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Sorrel Fiber as Reinforcement in Bio-composite

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ABSTRACT

Sorrel stem fiber is rich in cellulose content and exists in abundance as waste biomass throughout the world. Its low weight and high tensile strength makes it suitable for use as backbone in graft copolymerization to reinforce physico-chemico-thermo-mechanical resistance in the fiber. The paper explores the use of rare binary vinyl monomeric mixture to synthesis, characterize and evaluate the properties of graft copolymers for use as reinforcement in composite. Advanced analytical techniques were used to confirm the transformation in the modified fiber. The improved properties of the fiber and composite as compared to the native form make it an efficient technique for judicious use of renewable resource in scientific and industrial applications.

Key words: Sorrel, graft copolymer, crystallinity, thermal resistance

INTRODUCTION

Graft copolymerization is a commendable technique for transforming the properties of the raw fiber without changing its inherent traits. It is the chemical induction of copolymerization using biomass as the polymeric backbone. It imparts physical, thermal and chemical resistance to the fiber. Literature reveals the use of natural fibers such as ramie, flax, jute and pine needle in graft copolymerization because of their relatively high strength and stiffness for their viability in industries such as automobile, packaging and construction materials as it fulfills the economic and ecological requirements. Various polysaccharides have been modified for their use in metal ion-sorption, drug delivery and water absorption studies and as reinforcement in bio-composites. A lot of research on fiber, polymer and composite materials have yielded fruitful results (Aan *et al.*, 2011; Abdi *et al.*, 2010; Raja and Thilagavathi, 2011; Issaoui *et al.*, 2011; Abd El-Hady, 2011; Das *et al.*, 2011; Rocco, 2011; Adedayo, 2012).

Composites are the wonder materials that innovates the scientist for research in interdisciplinary fields due its dynamic performance and ever increasing demand. Phenoplasts have a wide range of applications nearly 41% of the phenol-formaldehyde (PF) resins are used in plywood manufacturing and about 14% in insulation. Many efforts have been made to further explore its mechanical potential by using variable reinforcements (Kaith *et al.*, 2003a, 2004; Kaith and Kalia, 2007; Kaith and Chauhan, 2008; Singha *et al.*, 2004).

Cellulose is the most abundant polymer that possesses both crystalline and amorphous regions. Crystallinity is correlated to the strength of the polymer. On grafting, the crystal lattice of the polymer changes but its strength may add to reinforce the structure. XRD, TGA, DTA and SEM are some helpful techniques for solid state characterization and evaluation of physico-chemico-thermal changes (Chauhan, 2009; Kaith *et al.*, 2003b).

Hibiscus sabdariffa (Sorrel) is found in abundance in nature through-out the world. The excellence of graft copolymerization provoked the thoughts to implement it onto the virgin

Hibiscus sabdariffa fiber. Ethyl acrylate (EA) was used as a principal monomer in binary vinyl monomeric mixtures with methyl methacrylate (MMA) to screen the change in the percentage grafting, morphology and physico-chemical behavior of the fiber. The raw fiber and the graft copolymers were then used as reinforcement in phenol-formaldehyde (PF) polymer matrix to form bio-composite and evaluated by comparative physico-chemico-thermo-mechanical accreditation (Thacker, 1997; Jirawut *et al.*, 2006). The paper highlights the use of the renewable natural fiber for graft copolymerization with rare but efficient binary vinyl monomeric mixture to obtain the advanced materials. It explores the transformation in the properties and uses it to form bio-composites that still remain unexplored.

MATERIALS AND METHODS

H. sabdariffa was refluxed with acetone for 72 h. The Merck chemicals: methyl acrylate, n-butyl acrylate, acrylonitrile, 4-vinyl pyridine, acrylic acid, acrylamide and vinyl acetate. Ceric ammonium nitrate, Phenol (s.d. fine-Chem. Pvt. Ltd.) Formaldehyde (CDH, India) and concentrated nitric acid were used as received. Weighing was carried-out on Libror AEG-220 (Shimadzu) electronic balance. FTIR of Perkin Elmer and SEM on Electron Microscopy Machine (LEO-435-25-20) were used. X-ray diffraction studies were performed on Bruker-D₈ Advance. Tensile and compressive strength were studied on Universal Testing Machine (Hounsfield, H25KS).

