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## **XRD and Physicochemical Evaluation of *Hibiscus sabdariffa* Cellulose-Butyl Acrylate-co-vinyl Monomer Graft**

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### **ABSTRACT**

Utilizing the renewable waste biomass to procure advanced materials has been the aim of research. Various reaction parameters were optimized for the graft co-polymerization of *Hibiscus sabdariffa* cellulose fiber using binary vinyl monomeric mixture. The graft co-polymers thus obtained were characterized by XRD technique and the results received the supporting evidence by various other advanced analytical techniques. The percentage crystallinity and crystallinity index were found to decrease with increase in grafting while a reduction in moisture absorption was observed. There was an increase in physico-chemico-thermal resistance in the graft copolymers. These graft copolymer can be used in various scientific applications.

**Key words:** Graft copolymerization, methyl acrylate, *Hibiscus sabdariffa*, binary mixture

### **INTRODUCTION**

Nowadays, whole world has focused its attention towards renewable and sustainable resources because of environment and health concerns. The development of new polymer, advanced materials from renewable raw materials in comparison to artificial fibers has been increased during last few years (Chauhan and Kaith, 2012; Aan *et al.*, 2011; Abd El-Hady, 2011; Abd El-Hady *et al.*, 2011; Abdi *et al.*, 2010; Raja and Thilagavathi, 2011; Issaoui *et al.*, 2011; Das *et al.*, 2011; Rocco, 2011; Adedayo, 2012). Natural fibers like pine needle, flax, jute and ramie are the most suitable reinforcement materials in industries such as automobile, packaging and construction materials. Low cost, easily availability and required properties (viz. low density, effective mechanical properties) makes them attractive in place of glass, carbon and other synthetic fibers. The constituents of natural fibers are cellulose, hemicelluloses, lignin and pectin with a small quantity of the extractives. The properties of bio-fibers mainly depend upon their origin, age, climatic conditions and extraction techniques. The presence of hydroxyl groups (polar group) in various constituents of lingo-cellulosic fiber reduces the utilities in many applications. In order to improve its adhesion, various techniques like graft copolymerization, chemical treatment (viz. mercerization, acetylation, benzoylation etc.) and treatment with various coupling agent can be used. Graft copolymerization is an efficient technique to impart desirable properties to backbone polymers (Kaith *et al.*, 2003, 2004).

Various workers have carried out the graft copolymerization onto different cellulosic backbone using vinyl monomers through various chemical and radiation techniques (Kaith *et al.*, 2003, 2004; Singha *et al.*, 2004; Bavan and Kumar, 2010; Zhu *et al.*, 2008). Sharma and Daruwalla (1977) reported grafting of Methyl Acrylate (MA) individually and in binary mixtures onto cotton yarn by

emulsion technique initiated by ceric ions. Okieimen and Idehen (1987) have carried out graft copolymerization of MA onto cellulose and thiolated holocellulose and reported decrease in incorporation of graft in latter. Graft copolymerization can be initiated by using chemical, ionic and radical initiator systems. Among these initiation systems the chemical initiation by grafting involving oxidizing agents such as potassium permanganate, potassium bromate, ceric ammonium nitrate, ozone hydroxyl radicals etc are promising from economic point of view. Different workers have studied the kinetics of graft copolymerization of vinyl monomers onto the starch and synthetic copolymers but few attempts seems to have been made to study kinetics of graft copolymerization of vinyl monomers onto natural fibers (Monier *et al.*, 2010; Sabaa and Mokhtar, 2002; Eromosele and Hamagadu, 1993; Mishra and Tripathy, 1981; Taghizadeh and Khosravy, 2003).

*Hibiscus sabdariffa* (Roselle) has attained dominance as a jute substitute and all attempts are being made to extend its cultivation in areas that are unfavorable for jute cultivation. As the fiber is virgin so it was selected to explore the scope of its viability as advanced material.

