

Optimization of fastness and tensile properties of cotton fabric dyed with natural extracts of Marigold flower (*Tagetes erecta*) by pad-steam method

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Abstract: A new approach was investigated for appraising the fastness properties while maintaining/ upgrading the tensile strength of 100% cotton, light weight fabric samples, dyed with natural colourant of Marigold flower (*Tagetes erecta*). Various dyeing formulas planned with the aid of different dyeing auxiliaries utilized as mordants, cross linkers, finishing agents and UV absorbers were applied by pad-steam (two-bath-two-stage) dyeing procedure. All the recipes were exercised both as pre and post treatments. Pretty yellow fabric samples resulted from the experiments having better colour fastness, enviable K/S value and adequate tensile strength. Some of the mordants (such as FeCl₃ and Al₂(SO₄)₃) produced samples of darker shades, entirely different from the yellow colour. The presence of CuSO₄ in the dyeing recipe produced sample with bright yellow shade. This mordant was excelled by all other dyeing auxiliaries in enhancing not only the colourfastness but also presenting greater colour strength (K/S value). Post treatment of a reactive UV absorber based on oxalinilide (UV-SUN) and fibre-reactive UV absorber Rayosan C (heterocyclic compound) imparted very good lightfastness (6 Blue Wool Standard). Crosslinking agent Fixapret CPF, methylation product based on glyoxalmonourein resulted in remarkable improvement in crocking and light fastness of cotton. Fixative Albafix WFF (quaternary ammonium compound) was also found effective in acquiring very good rub fast cotton samples.

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Introduction

Colouration of textiles is an area where constant exploration and innovation is essential for the assemblage of variety of reconcilable shades. Application of natural dyes has not attained any tremendous approval as procedures of dyeing have not been standardized and therefore, their shades are not reproducible. Scientific knowledge of chemistry on dyes and mechanism as well as technology of dyeing is inadequate. Natural dyes are chiefly mordant dyes, however, some acid, direct, pigment and vat types are also found. They are classified on the basis of structure and hue. In classification based on structure, the most commonly available natural dyes comprised of indigoids, anthraquinones, alpha naphthoquinones, flavones, dihydropyrans, anthocyanidin and carotenoids (Prabhu & Bhute, 2012).

The demand for eco-friendly products is always growing throughout the world. The present trend of utilization of synthetic dyes in the textile industry is switching over towards the use of naturally occurring colourants (Gulrajani et al., 2001). The use of natural dyes can play role in minimizing pollution and risk to human health (Ali et al., 2009). As compared to their synthetic counterparts, these dyes are easily biodegradable and highly compatible with the

environment (Zarkogianni et al., 2010). They are non-carcinogenic and non-toxic by nature that is why these colourants are believed to be safer. Their major shortcomings are non-uniform shades and poor to moderate colour fastness (Zarkogianni et al., 2010). Due to the low colour fastness compared with synthetic dyes and the complex extraction and storage procedures, their commercial use is still limited.

There are numerous plants that provide natural dyes, utilized in the textile industry. Some examples of plants used for producing natural dyes are; Alkanet, Balsam, Bougainvillea, Canna, Tulsi, Terminalia Arjuna, etc. (Vankar, 2013). Marigold flower (*Tagetes erecta*) is grown as a cut flower and a garden flower, in addition to being grown for its medicinal values. These flowers are rich in yellow to orange red colour and contain high ratio of lutein, a carotenoid pigment (Jothi, 2008). Yellow dyes generally produce pale shades and their wash fastness rating ranges between fair and excellent (Ratnapandian et al., 2012).

Natural dyes are conventionally applied by exhaust method that consumes extensive amounts of water and energy. They can however, be applied to cotton fabric by padding technique to produce appropriate light, washing and rubbing fastness (Kumaresan et al., 2013). They are lacking in having a

direct affinity for cotton fabric. In order to impart this quality, some suitable chemicals such as mordants are introduced to the dye bath. They are believed to set the colour and increase dye uptake by textile fibres. Surfactants are also needed in dyeing bath. They are used as wetting agents in the dyeing process for having dispersing and leveling properties. They play useful role in textile processing by wetting the fibre surface quickly and uniformly (Cristea & Vilarem, 2006).