Synthesis of the graft copolymers: Graft copolymerization of the monomer EA onto *H. sabdariffa* (Hs) was carried-out for the optimization of different reaction conditions like reaction time, reaction temperature, monomer concentration, concentration of initiator system and pH in order to obtain maximum graft yield. The fiber (0.5 g) was activated by swelling in 100 mL of the distilled water for 24 h. Ceric Ammonium Nitrate (CAN) and conc. HNO₃ mixture was added to the reaction medium under continuous stirring followed by the addition of a definite ratio of binary vinyl monomeric mixture. On completion of the reaction, the homopolymers of poly(MMA), were removed with water, acetone, chloroform and methanol were removed. The graft copolymers were dried at 50°C, till a constant weight was obtained (Kaith *et al.*, 2003a,b).

$$Pg = \frac{W_2 - W_1}{W_1} \times 100 \quad (1)$$

where, Pg refers to the percentage grafting while W₂, W₁ are the weights of the original fiber and functionalized fiber, respectively.

Preparation of the composites: Phenol-formaldehyde (P:F:0.75:1) matrix based composites were prepared by mixing fiber to resin (12.7:87.3). The mixture was then placed in the mold of a particular dimension: 40-80 mm (length), 5×5 mm (cross section). Degasification of sample was carried-out in Compression Molding Machine and the samples were kept for curing at 120°C for 10 min under 400 kg cm⁻² pressure. Composites thus prepared by reinforcing the raw fiber and its graft copolymers were subjected for different mechanical studies. In each case, the number of specimen used for the determination of mechanical properties was three. Tests were conducted under ambient laboratory conditions (Chauhan, 2009; Kaith and Chauhan, 2008).

PHYSICO-CHEMICAL EVALUATION

Moisture absorption study: Moisture absorbance (Mabs) 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 humidity ranging from 30-90% were taken. The percentage of moisture absorbance was calculated from the increase in initial weight in the following manner (Kaith *et al.*, 2003a; Kaith and Kalia, 2007; Singha *et al.*, 2004).

$$\text{Mabs (\%)} = \frac{W_f - W_i}{W_i} \times 100 \quad (2)$$

Acid and base resistance: 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.*, 2003a; Kaith and Kalia, 2007; Singha *et al.*, 2004):

$$\text{Weight loss (\%)} = \frac{W_i - W_f}{W_i} \times 100 \quad (3)$$

Mechanical evaluation (MOR): The analysis of the composites was done under ambient conditions by the methods as described below (Kaith and Chauhan, 2008; Chauhan, 2009).

Modulus of rupture: MOR was determined according to ASTM D 790 standard and was calculated using the following Eq. 4:

$$\text{MOR (N mm}^{-2}\text{)} = \frac{3PL}{2bd^2} \quad (4)$$

where, P = peak load, L = length of the sample, b = width of the sample and d = thickness of the sample.

Modulus of elasticity (MOE): MOE was determined according to ASTM D 790 standard and was calculated using the following Eq. 5:

$$\text{MOE (N mm}^{-2}\text{)} = \frac{P_1 L_3}{4bd^3 y} \quad (5)$$

where, b is the width of the sample, d is the thickness of the sample, P_1 is the load at the limit of proportionality and y is the rate of bending.

Stress at the limit of proportionality: Stress at the limit of proportionality (SP) was calculated by using the following Eq. 6:

$$\text{SP (N mm}^{-2}\text{)} = \frac{3P_1 L}{4bd^2} \quad (6)$$

Hardness: Hardness of the composites was tested using Rockwell Hardness tester (Balancing Instrument and Equipments Miraj Pvt. Ltd., Model-TSM) following ASTM D785 standard, using B-scale, 100 kg as a major load and 10 kg as a minor load with 1/16 inch indenter steel ball and was measured in HRB.

