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Dyeing of Grafted Jute Fibre with Reactive Dyes and its Improved Properties

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Abstract

Graft copolymerization of acrylic acid onto bleached jute fibre has been studied in aqueous solution with redox initiation system $K_2S_2O_8/FeSO_4$. The bleached and grafted jute fibres were dyed with reactive dyes viz. Reactive Red 2, Reactive Orange 14, Reactive Blue 4 and Reactive Brown 10. It has been observed that the percentage of dye exhaustion decreases with the increase of grafting percentage. The colour fastness of dyed fibre with light, acid and alkali perspiration, and washing and spotting tests, has been assessed. For all cases grafted fibre possesses better colour fastness. The tensile strength of grafted jute fibre with prolonged exposure to sunlight, to sunlight has been measured. The grafted fibre shows greater tensile strength than that of bleached fibre.

Keywords: Graft copolymerization, Reactive dyes, Dyeing, Colour fastness, Tensile strength.

1. Introduction

Natural cellulosic fibres provide a wide range of applications in multifarious products affecting every phase of our daily life for making coarse woven fabrics such as gunny sacks and bagging. The strongest potential for cellulose products originates from the combination of their low cost, biodegradability, renewability and high specific strength, especially in the bast fibres of jute. Recently, effort is being made in Bangladesh and India for commercial utilization of jute fibre to meet total fabric requirements of the country, as well as to minimize the import of synthetic fibres. Although jute fibre has high dimensional stability, one of the major problems that restrict the uses of jute products is that it suffers seriously from light-induced discolouration. Due to the presence of lignin and its complex structure, jute products show poor colour fastness and higher loss in tensile strength on exposure to sunlight, air and other external influences. For the proper use of jute, there is a need to eradicate these defects. This can possibly be done by chemical modification of jute fibre through grafting [1-5]. This enhances the desired properties of jute products.

Reactive dyes are often referred to as the king of dyes for the cellulosic fibre dyeing system. Their increased demand and popularity are for vast colour space, bright hues, good colour yield, flexibility of application method, good fastness on modern fastness tests, and lastly reasonable cost effectiveness. The colouration of jute fibre with reactive dyes is most suitable for its eco-friendly nature. In case of dyeing of jute with reactive dyes, anionic dyes react with hydroxyl groups of the fibre by either substitution or addition reactions. Many studies [6-7] have been devoted to improve the substantivity of cellulosic fibre for reactive dyes. A few efforts were made to improve the light fastness [8]. However, none to date has achieved significant success, since all suffer from one or more disadvantages, such as significantly reduced light fastness, unsatisfactory dye fixation efficiency or poor wet fastness, marked changes in hue etc. The objective of our study is to develop a new technique that minimizes the problems cited above.

In the present investigation, an effort has been made to improve colour fastness properties of reactive dyes applied onto acrylic acid grafted jute fibre. An assessment of light fastness, wet fastness, perspiration and breaking strength of dyed grafted jute fibre has been done. The results have been compared with that of bleached jute fibre.

2. Materials and Methods

The tossa variety (*Corchorcus olitorious*) [58.73% α-cellulose, 22.82% hemicellulose, 13.48% lignin, 0.98% pectic matter. 0.96% fatty and waxy matter and 3.03% aqueous extract] [9] of jute fibre was collected from a local market in Rajshahi, Bangladesh. Commercial grade acrylic acid monomer obtained from Riedel-de Haien, Germany was used without any treatment or purification. Reactive dyes M brand viz. Reactive Red 2 (C.I. No. 18200), Reactive Orange 14 (C.I. No. 19138), Reactive Blue 4 (C.I. No. 61205) and Reactive Brown 10 (C.I. No. 12225) manufactured by Sigma, USA were used without any purification. All other chemicals used were of laboratory reagent grade.