Colourfastness of a textile is an outstandingly important property of a dyed material and one of the important factors in determining the serviceability of an article. It refers to the resistance of colour to fade or change due to washing, rubbing, dry cleaning, and exposure to different light, heat or other treatments etc. (Samanta & Konar, 2011). Factors affecting the colourfastness properties include: the chemical and the physical state of dye; the chemical nature of the fibre; molecular structure of dye; the manner of bonding of dye to the fibre; the dye concentration; the amount of dye present in the fibre; the type of mordant; the presence of other chemicals/auxiliaries in the bath and the actual conditions prevailing during use. Most of the natural colourants have poor light fastness, an indigo and logwood being an exception (Ratnapandian et al., 2012; Lee et al., 2001). UV absorbers (UVAs) are colourless compounds that can be applied by any dyeing technique such as exhaust, pad batch or continuous method. They are applied on porous fabric to enhance the protective effect from UV while maintaining comfort of the fabric (Hussain, 2012). Some conservation experts have hypothesized that the utilization of UV absorbers could be useful methods in reducing fading (Oda, 2001).

In order for natural dyes to be used successfully on commercial scale, there is a need to properly adopt the suitable and standardized techniques. This can be accomplished through scientific knowledge on compatibility checking of selective natural dyes, standardization of dyeing methods, essential variables in dyeing bath and kinetics of dyeing etc. Regarding all these factors, information available in the literature is not clear and sufficient (Samanta & Agarwal, 2009). An attempt have been made through the present study to examine the possibility of dyeing cotton fabric by continuous dyeing procedure with extracts of Marigold flower natural dye using different dyeing auxiliaries. The research has also focused on investigating the fastness and tensile properties of cotton fabric in terms of comparison between pre-dyeing and post-dyeing treatments of all the above mentioned auxiliaries.

Material and methodology

With the purpose to formulate various recipes of Sun yellow (natural extracts of Marigold flower) for cotton fabric, dye baths were prepared by using various chemicals/auxiliaries such as mordants, cross linking agents, cationizing agents, UV absorbers and some fixative type of finishes. Light-weight, 100 % cotton, plain woven fabric was obtained in gray form from a textile mill. Commercial but laboratory scale procedure of desizing, bleaching and scouring was carried out on gray fabric to prepare it for dyeing. A yellow dye (powder of Marigold flower) was obtained from Sam Vegetable Colours Pvt. Ltd, India and was applied as it is. Different chemicals like mordants ($\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$, FeCl_3 , $\text{Al}_2(\text{SO}_4)_3$, CuSO_4 , $\text{K}_2\text{Cr}_2\text{O}_7$), cross linking agents, UV absorbers and cationizing/ finishing were used as received.

A two-bath-two-stage, pad-steam dyeing procedure (continuous dyeing) was adopted to apply various dyeing formulas. For padding, a 3-dip-3-nip treatment was accomplished following drying for 3 minutes at 120°C and then steaming the sample for 10 minutes at 100°C . All the chemicals were applied both as pre-treatment as well as post-treatment. However, the control sample was dyed by a recipe having no mordant, cross linker, UV absorber or any finishing agent. Dyeing bath for such sample was prepared by only a natural dye and required dyeing auxiliaries such as wetting agent and migration inhibitor etc. In case of pre-treatment the order of experiment was:

Pad (treatment) → Dry (air) → Steam → Pad (dye) → Dry (120°C) → Steam

In a post-treatment procedure, the sequence of operation was dyeing first and then padding the dyed sample with a specific chemical treatment.

Fabric Testing

Samples were analyzed for wash fastness (ISO 105- C10:2006) in terms of change in shade as well as staining on the adjacent white fabric by Grey-Scale. Test for rub or crocking fastness of dyed samples was carried out both on dry and wet sample using electronic crockmeter (105X- 12:2001). Findings were measured by Grey-scale for change in shade. Fastness to light was tested as per IS: 2454-1984 standards by exposing dyed samples to UV light in Shirley MBTF light fastness tester along with eight blue wool standards. Colour strength of dyed specimens was determined by computer-aided reflectance spectrophotometer (Kumaresan et al., 2013; Color iQC, 2012). Tensile strength and elongation testing of the dyed and control samples was performed on Testometric 220 D in accordance with standard procedures (ASTM, D5035-06, 2006) developed by American Society for Testing and Materials.

Results

Chemicals utilized as dyeing auxiliaries were able to improve the colour fastness of cotton sample when compared with untreated sample. Although,

some of them were partially successful in improving either wash, rubbing or crocking fastness, they failed to turn out the dyed material having excellent fastness to all the agencies.

Table 1: Dyeing auxiliaries used in this study for the colouration of cotton.

Name of substance	Description/chemical nature	Origin
Albafix WFF	Fixative, cationizing agent/ aqueous preparation of a polymeric, quaternary ammonium compound	Huntsman, Textile Effects
Dicrylan	Multipurpose polymer/polyurethane emulsion	Huntsman, Textile Effects
UV -SUN	Reactive UV absorber based on an oxalanilide	Huntsman, Textile Effects
Rayosan C	Fibre-reactive UV absorbers/ heterocyclic compound	Clariant International Ltd.
Fixapret CPF	Cross linking agent/ methylation product based on glyoxalmonourein	BASF chemical company
Fixapret F-ECO	Cross linker/ modified dimethyloldihydroxyethylene urea	BASF chemical company
Knitex RCT	Cross linking agent/modified dihydroxy ethylene urea	Huntsman, Textile Effects

Table 2: Fastness properties and tensile strength of cotton fabric dyed with natural extracts of Marigold flower by pad-steam method using various mordants.