## MATERIAL AND METHODS

**Plant materials:** *H. sabdariffa* was refluxed with acetone for 72 h. Monomers [Butyl Acrylate (BA) as principal monomer and its binary mixtures with Methyl Acrylate (MA), Acrylic Acid (AA) and Acrylamide (Am)] were used as received. Weighing was carried-out on Libror AEG-220 (Shimadzu) electronic balance. LEO Electron microscope (S.No.435-25-20) and Perkin Elmer instrument were used for the SEM and IR analysis. X-ray diffraction studies were performed on Bruker-D<sub>8</sub> Advance. Thermo Gravimetric Analysis (TGA) and Differential Thermal Analysis studies (DTA) were conducted in air on Thermal Analyzer (LINSEIS, L81-11) at a heating rate of 10°C min<sup>-1</sup>.

**Grafting:** Graft copolymerization of binary vinyl monomer mixture on to *H. sabdariffa* was done using butyl acrylate as a principal monomer in combination with the secondary monomers under optimized conditions as per the method reported earlier (Kaith *et al.*, 2003, 2004; Singha *et al.*, 2004):

$$P_g = \frac{W_f - W_i}{W_i} \times 100 \quad (1)$$

where,  $W_i$  and  $W_f$  are the weights of the original fiber and functionalized fiber, respectively.

## Characterization

**Fourier transformer infrared spectroscopy (FTIR) and scanning electron microscopy (SEM):** IR spectra of the ungrafted and the grafted fiber were taken with KBr pellets on Perkin Elmer Spectrophotometer. Since, the cellulose has non conducting behavior so it was gold plated to have an impact. Scanning was synchronized with microscopic beam in order to maintain the small size over large distance relative to the specimen. The resulting images has a great depth of the field. A remarkable three-dimensional appearance with high resolution was obtained on LEO Electron microscope (S.No.435-25-20).

**X-ray diffraction studies of graft copolymers (XRD):** X-ray diffraction studies were performed under ambient conditions, on Bruker using Cu K $\alpha$  (1.5418 Å) radiation, Ni-filter and scintillation counter at 40 kV and 40 mA on rotation between 13-25° at 2 $\theta$ -scale at 1 sec. step size and

increment of 0.01° with 0.5° or 1.0 mm of divergent and anti-scattering slit. The small particle size of each sample of *H. sabdariffa-g-poly* (Hs-g-poly) (BA-co-AA, BA-co-MA and BA-co-AAm) and ungrafted fiber was made. Each sample was homogeneously mixed prior to subjecting it for X-ray diffractometry.

The randomly oriented powdered sample with a uniform surface was exposed to X-rays from all the possible planes of the sample and then measuring the scattering angle of the diffracted X-rays with respect to the angle of the incident beam. The continuous scans were taken and different d-spacing's (Å) and relative Intensities (I) were obtained. The counter reading of highest peak intensity near 22.68° represents crystalline material and the peak near 15.00° in the halo-pattern corresponds to the amorphous material in cellulose. Degree of crystallinity and crystallinity index were calculated as per the following method (Mwaikambo and Ansell, 2002; Chauhan and Singh, 2011):

$$\text{Cr (\%)} = \frac{I_{22.68}}{I_{22.68} + I_{15}} \times 100 \quad (2)$$

$$\text{CI} = \frac{I_{22.68} - I_{15}}{I_{22.68}} \quad (3)$$

where, Cr (%) is percentage of crystallinity, CI is crystallinity index and  $I_{22.68}$ ,  $I_{15.00}$  are peak intensities of crystalline and amorphous content at 2θ-scale close to 22.68 and 15.00°, respectively.

### Physicochemical evaluation

**Moisture absorption study:** Moisture absorbance studies at various relative humidity levels were carried-out as per the method reported earlier. Moisture absorbance percentage was found out by placing a known weight ( $W_i$ ) of dry grafted and ungrafted samples in a humidity chamber for about two hours and then the final weight ( $W_f$ ) of the samples exposed to different relative humidities ranging from 30-90% were taken. The % moisture absorbance was calculated from the increase in initial weight in the following manner (Kaith *et al.*, 2003, 2004; Singha *et al.*, 2004; Chauhan and Singh, 2011; Chauhan and Kaith, 2012):