2.1 Bleaching

To remove dirty substances, jute fibre was scoured in a solution containing 3.5 gm Na_2CO_3 and 6.5 gm detergent per liter of water at 70°C in a large beaker. The fibre-liquor ratio was maintained at 1:50. Then, the fibre was bleached with 0.2% $NaClO_2$ solution buffered at pH 4 at 90-95°C for 90 min, maintaining the fibre liquor ratio of 1:50. To reduce chlorite action, the fibre was treated with 0.2% sodium metabisulphite solution for 15 min and then washed thoroughly with distilled water [10].

2.2 Grafting

Acrylic acid was dissolved in cold distilled water. The modifying baths were prepared by adding the required amount of monomer, initiator and catalyst. The fibre-liquor ratio was maintained at 1: 50. Modification was started at 30°C and then the temperature was slowly increased almost to 90°C for 30 min and continued for 240 min. After modification, the fibres were washed with hot distilled water and acetone to remove the suspended homopolymer.

The weight of dried fibre after and before modification was taken accurately. For precision a blank experiment was done which gives eventual weight loss during modification. The percentage of grafting was calculated according to the following formula [5].

$$\text{Percentage of grafting} = \frac{A - B}{B} \times 100$$

Where, A is the weight of the grafted fibre after modification; B is the weight of bleached jute fibre before modification.

2.3 IR spectroscopy

IR spectra of bleached and grafted jute fibres were recorded with Shimadzu IR-470 spectrophotometer (Shimadzu, Kyoto, Japan) using KBr pellet technique. The dried fibre samples were crushed to a size that was finer than 20 meshes before pelleting with KBr. The test KBr pellet contained about 1% powdered fibre [11].

2.4 Dyeing

The dye baths were prepared with 2.5% of reactive dyes with 5% $Al_2(SO_4)_3$, maintaining the fibre-liquor ratio at 1:40. Bleached jute and finished jute with an acrylic monomer system were put in a dyeing bath at 60°C for 50 min, the pH of the dye bath was adjusted to 6.8±0.2 with 0.5M Na_2CO_3 solution. In order to measure dyeability, as well as absorption of dye percentage of the treated and untreated fibre dyed, the samples with reactive dyes, the samples were examined in a Spectronic 20(colorimeter).

$$\text{Absorption of dye percentage} = \frac{Do - De}{Do} \times 100$$

Where, Do and De are the original and exhausted dye bath concentration, respectively.

2.5 Fastness Tests

The colour fastness of the dyed fibres to light and wet tests were assessed using AATCC test methods [12].

2.5.1 Light Fastness

The specimen of jute was exposed for a certain time, i.e., 12, 24, 36 and 72 hrs at 40 ^{0}C under an artificial light source (Daylight B01, James H, Heal and Company, Hamifax, England), equipped with Xenon arc and Mercury-Tangsten fluorescent, whose wave length range was similar to that of sunshine. The light fastness grades range from 1 (lowest light fastness) to 8 (highest light fastness). The changes were compared with the original specimen. The changes were assessed by the blue light fastness scale.

2.5.2 Acid and Alkali Perspiration

The acid perspiration test solution was prepared by 0.5 gm 1-Histidine monohydrochloride mono-hydrate ($C_6H_9O_2N_3HCl.H_2O$), 5 gm Sodium chloride (NaCl), 2.2 gm Disodiumhydrogen orthophosphate ($Na_2HPO_4.2H_2O$) and 1000 ml of distilled water where the pH was adjusted to 5.5 [13]. The jute specimen was cut to 10 cm and wetted in the above perspiration solution at room temperature, with a fibre-liquor ratio of 1:50 and time of 30 min. Then this composite test specimen was placed between two glass plates under a pressure of 5 kg and was heated in an oven for 4 hrs at 37 ± 2^0C. The specimen was removed and dried in open air. The contrast between the dyed bleached and dyed grafted jute sample was compared with the Grey scale.

