Sustainable dyeing of wool by natural dyes in conjunction with natural mordants

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Abstract:
Indians are known as precursor in natural dyeing art. Even though home-grown knowledge mode has been experienced in the past over the years, but applications of natural colourants have been reduced due to lack of methodical knowledge of extraction, dyeing procedures and documentation over generations. This leads to failure to commercialization of natural colourants. All the synthetic colourants being used for dyeing textiles nowadayshave dire environmental concerns due to their toxicity and non-biodegradability. They generate water pollution, are carcinogenic along with waste disposal problems. Natural colourants are rational solution to all these problems. Thus, it is obligatory to evolve suitable technology for extraction and sustainable applications of natural dyes on textiles. Present study is an approach to extract natural colourants from a variety of plants sources such as Kalanchoe-pinnata, papaya, peepal and banyan using specific extraction techniques to achieve maximum yield in k/s and antioxidant properties. These four natural extracts were tested for their dyeing potential on wool fabric. Dyeing was performed using three different mordanting techniques (pre, meta and post-mordanting) wherein different natural mordants such as harda, amla, pomegranate and orange as well as synthetic mordants such as alum, copper sulphate and ferrous sulphate were used to fix dye on to the textile substrates. A rainbow of natural colours was obtained with varied shades of each colour. Finding of the study shows that all the four natural extract give satisfactory wash and light colour fastness. The natural mordants give comparable results with synthetic mordants. Thus these natural extracts along with natural mordants can be explored at industrial scale for sustainable colouration of wool.

Keywords: *Natural dyes, natural and synthetic mordants, antioxidant, sustainable dyeing*

1. Introduction

Natural colourants have been used in textile, leather as well as food since prehistoric times. These colourants are obtained from natural substances such as animal and vegetable matter with no or very little chemical processing. Synthetic dyes were introduced in 1856 and being cheaper and easily available resulted in a drastic decline in the usage of natural colourants. However, in the present erathere has been a revival of interest in natural colourants due to their sustainable behavior [1] [2]. Environmentalists are always concerned about the use of synthetic colourants in textile industry as they cause waste disposaland water pollution problems [3]. Natural dyes do not cause any health hazards being biodegradable hence they can be easily used without much environment concerns.Despite this, use of natural dyes for dyeing textiles has been restricted mainly tocottage industries or at artisan level printers due associated problem with natural dyes such as lack in reproducibility, poor fastness and cumbersome extraction and application methods[4] [5]. Recently, many commercial printers have started using naturalprints to overcome the environmental damage caused by synthetic prints. Despite several limitations, there has been a trend to revive the art of natural colouring in recent years due to their distinct soothing aesthetic appeal. India being rich in biodiversity has more than 450 plants yielding dyes and pigments for food, textiles and allied industries[6] [7]. However, many of these plant colourants are not yet fully explored for their potential in dyeing textiles. Majority of these plants extracts are being used for medicinal purposes being having good antibacterial properties [7] [8]. The chemical constituents such as quinine, tannin, phenol, etc.present in plant extract provide colour as well as medicinal properties which can be also harvested for producing functional properties to textiles [9]. Thus, dyeing potential of different natural colourants extracted from varied plants i.e. banyan bark, peepal bark, papaya leaf and Kalanchoe-pinnata leaf were evaluated on wool fabric. Although, only dyeing studies of wool are discussed in this present research paper.

Natural dyes have poor fastness properties hence need mordanting. Pre, meta and post mordanting with heavy metal salts such as aluminum potassium sulphate, ferrous sulphate, copper sulphate, potassium dichromate, etc. is being used traditionally. Although, natural dyes are eco-friendly in nature but owing to use of associated

heavy metallic salts in mordanting step makes the dyeing process toxic. Thus, it is required to find out alternatives to heavy metallic salts. Hence, natural biomordants is also used in investigation to compare their behaviour in dyeing with metallic mordants.

2. Materials and Methods

2.1. Materials:

2.1.1. Textile Materials: Wool fabric was chosen for this study.

2.1.2. Plant Materials:

Four types of plant sources i.e., banyan bark, peepal bark, papaya leaf and Kalanchoe-pinnata leaf were explored for their dyeing potential.