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 and the formation of free radical sites where the monomeric chains get grafted. Graft yield and homopolymer formation have been found to be the functions of both the monomer and initiator concentration (Chauhan, 2009).

Optimization of the reaction parameters: The optimized reaction conditions using EA as principal monomer onto the fiber were: monomer conc.: 2.26×10^{-3} mol L⁻¹; CAN: 2.41×10^{-4} mol L⁻¹; HNO₃ conc.: 1.46×10^{-3} mol L⁻¹; pH of the medium: 7; time: 150 mins; temperature: 35°C that yielded Pg of 117.30 (+SD: 7.13; +SE: 4.12).

Effect of the binary monomer mixtures on percentage grafting: The use of ethyl acrylate as principal monomer for graft copolymerization onto *H. sabdariffa* fiber yielded a high Pg. It was due to high rate of propagation (K_p), low rate of termination (K_t), higher transfer rate constant (C_m) and higher reactivity of the monomer. MMA is an efficient monomer for grafting onto *H. sabdariffa* backbone, its combination with ethyl acrylate as comonomer results in high Pg (149.90) (Table 1). However, many other thermodynamic, rheological, stereochemical and physical factors effect the graft yield (Chauhan, 2009; Chauhan and Kaith, 2011; Brandrup and Immergut, 1975; Ham, 1964).

Characterization

X-ray diffraction studies of graft copolymers: X-ray diffraction studies were performed under ambient conditions, using Cu Kα (1.5418 Å) radiation, Ni-filter and scintillation counter at 40 kV and 40 mA on rotation between 13° to 25° at 2θ-scale and 1 sec. step size. Increment of 0.01° with 0.5° or 1.0 mm of divergent and anti-scattering slit and calibrated by corundum. The continuous scans were taken and different d-spacing and relative intensities (I) were obtained (Table 2). The

Table 1: Effect of the binary mixtures on Pg using EA as a principal monomer

Sample	Binary mixture (10 ⁻³ mole L ⁻¹)	Mean Pg	±SD	±SE
Hs-g-poly (EA+MMA)	2.26+0.92	99.00	4.41	2.54
	2.26+1.84	140.06	2.60	1.50
	2.26+2.76	149.90	7.15	4.13
	2.26+3.68	110.10	2.57	1.48
	2.26+4.60	82.80	2.60	1.50

Pg: Percentage grafting

Table 2: The Cr (%) and CI of the *H. sabdariffa* fiber and its graft copolymers

Sample	Pg	2θ scale		Cr (%)	CI
		I ₁₅	I _{22.68}		
<i>H. sabdariffa</i>	-	40	136	77.20	0.70
Hs-g-poly (EA)	117.30	10	29	74.35	0.65
Hs-g-poly (EA-co-MMA)	149.90	10	16	62.50	0.37

Pg: Percentage grafting

counter reading of highest peak intensity near 22.68° represents crystalline material and the peak near 15.0° in the halo-pattern corresponds to the amorphous material in the cellulose. Degree of crystallinity and crystallinity index were calculated as per the Eq. 7 and 8 (Chauhan and Kaith, 2011):

$$\text{Cr (\%)} = \frac{I_{22.68}}{I_{22.68} + I_{15.00}} \times 100 \quad (7)$$

$$\text{CI} = \frac{I_{22.68} - I_{15.00}}{I_{22.68}} \quad (8)$$

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

As is evident from Table 2 that with increase in Pg in Hs-g-poly(EA-co-MMA), there is decline in % Cr and CI [62.50 and 0.37], respectively. In case of raw *Hibiscus sabdariffa* fiber the % Cr and CI are higher [% Cr: 77.20, CI: 0.70] and the incorporation of the monomer chains to the back-bone has impaired the crystallinity. Therefore, on grafting the percentage crystallinity decreases rapidly with reduction in its stiffness and hardness. 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 in the fiber. It could be due to dis-orientation of the cellulose crystalline lattice to the fiber axis during grafting, as seen in Table 2 (Chawala, 2002; Chauhan and Kaith, 2011). Figure 1 shows the halo pattern of the PF resin that decreases in intensity by the incorporation of the cellulosic grafted fiber as reinforcement due to accumulation of the amorphous content.