For alkali perspiration, solution was prepared with 0.5 gm 1-Histidine mono-hydrochloride mono-hydrate ($C_6H_9O_2N_3HCl.H_2O$), 5 gm Sodium chloride (NaCl), 2.5 gm Disodiumhydrogen orthophosphate ($Na_2HPO_4.2H_2O$) and 1000 ml of distilled water where the pH was adjusted to 8.0 by adding 0.1 M NaOH solution[13]. Then the same procedure was followed as with acid perspiration.

2.5.3 Washing

The dyed jute specimens were washed in a hot washcator at 50, 70 and 90^0 C for 30 min using the detergent solution (5 g/L) to a fabric liquor ratio of 1:50[12]. The change in colour of the specimen and the staining of the adjacent fabric were assessed by the Grey scale.

2.5.4 Spotting

The specimens were spotted at room temperature with the acids and alkalies by a glass rod; a spot of diameter of approximately 20 mm was formed. Then the specimen was dried at room temperature without rinsing. The contrast between the treated and untreated sample was compared with the Grey scale [14-15]. Acid and alkali spotting were assessed by the solutions: Acetic acid, 300 gm/L; Sulfuric acid, 50 gm/L; Tartaric acid, 100 gm/L; Sodium carbonate, 100 gm/l; Sodium hydroxide, 50 gm/L; Ammonia solution, 10%.

2.6 Breaking Strength Test

A torsee's Schopper typә-05-100 tensile tester was used for measuring the tensile strength of jute fibre. The tensile strength of each specimen was measured according to International Standard (Strip method) [16].

Table. 1 Effect of grafting of bleached jute fibre

No. of Exp	Grafting conditions					Graft yield %
	Monomer conc. mol/L	Initiator conc. mol/L	Catalyst conc. mol/L	Time min	Temp °C	
1	0.056	0.0037	0.0036	90	60	4.4
2	0.222	0.0037	0.0036	90	60	12.0
3	0.139	0.003	0.0036	90	60	6.0
4	0.139	0.0052	0.0036	90	60	14.4
5	0.139	0.0052	0.0014	90	60	8.0
6	0.139	0.0052	0.005	90	60	10.2

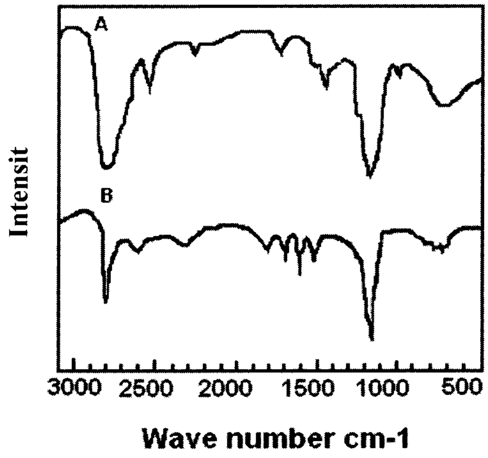

Fig. 1 IR spectra of (A) Bleached jute fibre and (B) Grafted (14.4%) jute fibre.

3. Results and Discussion

The graft copolymerization of acrylic acid onto bleached jute fibre initiated by potassium persulphate ($K_2S_2O_8$) in catalytic influence of $FeSO_4$ may be pictured as involving generation of free-radical ($^{\bullet}OH$ and $SO_4^{-\bullet}$) species and to some extent by Fe(III) ions on jute fibres. The free radical species and Fe(III) ions are produced from the redox reaction between $K_2S_2O_8$ and Fe(II). The macroradicals so formed subsequently attack the monomer leading to chain initiation of grafting. Table 1 shows that the percent graft yield depends on the various conditions of grafting which are discussed in a previous paper [17]. For evidence of monomer loading with fibre, IR spectroscopy was done. From the Fig. 1, it is seen that the spectra of grafted fibre is much more similar to that of bleached fibre. But at 1733 cm^{-1} and 1650 cm^{-1} additional peaks were was found which are in the region of ester and carboxylic groups, respectively. A possible mechanism is that the ester linkage of grafted fibre is due to the reaction of -COOH group of acrylic acid and -OH group of cellulose with another carboxylic group acquired from grafted acrylic acid.