2.1.3. Mordants:

Three chemical mordants such as aluminum potassium sulphate, ferrous sulphate, copper sulphate and four natural mordants such as amla, harda, pomegranate rind and orange peel were taken for this study.

2.2 Preparation of dye solution and dyeing:

Using optimized conditions of extraction i.e., MLR, 1:30, pH 5 and time 90 min at 100^0C, the dye was extracted from Kalanchoe-pinnata and papaya leaves, peepal and banyan barks. All fabrics were cut into 20×20cm size samples and dyed for optimizing dyeing parameters i.e. MLR, pH, and temperature of the dye bath as well as time of dyeing for obtaining maximum K/S value. Further, the dyed fabrics were mordanted with four natural mordants such as amla, harda, orange peel and pomegranate to improve fastness properties. Similarly, dyed fabrics with optimized dye recipes were also mordanted with three chemical mordants i.e., alum, copper sulphate and ferrous sulphate to compare their fastness behavior with natural mordants.

2.3 Characterization of Dye Extractand dyed fabrics:

Antioxidant Property: Anti- Diphenyl-2-picryl-hydrazyl (DPPH) assay which is used to measure anti-oxidant property of dye extract was carried out to calculate the Free Radical Scavenging Activity (RSA). Anti-oxidant property is a measure of the capacity of extracts to scavenge the stable free radicals of DPPH, samples of 0.20ml volumes of extracts were added to 3.8 ml of 0.1 mM DPPH solution in ethanol as reported [8]. Samples were put in dark for 30 minutes to complete the reaction at room temperature for decolourizing the solution. Further, de-colourization was assessed on spectrophotometer at 517 nm wavelength and RSA percentage was calculated using the formula 1:

Radical Scavenging Activity (%) = 1- $(Absorbance_{sample})/(Absorbance_{control})\times 100$ (1)

Where: - $Absorbance_{sample}$ refers to the absorbance of the solution having dye extract and $Absorbance_{control}$ refers to the absorbance of the solution having the de-ionized water.

2.4 Anti-microbial behavior evaluation of dye and dyed fabrics:

Extracted dye and dyed fabrics were evaluated for anti-microbial behavior using AATCC-100 method using gram positive(S-aurous) and gram negative (E-coli) bacteria at IIT, Delhi using quantitative assessment of anti-microbial behavior. Equation 2 was used to calculate bacterial reduction percentage.

Bacterium Reduction (%) = $\frac{A-B}{A}\times 100$ (2)

Where, A represents bacterial colonies for the control after 24 h incubation time

B represents bacterial colonies for sample after 24 h incubation time

2.5 Analysis of Colour co-ordinates of dyed fabrics:

All the dyed samples were assessed for measuring colour co-ordinates (L, a, b and K/S) using Premier Colour scan computer colour matching system at D65 illuminant/10^0 observer.

2.6 Evaluation of fastness properties of dyed fabrics

The light fastness, rubbing fastness (wet and dry) and washing fastness of the dyed fabric samples were evaluated as per ISO 105-BO2:2002, ISO-105-X12and IS: 3361:79, methods respectively.

3 Results and discussion

K/S value was taken as the optimization criteria for different dyeing variables and the results are shown in Table 1.

Table 1. Optimized dyeing conditions for Wool fabric four all extracts:

Extracts	Variables			
	Time(min)	pH	Temperature(^{0}C)	MLR
Banyan bark	90	3	90	1:30
Peepal bark	90	3	90	1:30
Papaya leaf	90	3	90	1:30
Kalanchoe-	90	5	90	1:30

pinnata leaf				

It is clear from Table 1 that all four extract dyed protein fabric show maximum K/S values in acidic medium.

3.1 Anti-oxidant Activity of the Extracts:

All natural species of plants have rich phenolics, carotenoids, flavonoids and secondary metabolites in their chemical structure which contribute towards anti-oxidant behavior. All four extracts were tested for anti-oxidant activity against the free radicals by DPPH because its chemical reaction is very easy to perform [9, 10]. The findings of the study are as follows:

Ascorbic acid calibration curve equation

$y = -0.4892x + 0.7801$

Where y is absorbance value and x is amount of Ascorbic Acid

Resultant value of antioxidant for all extracts is as shown in Table 2.