Method	Chemical/ Mordant	Wash Fastness		Crocking Fastness		Light Fastness	K/S Value	% change in tensile strength
		Change in shade	Staining on fabric	Dry rubbing	Wet rubbing			
Pre mordanting	Al ₂ (SO ₄) ₃	3	4	3-4	3	6	10.26	-26.23
	CuSO ₄	4	4-5	4-5	3-4	5	7.12	-12.68
	FeCl ₃	3	4	4-5	2-3	5	5.52	-24.82
	K ₂ Cr ₂ O ₇	3	4	3	2-3	5	5.21	14.60
	KAl(SO ₄) ₂ ·12H ₂ O	3	3-4	4-5	3	4	4.29	-16.02
Post mordanting	Al ₂ (SO ₄) ₃	2-3	4	4	4	4	3.36	-5.11
	CuSO ₄	3-4	4-5	4-5	3-4	4	8.57	-3.17
	FeCl ₃	3	4	4-5	3	5	7.41	-29.23
	K ₂ Cr ₂ O ₇	3	4	3-4	3-4	4	4.18	4.75
	KAl(SO ₄) ₂ ·12H ₂ O	3	4	4-5	3-4	4	3.01	0.88
No mordanting	Control sample	3	4	4	3	3	4.29	--

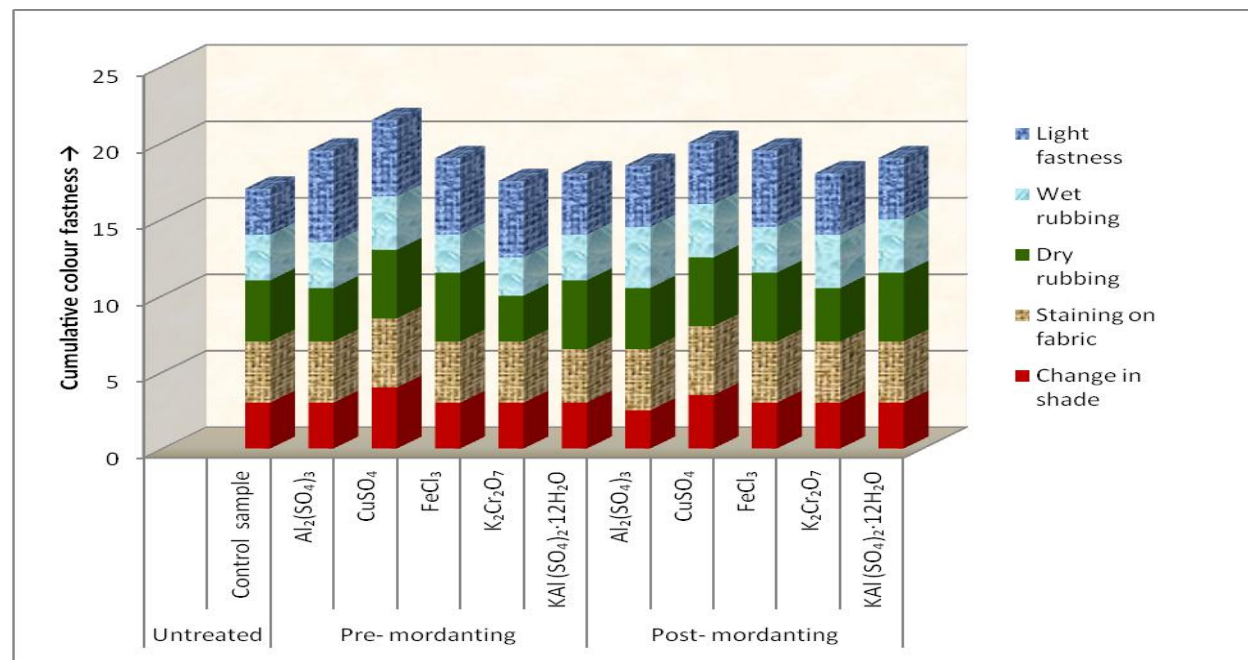


Figure 1: Cumulative colour fastness of cotton samples dyed with natural extracts of Marigold flower by pad-steam method using various mordants.

Table: 3 Fastness properties and tensile strength of cotton fabric dyed with natural extracts of Marigold flower by pad-steam method using various finishes and UV absorbers.

