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Dyeing and thermal behavior of jute fibre grafted with nitrile monomer

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Abstract

Modification of bleached jute fibre was carried out by graft co-polymerization with the vinyl monomers, acrylonitrile and methacrylonitrile. A $K_2S_2O_8/FeSO_4$ redox system was used in the presence of air. The graft yield and grafting efficiency increased with the increase of monomer, initiator and reaction time up to certain values, thereafter it decreased. This is due to the increasing rate of homopolymerization, rather than grafting, at the higher monomer concentration. The maximum graft yield with acrylonitrile and methacrylonitrile, under optimized conditions, was 19.78 and 43.15%, respectively. The graft yield of methacrylonitrile is greater than that of acrylonitrile. This is due to the $-CH_3$ group present in methacrylonitrile. The inclusion of nitrile monomer onto the treated fibre was identified by Fourier Transform Infrared Spectroscopy. The surface morphology and thermal behavior of the modified fibre was investigated by Scanning Electron Microscopy and Thermogravimetric Analysis. Modification of jute fibre improved the thermal stability, breaking strength as well as dyeability of the fiber.

Keywords: Jute, Grafting, Nitrile monomer, Thermal stability

Introduction

Jute fibre is the most important cash crop and the biggest foreign exchange earner of Bangladesh. It is composed of 60–70% α -cellulose, 15–20% hemicelluloses, 5–10% lignin and 3–5% pectins, together with some fatty acid and waxy matter (Alam and Khan 2007). In recent years, it has been threatened by increasing competition from cheap synthetic fibres. Due to environmental issues, many ecofriendly alternative fibres and chemical modifications have been considered by researchers (Mishra et al. 2001; Gonaslvcs and Mungara 1996). So it is necessary to improve the quality of jute fibre for commercial utilization, to meet total fabric requirements of the importing countries as well as to minimize the import of competing synthetic fibres.

Various chemical treatments have been used to improve the physico-chemical performance of natural fibres in the past (Ouajai et al. 2007; Mohanty et al. 2000; Dong et al. 2015). Although a lot of work has been reported on graft co-polymerization of vinyl monomers onto cellulosic and other textile fibre (Mondal 2013; Bakr et al. 2015), much less has been reported on grafting of nitrile monomers onto jute fibre. Yet jute is an important lignocellulose fibre.

Depending on the chemical structure of the monomer grafted onto cellulose, graft copolymer gain new properties such as improved elasticity, hydrophobic character

(Bicak et al. 1999; Okieimen 1987), heat resistance (Samal et al. 1988; Misra et al. 1987; Huque et al. 1980), resistance to microbial attack (Mcdowall et al. 1984), abrasion resistance (Bianchi et al. 2000). Grafting also changes the mechanical properties of grafted polymer varied with the amount of grafting (Ouajai et al. 2004; Fatimah et al. 2014; El-Shekeil et al. 2014) and the grafting led little degradation effect on mechanical properties. Hence, it is expected that the properties of this fibre on modification by grafting, can be improved for use in the manufacture of high-quality products.

Therefore chemical modification, through graft co-polymerization, of nitrile monomer on to jute fibres, has been receiving considerable interest in recent years. The grafting was determined on the basis of the increased weight of the fibre treated. In the present work, attempts have been made to modify the jute fibre with acrylonitrile (AN) and methacrylonitrile (MAN) monomers, initiated under a potassium persulphate ($K_2S_2O_8$) and ferrous sulphate ($FeSO_4$) redox system. The study attempted to determine the optimum grafting conditions for such modification. Some physico-chemical characteristics of the grafted fibre were investigated.

Methods

Materials

Raw jute fibre (*Corchorus olitorius*, *Tossa Variety*) was collected from Rajshahi Jute Mill Ltd., Bangladesh. Glacial acetic acid, anhydrous sodium acetate, sodium chlorite, acrylonitrile, methacrylonitrile, potassium persulphate, ferrous sulphate and the dyestuffs were purchased from BDH, England.

