

Evaluation of Sereni Fiber Reinforced Composite

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ABSTRACT

Sereni stem fiber, due to low weight and high tensile strength was selected as backbone for graft copolymerization with binary vinyl monomeric mixture to explore its effect on percentage grafting, properties and the behavior of the modified fiber. The graft co-polymers were reinforced in a phenoplast matrix to form fiber reinforced composites and were characterized by XRD, TGA, DTA, SEM and FTIR techniques. Moisture absorbance, chemical resistance in acid and base and assessment of flexural strength, young's modulus, stress at the limit of proportionality, and hardness of the composite were studied in comparison to the phenoplast.

Keywords: Sereni, graft- copolymers, crystallinity, thermal resistance.

INTRODUCTION

Graft copolymerization is a technique used to incorporate desired properties into a textile fiber without affecting its basic properties [1]. It imparts additional properties such as thermal and chemical resistance to the naturally occurring back-bone for use in various fields [2,3]. Natural fibers such as flax, jute, ramie and pine needle are suitable as reinforcement materials because of their relatively high strength and stiffness in industries such as automotive, packaging and construction materials [4], to satisfy economic and ecological aspects.

Various polysaccharides such as cellulose, starch, chitosan, guar-gum and psyllium have been modified for use in metal ion-sorbition, drug delivery and water absorption studies. Moreover, these polymeric materials have been extensively used in agricultural and membrane technology. Studies have shown that these natural polymers have immense significance in water treatment processes [5-14] due to biodegradability and cost effectiveness.

Sereni (*Hibiscus sabdariffa*) has attained prominence as a jute substitute and attempts are being made to extend its cultivation in areas which are not favorable

for jute cultivation. The fiber is rich in cellulose content (73.9%) but it lacks therapeutic potential. Due to its light weight and high tensile strength, it could be used as backbone for graft copolymerization [15]. The versatility of graft copolymerization inspired the thoughts to implement it onto virgin *Hibiscus sabdariffa* stem fiber by using effective butyl acrylate (BA) as principal monomer with acrylic acid (AA), acrylamide (AM) and 4-vinyl pyridine (4-VP) as secondary monomer to screen the change in physico-chemio-thermal properties, that still remained unnoticed; and then use them as reinforcement in a phenol-formaldehyde polymer matrix for physico-chemico-mechanical evaluation.

EXPERIMENTAL

Materials

H. sabdariffa fiber was obtained from the Department of Agronomy, Chaudhary Sarwan Kumar Himachal Krishi Vishwavidyalaya, Palampur (H.P.) India. Monomers (Merck) and ceric ammonium nitrate (s. d. fine-Chem, Pvt. Ltd, Mumbai, India) were used as received. *H. sabdariffa* fiber was purified through soxhlet extraction in acetone for 72 hours.

Method

Graft Copolymerization

Graft copolymerization of the vinyl monomer onto *H. sabdariffa* 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 the maximum graft yield. The fiber (0.5g) was activated by swelling in 100 ml of the distilled water for 24 hrs. Mixture of ceric ammonium nitrate (CAN) and conc. HNO₃ mixture were slowly added to the reaction medium in definite ratio of binary vinyl monomeric mixture. The reaction was carried-out at a definite temperature for a particular time interval. On completion of the reaction, homo-polymer like poly(BA), poly(BA-co-AA), poly(BA-co-AM) and poly(BA-co-4VP) were removed on extraction with chloroform, acetone, methanol and water. The graft

co-polymer was dried at 50°C, until a constant weight was obtained. The percent grafting (P_g) was shown in Eq. (1) [1-4]:

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

where, W_f = final weight of the fiber, W_i = initial weight of the fiber.

Modulus of Rupture and Modulus of Elasticity (MOR, MOE)

MOR (flexural strength), MOE (young's modulus) were determined according to ASTM D 790 and were calculated using the Eq. (2) and Eq. (3) [16]:

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

$$MOE = \frac{P_1L^3}{4bd^3y} \text{ N/mm}^2 \quad (3)$$

where, P = peak load, L = length of the sample, b = width of the sample and d = thickness of the sample, P_1 = load at the limit of proportionality and y = rate of bending.

Stress at the Limit of Proportionality

Stress at the limit of proportionality was calculated by using the Eq. (4) [16]:

$$SP = \frac{3P_1L}{2bd^2} \text{ N/mm}^2 \quad (4)$$

where, P_1 = load at the limit of proportionality, L = length of the sample, b = width of the sample and d = thickness of the sample.

