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Printing of Lyocell fabric with Rubia Cordifolia and Acacia catechu using Guar gum and Chitosan as Thickening Agent

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In the present work the natural madder dye (Rubia Cordifolia) and Kattha (Acacia catechu) was applied on the regenerated cellulosic fabric-Lyocell through hand screen printing method. In the first part of the study printing paste was developed, using various ratios of guar gum and chitosan for printing with Madder and Kattha. The printed fabric was then characterized with respect to colourfastness and antimicrobial activity against *S. aureus* and *E. coli.* For durability tests, both the dyes showed a moderate to good light fastness and fastness to perspiration and good to excellent fastness to washing.

Keywords: Acacia catechu, Chitosan, Guar gum, Lyocell, Rubia cordifolia

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Nowadays, consumers are frequently moving from synthetic to nature-based products**,** they are demanding eco-friendly, non-toxic and hygienic textile and the consumption of antimicrobials is increasing day by day. Precise research and development activity are trying to keep steps for developing various and effective solutions which are safe for the human being and environment. Vegetable dyed and printed materials is not extensively available to the mass consumer (Mishra, 2007)[1]

Dyes obtained from natural sources are not only used to impart colour to an infinite variety of materials such as textile, paper, wood, etc. but also, they are widely used in cosmetic, food and pharmaceutical industry. They have wide range of medicinal importance in pharmaceutical industry (Chengaiah et al., 2010)[2] Variety of plants used for dye extraction are categorised under medicinal dyes and some of these have recently been found to have antimicrobial activity also (Siva, 2007)[3] In India there are more than 450 plants that yield dye and in addition to their dye yielding characteristics many of these plants are bioactive in nature (Kesari & Siyamak, 2014)[4]

There is an increasing affection towards natural materials whose apparent sources are chitosan from animal sources and guar gum and vegetable dyes from plant sources.

In the present study, two of the natural dyes from plant origin, Red dye with their tint and shades have been chosen which also have some of the medicinal and bioactive properties these are: Madder (*Rubia* cordifolia*)*, Kattha *(Acacia catechu)*.

Madder consists of the groundup dried roots of a plant Rubia cordifolia. It is one of the best and fastest dyes which is used in combination with other dyes to produce compound colours. The roots contain approximately 1.9% of dye, present in the free form or bound as the glucoside (Gupta, 2004)[5]. The colour producing principle of Madder is chiefly alizarin and purpurin. Madder dyes are hydroxy-anthraquinones which are extracted from the root/bark of various Rubiacease (Chengaiah et al., 2010)[2]. It contains the antimicrobial activity. It has medicinal qualities as it was used anciently to remedy health problems. It is mainly used for urinary tract problems. The roots are the alternative source of astringent, antiphlogistic and antiseptic (Teli, 2006)[6].

Catechu is an extract of acacia trees used variously as food additive, astringent tannin and dye. It is extracted from various species of Acacia, but specially senegalia catechu. It is also known as cutch, black cutch and katha. Catechu dyed fabric imparts many medicinal effects it is known that dyed fabric

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with natural colour impart some or all the activities (Bhattacharya, 2000)[7]. Successive treatment of catechu with ether and absolute alcohol abstracts the two principle constitutes namely from 13 to 33% of crude catechn, also catechnic acid and from 22 to 50% of pecular tannic acid called catechu-tannic acid (Chengaiah et al., 2010)[2.] Catechu has antimicrobial activity. Its main properties are to drain dampness, stop bleeding, clear the lungs and transform phlegm. Some cultures use catechu as a type of mouth wash and to treat oral ulcers. Externally catechu can treat conditions such as haemorrhoids and eczema. It has significant antioxidant and antimicrobial effects.

Lyocell Fabric: The name lyocell, was given in 1989 for solvent-spun fibres. Which owes its genesis to the Greek word lyein which means *Dissolve* from which comes Lyo and to cell from the cellulose. This name was recognized as the generic name by *International Bureau for the standardization of Rayon and Synthetic Fibres, Brusels* and *the Federal Trade Commission (USA)* (Chavan and Patra 2004). It is the first in a new generation of cellulosic fibres. Its development was made by the desire for cellulosic fibre which exhibited an improved cost and performance profile as compared to viscose rayon (Goswami et al., 2009)[8]. Another main driving force was the continuing demand for industrial processes to become environmentally responsible and utilise renewable resources as their raw material. As a result, lyocell meets both the qualities (Periyasamy et. al., 2011)[9]. It has all the benefits of being a cellulosic fibre, which is fully biodegradable, it is absorbent and handle can be changed significantly by the use of enzymes or chemical finishing techniques (Jayalakshmi, 2011)[10]. The physical properties of the lyocell also results in its excellent blending characteristics with fibres such as linen, cashmere, silk and wool.