Sample preparation

The collected jute fibre were carefully combed, blended and washed with 6.5 g of detergent and 3.5 g of soda per litre at 75°C for 30 min (Farouqui and Mondal 1989). The jute fibre was bleached, with a sodium chlorite solution of concentration 0.5%, at pH 4 and at 80–90°C, for 90 min (Farouqui and Mondal 1989). Then the bleached jute fibre was grafted with acrylonitrile and methacrylonitrile monomers.

Method of grafting

The graft copolymerization of jute fibre was carried out in a 100 mL stoppered Erlenmeyer flask. Polymerization was done with 240–330% monomer, 18–25% $K_2S_2O_8$ as initiator and 1–6% $FeSO_4$ as catalyst, based on the weight of the fibre. Polymerization took 30–150 min, at 60–110°C in the fibre–liquor ratio of 1:50. At the end of the desired period, the jute fibre was washed repeatedly three times with warm distilled water and dried (Hebiesh et al. 1971; EL-Rafie et al. 1974). Graft yield and grafting efficiency were calculated according to the following formula:

$$\text{Graft yield, \%} = \frac{W_1 - W_0}{W_0} \times 100$$

$$\text{Grafting efficiency, \%} = \frac{W_1 - W_0}{W_2} \times 100$$

where W_1 is the weight of the grafted jute fibre after modification, W_0 is the weight of ungrafted jute fibre before modification and W_2 is the weight of monomer used.

IR spectroscopy

Fourier Transform Infrared Spectroscopy (FTIR) spectroscopy analysis was performed, using a Perkin Elmer 100 infrared spectrometer. The fibres and KBr (potassium bromide) were dried in an oven at 105°C, until they were moisture-free. Then about one percent fibre was mixed with dried KBr, ground into powder, using a mortar pestle (Mondal 2013). The infrared spectra of the samples were recorded with FTIR between 400 and 4,000 cm^{-1} .

Thermal analysis

Thermogravimetric analysis was used to characterize the thermal decomposition rate and the thermal stability of the raw and grafted fibre. The samples, approximately 10 mg each, were heated from 30 to 600°C. Heating was done in an inert atmosphere (argon), at a rate of 20°C/min, in a Seiko-extar-TG/DTA-6300, Tokyo, Japan.

Scanning electron microscopy

Scanning electron microscopy (SEM) was performed. An SEM (FEI Quanta Inspect, Model: S50, Kyoto, Japan) was used to observe the surface microstructure of the treated and untreated fibres.

Method of dyeing

Dyeing of bleached and modified jute fibres was carried out with direct and reactive dyes. The dye concentration of reactive and direct dyes were 3 and 1%, and the electrolyte concentration for the dyes were 7 and 25% respectively.

Tensile strength measurement

The tensile strength of the jute fibres was measured, using a tensile strength tester (Seri-graph machine 4004, Japan). Jute fibres were cut into equal pieces, 30 cm in length, and the length of each specimen between the jaws was maintained at 10 cm. One twist per 2 cm was given, along the length of the fibre between the jaws of the machine. The average breaking load (the mean of 10 readings) of the fibre was measured in kg/Yarn. Tenacity was calculated by the formula 9 (ISO 5081-1977(E) 1997).

$$\text{Tenacity} = \frac{\text{Average breaking load}}{\text{Denier}} \times \frac{\text{g}}{\text{Denier}}$$

where,

$$\text{Denier} = \frac{\text{Weight of the sample in grams}}{\text{Length of the sample in meters}} \times 9,000.$$

Method of sunlight exposure

The jute sample was exposed directly, on a flat board, to sunlight, without any protection from weathering but was protected from rain, snow etc. At the same time and in the same place, bleached and grafted jute fibres were exposed to sun on the roof of a building in the months of April to July for 7 h each day. This continued for a total of 350 h.

Method of heating

Jute sample was placed in an electric oven, in the presence of air, at 30–160°C for 3 h. Its breaking strength was then measured as described above.