3.1 Dye Exhaustion

From Table 2, the dye exhaustion of bleached, 4.4, 6.0, 8.0, 10.2, 12.0 and 14.4% acrylic acid grafted fibre is observed. From the above, the order of dye exhaustion percentage is bleached jute fibre >4.4%>6.0%>8.0%>10.2%> 12.0%>14.4% acrylic acid grafted fibre. A possible explanation of such dye exhaustion variation is that dye absorption by the fibre depends on the availability of the total external surface of the fibre pores or cavities in a fixed amount of fibre and the attractive forces between the fibre and dye ions [9]. The bleached fibre has more available pores or cavities than modified fibre. The attractive force between dyes and fibre reduces in grafting due to an increase in hydrophobicity. So, dye exhaustion of bleached fibre is greater than modified jute fibre. Another possible reason is that, the grafted fibre is more acidic than bleached fibre and the pH of reaction medium is slightly acidic. Thus, bleached fibre shows greater dye exhaustion than modified fibre. The order of dye exhaustion of the reactive dyes are: Reactive Red 2> Reactive Blue 4> Reactive Orange 14>Reactive Brown 10. The greater dye absorption of Reactive Red 2 and Reactive Blue 4 as compared with Reactive Orange 14 and Reactive Brown 10 may be due to the presence of more and stronger covalent bonds between the triazinyl groups of those reactive dyes and the jute fiber.

Table. 2 Effect of dyeing of bleached and grafted jute fibre

% grafting fibre	Dye exhaustion %			
	R.R. 2	R.O. 14	R.Bl. 4	R.Br. 10
Bleached	94	86	91	82
4.4	90	81	86	75
6.0	88	78	82	72
8.0	85	73	77	68
10.2	83	69	75	65
12.0	82	66	74	64
14.4	81	64	72	63

R.R.-Reactive Red; R.O.-Reactive Orange; R.Bl.-Reactive Blue; R.Br.- Reactive Brown.

3.2 Colour Fastness

Table 3 summarizes the light fastness properties of dyed fibres. In this assessment the fastness of dyed grafted jute fibre was better than that of dyed bleached fibre. This is probably due to more lignin presence in bleached jute fibre. The hydroxyl group of lignin is highly sensitive to the action of light. When UV-light falls upon dyed jute fibre, the phenolic hydroxyl groups of lignin creates free radicals. These free radicals undergo transformation into quinonoid structures and show yellowing on the surface of fibre, thus causing easy fading of dyed bleached jute fibre [18]. In grafting, the reactive sites of jute i.e. lignin are blocked. So, the grafted fibre shows better colour fastness.

In acid, hydrocellulose was produced by the action of dilute acids. This results in the cleavage of the chains by hydrolysis. Hydrolysis decreases the tensile strength of the fibre and breaks down the covalent bonds between the fibre and the reactive dyes. It was observed from Table 3 that the colour fastness of dyed grafted fibre was more prominent than dyed bleach fibre. The presence of more acidity in grafted fibre reduces the hydrolysis as well as colour fastness. A reverse phenomenon was observed in the alkali perspiration test. Grafted fibre reacted strongly with the alkali perspiration solution as compared with the bleached jute fibre.

The colour fastness of dyed jute fibre has been studied at 50, 70 and 90^{0}C in detergent solution and the results are tabulated in Table 3. The wash fastness depends upon the physical and chemical properties of the fibre, the class of the dyes and their forces of interaction and their interaction with soap solution. It was evident that wash fastness decreases with the increase of temperature, It seems that at higher temperature, dissolution of the dye particles from the fibre surface takes place and hence more dye was easily washed off the fabric. In that case grafted jute fibre possesses better colour fastness to washing, because in grafting the fibre becomes hydrophobic in nature. So, grafted fibre has less shrinkage in water.