FTIR and SEM studies: In case of ethyl acrylate as a principal monomer, additional peak appeared due to EA (carbonyl stretch) at 1734 cm^{-1} and other incorporated groups of secondary monomers were witnessed at 1735.0 cm^{-1} (>C = O stretch) in Hs-g-poly(EA-co-MMA).

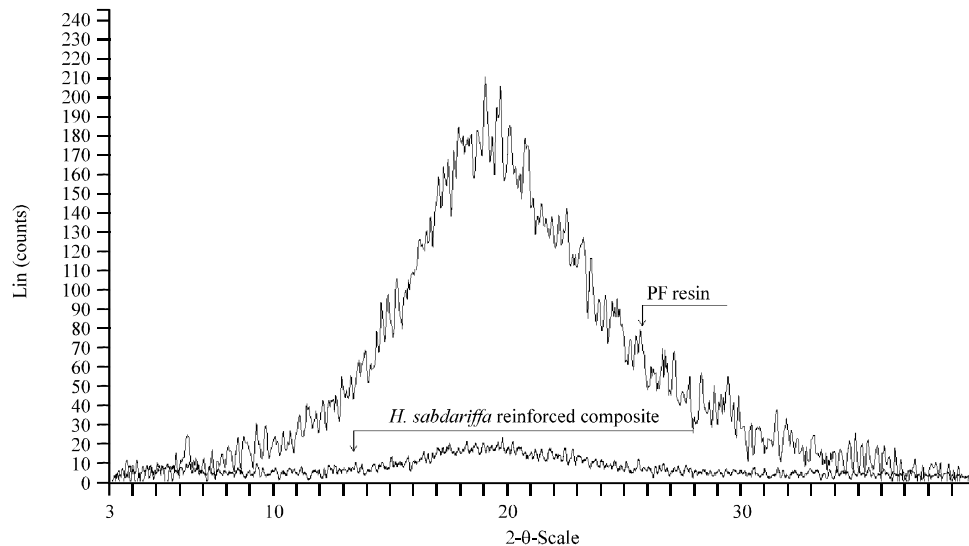


Fig. 1: The XRD overlay of PF resin and *H. sabdariffa* reinforced composite

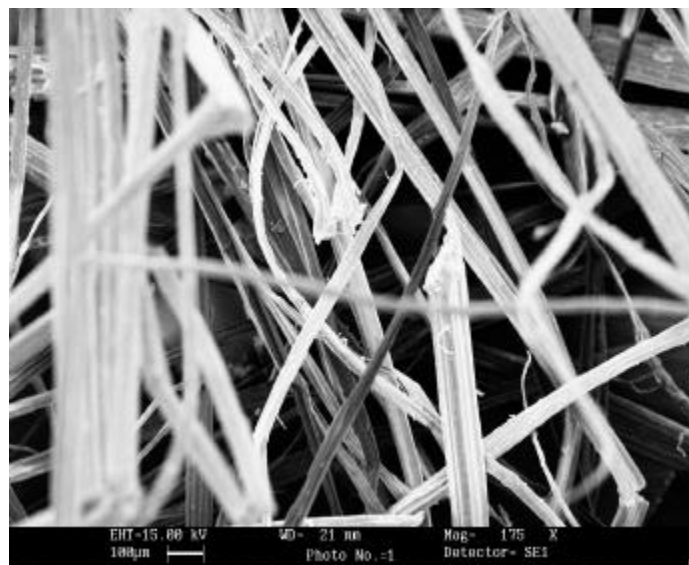


Fig. 2: SEM of raw *H. sabdariffa*

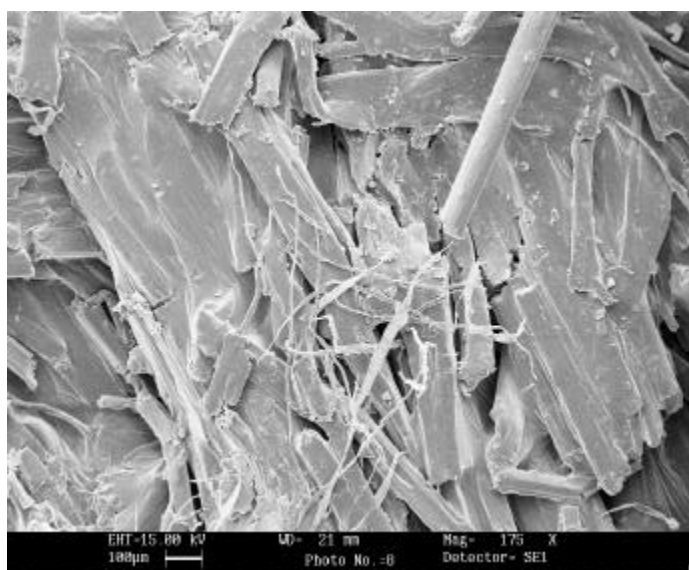


Fig. 3: SEM of highest grafted copolymer

The IR spectrum of the PF resin showed peaks at 3422.0 cm^{-1} (-OH stretch), 2920.2 cm^{-1} (C-H aromatic stretch), 1609.9 cm^{-1} and 1508.6 