Type of Treatment		Wash Fastness		Croaking Fastness		Light Fastness	K/S Value	% Change in tensile strength
Chemical/ product	Method	Change in shade	Staining on fabric	Dry rubbing	Wet rubbing			
Albafix WFF	Pre cationizing	3	4	4	3-4	3	5.33	6.69
	Post cationizing	3	4	4-5	3-4	4	5.70	4.75
Dicrylan	Pre finishing	3	4-5	4	3-4	4	5.03	3.17
	Post finishing	3-4	4-5	4-5	4	4	5.32	5.63
UV –SUN	Pre treatment	3	4-5	4-5	3-4	5	3.06	3.52
	Post treatment	3	4	4-5	4	6	4.45	-11.97
Rayosan C	Pre treatment	3	3-4	4	4	5	4.56	3.87
	Post treatment	3	4	4-5	4-5	5	4.87	-16.02
Control sample	Untreated	3	4	4	3	3	4.29	--

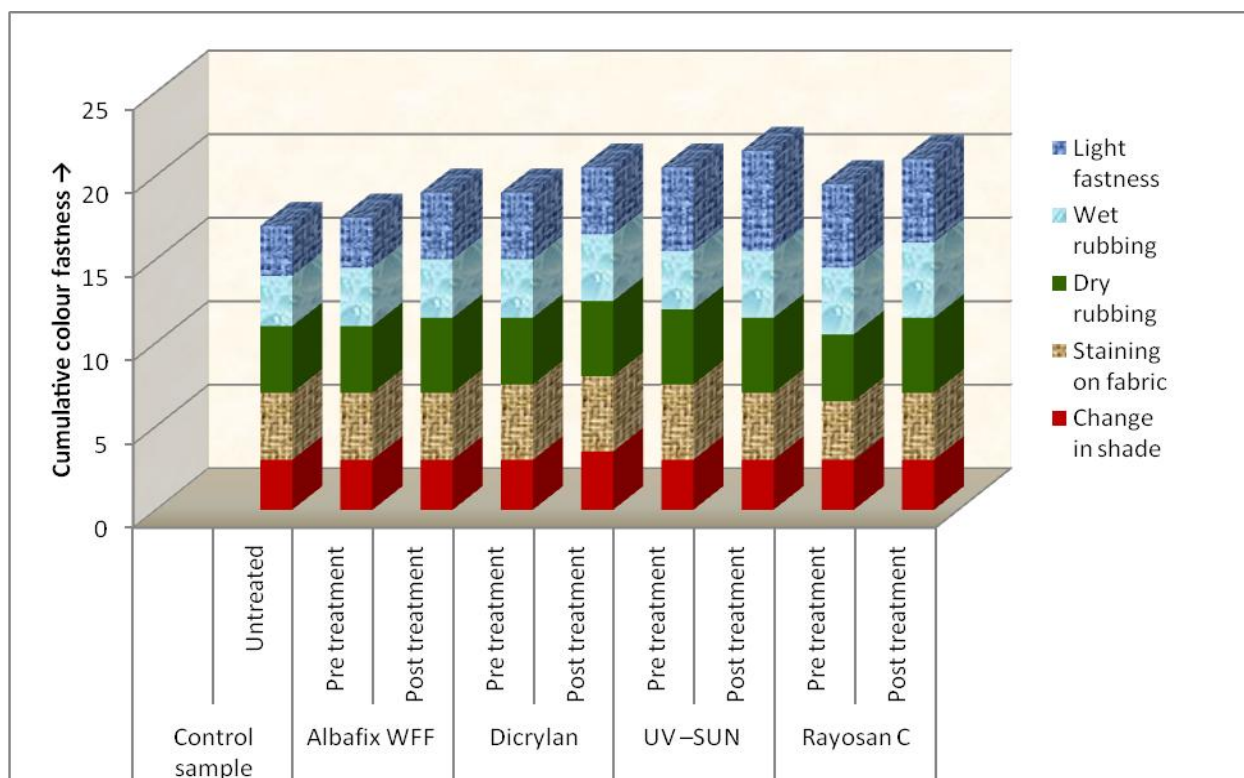


Figure 2: Cumulative colour fastness of cotton samples dyed with natural extracts of Marigold flower by pad-steam method using various finishing agents and UV absorbers.

Table: 4 Fastness properties and tensile strength of cotton fabric dyed with natural extracts of Marigold flower by pad-steam method using various cross linking agents.