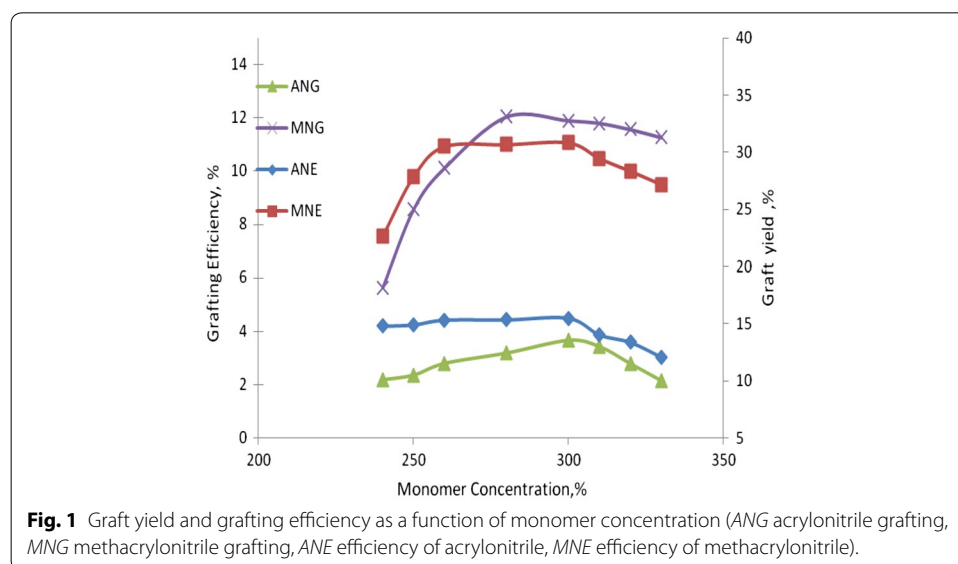
Results and discussion

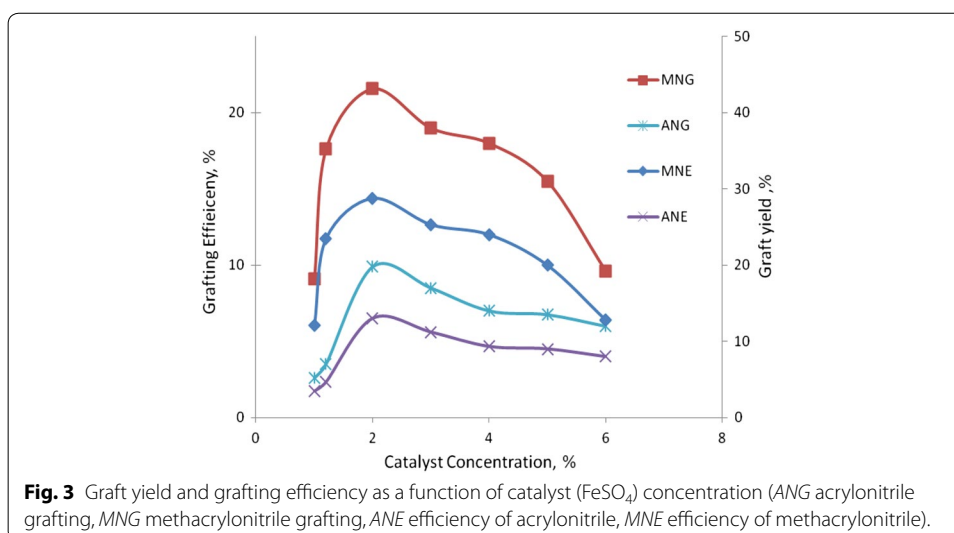
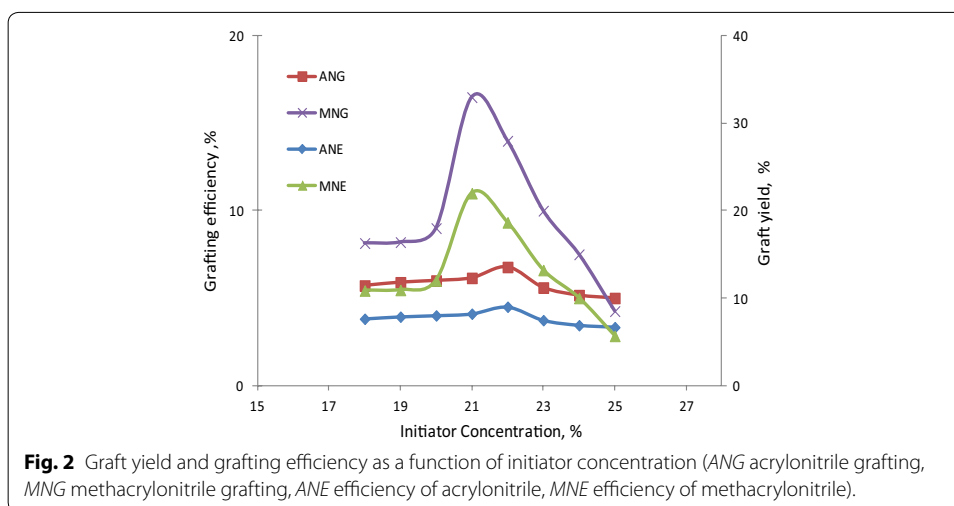
Optimization of graft copolymerization