cm^{-1} (C = C stretch). Other peaks were found at 1465.0 cm^{-1} (CH_2 bend) and 1235.0 cm^{-1} (C-O stretch of phenol), respectively.

SEM of the raw and the highest grafted graft copolymer clearly distinguish the change in morphology brought about by grafting, depending on the Pg (Fig. 2, 3) (Singha *et al.*, 2004).

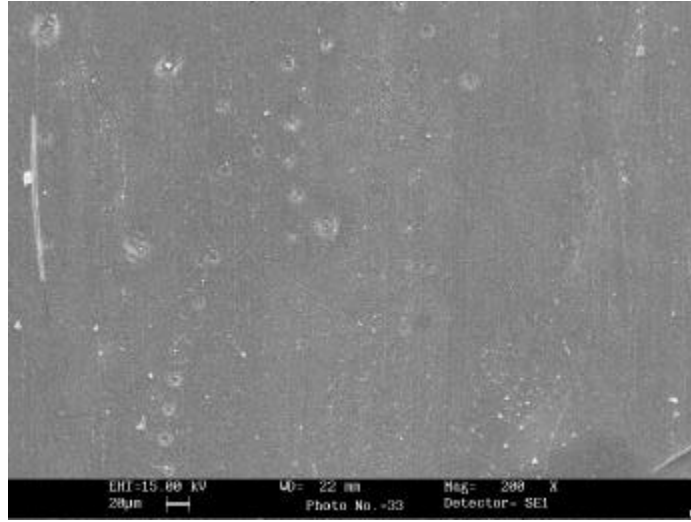


Fig. 4: SEM of raw PF resin

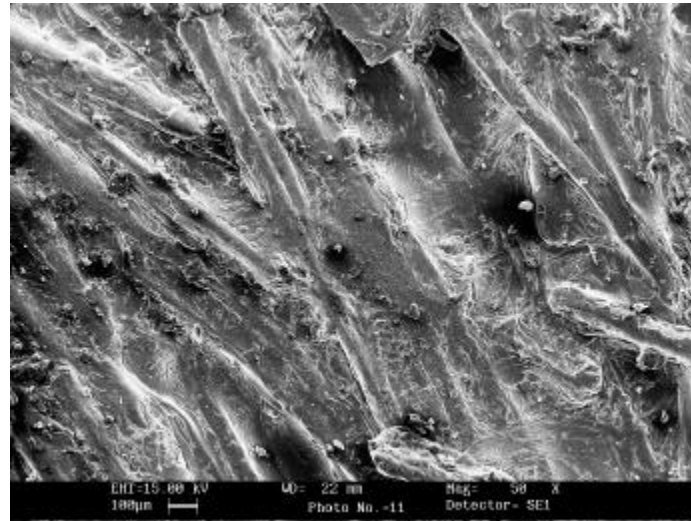


Fig. 5: SEM of *H. sabdariffa* reinforced PF composite

Since, the bio-composites had non-conducting behavior, so they were gold plated to have an impact. The SEM results helps to distinguish between the phenoplast and the fiber reinforced composite (Fig. 4, 5).