Type of Treatment		Wash fastness		Croaking fastness		Light fastness	K/S Value	% change in tensile strength
Chemical/ product	Method	Change in shade	Staining on fabric	Dry rubbing	Wet rubbing			
Fixapret CPF	Pre treatment	3	3-4	4	3	4	4.23	-14.79
	Post treatment	3	3-4	4	3-4	5	4.89	10.56
Fixapret F-ECO	Pre treatment	3	4	4	3	4	3.13	- 15.85
	Post treatment	3	3-4	3-4	3-4	5	4.01	4.75
Knittex RCT	Pre treatment	3	3-4	4	3-4	4	3.57	12.68
	Post treatment	2-3	3	3-4	3-4	4	4.11	0.70
Control sample	Untreated	3	3-4	4	3	3	4.29	--

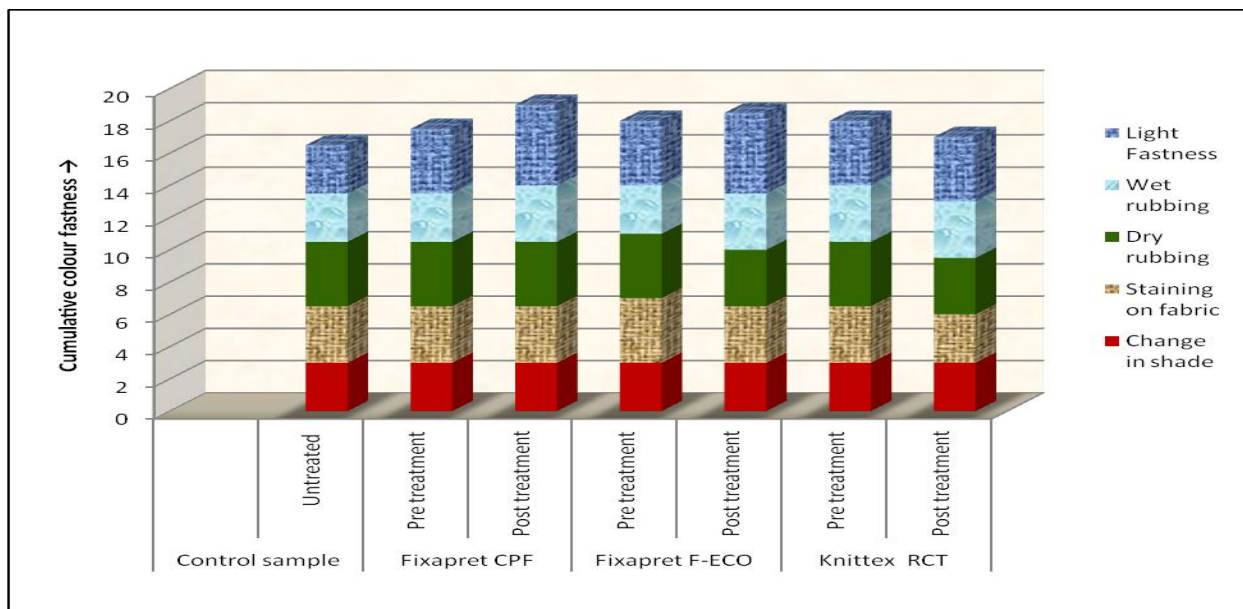


Figure 3: Cumulative colour fastness of cotton samples dyed with natural extracts of Marigold flower by pad-steam method using various cross linking agents.