Hardness

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

Characterization

IR spectra of the *H. sabdariffa* and its graft copolymers were recorded with a Perkin Elmer Fourier Transform Infrared (FTIR) spectrophotometer using KBr pellets (Sigma Aldrich). Scanning Electron Micrographs (SEM) of *H. sabdariffa* and its graft copolymers were obtained by using the Electron Microscopy Machine (LEO 435-25-20). Thermo-gravimetric and differential thermal analysis were performed on a thermal analyzer (LINSEIS, L-81 11). X-ray diffraction studies were performed on an X-ray diffractometer (Bruker D8 Advance) under ambient conditions using $\text{Cu K}\alpha$ (1.5418 Å) radiation, Ni-filter and scintillation counter as detector at 40 KV

and 40 mA on rotation between 13° to 25° at 2θ scale at 1 sec. step size and increment of 0.01 degree with 0.5° or 1.0 mm of divergent and anti-scattering slit. The sample holder was made of PMMA and consisted of round central cavity. Outer diameter of the holder was 50 mm, central cavity had a diameter of 25 mm that retained the sample height up to 1 mm and the holder height was 8.5 mm. Corundum was the reference sample and quartz was the standard used to verify and calibrate the instrument.

The continuous scans were taken and different d-spacing and relative intensities (I) were obtained. Results are depicted in *Figure 1*. Crystallinity index (C.I.) measures the relative ratio of the crystalline and amorphous regions in fiber, using the wide angle X-ray diffraction counts at 2θ-scale close to 22.68° and 15°. The counter reading at the peak intensity of 22.680 represents the crystalline material and the peak intensity at 15° corresponds to the amorphous material in cellulose. Percentage Crystallinity (% Cr) and Crystallinity index (C. I.) were calculated as shown in Eq. (5) and Eq. (6) [1]:

$$\% \text{ Cr} = \left[\frac{I_{22.68}}{I_{22.68} + I_{15}} \right] \times 100 \quad (5)$$

$$\text{C. I.} = \left[\frac{I_{22.68} - I_{15}}{I_{22.68}} \right] \quad (6)$$

where, $I_{22.68}$ and I_{15} are the crystalline and amorphous intensities at 2θ-scale close to 22.68° and 15°, respectively.

Physico-Chemical Evaluation

Moisture Absorption Study

Moisture absorbance was found by placing a known weight (W_i) of dry grafted and ungrafted samples in humidity chamber for about twelve 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 by the Eq. (7) [1-3]:

$$\text{Mabs (\%)} = \left[\frac{(W_f - W_i)}{W_i} \right] \times 100 \quad (7)$$

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 1N HCl and 1N NaOH and the final weights (W_f) of the samples were noted down after 72 hours [1-3]:

$$\text{Weight loss (\%)} = \left[\frac{(W_i - W_f)}{W_i} \right] \times 100 \quad (8)$$

Preparation of the Resin

Phenol-formaldehyde resin was prepared as per the method reported earlier [16]. Different ratios of P:F

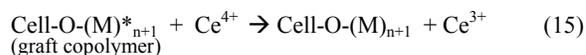
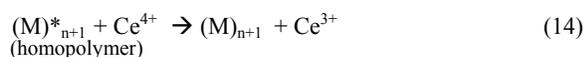
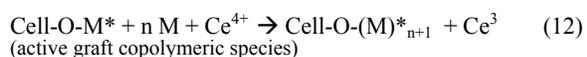
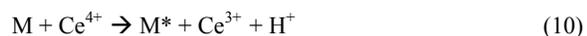
were optimized with respect to their mechanical behavior so as to achieve better mechanical properties. A P-F ratio of 0.75: 1.0 was found to be the optimum ratio exhibiting better mechanical properties.

Preparation of the Composites

The *H. sabdariffa* stem fiber was mixed with P-F resin, using different fiber: resin ratios. The mixture was placed in the mold with dimensions: 40- 80 mm length, 5 x 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 minutes under 400 kg/cm² pressure. Fiber: resin ratio of 12.7: 87.3 was found to exhibit better mechanical properties. In each case, the number of replications used for the determination of mechanical properties was three. Tests were conducted under ambient laboratory conditions (35°C and 50% RH) [16].