The effect of the monomer concentration (AN and MAN) on polymerization of bleached jute fibre in the presence of a $K_2S_2O_8/FeSO_4$ redox system, is shown in Fig. 1. The graft yield increased with the increase of monomer concentration for both AN and MAN. After a certain value, it decreased with a further increase in the monomer concentration (Samal et al. 1986, 1989). The latter is due to the increasing homopolymerization rate rather than to grafting at the higher monomer concentration. The maximum graft yield was obtained when the bleached jute fibre was treated with 300% AN and 280% MAN. Their grafting efficiencies are 4.50 and 11.00%, respectively.

Figure 2 shows that the graft yield and grafting efficiency increased with the increase of $K_2S_2O_8$ concentration as initiator, up to 22% for AN and 20% for MAN. The increasing trend of the graft yield might be the result of the formation of a large number of grafting sites on the cellulose backbone of the jute fibre. At the high initiator concentration, a large number of activated monomers become available. These turn to the formation of homopolymers, causing a shortage of monomer in the vicinity of the cellulose macroradicals. Hence, the graft yield and grafting efficiency start to decrease. The retarding effects of graft yield and grafting efficiency at higher concentration of $K_2S_2O_8$ may be due to the predominance of homopolymerization over grafting. The termination of growing grafted chain is caused primarily by free radicals, resulting from the decomposition of excess initiator and production of excess Fe(III) (Mukhopadhyay et al. 1975; Misra et al. 1987; Mohanty et al. 1986).

Figure 3 shows that the percent graft yield increased with the increase of $FeSO_4$ concentration, up to 2%, for both AN and MAN. Thereafter, percent graft yield decreased.