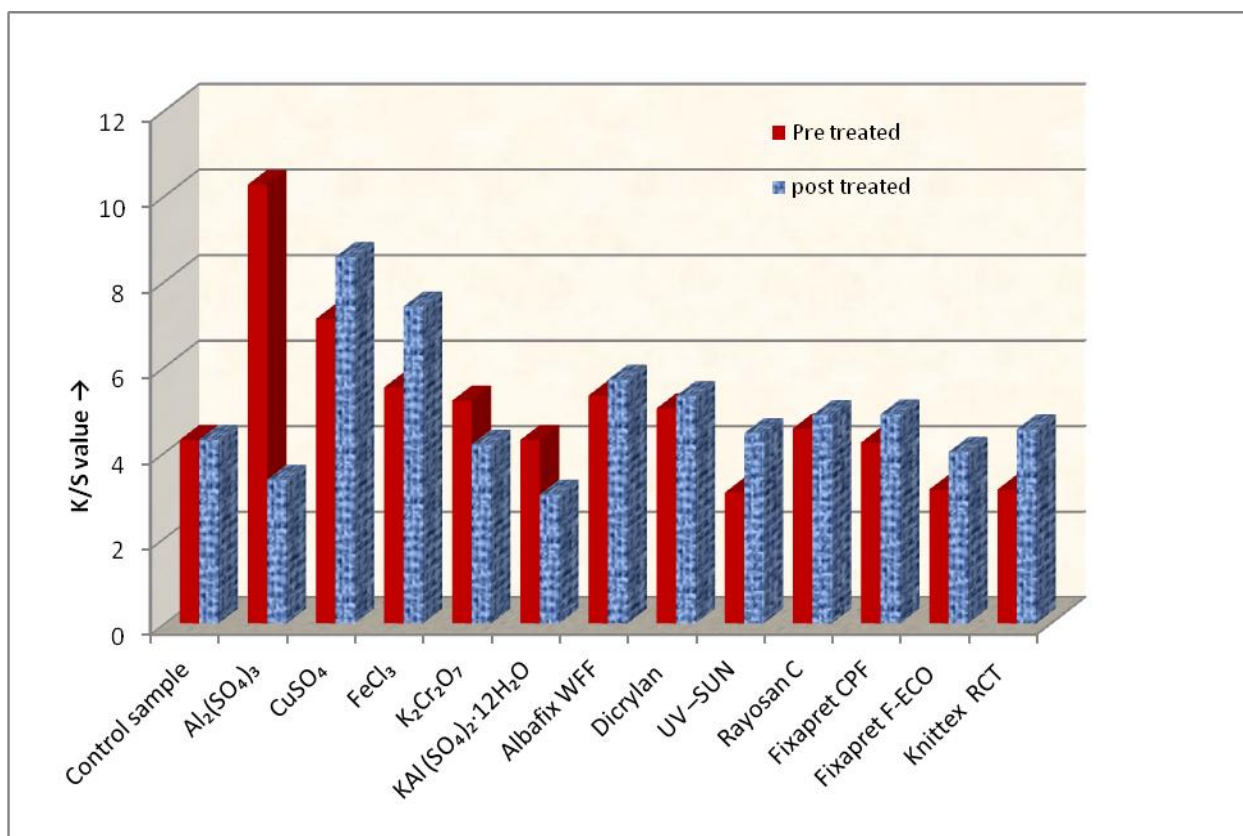


Figure 4: Colour strength of cotton samples dyed with natural extracts of Marigold flower by pad-steam method using various dyeing auxiliaries.

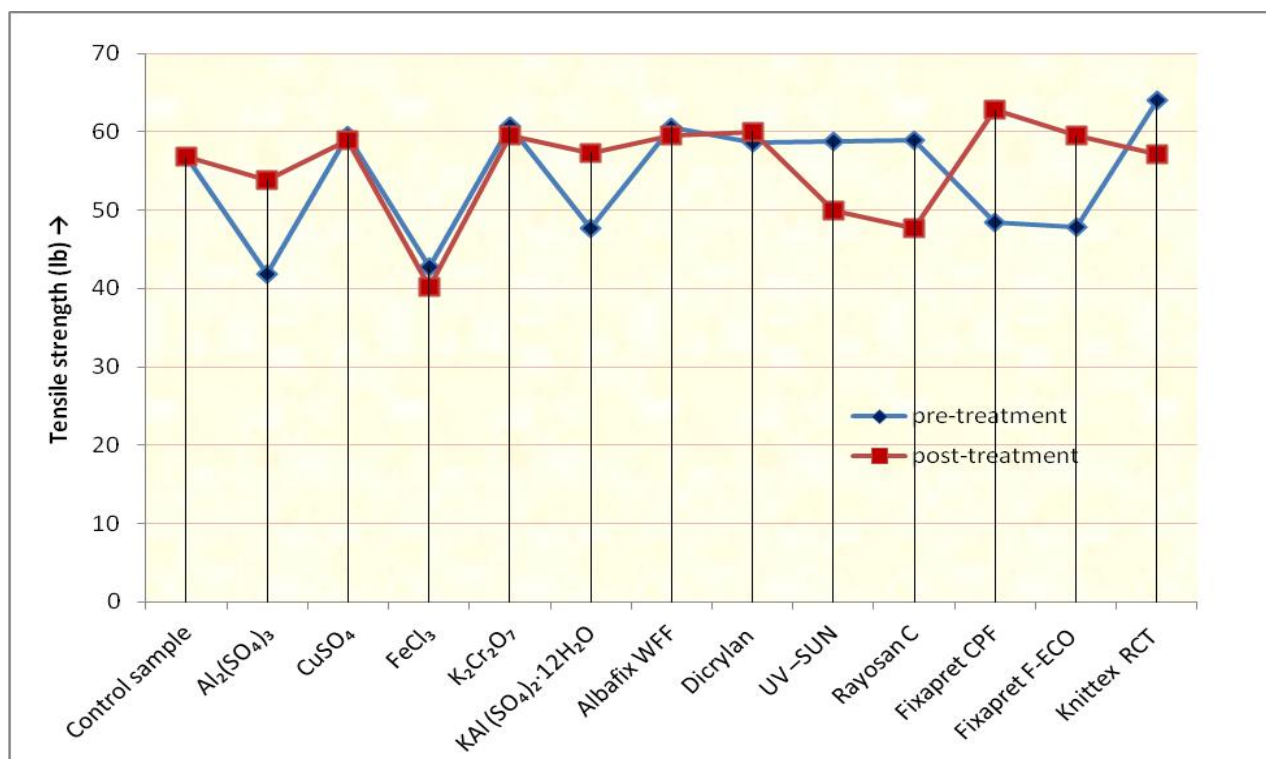


Figure 5: Tensile strength of cotton samples dyed with natural extracts of Marigold flower by pad-steam method using various dyeing auxiliaries.