RESULTS AND DISCUSSION

Ceric ammonium nitrate used as source of ceric ion in presence of concentrated nitric acid plays an important role during graft copolymerization Eq. (10) and Eq. (15). Because of large size [Ce-O-Ce]⁶⁺ ion it is unable to form complex with the fiber whereas, in presence of HNO₃ more of Ce⁴⁺ and [Ce (OH)₃]³⁺ ions were formed and these ions easily undergo complex formation with the hydroxyl groups of fiber. Ceric ion forms the chelate complex with the cellulose molecule through C-2 and C-3 hydroxyl groups of the anhydro glucose unit. Transfer of the electron from the cellulose molecule to Ce (IV) would follow, leading to its reduction to Ce (III), breakage of the bonds at C-2 and C-3, resulting in the formation of the free radical sites ceric ion forms complex with the cellulose through C-2 and C-3 hydroxyl groups of the anhydro glucose unit. Graft yield and homo-polymer formation Eq. (14) have been found to be the functions of both the monomer and initiator concentration [1].



where, Cell-OH = *H. sabdariffa* backbone and M = monomer

Optimization of Different Reaction Parameters and Effect of Binary Vinyl Monomeric Mixture

The optimized parameters for the graft copolymerization of BA (as a principal monomer) onto *H. sabdariffa* fiber were: monomer conc.: 1.40 x 10⁻³ mol L⁻¹; CAN: 2.27 x 10⁻⁴ mol L⁻¹; HNO₃ conc.: 1.46 x 10⁻³ mol L⁻¹; pH of the medium: 7; time (mins.): 120; temperature (°C): 35 that yielded Pg of 66.80 (±SD: 6.10; +SE: 3.52).

Butyl acrylate as a principal monomer had relatively high reactivity, suitable chemical properties (K_t, K_p /K_t, C_M) needed for grafting onto *H. sabdariffa* backbone. 4-VP due to an unfavorable reactivity ratio deteriorates its Pg even at high concentrations. AA as comonomer had high solubility in the reaction medium that decreases the Pg (24.95). AM had higher polarity that effects the grafting (Pg:12.80) [17,18] (Table I). However, many other factors influence 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 the initiator and the monomer. Elevated temperature favors the degradation, reduces the initiator half life, modifies the rate or specificity of the reaction, and influences the solubility and rheological parameters [17-19].

Characterization

FTIR and SEM

FTIR spectrum of the *H. sabdariffa* showed a broad peak at 3424.0 cm⁻¹ (-OH group) and peaks at 2924.7 cm⁻¹, 1246.9 cm⁻¹ and 1032.0 cm⁻¹ were observed due to -CH₂, C-C and C-O stretching, respectively. However, in case of *Hs-g-poly(BA)* a peak (due to >C=O groups) appeared at 1738.0 cm⁻¹ whereas, an additional peak at 1638.1 cm⁻¹ (vinyl group) in *Hs-g-poly(BA-co-4VP)*; 2928.6 cm⁻¹ (-OH group) in *Hs-g-poly(BA-co-AA)* and at 3442.0 cm⁻¹ (N-H stretch) in *Hs-g-poly(BA-co-AM)* were observed.

In SEM, the cellulose was gold plated to have an impact. It is quite evident from the *Figures 1a-1b* that there has been a sufficient deposition of polyvinyl

monomers onto fiber. Comparison of the scanning electron micrographs of the raw *H. sabdariffa* fiber with the graft copolymers reveals the distinction between the ungrafted and grafted samples depending upon the Pg. Similarly, the SEM of the P-F resin and *Hs*-reinforced-PF composite distinguish the change in morphology brought about by the reinforced fiber in Figures 1c-1d.

TABLE I. Effect of the binary mixtures on Pg using BA as a principal monomer.

Sample	Binary mixture (10 ⁻³ mole L ⁻¹)	Mean Pg	+SD	+SE
<i>Hs</i> -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
<i>Hs</i> -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
<i>Hs</i> -g-poly (BA+4VP)	1.40+0.45	05.80	+0.48	+0.27
	1.40+0.91	15.85	+1.74	+1.00
	1.40+1.37	25.00	+3.57	+2.06
	1.40+1.83	21.20	+1.78	+1.02
	1.40+2.29	16.40	+1.79	+1.03

XRD Studies

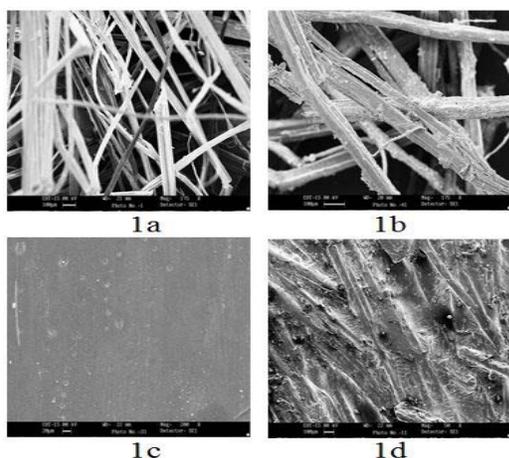


FIGURE 1. SEM of 1a. *Hibiscus sabdariffa*. 1b. *Hs*-g-poly(BA) 1c. P-F resin. 1d *Hs*-r-PF composite.