$$\text{Moisture absorbance (\%)} = \frac{W_f - W_i}{W_i} \times 100 \quad (4)$$

**Acid and base resistance:** Acid and base resistance studies were carried-out as per the method reported earlier. Acid and base resistance was studied by placing a known weight ( $W_i$ ) of dry grafted and ungrafted samples in fixed volume (50 mL) of 1 N HCl and 1 N NaOH and the final weights ( $W_f$ ) of the samples were noted down after 72 h (Kaith *et al.*, 2003, 2004; Singha *et al.*, 2004; Chauhan and Singh, 2011; Chauhan and Kaith, 2012):

$$\text{Weight loss (\%)} = \frac{W_i - W_f}{W_i} \times 100 \quad (5)$$

## RESULTS AND DISCUSSION

Ceric ion forms complex with the cellulose through C-2 and C-3 hydroxyl groups of the anhydroglucose unit. Transfer of the electron from the cellulose molecule to Ce (IV) would follow,

leading to its reduction to Ce(III), breakage of -OH bonds at C-2 and C-3 bond and the formation of the free radical sites where the monomeric chains get grafted. Graft yield and homo-polymer formation have been found to be the functions of both the monomer and initiator concentration (Chauhan and Singh, 2011; Chauhan and Kaith, 2012).

**Optimization of the reaction parameters:** During the graft copolymerization of BA (as a principal monomer) onto *H. sabdariffa* fiber, the different optimized reaction parameters to obtain the maximum graft yield (66.80%) were: temperature (°C), 35; time (min), 120; CAN (mol L<sup>-1</sup>), 2.27×10<sup>-4</sup>; HNO<sub>3</sub> (mol L<sup>-1</sup>), 1.46×10<sup>-3</sup>; BA (mol L<sup>-1</sup>), 1.40×10<sup>-3</sup> and pH, 7.0.

**Effect of the binary monomer mixtures on percentage grafting:** Graft copolymerization of different binary mixtures: BA+MA, BA+AA and BA+AAM using BA as a principal monomer under optimized reaction conditions showed 44.95%, 24.95% and 12.80% grafting, respectively (Table 1). Butyl acrylate as a principal monomer resulted in high Pg (66.80%) due to its chemical reactivity, high rate of propagation (K<sub>p</sub>), high rate of propagation to termination (K<sub>p</sub>/K<sub>t</sub>) and high radical transfer rate constant (C<sub>m</sub>). However, methyl acrylate as a comonomer due to relatively similar nature and properties has lesser diffusibility into the reaction medium that decreases its Pg. AA as a comonomer due to high solubility in the reaction medium forms hydrogen bond that results in decreased Pg. In case of AAM as a comonomer with butyl acrylate, the change in free radical transfer rate constant, polarity of the monomer mixture and overall composition affects the flocculation property of the monomer leading to significant decrease in Pg. However, many other factors also determine the graft yield like the type of fiber, swelling, number of active sites, the nature and amount of the solvent and temperature of polymerization strongly influence the reactivity ratios. In absence of monomer rich phase, the diluents will compete with the monomers for adsorption sites. The amount of adsorption will depend upon the total amount of surface area present and this in turn, is dependent upon the rate of stirring. Physical factors like mixing efficiency determines the melt temperature, the pressure, the rheological properties, solubility of

Table 1: Graft copolymerization of different binary vinyl monomeric mixture onto *H. sabdariffa* fiber

Sample	Binary mixture	Pg	±SD	±SE
Hs-g-poly (BA+MA)	1.40+0.55	35.00	3.57	2.06
	1.40+1.10	44.95	1.78	1.02
	1.40+1.65	34.00	3.56	2.06
	1.40+2.20	27.90	3.59	2.07
	1.40+2.75	23.15	4.40	2.58
Hs-g-poly (BA+AA)	1.40+0.72	24.95	1.76	1.02
	1.40+1.45	22.65	3.54	2.05
	1.40+2.17	19.25	6.07	3.51
	1.40+2.91	18.00	6.26	3.61
	1.40+3.62	15.00	4.38	2.53
Hs-g-poly (BA+Am)	1.40+1.76	12.80	1.74	1.00
	1.40+3.50	10.81	3.58	2.06
	1.40+5.25	9.62	0.48	0.27
	1.40+7.00	9.00	0.86	0.49
	1.40+8.75	6.00	0.50	0.28