Thermal analysis: In the thermogram, the TGA of ungrafted and grafted *H. sabdariffa* have been studied as a function of % wt. loss vs. temperature. Cellulosic in *H. sabdariffa* degrades by dehydration, glycogen formation and depolymerization. In case of *H. sabdariffa*, two-stage

Table 3: TG-DTA analysis of *H. sabdariffa*, its graft co-polymers and phenoplast

Sample	Pg	TGA		Residue left (%)	DTA peaks °C (μV)
		IDT	FDT		
<i>H. sabdariffa</i>	-	225.7	463.0	20.00	327.9 (18.0), 422.7 (14.0)
(EA)	117.30	312.4	500.0	06.70	361.2 (3), 432.5 (29)
(EA-co-MMA)	149.90	339.0	542.7	28.33	152 (1.2), 341.8 (3.3), 399.4 (17)
Phenoplast	-	500.0	600.0	-	551.0 (150)
Hs-r-PF composite	-	540.0	680.0	10.00	610.0 (164)

IDT: Initial decomposition temperature, FDT: Final decomposition temperature, Pg: Percentage grafting

decomposition has been found, the former stage is attributed to loss by dehydration and volatilization whereas, the later stage is attributed to loss by depolymerization, delignification and oxidation of the char. Graft copolymers showed two stage decomposition. The first stage refers to the loss of moisture, decarboxylation and chain scissions while the second stage pertains to the breaking up of covalent bonds that has raised the FDT. Thus, it is evident from the TGA data that grafted fiber is thermally more stable than the raw fibers. The raw fiber had low IDT: 225.7 and FDT: 463.0. It may be due to the incorporation of poly(vinyl) chains on backbone polymer either through covalent bonds or mechanically, confirming the additional strength to the fiber as seen in Table 3 (Ouajai and Shanks, 2005).

In DTA studies (Table 3), *H. sabdariffa* has been found to exhibit two major exothermic peaks whereas, graft copolymers exhibited some minor and major exothermic peaks. The first and second transition peaks revealed the dehydration, adsorption and oxidation of the semi-crystalline host and the major peak signifies the fusion and irreversible dissociation of the crystallites (Chawala, 2002).

In case of PF resin the higher initial and final decomposition temperature could be due to three dimensional network structure of the thermoset. The DTA showed prominent exothermic peaks at 551°C (150 μV) that corresponds to the major decomposition due to irreversible decomposition of the crystallites between 500-600°C as a function of temperature, respectively. The IDT: 540, FDT: 680 of fiber reinforced composites have increased due to strong fiber matrix interaction. The grafted monomer incorporated the covalent bonds that served as coupling agent to elevate the decomposition temperature (Ouajai and Shanks, 2005; Chawala, 2002; Chauhan and Kaith, 2011).

Physico-chemical evaluation: The graft fiber reinforced composite showed a marked decrease in moisture absorbance at variable humidity levels [0.3% moisture absorbance at 85-90% RH] and chemical resistance against 1 N HCl and 1 N NaOH for 72 h (1% wt. loss in 1 N HCl and no loss in 1 N NaOH) due to intricate three dimensional network of PF resin that formed intricate complex with the grafted fiber and the monomer acted as a coupling agent to reinforce the strength in comparison to the micro porous surface of the phenoplast since it is prone to moisture and chemical effect whereas the native fiber had poor physico-chemical resistance [wt. loss of 55 and 43% in 1 N HCl and 1 N NaOH while percentage moisture absorbed of 2.5 at 85-90% RH] (Table 4) (Singha *et al.*, 2004; Chauhan, 2009).