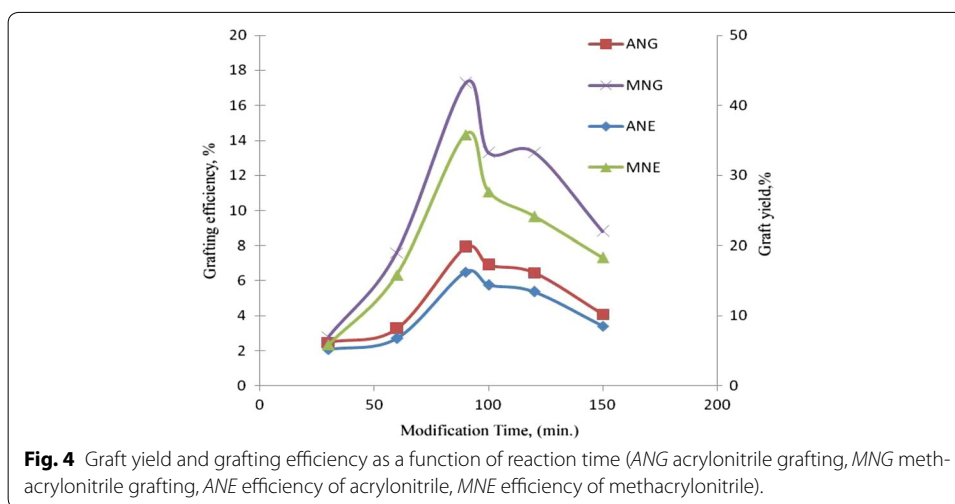




As the catalyst concentration increases, a greater number of Fe(II) ions is produced. Also, more reactive sites on the jute surface develop. Hence, grafting increases up to a certain concentration of catalyst. Beyond this concentration, graft yield and grafting efficiency decline. This is probably due to the formation of Fe(III) ions, which promote the premature termination of the growing grafted chain. Homopolymerization is also enhanced in presence of FeSO_4 (Salam 2005). The effective concentrations of FeSO_4 are 2% for both AN and MAN, and the corresponding graft yield and grafting efficiency rates are 17.78, 43.15 and 6.5, 14.38% respectively.

Figure 4 shows that the graft yield increased with the increase of reaction time, up to 90 min for both AN and MAN. During this span of time, the activated monomer combined with the active sites of the fibre matrix. After 90 min, the monomers found more reactive sites on the fibre backbone of cellulose and thus became homopolymerized (EL-Rafie 1994).

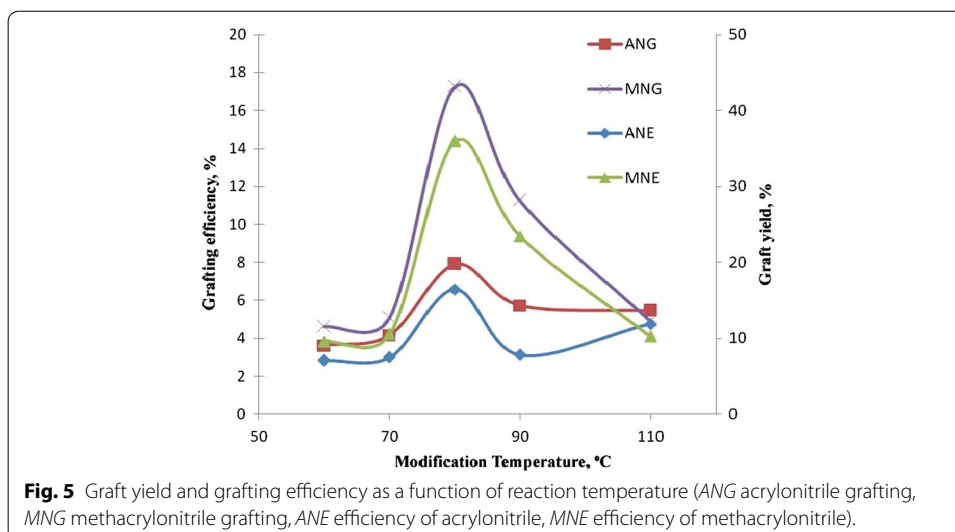
Again, at longer reaction times, the graft yield decreased. This could be due to the partial dissolution of the grafted fibre (Hebeish and Mehata 1968). The optimum



polymerization time was 90 min for both AN and MAN, while the corresponding graft yields were 19.78 and 43.15% respectively.

The effect of polymerization temperature on grafting of AN and MAN onto bleached jute fibre is shown in Fig. 5. It is seen that the grafting rate is low at low temperature, but at 80°C, the graft yield and grafting efficiency peaked. The graft yield and grafting efficiency are 19.78, 6.59% for AN and 43.15, 14.38% for MAN, respectively, within an 80–90°C temperature range. The increase in graft yield and grafting efficiency up to optimum temperature may be ascribed as increase in the rate of production of active free radicals. These free radicals increase the number of grafting sites on the fibre backbone at a higher rate. Increase of temperature increases the rate of diffusion of monomer into the jute matrix, where grafting is also initiated by complex monomers (Rahman 1970).