Effect of mordanting on colour fastness

The results obtained from the application of various mordants along with the natural dye from Marigold flower on cotton fabric are included in Table 1. Pre mordanting with various chemicals could not increase the wash fastness of cotton dyed with natural extracts of Marigold flower. Application of copper sulfate slightly enhanced the wash fastness to 3-4 on Grey-Scale (GS) which is considered to be fair performance. Staining on the adjacent white fabric was rated as 4 GS from the pre treatment of mordants. All the mordants utilized in this study proved to be good in respect of crocking fastness, mainly dry rubbing. The pre treatment of FeCl₃ and K₂Cr₂O₇ produced samples with the wet rubbing of only 2-3 GS. Moreover, no considerable difference between the behavior of pre and post mordanting operations has been observed specially in the wash fastness. Maximum fastness to rubbing was exhibited by cotton sample dyed with recipe having CuSO₄ as pre mordanting auxiliary thereby, producing dry rubbing equivalent to 4-5 GS (very good performance) and wet rubbing of 3-4 GS which is considered to be fair fastness.

Effect of UV absorbers and finishing agents on colour fastness

UV absorbers UV-SUN based on oxalnilide and Rayosan C, a heterocyclic compound demonstrated only satisfactory results of wash fastness when applied on cotton both as pre treatment and post treatment. Their rubbing fastness performance was good in both cases. While in case of light fastness, the UV absorbers have made tremendous improvement in the performance of cotton samples. The post treatment of UV-SUN resulted in the fastness rating equivalent to Blue Wool Standard no. 6, which is considered to be very good performance.

Pre and post cationizing (with Albafix WFF quaternary ammonium compounds) of cotton dyed with this natural yellow colourant played no role in improving the performance of wash fastness, however, this treatment managed to manifest very good dry rubbing and fair- good wet rubbing fastness. Post cationizing also made a little enhancement in the light fastness, forwarding it to standard no. 4 (satisfactory performance) in comparison with only standard 3 in case of untreated sample. The performance of a dyed sample from pre treatment of Dicylan finish (polyurethane emulsion) was not better than the performance of cationized cotton. However, the post treatment of this product was able to produce sample with fair wash fastness, good dry

crocking, fair well rubbing and satisfactory light fastness performance.

Effect of cross linking chemicals on the colour fastness

Cross linking did better job in enhancing the colour fastness of cotton fabric dyed with natural dye from Marigold flower. The data in Table 3 clearly depicts that although they have been able to raise the fastness performance of dry rubbing (up to 4 GS) and light fastness (up to standard 5) with some post treatments, the entire list of cross linking chemicals utilized in this study failed to bring about further improvement in wash fastness of the samples dyed with extracts of *Tegetes erecta* flower.

Analysis of cumulative colour fastness

The data related to cumulative colour fastness of samples dyed with natural dye from the extracts of Marigold flower is displayed through Figure 1 which clearly illustrates that the sample dyed with recipe having CuSO_4 as pre mordant, exhibited excellent performance of fastness properties. The same trend was also found in the dyed sample which was pre mordanted with CuSO_4 . This best performance of colour fastness was followed by aluminum sulfate applied both as pre and mordants in the dyeing recipe. Ferric chloride in the dyeing recipe also produced good results of colour fastness performance on cotton sample particularly, when it was utilized as pre mordant. Alum applied as post mordant was also able to slightly increase the fastness; however, the contribution of $\text{K}_2\text{Cr}_2\text{O}_7$ in this regard was negligible.

Cumulative colour fastness performance of the samples treated with various UV absorbers and finishing agents is detailed in Figure 2. All the UV absorbers and other fixative types of finishes did their job in appraising the fastness properties, however, the post treatment of UV absorber UV-SUN proved to be the best. This best performance was followed by a UV absorber Rayosan C when used as post treatment. The least effective role was played by cationizing of the cotton fabric both with pre and post treatment/finishing operation. Treatment with Dicylan was more successful in enhancing the colour fastness of cotton fabric dyed with Marigold flower dye. Cross linkers improved the colour fastness of cotton samples dyed with natural yellow dye (Figure 3) particularly the post treatment of fixapret CPF, a methylation product based on glyoxalmonourein and fixapret F-ECO (modified dimethylodihydroxyethylene urea). Treatment of knittex RCT could not play any role in bringing any further increase in the colour fastness and its performance was just equal to un treated sample, the

one which was dyed with recipe having no cross linker or any other dyeing auxiliary.