Pg: Percentage grafting, BA, MA, AA, Am are monomers used

the initiator and the monomer. Elevated temperature favors the degradation, reduces the initiator half life, modifies the rate or specificity of the reaction, influences the solubility and rheological parameters (Fried, 2005; Brandrup and Immergut, 1975; Ham, 1964).

### Characterization of graft-copolymers

**Fourier transformer-infrared spectroscopy:** *H. sabdariffa* fiber when analyzed by FTIR depicted a broad peak at  $3422.0\text{ cm}^{-1}$  (bound-OH groups) and at  $2924.8$ ,  $1454.0$  and  $1055.0\text{ cm}^{-1}$  ( $-\text{CH}_2$ , C-C and C-O stretching, respectively). However, in case of *H. sabdariffa*-g-poly(BA-co-MA): sharp peak at  $1737.8\text{ cm}^{-1}$  due to  $>\text{C}=\text{O}$  stretch of methyl acrylate while in Hs-g-poly(BA-co-AA) a peak at  $2928.6\text{ cm}^{-1}$  (due to OH stretch of COOH gp.) was observed, whereas *H. sabdariffa*-g-poly(BA-co-AAm) exhibited peaks at  $3442.0\text{ cm}^{-1}$  ( $-\text{NH}$  group of AAm) in addition to the  $>\text{C}=\text{O}$  group of butyl acrylate in all the cases.

**Scanning electron microscopy:** The cellulosic fiber lying at a distance in raw sample (Fig. 1) started forming bundles in graft co-polymers i.e. Hs-g-poly(BA-co-MA), Hs-g-poly(BA-co-AA) and Hs-g-poly(BA-co-AAm) and a distinct morphological differentiation between backbone and graft copolymers was observed (Fig. 2-4).

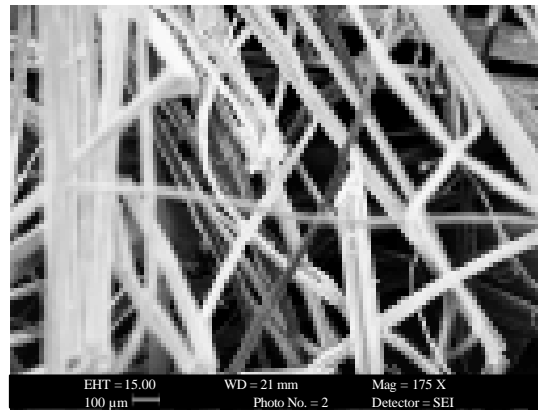


Fig. 1: SEM of *Hibiscus sabdariffa* fiber

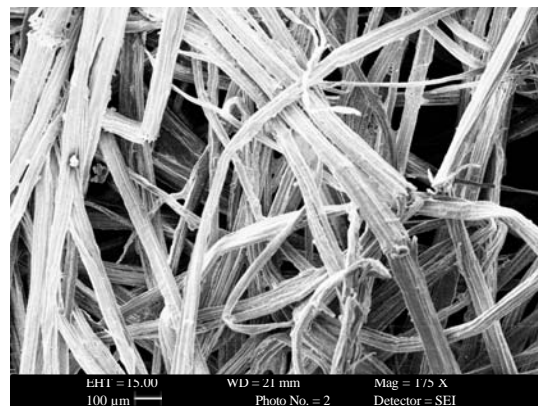


Fig. 2: SEM of Hs-g-poly(BA-co-MA)