As is evident from the XRD studies in Table II, the percentage crystallinity and crystallinity index were found to decrease with increase in percentage grafting. The incorporation of monomer moiety at the backbone impairs the natural crystallinity and increased the amorphous content of the fiber. With increase in Pg the crystallinity index decreased along-with the reduction in stiffness and hardness. Crystallinity index (C.I.) is the quantitative measure of the orientation of the crystal lattice to the fiber axis; therefore, lower crystallinity index in case of graft copolymers stands for poor order of crystal lattice in the fiber. The dis-orientation of the crystal lattice during grafting resulted in decrease in crystallinity index. This clearly indicates that the cellulose crystals are better oriented in *H. sabdariffa* fiber rather than *Hs*-g-poly(BA) and its binary graft copolymers [1].

Thermal Evaluation

In the thermogram, the TGA of ungrafted and grafted *H. sabdariffa* have been studied as a function of % wt. loss vs. temperature (Figures 2 & 3, Table III). Cellulosic in *H. sabdariffa* degrades by dehydration, glycogen formation and depolymerization. In case of *H. sabdariffa*, two-stage decomposition has been found, the former stage is attributed to loss by dehydration, volatilization whereas, the later stage is attributed to loss by depolymerization, delignification and oxidation of the char [20]. 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. Thus, it is evident from the TGA data that increase in FDT of the grafted fiber is due to the incorporation of poly (vinyl) chains on backbone polymer either through covalent bonds or mechanically, confirming the additional strength to the fiber [20-23].

TGA studies had strongly been supported by the DTA evaluation pattern, the fiber was 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 (Table III) [20- 23]. In case of raw *H. sabdariffa* fiber initial combustion takes place at lower temperature as compared to that of graft co-polymers. It could be due to the fact that the hydroxyl groups of the native form have been replaced by the covalent bonds during grafting.

Physico-Chemical Evaluation

Moisture Absorbance

It was found that graft co-polymerization of vinyl monomer onto *H. sabdariffa* had a great impact on the moisture absorbance behavior (Table IV). It was observed that with increase in graft yield, there was a decrease in percent moisture absorbance due to blocking of sites vulnerable for moisture absorbance

with vinyl monomer chains thereby, converting the fiber less sensitive to moisture [1-3]. Similarly, the graft fiber reinforced composite showed a marked decrease in moisture absorbance due to strong intricate bonds between the resin and the fiber. Moreover, the monomer incorporated on the fiber acted as coupling agent that decreased the moisture absorbance as compared to phenoplast.

TABLE II. The % Cr and C. I. of the *H. sabdariffa* fiber and its graft copolymers.

Sample	Pg	20 Scale		%Cr	C.I.
		I ₁₅	I _{22.68}		
<i>H. sabdariffa</i>	-	40	136	77.20	0.70
<i>Hs-g-poly</i> (BA)	66.80	28	50	64.10	0.44
<i>Hs-g-poly</i> (BA-co-AM)	12.80	40	119	74.84	0.66
<i>Hs-g-poly</i> (BA-co-AA)	24.95	38	111	75.00	0.65
<i>Hs-g-poly</i> (BA-co-4VP)	25.00	35	104	74.82	0.66

where, % Cr= percentage of crystallinity, C. I.= crystallinity index.

TABLE III. Thermogravimetric-Differential Thermal Analysis of *H. sabdariffa* and its graft copolymers.

Graft copolymer	Pg	TGA			DTA
		IDT	FDT	% Residue left	Peaks in °C(μV)
<i>H. sabdariffa</i>	-	225.7	463.0	20.00	139.7 (6), 327.9 (18.0), 422.7 (14)
(BA)	66.80	301.6	489.5	13.40	150 (3), 368.1 (8), 397.5 (22)
(BA-co-AM)	12.80	233.0	470.0	16.00	150 (1.9), 358.3 (3.8), 430.2 (21)
(BA-co-AA)	24.95	243.0	475.0	14.00	145.0(9), 349.0(27), 391.0(29)
(BA-co-4VP)	25.00	248.0	474.0	18.00	150.0(3), 360.0(14), 398.0(31)

where, IDT = initial decomposition temperature, FDT= final decomposition temperature

TABLE IV. Chemical resistance and moisture absorbance studies of graft copolymers vis-à-vis back bone.