The decrease in grafting efficiency and graft yield, beyond optimum temperature, may be attributed to the increase in activation energy for graft copolymerization. As



discussed above, premature termination of growing grafted chains by excess Fe(III) ions, produced on the oxidation of Fe(II) ions, can also be a factor,

Dyeing behavior of the grafted jute fibre

The exhaustion of direct dyes by bleached and grafted jute fibres is listed in Table 1. The dye exhaustion of the bleached jute fibre was higher than that of the modified fibre. The dye exhaustion decreased with an increase in the percent graft yield which occurred due to increase in hydrophobicity of grafted fibre. The dye absorption of fibre depends on the availability of the total external surface of the fibre pores or cavities in a fixed amount of the fibre and the attractive force between the fibres and dye ion. The bleached jute fibre had more available pores or cavities than the modified jute fibre as these, on grafting, were blocked. Hence, the dye exhaustion of the grafted jute fibre was less than that of the bleached jute fibre. The hydrophobic nature of the grafted jute fibre may also have increased with increases in the grafting amount. In such a case, the dye exhaustion of the bleached jute fibre would be higher than that of the grafted jute fibre.

FTIR spectra of grafted jute fibre

The infrared spectra of the bleached and grafted jute fibres are shown in the Fig. 6. The IR spectra of bleached and grafted fibre were more or less similar. The FTIR spectra of the bleached and grafted jute fibre exert absorption peaks at $3,200\text{--}3,600\text{ cm}^{-1}$ due to

Table 1 Effect of dye absorption on dyeing of bleached and modified (AN and MAN) jute fibres

Dyes	Name of dye	Dye exhaustion, %		
		Bleached fibre	AN-grafted fibre	MN-grafted fibre
Reactive dyes	Reactive Orange 14	47.18	46.00	44.4
	Reactive Brown 10	46.40	40.28	38.23
Direct dyes	Direct Orange 31	83.18	82.40	81.13
	Direct Blue 1	94.15	90.38	89.23

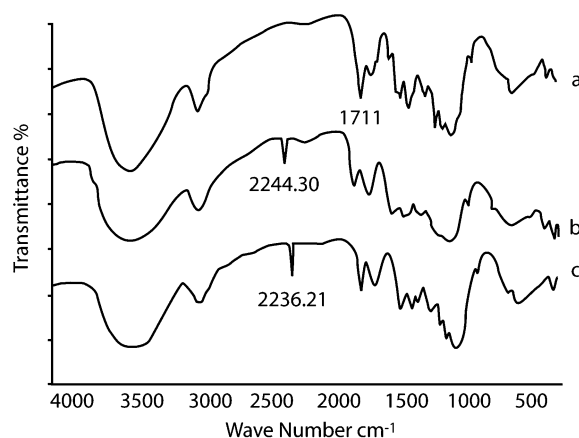


Fig. 6 FTIR spectra of (a) bleached jute, (b) acrylonitrile and (c) methacrylonitrile modified jute fibre.

the -OH vibration, $2,820\text{--}3,050\text{ cm}^{-1}$ result from the C-H stretching vibration, and the peak at $1,163\text{ cm}^{-1}$ due to the antisymmetric bridge C-O-C stretching's vibration of the cellulose pyranose ring. The presence of an absorption band near $1,711\text{ cm}^{-1}$ in the bleached jute fibre was probably due to the C=O stretching of the ester group in celluloses (Tsuboi 1957). The new additional peaks in the grafted jute fibre were appeared at around $2,240$ and $2,236\text{ cm}^{-1}$ for AN and MAN respectively indicated the presence of nitrile ($\text{-C}\equiv\text{N}$) group (Mondal et al. 2004; Khullar et al. 2008; Patra and Singh 1994).

Determination of moisture content

The moisture content of bleached fibre is 7.3% and that of modified fibre with AN and MAN fibres are 2.84 and 4.01%, at 105°C , respectively. These were measured by a moisture analyzer (Model: R.A 120-3, Kern, Germany).