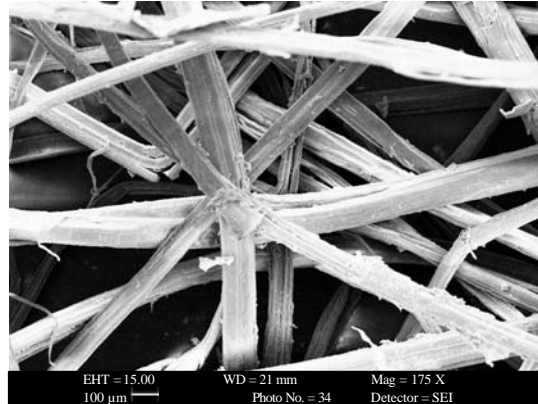


Fig. 3: SEM of Hs-g-poly(BA-co-AA)



Fig. 4: SEM of Hs-g-poly(BA-co-Am)

Table 2: Percentage crystallinity (Cr) and crystallinity index (CI) of the grafted and raw *H. sabdariffa* fiber

Sample	Pg	2θ		Cr (%)	CI
		I <sub>15.00</sub>	I <sub>22.68</sub>		
<i>H. sabdariffa</i> raw	-	40.00	136.0	77.20	0.70
Hs-g-poly(BA-co-Am)	12.80	40.00	119.0	74.84	0.66
Hs-g-poly(BA-co-AA)	24.95	38.00	111.0	75.00	0.65
Hs-g-poly(BA-co-MA)	44.75	28.00	75.00	72.81	0.62

Pg: Percentage grafting

**X-ray diffraction studies of graft copolymers:** It is evident from the results that there was decline in Cr (%) and CI, Fig. 5 clearly shows that the crystallinity and crystalline index of the fiber decreases from ungrafted to highest grafted. The results obtained are briefly tabulated in Table 2. The Cr (%) and CI from 77.20, 0.70 in raw fiber decreases to 72.81, 0.62 in Hs-g-poly(BA-co-MA) with Pg: 44.75. Crystallinity index gives a quantitative measure of the orientation of the cellulose crystals in the fibers. A lower crystalline index in case of graft co-polymers means poor order of arrangement of cellulose crystals with respect to the fiber axis. This is due to dis-orientation of the cellulose crystalline lattice to the fiber axis during grafting. Since, the incorporation of monomer

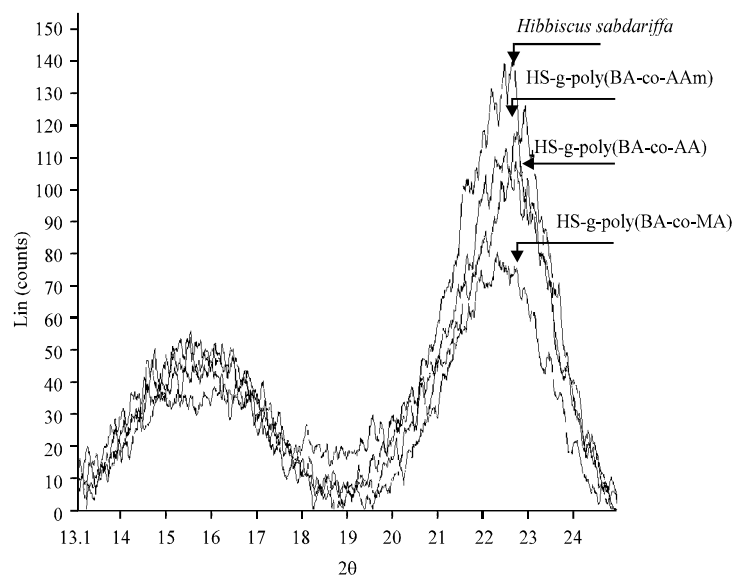


Fig. 5: XRD overlay pattern of the raw *H. sabdariffa* and its graft copolymers with binary vinyl monomeric mixtures

monomer moiety in the backbone impairs the natural crystallinity of the fiber, therefore, graft copolymerization of vinyl monomers onto *H. sabdariffa* fiber resulted in impaired crystallinity and increased the amorphous region of the fiber. Thus, with increase in percentage grafting, the percentage crystallinity and crystallinity index decreased along-with reduction in stiffness and hardness. The Cr (%) and CI of *Hs* raw (77.20; 0.70) gets reduced (72.81; 0.62) in Hs-g-poly(BA-co-MA), respectively (Chauhan and Kaith, 2012; Chauhan and Singh, 2011).