Table 2. Anti-oxidant characteristics of all extracts:

Extracts	Antioxidant property	
	Absorbance of extract	Anti-oxidant Assay equivalent to Ascorbic Acid
Banyan bark	0.629	0.308
Peepal bark	0.526	0.519
Papaya leaf	0.523	0.525
Kalanchoe-pinnata leaf	0.445	0.685

It can be seen that among all four extracts Kalanchoe-pinnata leaf has maximum Anti-oxidant property so it can be used in cosmetics and finishing of facial wipes etc. for textile application [11].

3.2 Anti-microbial Property:

All four extracts and the dyed protein fabric showed very good anti-microbial property against E-coli (gram-) and S-aurous (gram+) bacteria as mentioned in Table 3.Although, papaya leaf extract has maximum bacterial reduction %.

Table 3. Bacterial Reduction % of all extracts and dyed fabric

Bacteria	Bacterial Reduction %							
	Banyan bark		Peepal bark		Papaya Leaf		Kalanchoe-pinnata Leaf	
	Extract	Fabric	Extract	Fabric	Extract	Fabric	Extract	Fabric
S-aurous	96.06	92.16	96.16	92.79	96.92	94.58	96.82	93.49
E-coli	95.50	90.89	95.63	91.52	96.93	94.19	95.76	92.62

Thus, all these four natural extracts can be used efficiently in medical textiles being excellent antibacterial properties.

3.3 Colour measurement using Computer Colour matching system:

The colour co-ordinate values and shades of wool fabric dyed with Banyan and Peepal bark extract in conjunction with various mordants and mordanting techniques are shown in Table 4. Whereas, the colour co-ordinate values and shades of wool fabric dyed Papaya and Kalanchoe-pinnata leaf extracts in conjunction with various mordants and mordanting techniques are indicated in Table 5.

It can be seen from the Table 4 and 5 that all the dyed samples with different mordants and mordanting techniques exhibit different shades. It can be observed that alum does not have much effect on colour, ferrous sulphate gives tones of grey and copper sulphate changes shades into greenish tone. Pomegranate peel has large amount of tannins hence highlights its own shades in combination with extracts. Harda powder along with extracts modifies the shades of dyed fabrics up to a little extent while Orange peel and Amla powder don't affect the actual shade obtained with true extract. Wool fabric being coarse absorbs large quantity of dye resulting in

dark shades. Although, it can be observed that nospecific particular trend was observed for any mordant and mordanting technique.

Table 4. Colour co-ordinates of wool dyed with Banyan and Peepal bark extracts

S. No	Name	Wool dyed fabric with Banyan bark extract					Wool dyed fabric with Peepal bark extract				
		L*	a*	b*	k/s	Shades	L*	a*	b*	k/s	Shades
Undyed Wool		87.8	-1.2	11.7	0.3		87.8	-1.2	11.7	0.3	
W/O Mordant		58.1	11.0	19.6	2.5		89.9	21.3	23.19	2.7	
1	Al Pre	54.6	13.8	22.6	3.6		55.1	9.7	21.5	3.9	
2	Al Meta	62.9	9.4	21.7	2.2		61.8	6.7	21.1	3.6	
3	Al Post	59.8	11.4	20.3	2.4		57.8	9.1	20.4	3.1	
4	Cu Pre	44.7	10.9	17.3	6.0		48.6	6.0	19.8	5.7	
5	Cu Meta	47.2	6.2	18.4	5.7		51.2	3.9	21.8	6.0	
6	Cu Post	45.6	8.4	18.2	6.2		47.2	4.6	18.8	6.1	
7	Fe Pre	44.3	6.1	10.6	4.6		42.9	3.0	10.0	5.3	
8	Fe Meta	42.8	1.3	7.0	4.4		45.8	1.8	10.4	4.9	
9	Fe Post	44.0	3.1	9.5	4.5		44.4	2.6	13.1	5.6	
10	H Pre	52.3	9.9	25.0	6.2		50.1	8.5	23.2	6.9	
11	H Meta	54.6	8.7	26.0	5.8		53.7	7.3	25.7	6.6	
12	H Post	57.1	9.0	29.5	5.8		54.9	7.6	26.1	6.0	
13	P Pre	50.3	10.0	23.0	6.0		51.8	8.1	22.6	6.0	
14	P Meta	51.3	7.1	23.2	6.4		52.7	7.3	23.8	6.2	
15	P Post	54.9	9.8	27.5	6.0		53.5	7.8	24.5	6.0	
16	O Pre	51.1	11.1	20.3	4.2		56.1	9.0	19.6	3.5	
17	O Meta	53.7	9.2	20.8	4.0		56.4	7.9	20.1	3.4	
18	O Post	55.7	9.8	22.0	3.7		56.4	8.4	19.9	3.3	
19	A Pre	52.8	13.2	19.6	3.5		50.7	8.4	19.4	4.8	
20	A Meta	53.9	9.7	18.3	3.3		54.1	7.7	21.0	4.3	
21	A Post	57.5	10.6	18.8	2.6		53.6	7.5	20.5	4.3	