Table 3 shows the colour fastness of dyed bleached and grafted fibre in the spotting test. In acid spotting, the colour of grafted jute fibre is unchanged, but the colour seriously changed with alkali spotting. Because grafted fibre is much more acidic, it reacts greatly with alkali.

Table. 3 Colour fastness of dyed bleached and dyed grafted jute fibre.

Fastness assessment		Fastness grade							
		R.R. 2		R.O. 14		R.Bl. 4		R.Br. 10	
		BL	GR	BL	GR	BL	GR	BL	GR
Light fastness (Exposure time)	12 hrs	6-7	8	6	7	6-7	7-8	5-6	7
	24 hrs	5-6	7	5	5-6	5-6	6-7	4-5	6
	36 hrs	4	5-6	3-4	4-5	3-4	5	3	4-5
	72 hrs	3	4-5	2	3-4	3	4	2	3-4
Acid Perspiration	Shade	4	5	3-4	4-5	3-4	4-5	3	4
	Staining	3-4	4	3	4	3	4	2-3	3-4
Alkali Perspiration	Shade	4	3	4	3	3	3	3	2
	Staining	3	2	3	2	3	2	2	2
Washing (temperature)	50°C	4	5	4	5	4	5	4	5
	70°C	3-4	4-5	3	5	3-4	4	3	4
	90°C	3	4	2-3	4	2-3	4	2	3-4
Spotting	Acetic acid	5	5	5	5	5	5	4-5	5
	Sulfuric acid	4-5	5	4	5	4	5	4-5	5
	Tartaric acid	5	5	5	5	5	5	4-5	5
	Sodium carbonate	4-5	4	4	4-5	4-5	4	4	4-5
	Sodium hydroxide	3-4	2	3	2-3	3	2	3	3
	Ammonia solution	4	2-3	3-4	3	3-4	2-3	3-4	3-4

BL-Bleached jute fibre; GR-Grafted (14.4%) jute fibre;
R.R.-Reactive Red; R.O.-Reactive Orange; R.Bl.-Reactive Blue; R.Br.- Reactive Brown.

3.3 Effect of Sunlight (UV) on the Tenacity of Jute Fibre

From Table 4, it is seen that the loss in tenacity of dyed bleached jute is greater than dyed modified jute fibre. Graft copolymerization of vinyl monomer onto jute fibre has been considered as a powerful method to substantially improve some intrinsic properties. The affinity of graft polymerized jute fibre towards moisture has been found to decrease with an increase of polymer loading onto jute fibre. So, it is thought that the reduction of affinity of jute towards moisture by graft copolymerization might render it less susceptible to the degrading action of UV-light [2].

Table. 4 Effect of sunlight on tenacity of bleached and grafted jute fibre

Exposure Period, hrs	Tensile strength, g/denier							
	R.R. 2		R.O 14		R.Bl. 4		R.Br. 10	
	BL	GR	BL	GR	BL	GR	BL	GR
0	2.70	2.90	2.62	2.85	2.68	2.88	2.75	2.84
300	1.65	2.15	1.70	2.30	1.70	2.34	1.66	2.11

BL-Bleached jute fibre; GR-Grafted (14.4%) jute fibre;
R.R.-Reactive Red; R.O.-Reactive Orange; R.Bl.-Reactive Blue; R.Br.- Reactive Brown.

4. Conclusions

The dyeing fastness properties of bleached and grafted jutted jute fibre has been evaluated using Reactive dyes viz. Reactive Red 2, Reactive Blue 4, Reactive Orange 14 and Reactive Brown 10. Positive results are obtained in all cases such as light fastness, wash fastness, perspiration and spotting tests. The loss in breaking strength of dyed bleached fibre is higher than that of dyed grafted fibre. The colour fastness properties of reactive dyes are in the order of: Reactive Red 2>Reactive Blue 4> Reactive Orange 14>Reactive Brown 10.

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