**Thermogravimetric and differential thermal analysis (TG-DTA) of graft co-polymers:** The sample, at the heating rate of 10°C per min was scanned for 60 min up to 600°C. Thermogravimetric analysis of raw *H. sabdariffa* fiber and its graft co-polymers was carried-out as a function of weight loss versus temperature. The degradation occurs in various forms like deacetylation, dehydration, decarboxylation and chain scissions. The thermograms of grafted fiber depicted two phase decomposition, the first stage signifies the breakdown of cellulose and it shifts to higher temperature as compared to raw fiber while the second stage was related to the degradation of the grafted poly vinyl chain. The shift to higher temperature could be accounted due to increase in the covalent bonds in the graft copolymers varying with the Pg. In case of *H. sabdariffa* fiber, the major weight loss occurs in the first stage due to cellulosic decomposition followed by the oxidation of the char. In case of raw fiber, both Initial Decomposition Temperature (IDT) and Final Decomposition Temperature (FDT) are lower i.e., 225.7°C and 463.0°C, respectively, as compared to those of graft co-polymers. Thus, thermal resistance of the backbone could be increased through graft copolymerization with different vinyl monomeric mixtures (Table 3) (Ouajai and Shanks, 2005; Princi *et al.*, 2005).

Further, TGA studies have strongly been supported by the DTA evaluation pattern (Table 3). The thermogram of the *H. sabdariffa* fiber have shown two major exothermic peaks at 327.9°C (18 μV) and 422.7°C (14 μV). However, in case of Hs-g-poly(BA-co-MA), Hs-g-poly(BA-co-AA) and Hs-g-poly(MA-co-AAm) the major exothermic peaks were observed at elevated temperature with higher energy release that again endorses the higher thermal stability of the graft copolymers as



Table 3: Thermo-gravimetric and differential thermal analysis of *Hibiscus sabdariffa* and its graft co-polymers

Sample	Pg	TGA		DTA
		IDT	FDT	Observed peaks in °C [μV]
Raw <i>H. sabdariffa</i>	-	25.7	463.0	139.7 (6), 327.9 (18.0), 422.7 (14.0)
Hs-g-poly(BA-co-Am)	12.80	235.0	475.0	145.0 (6.5), 350.0 (16.0)
Hs-g-poly(BA-co-AA)	24.95	260.0	504.0	151.0 (7.1), 381.0 (30.8)
Hs-g-poly(BA-co-MA)	44.75	280.0	489.9	210.0 (1.9), 386.3 (3.8), 451.2 (21.0)

Pg: Percentage grafting, IDT and FDT: Initial and final decomposition temperature

Table 4: Chemical resistance and moisture absorbance studies of graft copolymers vis-à-vis back bone

Graft	Pg	Chemical resistance (%)		Moisture absorbance (%) after 12 h			
		wt. loss after 72 h (%)		Relative humidity (%)			
		1 N HCl	1 N NaOH	30-35	50-55	60-65	85-90
<i>H. sabdariffa</i>	-	55.0	43.0	0.5	0.8	1.8	2.5
BA	66.80	37.0	26.0	-	0.1	1.3	1.6
BA-co-Am	12.80	53.0	40.0	0.4	0.6	1.7	2.5
BA-co-AA	24.95	50.0	38.0	0.2	0.3	1.3	2.1
BA-co-MA	44.95	29.0	25.0	-	0.1	0.7	1.5

Pg: Percentage grafting

compared to the raw *H. sabdariffa*. The first transition minor peak revealed the dehydration, adsorption and oxidation from the semi-crystalline host and the two major peaks signifies the fusion and the irreversible dissociation of the crystallites. Moreover, most of the hydroxyl groups of the native form have been replaced by the covalent bonds through incorporation of poly (vinyl) chains into backbone thereby leading to higher thermal stability (Ouajai and Shanks, 2005; Princi *et al.*, 2005).