Effect of dyeing auxiliaries on relative colour strength (K/S value)

A greater K/S value was observed from the recipe of aluminum sulfate. Consistency has been noticed in the behavior of CuSO_4 in yielding samples with greater colour strength when applied both as pre and post dyeing. Ferric chloride also made better progress in the colour strength of cotton. Other auxiliaries were not able to acquire any additional value of colour depth, particularly the cross linkers. The K/S value attained by them was almost equal to the recipe having no crosslinker at all.

Effect of dyeing auxiliaries on tensile strength

Most of the auxiliaries resulted in changing the tensile strength of the dyed specimens. Some of the dyeing auxiliaries add to the tensile strength of the cotton fabric after dyeing as compared to the tensile strength of untreated sample. Maximum gain in tensile strength of 14.6% was achieved from the pre treatment of $\text{K}_2\text{Cr}_2\text{O}_7$ followed by 12.68% rise in tensile strength of dyed cotton from recipe having pre treated knittex cross linker. A post treatment of cross linker fixapret CPF also produced dyed samples with increased tensile strength of up to 10.56%. On the other hand, a mordant FeCl_3 had adversely affected the tensile strength of the dyed cotton sample. The behaviour of other mordants utilized in this study was also similar to that of FeCl_3 .

Discussion

Mordants employed in this study proved to be good in respect of crocking fastness, mainly dry rubbing. However, wet rubbing of dyed samples was not satisfactory; particularly with the pre treatment of FeCl_3 and $\text{K}_2\text{Cr}_2\text{O}_7$. According to Wanyama et al. (2010), metal mordants are necessary in dyeing cotton fabrics with natural dyes and also, they influence the colour. Shades produce while using mordants depends on the nature of mordant and dye complex which is formed during the process. Colour strength expressed as K/S values are also dependent on mordants and type of natural dye. Different mordants also cause variations in hue and changes in K/S Values. The appearances of different colours, their strength and stability on the cotton fabrics largely depend on the choice of mordant used and the nature of the textile fibre. From their study, they have also concluded that ferrous sulphate mordant provides dark shades. In the present study also, darker shades were observed from the treatment of ferric chloride utilized as mordant. Various shades/colours can be obtained by using different

mordants on natural dyes (Moiz et al., 2010) and the colour fastness properties also can be enhanced by various treatment practices.

Among all the chemicals applied as mordants in this study, copper sulfate presented best results of improved colour fastness. Beside CuSO₄, no other chemical could surpass the wash fastness rating of the sample dyed without mordant i.e. control sample. Post treatments were better in many respects in comparison with pre treatments of various dyeing auxiliaries, as they offer higher K/S values and enhancement in tensile strength. In an earlier work, while dealing with natural dyes and various methods of mordanting, Moiz et al. (2010) concluded that among the three dyeing method studied, post-mordanting method bestowed the better depth of shade as well as superior wash and light fastness. Among all mordants/cases best outcomes were attained from copper salts as mordant. Poor light fastness of some of the natural dyes is due to photooxidation of chromophores (Grover & Patni, 2011) which can be prevented or minimized by mordants, forming dye-transition metal complex. According to Grover & Patni (2011), dyed cotton fabrics further need to be treated with cationic dye-fixing agents and also with an UV-absorber compound, respectively, to improve the wash and light fastness of these dyed cotton fabrics.

The good fastness of colour presented by Marigold flower dye with the utilization of various dyeing auxiliaries may be attributed to the two hydroxyl groups (Jothi, 2008). Using iron and copper sulphate on the samples dyed with Marigold flower extract as a mordant produced good light fastness. As discussed earlier by Jothi (2008), this may be due to the development of a complex with transition metal which is responsible for protecting the chromophores from photolytic degradation and due to the sorption of photons by chromophoric group which scatter their energy by resonating within the six member ring, thereby providing UV protection to the dye.

Conclusion

Marigold flower dye applied on cotton revealed good to excellent fastness properties. Colourfastness of apparels is an important property which is directly related to the quality and serviceability of commercial products. In the present study, cotton samples were dyed with natural colourant from Marigold flower and attempt was made to formulate such dyeing recipes which may be able to enhance fastness properties of this dye on cotton. A continuous dyeing technique was adopted for colourant application. The use of UV absorbers promoted the poor light fastness of this dye. Cross linking agents and fixative type of finishes made their

way in improving the crocking and light fastness of cotton samples. It can be concluded that a dye from Marigold flower is a promising natural colourant. Its application by pad-steam method can explore ways to develop new range of eco-friendly dyeing for cotton.

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