Thermal stability

Figure 7a–c represents the thermal stability properties of bleached, AN and MAN modified jute fibres. Each of the figure represents two thermogram curves, namely TGA and DTG. From Fig. 7a–c, it can be seen that the loss in weight is around 66.2% at 373.5°C , 55.7% at 384°C and 62.1% at 389°C for bleached, AN-modified and MAN-modified jute fibres, respectively. The main peak at about 355°C was due to the decomposition of α -cellulose (Fig. 7a). From the DTG curve in Fig. 7a–c, the rate of decomposition of bleached jute fibre was higher than that of AN- and MAN-modified jute fibres. Thus, the thermal stability of AN- and MAN-modified jute fibres are as high as 364 and 357°C , respectively. These are higher than that of the unmodified fibres. This can be explained by the incorporation of nitrile monomer on to the jute fibre.

Surface morphology

Figure 8a–c shows the SEM micrograph of unmodified, AN-modified and MAN-modified jute fibres, respectively. The untreated cotton fibre shows the presence of a large number of micro-pores on its surface. After AN and MAN treatment, the jute fibre surfaces are coated with an outer layer of nitrile monomer (Rashidi et al. 2004) as observed in Fig. 8a, b. The ruptured surface of the modified fibre indicates the excess deposition of the nitrile monomer on the fibre hair, present in the jute fibre.

Determination of breaking strength of the bleached and modified jute fibres

It can be observed from Table 2 that the breaking load of modified jute fibre is higher than that of the bleached fibre. It is also seen that the breaking load of MAN-modified fibre is higher (19.20 kg/Yarn) than that of AN-modified fibre (17.5 kg/Yarn). The difference may be due to the fact that poly-methacrylonitrile is a rigid polymer and poly-acrylonitrile is a soft polymer (Mondal et al. 2004).

Conclusions

In the present work, we have presented the graft copolymerization of nitrile monomers on to bleached jute fibre was carried out in the presence of a redox system, in an aqueous medium. The effect of the graft yield of the nitrile monomers on the jute fibre

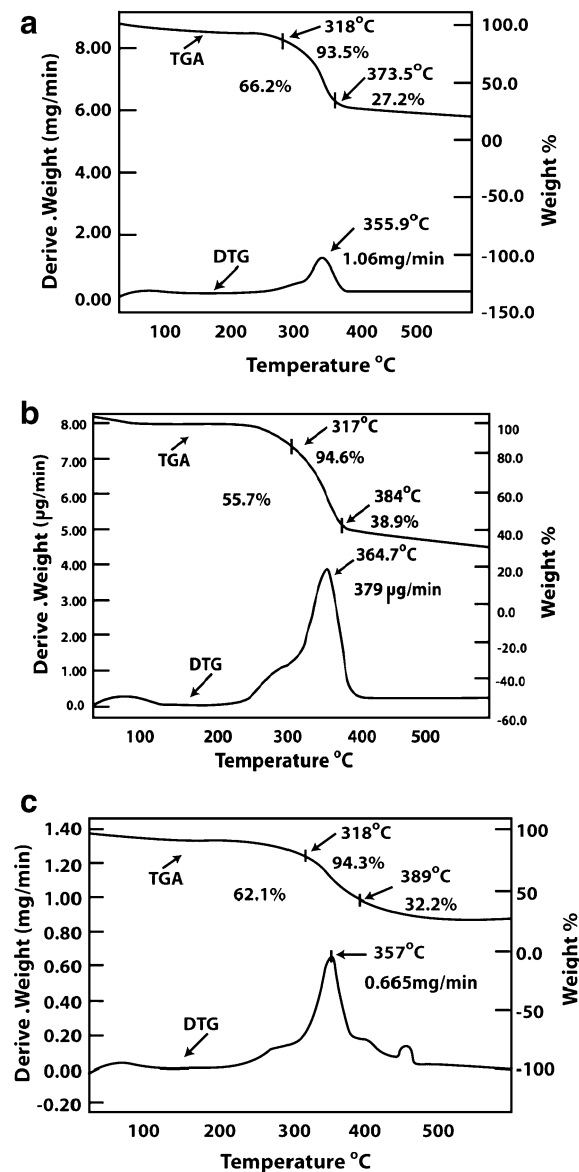


Fig. 7 TGA and DTG of **a** bleached jute fibre, **b** acrylonitrile grafted jute fibre and **c** methacrylonitrile grafted jute fibre.