Mechanical evaluation of the bio-composites: Amongst all the graft copolymers synthesized, only the three highest grafted copolymers obtained in binary mixtures and the raw fiber were used as reinforcement in the polymer matrix based composites for the mechanical evaluation, in reference to the phenoplast and Hs-reinforced PF composite.

Table 4: Chemical resistance and moisture absorbance studies of the samples

Sample	Pg	Chemical resistance (%) (wt. loss % after 72 h)		Moisture absorbance (%) at different RH after 12 h			
		1 N HCl	1 N NaOH	30-35	50-55	60-65	85-90
Hs-g-poly-							
<i>H. sabdariffa</i>	-	55.0	43.0	0.5	0.8	1.8	2.5
(EA)	117.3	-	-	-	-	-	0.2
(EA-co-MMA)	138.5	-	-	-	-	-	0.5
Phenoplast	-	04.0	02.0	-	-	0.3	0.5
<i>Hs-r-PF-composite</i>	-	01.0	-	-	-	-	0.3

Pg: Percentage grafting

Table 5: Mechanical studies of graft copolymers reinforced PF composite vis-à-vis PF resin

Composite	Strength test			
	MOR (N mm ⁻²)	MOE (N mm ⁻²)	SP (N mm ⁻²)	Hardness (HRB)
Phenol-formaldehyde	43.20	496.12	38.40	Brittle
<i>H. sabdariffa-r-PF</i>	69.99	614.40	57.60	67.00
Hs-g-poly(EA)-r-PF	82.04	898.56	74.24	75.00
Hs-g-poly(EA-co-MMA)-r-PF	78.00	680.00	70.00	78.00

r-PF: Reinforced phenol-formaldehyde, MOR: Modulus of rupture, MOE: Modulus of elasticity, SP: Stress at the limit of proportionality

Thus, it is evident from the results (Table 5) that *H. sabdariffa* fiber as reinforcement has improved the mechanical properties of the PF composites. The better mechanical behavior of Hs-g-poly(EA)[MOR: 82.04, MOE: 898.56, SP: 74.24 in N mm⁻²] and Hs-g-poly(EA-co-MMA) [hardness: 78 HRB] graft copolymer reinforced composite could be accounted due to compatible fiber-matrix interaction and the orientation of the fiber. The better mechanical strength in composites reinforced with graft copolymers in comparison to the raw fiber could be due to the reinforced strength provided by the monomer incorporated during graft copolymerization and the monomer itself acted as the coupling agent. The PF composite had still lesser strength [MOR: 43.20, MOE: 496.12, SP: 38.40 and hardness: brittle] due to lack of tensile strength and reinforcement. However, some deviations in the results could be justified by the factors like nature, amount of matrix and fiber, orientation, distribution of the fiber with respect to the matrix axis, form of reinforcement used (woven or non-woven, grafted or ungrafted), strength of the interfacial bond between the fiber and matrix, length of the fiber (continuous or discontinuous), aspect ratio that on mere imbalance may lead to debonding and cracking (Chawala, 2002; Chauhan and Kaith, 2011; Chauhan, 2009).

CONCLUSIONS

Graft copolymerization of ethyl acrylate and its binary vinyl monomeric mixture onto *Hibiscus sabdariffa* fiber improved the properties, behavior of the fiber. The raw fiber showed the highest percentage crystallinity and crystallinity index in comparison to its graft copolymers that decreased continuously with increase in Pg. However, the cellulose form I remained unchanged after graft copolymerization. The use of these physico-chemico-thermally modified graft copolymers could be of numerous applications in transport, packaging, aerospace and insulator in transformers. When these graft copolymers were used in phenoplast as reinforcement they increased the physico-

chemico-mechanical strength. These novel materials were found to be physico-chemico-thermally resistant for better scientific application and advancement of technology. It is one of the wise and economic chemical technique to utilize the renewable waste biomass.

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