### Physical and chemical properties of the graft co-polymers

**Moisture absorbance behavior:** It is evident from the Table 4 that the moisture absorbance behavior has been greatly affected by graft copolymerization this was due to incorporation of the hydrophobic organic moieties replacing the free hydroxyl groups at C-2, C-3 and C-6. Thus, with increase in Pg, there was decrease in the percent moisture absorbance i.e., Hs-g-poly(BA-co-MA)<Hs-g-poly(BA-co-AA)<Hs-g-poly(BA-co-Am) (Kaith *et al.*, 2003, 2004; Singha *et al.*, 2004).

**Chemical resistance:** Graft co-polymerization imparts resistance to the natural fiber against the alkaline and acidic medium. It has been observed that increase in Pg enhanced the chemical resistance. This could be due to the fact that incorporation of hydrophobic moieties through graft copolymerization in natural fiber decreased the chemical sensitivity for acid-base and resulted in higher chemical stability. The chemical resistance decreased in the following order Hs-g-poly(BA-co-MA)>Hs-g-poly(BA-co-AA)>Hs-g-poly(BA-co-Am) (Table 4) (Kaith *et al.*, 2003, 2004; Singha *et al.*, 2004).

### CONCLUSION

Graft co-polymerization of butyl acrylate and its binary mixtures with methyl acrylate, acrylic acid and acrylamide resulted in the incorporation of desired features in the *H. sabdariffa* fiber

while sustaining its inherent characteristics. Graft copolymerization could yield the end product with enhanced moisture and chemical resistance along with higher thermal stability. Moreover, a complete morphological transformation was observed on graft copolymerization that was important technologically as seen in the XRD results. The XRD results were supported by characterization and justified by the evaluation of the physicochemical behavior of the grafted fiber. The graft copolymers thus obtained are the better means for the utilization of the waste biomass in the advancement of technology.

## REFERENCES

- Aan, M.P.S., M. Krishna, H.N.N. Murthy and S.K. Rai, 2011. Thermal, mechanical, morphological and flame retardance properties of DABA modified novolac/CFRP composites. *Asian J. Mater. Sci.*, 3: 20-30.
- Abd El-Hady, R.A.M., 2011. The effect of aesthetic beauty design on the performance properties of jacquard weft knitted fabrics. *Asian J. Textile*, 1: 74-86.
- Abd El-Hady, R.A.M. and R.A.A. Abd El-Baky, 2011. Enhancing the functional properties of sportswear fabric based carbon fiber. *Asian J. Text.*, 1: 14-26.
- Abdi, S., Y. Ourova and O. Menchi, 2010. Development and characterization of metal matrix composite steel/Cu-Zn. *Asian J. Mater. Sci.*, 2: 147-153.
- Adedayo, A.V., 2012. Factors influencing semi-solid microstructure. *Asian J. Mater. Sci.*, 4: 1-12.
- Bavan, D.S. and G.C.M. Kumar, 2010. Potential use of natural fiber composite materials in India. *J. Reinf. Plast Compos.*, 29: 3600-3613.
- Brandrup, J. and E.H. Immergut, 1975. *Polymer Handbook*. Wiley Interscience, John Wiley and Sons, New York, Pages: 105.
- Chauhan, A. and B. Singh, 2011. X-Ray powder diffraction studies to accredit the morphological transformations in *Hibiscus sabdariffa* graft-copolymers. *Int. J. Polym. Anal. Charact.*, 16: 319-328.
- Chauhan, A. and B. Kaith, 2012. Accreditation of novel roselle grafted fiber reinforced bio-composites. *J. Eng. Fibers Fabrics*, 7: 66-75.
- Das, B.R., S.M. Istiaque and R.S. Rengasamy, 2011. Migration behavior of polyester/viscose blended rotar spun yarns. *Asian J. Textile*, 1: 27-34.
- Eromosele, I.C. and T.J. Hamagadu, 1993. Graft copolymerization of methylmethacrylate onto Caesarweed fibers by the potassium permanganate-toluene redox system. *J. Applied Polym. Sci.*, 50: 645-649.
- Fried, J.R., 2005. Conformation, Solution and Molecular Weight. In: *Polymer Science and Technology*, Fried, J. (Ed.). 2nd Edn., Prentice Hall, Upper Saddle River, NJ., USA.
- Ham, G., 1964. *High Polymer, Copolymerization*. Interscience Publishers, USA.
- Issaoui, C., A.H. Said and S. Roudesli, 2011. A polyanizidine coated textile: Elaboration and characterization. *Asian J. Textile*, 1: 98-105.
- Kaith, B.S., A.S. Singha and K.S. Sharma, 2003. Graft copolymerization of flax fibers with binary vinyl monomer mixtures and evaluation of swelling, moisture absorbance and thermal behavior of the grafted fibers. *J. Polym. Mater.*, 20: 195-195.
- Kaith, B.S., A.S. Singha and K.S. Sharma, 2004. Synthesis of graft copolymers of binary vinyl monomer mixtures and flax fiber using FAS-KPS Redox system international. *J. Chem. Sci.*, 2: 37-43.