depends on the parameter variables and on the jute fibre as well. The chemical attachment between the nitrile monomer and hydroxyl group of cotton fibres was evaluated by FTIR. The grafted fibre showed improved physicochemical properties like tensile

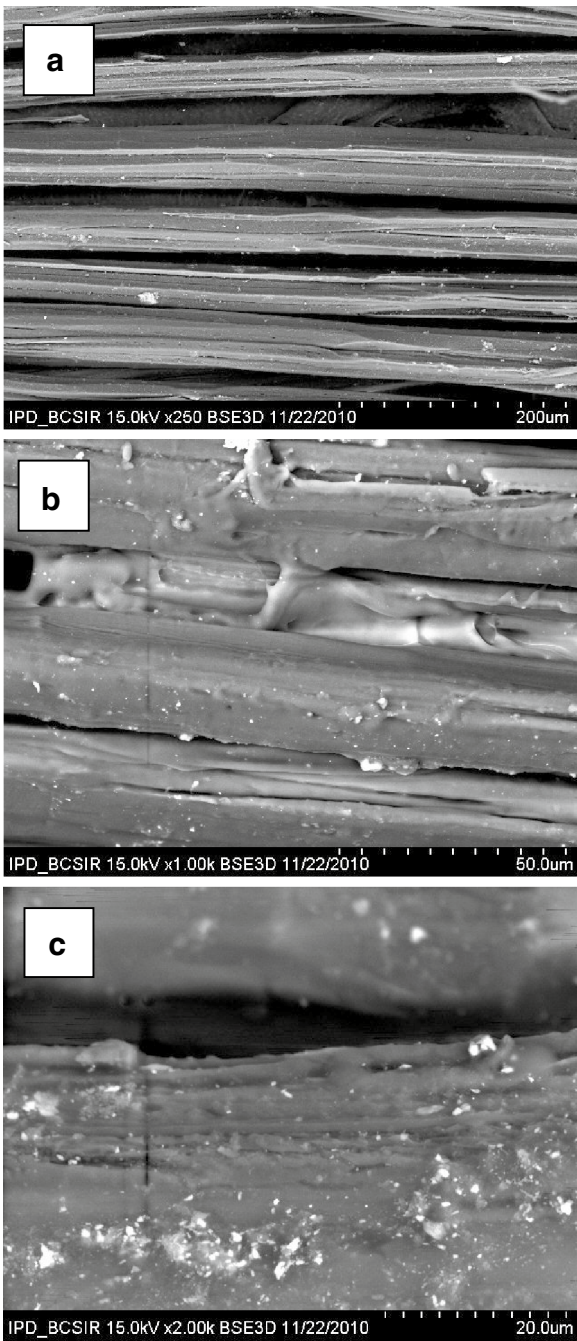


Fig. 8 SEM of **a** bleached fibre, **b** acrylonitrile grafted jute fibre and **c** methacrylonitrile grafted jute fibre.

Table 2 Breaking strength of bleached and modified fibres

Bleached fibre	Modified fibre	
	AN-grafted fibre	MN-grafted fibre
Breaking load (kg/Yarn)		
16.02 ± 0.42	17.5 ± 0.39	19.20 ± 0.56

Results are expressed as mean ± standard deviation; n = 4.

strength, moisture absorption and thermal stability. Thus a new type of jute fibre was obtained through graft polymerization. Such a process can make jute more suitable, as an input, in the manufacture of garments, home textiles etc.

Authors' contributions

IHM designed the paper, performed the literature survey and completed the paper. KI performed the experiments and analyzed the data in consultation with IHM. All authors read and approved the final manuscript.

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Compliance with ethical guidelines

Competing interests

The authors declare that they have no competing interests.

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