Chitosan: A cationic natural biopolymer obtained by alkaline N-deacetylation of chitin (Kurita 1998). It is the most copious and revitalize natural polymer after cellulose. It comprises copolymer of glucosamie and N-acetyl glucosamine (Illum 1998) and exhibits many unique properties such as non- toxicity, biocompatibility and biodegradability. The reaction of chitosan is considerably more versatile than cellulose due to the presence of NH2 groups. It is a remarkable biomaterial because of its numerous biological and immunological activities (Lim and Hudson 2003)[11]. In particular, its non-toxic and biodegradable properties have attracted considerable attention for biomedical, textile and chemical industrial applications. It also possess the properties for textile dyeing and finishing as a substitute for various other chemicals traditionally used in textile processing and also been used as mordant.

Guar Gum (*Cyamopsis tetragonoloba*): Guar gum is the powdered endosperm of the seeds of cymopsis tetragonoloba which is an annual legume. Guar is being grown for seed, green fodder, vegetable and green manuring. For obtaining the gum, the hull (seed coat, testa or husk) is removed by grinding the soaked seed and then the naked seed is broken down into two portions, gum-splits (endosperm) and grits or germ (embryo or cotyiedon). (Mudgil et. al., 2014)[12] The difference in hardness of the germ and the endosperm is utilized in multistage differential grinding and sifting. The germ and seed coat together constitute the guar meal, which is obtained as a by-product during the manufacturing of gum. The endosperm is ground to fine particles and marked as guar gum of consumer.

Materials and Method

Matrials

For the present study regenerated cellulosic fabric-Lyocell fabric has been used. Fabric was purchased from HP Singh agency, New Delhi and two of the natural dye sources of Red dye (tint and shades) Madder *(Rubiacordifolia),* Kattha *(Acacia catechu)* which contains medicinal properties were selected. Dyes were purchased from Sodhani Biotique, Jaipur.

Guar gum and chitosan (from sigma aldrich) were used as a thickener for making printing paste. Two categories of recipes were developed first one with using only guar gum and second category of recipe were prepared by blending guar gum and chitosan at various ratio such as: 1) 50% guar gum+50% chitosan, 2) 75% guar gum+25% chitosan, 3) 25% guar gum+75% chitosan

Methods

Extraction: Purchased dye sources were in powdered form, so extraction process was done for making printing paste. For that Alcoholic extraction method was adopted.

Printing recipe: Printing of the fabric, with selected natural dyes was carried out through direct style of printing method (screen printing) on Lyocell fabric with two thickeners viz. Guar gum and Chitosan (Abdou et al., 2013)[13].

Blend formation: Chitosan was blended with guar gum on different ratio (50% gg+50% ch, 25% gg+75% ch and 75% gg+25% ch).

Lyocell fabric was printed with above prepared paste through screen printing method, dried and then steaming process was done for the fixation of the print on the fabric.

Preparation of the fabric: Scouring of the fabric was done to remove any kind of solid impurities such as oil, fats and any other finishing treatment given to the fabric at the time of manufacturing. The fabric was scoured with 2 gpl caustic soda maintaining the MLR 1:20 for 1 h. at 80°C. The scoured fabric was thoroughly rinsed with water to ensure complete removal of soap and dried at room temperature.

Printing: After preparation of the fabric and printing paste of various blend ratio and pure guar gum paste printing of the fabric was done (Fig. 1-4). Screen printing method was selected in the present study.

Lyocell fabric printed with selected natural dyes

After treatment of the fabric: To fix the print on the fabric its steaming process was done. For steaming process gas steamer was used in which fabric was kept to get steamed for 60 min at 100°C to 200°C.

Characterization of the printed fabric: Colourfastness to washing (ISO 105 C06 (ISO test no. 3), perspiration (ISO 105 E01) and sunlight (IS: 686-1957) and antimicrobial properties (AATCC 147 test method) were assessed. Gray scale was used for the assessment of the colourfastness (Table 1).