Abbreviations: Al: Alum, Fe: Ferrous Sulphate, Cu:Copper sulphate, H:HArda, A:Amla, O:Orange Peel, P:Pomegranate Peel, Pre: Pre-mordanting, Meta: Meta-mordanting, Post: Post-mordanting

Table 5. Colour co-ordinates of wool dyed with Papaya and Kalanchoe-pinnata leaf extracts

S. No	Name	Wool dyed fabric with Papaya leaf extract					Wool dyed fabric with Kalanchoe-pinnata leaf extract				
		L*	a*	b*	k/s	Shades	L*	a*	b*	k/s	Shades
Undyed Wool		87.8	-1.2	11.7	0.3		87.8	-1.2	11.7	0.3	
W/O Mordant		67.0	2.7	23.9	2.5		66.5	2.8	16.0	1.9	
1	Al Pre	62.2	2.7	24.5	3.6		83.8	-1.6	12.2	0.4	
2	Al Meta	68.6	1.2	28.2	2.8		72.2	-0.5	19.7	1.6	
3	Al Post	68.4	0.3	20.8	1.9		66.7	-1.3	19.2	2.0	
4	Cu Pre	49.3	2.5	21.9	6.6		66.4	-5.9	19.4	2.0	
5	Cu Meta	50.8	-0.1	24.1	7.0		52.0	-0.4	25.3	6.7	
6	Cu Post	52.9	-1.7	22.6	5.7		48.9	1.1	20.1	6.2	
7	Fe Pre	52.8	3.6	21.1	5.1		68.2	3.6	16.7	1.4	
8	Fe Meta	43.2	-0.1	12.9	6.4		44.4	-0.9	8.5	4.9	
9	Fe Post	47.8	2.6	18.1	6.0		43.3	0.2	7.3	4.6	
10	H Pre	54.1	3.5	26.3	8.0		59.6	4.8	29.2	5.9	
11	H Meta	58.7	3.6	27.9	6.5		58.9	4.8	27.6	5.9	

12	H Post	57.8	3.5	29.6	7.1		57.9	5.6	30.0	6.9	
13	P Pre	51.7	5.0	24.1	7.7		57.1	1.2	25.1	5.9	
14	P Meta	56.4	5.7	28.0	7.0		57.7	3.8	28.7	6.4	
15	P Post	56.0	6.7	29.3	7.2		59.5	4.1	32.2	7.3	
16	O Pre	60.9	1.7	20.8	3.1		79.1	0.5	20.0	0.9	
17	O Meta	64.7	2.6	21.5	2.5		67.8	2.6	18.2	1.8	
18	O Post	66.5	2.7	20.0	2.1		68.2	1.8	18.6	1.8	
19	A Pre	54.3	3.8	20.0	4.8		60.3	5.2	22.6	3.3	
20	A Meta	58.7	4.0	20.6	3.7		62.3	2.9	19.6	3.0	
21	A Post	56.4	4.1	18.8	3.7		60.3	3.9	21.9	3.8	

Abbreviations: Al: Alum, Fe: Ferrous Sulphate, Cu:Copper sulphate, H:HArda, A:Amla, O:Orange Peel, P:Pomegranate Peel, Pre: Pre-mordanting, Meta: Meta-mordanting, Post: Post-mordanting

3.4 Colour Fastness analysis:

Tables 6 to Table 9 show results of colour fastness ratings of the wool dyed fabric with banyan bark, peepal bark, papaya leaf and Kalanchoe-pinnata leaf, respectively. It can be observed from these tables that all the dyed fabrics show satisfactory to good wash fastness. Chemical mordants form H-bond or coordinate bonds with dye and fabric resulting in satisfactory to good wash fastness properties. Although, it can be observed from these tables that rubbing fastness was in generally poor to satisfactory except the Kalanchoe-pinnata dyed wool fabric. Poor rubbing fastness may be occurred due to deposition of natural dyes molecules more on fabric surface instead of penetration inside the interiors of the fabric. Use of appropriate leveling agents may reduce this problem and can improve the rubbing fastness properties.

