20	6.30	3.10	4.60	5.70
	S-2	8.90	6.70	4.00	6.40	2.80	6.20	4.50
	S-3	8.20	8.00	4.40	5.60	3.00	5.10	5.10
	S-4	6.90	6.90	4.50	5.60	3.40	5.00	4.90
	S-5	7.80	7.00	4.70	5.30	2.70	5.10	5.00
	S-6	7.50	7.40	5.30	4.90	3.50	4.90	4.40
	S-7	7.00	6.50	4.00	5.50	3.80	5.70	4.00
	S-8	6.00	6.90	4.60	5.10	3.10	5.80	4.90
	Average	7.40	7.14	4.46	5.59	3.18	5.30	4.81
	Standard Deviation	0.90	0.52	0.43	0.53	0.37	0.54	0.51
	CV %	12.17	7.22	9.58	9.49	11.63	10.19	10.68
	% Change	-	3.51	39.73	24.46	57.03	28.38	35.00
	EXTENSION % (Warp)	S-1	15.00	13.33	3.33	3.33	6.67	8.33
S-2		13.33	13.33	3.33	3.33	6.67	6.67	6.67
S-3		13.33	13.33	3.33	3.33	6.67	8.33	6.67
S-4		10.00	13.33	3.33	3.33	6.67	8.33	8.33
S-5		13.33	13.33	3.33	3.33	6.67	8.33	6.67

	Average	13.00	13.33	3.33	3.33	6.67	8.00	7.00
	Standard Deviation	1.83	0.00	0.00	0.00	0.00	0.75	0.74
	CV %	14.04	0.00	0.00	0.00	0.00	9.32	10.60
	% Change	-	-2.54	75.02	75.02	48.70	38.46	46.15
EXTENSION % (Wett)	S-1	16.67	21.67	3.33	6.67	8.33	10.00	8.33
	S-2	25.00	21.67	3.33	6.67	8.33	8.33	8.33
	S-3	16.67	21.67	3.33	6.67	10.00	10.00	10.00
	S-4	23.33	21.67	3.33	6.67	8.33	10.00	8.33
	S-5	21.67	21.67	3.33	6.67	8.33	10.00	8.33
	S-6	20.00	21.67	3.33	6.67	8.33	10.00	10.00
	S-7	18.33	21.67	3.33	6.67	8.33	10.00	8.33
	S-8	20.00	21.67	3.33	6.67	8.33	10.00	8.33
	Average	20.21	21.67	3.33	6.67	8.54	9.79	8.75
	Standard Deviation	3.01	0.00	0.00	0.00	0.59	0.59	0.77
	CV %	14.91	0.00	0.00	0.00	6.90	6.02	8.84
	% Change	-	-7.22	83.52	48.70	35.92	23.08	56.70

B. Stain Reduction

As described in ‘Materials and Methods’ section, stain reduced samples were evaluated by three people independently as per the laid out methodology. The ratings provided by them have been listed in table 3.

Table 3: Ratings given to various Stain-reduction samples

STAIN/SAMPLE	Evaluator	Control	Water Treatment	Enzyme Treatment	Home Laundry	Reetha Treatment	Dry Cleaning	Ultrasonic Wash 5mins	Lab. Reagents
	CODE	1	2	3	4	5	6	7	8
BLOOD	Evaluator 1	-	A	B-C	B	B-C	A	A	D
	Evaluator 2	-	A	C	B	C	A	A	D
	Evaluator 3	-	B-A	C	C	C-D	A	C	D-E
	Concurrent Reading	-	A	C	B	C	A	A	D
MOULD	Evaluator 1	-	A-B	D	A-B	B-C	D	A-B	D-E
	Evaluator 2	-	A	C-D	A	A-B	C-D	C	A-B
	Evaluator 3	-	C-D	C	D	D	C	A	D
	Concurrent Reading	-	A	C-D	A	B	C-D	A	D
RUST	Evaluator 1	-	A-B	A-B	A-B	A-B	B-C	A-B	A-B
	Evaluator 2	-	A-B	A-B	A-B	A	A-B	A	C-D
	Evaluator 3	-	C	C	C	C	D-C	C	C
	Concurrent Reading	-	A-B	A-B	A-B	A	B-C	A	C

A=UNCHANGED, B=SLIGHT STAIN REMOVAL, C=CONSIDERABLE STAIN REMOVAL, D=STAIN ALMOST REMOVED, E=STAIN COMPLETELY REMOVED

It can be seen from the Table 3. that spot treatments by laboratory reagents have produced most satisfactory results in most of the cases. In case of blood stain, enzyme and *reetha* treatment results in considerable stain removal but not as much as by Thioglycolic

acid. Considering, that acidic laboratory reagent would be harsher than the other two, the conservator could choose between any of the three treatments depending on the condition of the artefact. The situation looks similar in case of mould stain. Also, for the rust stain, only dry-cleaning provides an option closer to reduction achieved by laboratory reagent. Thus, conservator can choose between the two, in discussion with the stake-holders.

V. CONCLUSIONS

- A. Lab reagents have most profound impact on rust, mould and blood stains. However, they cannot be recommended for museum textiles because of their harsh impact on fabric substrate.
- B. Dry-cleaning or cleaning by organic reagents is the most efficient technique for removing mould & rust stains with minimum loss to fabric strength.
- C. Blood stains are best removed by *Reetha* treatment or enzyme wash with minimum damage to fabric strength.
- D. Enzyme treatments have the capability to become safe and efficient tools for conservators, aiding in effective stain management of textile artefacts. However, enzyme treatment processes need to be optimized for museum textiles with future research.

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