- Mishra, M.K. and A.K. Tripathy, 1981. Grafting vinyl monomers onto natural fibers. Graft copolymerization of methyl methacrylate onto wool and silk using peroxydiphosphate-tartaric acid redox system. *Eur. Polym. J.*, 17: 1225-1226.
- Monier, M., N. Nawar and D.A. Abdel-Latif, 2010. Preparation and characterization of chelating fibers based on natural wool for removal of Hg(II), Cu(II) and Co(II) metal ions from aqueous solutions. *J. Hazard. Mater.*, 184: 118-125.
- Mwaikambo, L.Y. and M.P. Ansell, 2002. Chemical modification of Hemp, Sisal, Jute and Kapok fibers by Alkalization. *J. Applied Polym.*, 84: 2222-2234.
- Okieimen, F.E. and K.I. Idehen, 1987. Note graft copolymerization of methyl methacrylate on thiolated holocellulose. *J. Macromol. Sci. A Chem.*, 24: 1381-1384.
- Ouajai, S. and R.A. Shanks, 2005. Composition, structure and thermal degradation of hemp cellulose after chemical treatments. *Polym. Degrad. Stability*, 89: 327-335.
- Princi, E., S. Vicini, E. Pedemonte, A. Mulas, E. Franceschi, G. Luciano and V. Trefiletti, 2005. Thermal analysis and characterization of cellulose grafted with acrylic monomers. *Thermochim. Acta*, 425: 173-179.
- Raja, A.S.M. and G. Thilagavathi, 2011. Influence of enzyme and mordant treatments on the antimicrobial efficacy of natural dyes on wool materials. *Asian J. Textile*, 1: 138-144.
- Rocco, F., 2011. Technical textile based on regenerated wool: Process and test of a new generation of fabrics. *Asian J. Textile*, 1: 42-49.
- Sabaa, M.W. and S.M. Mokhtar, 2002. Chemically induced graft copolymerization of itaconic acid onto cellulose fibers. *Polymer Testing*, 21: 337-343.
- Sharma, V.N. and E.H. Daruwalla, 1977. Thermal behavior of cotton grafted vinyl monomers individually and in mixture compositions. *J Applied Polm. Sci.*, 21: 331-341.
- Singha, A.S., B.S. Kaith and S. Kumar, 2004. Evaluation of physical and chemical properties of FAS-KPS induced graft co-polymerization of binary vinyl monomer mixtures onto mercerized flax. *Int. J. Chem. Sci.*, 2: 472-472.
- Taghizadeh, M.T. and M. Khosravy, 2003. Kinetics and mechanism of graft copolymerization of vinyl monomers (Acrylamide, Acrylic acid and Methacrylate) onto starch by potassium Dichromate as redox initiator. *Iran Polym. J.*, 12: 497-505.
- Zhu, Z.H., K. Ohgo and T. Asakura, 2008. Preparation and characterization of regenerated *Bombyx mori* silk fibroin fiber with high strength. *eXPRESS Polym. Lett.*, 2: 885-889.