The results of the study show that all the dyed samples give good light fastness rating or there is increase in darkness of the dyed wool samples instead of fading. The increase in colour depth of some wool dyed sample is due to oxidation of aromatic constituents of natural colourants [12]. It can be also analyzed that natural mordants are also comparable to heavy metal based mordants. Thus, all the four plants extract as well as natural mordants have very good potential in dyeing and finishingof textiles in an eco-friendly way.

Table 6. Fastness ratings for dyed wool fabric with Banyan bark extract:

Applied Mordant	Mordanting Technique	Wash Fastness			Rubbing Fastness		Light fastness
		Fading	Staining		Dry	Wet	
			Cotton	Wool			
Without mordant		3/4	4	3/4	3	3	7
Harda	Pre	3/4	4	3/4	1-2	1-2	Darker
	Meta	3/4	3/4	3/4	1-2	1-2	Darker
	Post	3/4	3/4	3/4	1-2	1-2	Darker
Orange peel	Pre	3/4	3/4	3/4	3	2-3	Darker
	Meta	4	4	4	2-3	2	Darker
	Post	4	4	4	3	2-3	Darker
Pomegranate peel	Pre	3/4	3/4	3/4	3	2-3	Darker
	Meta	3/4	3/4	3/4	2-3	2	Darker
	Post	4	4	4	3	2-3	Darker
Amla	Pre	3/4	3/4	3/4	2-3	3	7
	Meta	4	4	4	2-3	2	6
	Post	3/4	3/4	3/4	4	3-4	7
Alum	Pre	4	4	3/4	1-2	1-2	7

	Meta	4/5	4	4	1-2	1-2	7
	Post	4	4	3/4	2	2	7
$CuSO_4$	Pre	3/4	4	3/4	1-2	1-2	7/8
	Meta	4	4	4	1-2	1-2	7/8
	Post	4	4	4	2	2	7/8
$FeSO_4$	Pre	3/4	4	3/4	2	1-2	Darker
	Meta	3/4	3/4	3/4	1-2	1-2	Darker
	Post	3/4	3/4	3/4	1-2	1-2	Darker

Table 7. Fastness ratings for dyed wool fabric with Peepal bark extract:

Applied Mordant	Mordanting Technique	Wash Fastness			Rubbing Fastness		Light fastness
		Fading	Staining		Dry	Wet	
			Cotton	Wool			
Without mordant		4	4	3/4	2-3	2	6
Harda	Pre	4	4	3/4	2-3	2-3	Darker
	Meta	3/4	3/4	3/4	2	2	Darker
	Post	4	4/5	4	2-3	2	Darker
Orange peel	Pre	4	4/5	4	2-3	2-3	7
	Meta	3/4	4	3/4	1-2	1-2	7
	Post	3/4	4	3/4	2	2	7
Pomegranate peel	Pre	3/4	4	3/4	1-2	1-2	Darker
	Meta	4	4/5	4	1-2	1-2	Darker
	Post	4	4/5	4	2	2	Darker
Amla	Pre	4	4/5	4	1-2	1-2	7
	Meta	3/4	4	3/4	1-2	1-2	7
	Post	4	4/5	4	2-3	2-3	7
Alum	Pre	3/4	3/4	3/4	1-2	1-2	6
	Meta	4	4	4	2	2	6
	Post	3/4	3/4	3/4	2-3	2-3	6
$CuSO_4$	Pre	4	4	4	1-2	1-2	6
	Meta	3/4	3/4	3/4	2	2	6
	Post	3/4	3/4	3/4	1-2	1-2	6
$FeSO_4$	Pre	3/4	3/4	3/4	1-2	1-2	6
	Meta	4	4	4	2	2	6
	Post	3/4	3/4	3/4	2-3	2-3	6

Table 8. Fastness ratings for dyed wool fabric with Papaya leaf extract:

Applied Mordant	Mordanting Technique	Wash Fastness			Rubbing Fastness		Light fastness
		Fading	Staining		Dry	Wet	
			Cotton	Wool			
Without mordant		4	4/5	4/5	3	3	7
Harda	Pre	3/4	3/4	3/4	1-2	1-2	Darker
	Meta	3/4	3/4	3/4	1-2	1-2	Darker
	Post	4	4	4	-2	1-2	Darker
Orange peel	Pre	4	4/5	4/5	3	2-3	7/8
	Meta	4/5	4/5	4/5	2-3	2	7/8
	Post	4	4/5	4/5	3	2-3	7/8
Pomegranate peel	Pre	3/4	3/4	3/4	3	2-3	Darker
	Meta	4	4	4	2-3	2	Darker

	Post	3/4	3/4	3/4	3	2-3	Darker
Amla	Pre	3/4	4	3/4	2-3	3	Darker
	Meta	4	4/5	4	2-3	2	Darker
	Post	4	4/5	4	4	3-4	Darker
Alum	Pre	4/5	4/5	4/5	1-2	1-2	7/8
	Meta	4	4	4	1-2	1-2	7/8
	Post	4	4	4	2	2	7/8
$CuSO_4$	Pre	4/5	4/5	4/5	1-2	1-2	7/8
	Meta	4/5	4	4	1-2	1-2	7/8
	Post	4/5	4	4	2	2	7/8
$FeSO_4$	Pre	3/4	3/4	3/4	2	1-2	Darker
	Meta	3	3/4	3/4	1-2	1-2	Darker
	Post	3	3/4	3/4	1-2	1-2	Darker

Table 9. Fastness ratings for dyed wool fabric with Kalanchoe-pinnata leaf extract:

Applied Mordant	Mordanting Technique	Wash Fastness			Rubbing Fastness		Light fastness
		Fading	Staining		Dry	Wet	
			Cotton	Wool			
Without mordant		4	4/5	4/5	4/5	4	Darker
Harda	Pre	4	4/5	4/5	4/5	3	Darker
	Meta	4/5	4/5	4/5	4/5	3/4	Darker
	Post	4/5	4/5	4/5	4/5	4	Darker
Orange peel	Pre	4	4/5	4/5	4	3/4	No change
	Meta	4	4/5	4/5	4	3/4	Darker
	Post	3/4	4/5	4/5	4/5	4	Darker
Pomegranate peel	Pre	3/4	4/5	4/5	3	2/3	Darker
	Meta	3/4	4/5	3/4	4/5	3/4	Darker
	Post	4	4/5	4	4	3/4	Darker
Amla	Pre	4	4/5	4	4/5	4	No change
	Meta	4	4/5	4/5	4/5	3/4	No change
	Post	3/4	4/5	4	4/5	4	Shade change
Alum	Pre	4	4/5	4/5	4/5	4	No Change
	Meta	4	4/5	4/5	4/5	4	Darker
	Post	4	4/5	4/5	4/5	4	Darker
$CuSO_4$	Pre	4	4/5	4/5	4/5	3/4	No Change
	Meta	3/4	4/5	4	3/4	3	No Change
	Post	4	4/5	4	3	2/3	No Change
$FeSO_4$	Pre	3/4	4/5	4/5	4/5	4	Darker
	Meta	3/4	4	4	2/3	1/2	Darker
	Post	3/4	4	3/4	2/3	2	Shade change

Conclusion

All the studied four plant extracts such as banyan bark, peepal bark, papaya leaf and Kalanchoe-pinnata have very good amount of anti-oxidant contents which make them effective colouring and finishing agents for of textiles.Finding of the study shows that all the plant extracts have good affinity towards wool. These plants extracts in conjunction with different natural and chemical mordants give beautiful and wide colour spectrum to wool fabric. It is pertinent to mention that natural mordants showncomparable results of dying and colour

fastness to chemical mordants. Hence, natural mordants provide an eco-friendly alternative to toxic heavy metal based chemical mordants. Beside these, dyed wool fabrics with these four natural extracts possess very high bacterial reduction % leading their application in medical and functional textiles.

In summary, all the four natural extracts in conjunction with natural mordants has good potential in sustainable dyeing of wool fabric with additional antibacterial properties.

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