Results and Discussion

In present study, an attempt has been made to prepare a total eco-friendly product using natural dyes and natural thickener. Assessment of the developed samples was done on various aspects such as- colourfastness to washing, perspiration and sunlight and antimicrobial properties of the printed fabric.

Table 2 shows that after assessment of colour fastness to washing using gray scale it was observed that Lyocell fabric printed with Madder dye with the ratio of thickener 25% GG+75% CH found to have a considerable change in colour rated (2) on gray scale, while for colour staining on wool the ratio 75% GG+25% CH had slightly to no change (4-5) on gray scale and on cotton no staining (5) was found with all the developed recipes.

Whereas fabric printed with Katha dye with three of the ratios 25% GG+75% CH, 50% GG+50% CH and 100% Guar gum was rated on 4-5 on gray scale

Table 1 — Gray scale for Evaluation

Rating for Change in Colour	Rating for Colour Staining
5- Negligible or no change	5- Negligible or no staining
4- Slightly Changed	4- Slightly stained
3- Noticeable Changed	3- Noticeable stained
2- Considerably Changed	2- Considerably stained
1- Much Changed	1- Heavily Stained

Table 2 — Assessment of Fastness to washing (ISO 105 C06 (ISO test no. 3)

S.no	Guar Gum, Guar Gum: Chitosan ratio	Dye	Washing Fastness		
			CC	CS	
				Wool	Cotton
		Madder			
1	75% (GG) +25% (CH)		1-2	4-5	5
2	25% (GG) +75% (CH)		2	4	5
3	50% (GG) +50% (CH)		1	4	5
4	100% Guar Gum		1	4	5
		Katha			
5	75% (GG) +25% (CH)		3-4	4	5
6	25% (GG) +75% (CH)		4-5	4-5	5
7	50% (GG) +50% (CH)		4-5	4-5	5
8	100% Guar Gum		4-5	4-5	5

Fig. 1 — Katha dye; Fig. 2 — Katha dye; Fig. 3 — Madder dye; Fig. 4 — Madder dye

which is slightly to no change in colour while in colour staining condition was also found to have slightly to no staining on wool fabric whereas on cotton it was rated on point 5 which shows no staining was found on the fabric (Klaichoi et al., 2014)[14].

From the Table 3 we can interpret the fastness to perspiration of the printed Lyocell fabric with the developed recipes of natural dyes and thickener. Fastness to perspiration was assessed on both acidic and alkaline nature of perspiration.

For the acidic perspiration it was observed that fabric printed with Madder dye had slight change in colour rated (4) on gray scale with the ratio 100% Guar gum elsewhere between the range of 3-4 to 3. While on colour staining part no staining (5) was found on wool fabric with the ratio 100% Guar gum and on cotton same rating was observed with the ratio 25% GG+75% CH.

While with the alkaline solution for change in colour best result was with the ratio 100% Guar gum which is similar to the acidic solution. For colour staining part no staining (5) on wool fabric was found with two of the ratios 25% GG+75% CH and 100% guar gum while same result was found with the ratio 100% Guar gum on cotton.

Fabric printed with Katha dye found to have no change in colour (5) with three of the ratio except the ratio 75% GG+25% CH with the acidic solution while with alkaline solution all the four ratios had slight to no change in colour (4-5). Where for colour staining it was found that two of the ratio 75% GG+25% CH and 25% GG+75% CH had no staining (5) on wool whereas on cotton all the ratio was rated on (4-5) with the acidic solution. Result observed for the alkaline solution for colour staining on wool was best with two of the ratios 25% GG+75% CH and 50% GG+50% CH, while on cotton slightly to no change was found with two of the ratios 75% GG+25% CH and 25% GG+75% CH.

From Table 4, we can conclude the fastness of the printed Lyocell fabric wit natural dyes towards sunlight. It was observed after the procedure that fabric printed with Madder dye had Good (5) sunlight fastness with two of the ratios 75% GG+25% CH and 100% Guar gum paste. It was found that fabric printed with Katha dye had Good fastness (5) towards sunlight with all the developed recipes.

Table 5 shows the antimicrobial activity of the printed Lyocell fabric with selected natural dyes and thickeners ratios. Grey Lyocell fabric shows no antimicrobial activity with both the bacteria *E. coli* and *S. aureus*. It was observed that fabric printed with Madder dye had antimicrobial activity with two of the ratios 25% GG+75% CH with *E. coli* and 75% GG+25% CH with *S. aureus*. Katha dye found to have antimicrobial activity with the ratio 75% GG+25% CH and 25% GG+75% CH with the bacteria *E. coli* while no such activity was found with *S. aureus*. (Fig. 5-7)

Table 4 — Colour Fastness to Sunlight (IS: 686-1957)

S.no	Guar Gum, Guar Gum: Chitosan ratio	Dye	Sunlight Fastness
		Madder	
1	75% (GG)+25% (CH)		5
2	25% (GG)+75% (CH)		4
3	50% (GG)+50% (CH)		4-5
4	100% Guar Gum		5
		Katha	
5	75% (GG)+25% (CH)		5
6	25% (GG)+75% (CH)		5
7	50% (GG)+50% (CH)		5
8	100% Guar Gum		5

Table 3 — Assessment of Fastness to Perspiration (IS 971, ISO 105 E01)

S.no	Guar Gum, Guar Gum: Chitosan ratio	Dye	Acid			Alkaline		
			CC	CS		CC	CS	
				Wool	Cotton		Wool	Cotton
		Madder						
1	75% (GG)+25% (CH)		3	4	4-5	3	4	4-5
2	25% (GG)+75% (CH)		3	4-5	5	3	5	4-5
3	50% (GG)+50% (CH)		3-4	4-5	4-5	3-4	4-5	4-5
4	100% Guar Gum		4	5	4-5	4	5	5
		Katha						
5	75% (GG)+25% (CH)		3	5	4-5	4-5	4-5	4-5
6	25% (GG)+75% (CH)		5	5	4-5	4-5	5	4-5
7	50% (GG)+50% (CH)		5	4-5	4-5	4-5	5	4
8	100% Guar Gum		5	4-5	4-5	4-5	4-5	4


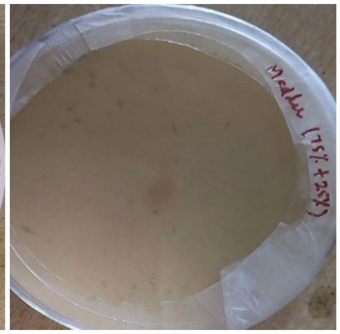
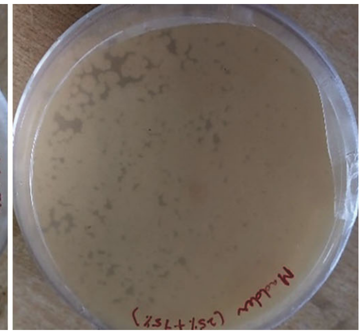

Fig. 5 — Katha dye; Fig. 6 —Madder dye; Fig. 7 —Madder dye

Table 5 — Antimicrobial Activity of Grey and Printed Lyocell Fabric

S.no	Guar Gum, Guar Gum: Chitosan ratio	Dye	Antimicrobial Activity	
			E. coli	*S. aureus*
	Grey Fabric	Madder	-	-
1	75% (GG) +25% (CH)		-	+
2	25% (GG) +75% (CH)		+	
3	50% (GG) +50% (CH)			
		Katha		
4	75% (GG) +25% (CH)		+	-
5	25% (GG) +75% (CH)		+	-
6	50% (GG) +50% (CH)		-	

Conclusion

Conclusion can be drawn from the present research work that both the dye sources Madder and Katha had affinity on both the regenerated cellulosic fabric, Lyocell. Out of four developed recipe of various blend ratio three of the ratios (75% GG+25% CH, 25% GG+75% CH and 50% GG+50% CH) had best results on all the parameters of assessment. Fabric printed with Katha dye with the ratio (75% GG+25% CH and 25% GG+75% CH performed best among all. Fabric printed with both the dyes with three of the ratios of thickener 75% GG+25% CH, 25% GG+75% CH and 50% GG+50% CH gave antimicrobial activity. These value addition on the regenerated cellulosic fabrics with not so common colouring and thickening agents can be additionally implemented with dye of the fabric before printing to enhance the fastness properties of the dye. These developed dye-thickener recipes developed in the study can control the growth of microbes on the fabric, which can be used as an alternative for the harmful synthetic dyes, therefore, these dyes can be used for kids clothing, smart textiles, medical textile and for carpet manufacturing.

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