Graft copolymer	Pg	% Chemical Resistance		% Moisture Absorbance			
		% wt. loss after 72hour		at different RH after 12 hours			
<i>Hs-g-poly-</i>		1N HCl	1N 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	-	-	0.5	1.1
(BA-co-AM)	12.80	50.0	40.0	0.1	0.4	1.3	2.2
(BA-co-AA)	24.95	46.0	36.0	0.1	0.4	0.9	1.9
(BA-co-4VP)	25.00	46.0	35.0	-	0.4	0.7	1.9

Chemical Resistance Studies

It has been observed that acid- base resistance of the fiber increased with increase in percent grafting. This is due to the fact that poly (vinyl) chains grafted onto *H. sabdariffa* fiber had less affinity for 1 N HCl and 1N NaOH as compared to the free hydroxyl groups present in ungrafted fiber. Therefore, the resistance of fiber towards acid-base was found to increase with the incorporation of poly (vinyl) chains on the active sites of the backbone [1-3] (Table IV). Similarly, the graft fiber reinforced composite complex showed a marked decrease in chemical resistance due to strong fiber

matrix complex moreover, the monomer acted as a coupling agent to reinforce the strength in composite as compared to the phenoplast.

Mechanical Evaluation

It is well evident from the results (Table V), that *H. sabdariffa* graft copolymer and the raw fiber as reinforcement have improved the mechanical properties of the P-F composites. The better mechanical behavior could be accounted by the favorable fiber-matrix interaction, compatibility of the

vinyl monomers that served as coupling agent and the orientation of the fiber. However, some deviation in the results could be justified by other governing factors like nature and 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 [23-25]. These composites were also found to be physico-chemico-thermally stable and efficient [25].

TABLE V. MOR, MOE, SP and Hardness studies of graft copolymers reinforced P-F composite vis-à-vis P-F 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</i> -r-PF	69.99	614.40	57.60	67.00
<i>Hs</i> -g-poly(BA)-r-PF	75.16	720.60	66.00	70.00
<i>Hs</i> -g-poly(BA-co-AM)-r-PF	72.00	642.00	59.00	69.00
<i>Hs</i> -g-poly(BA-co-AA)-r-PF	73.00	780.00	60.00	68.00
<i>Hs</i> -g-poly(BA-co-4VP)-r-PF	73.00	690.90	61.00	70.00

where, -r-PF refers to reinforced Phenol-formaldehyde

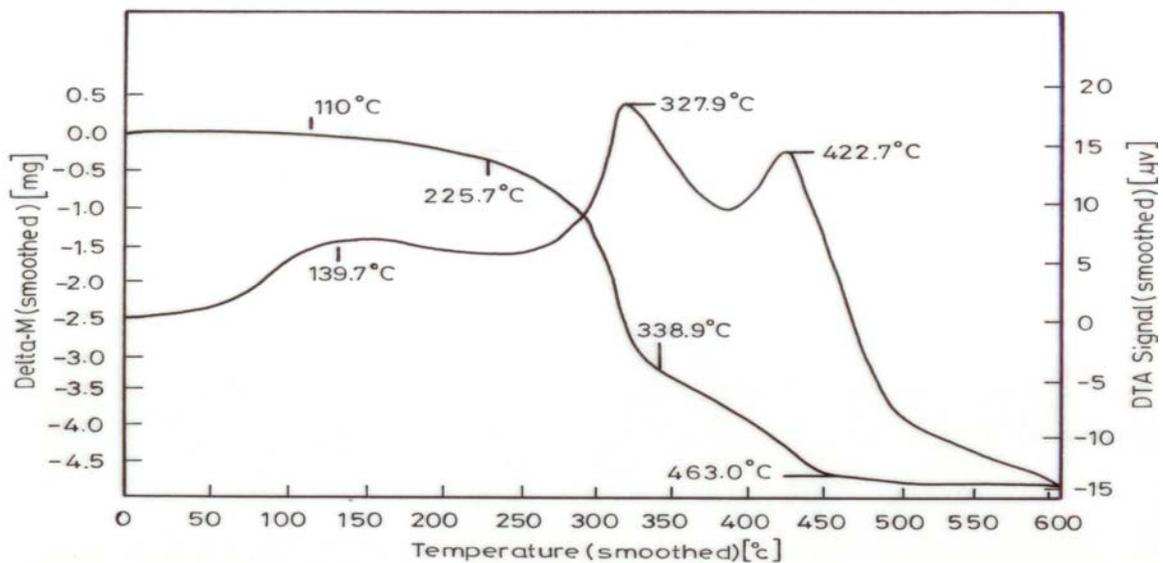


FIGURE 2. The TG-DTA of *Hibiscus sabdariffa*.

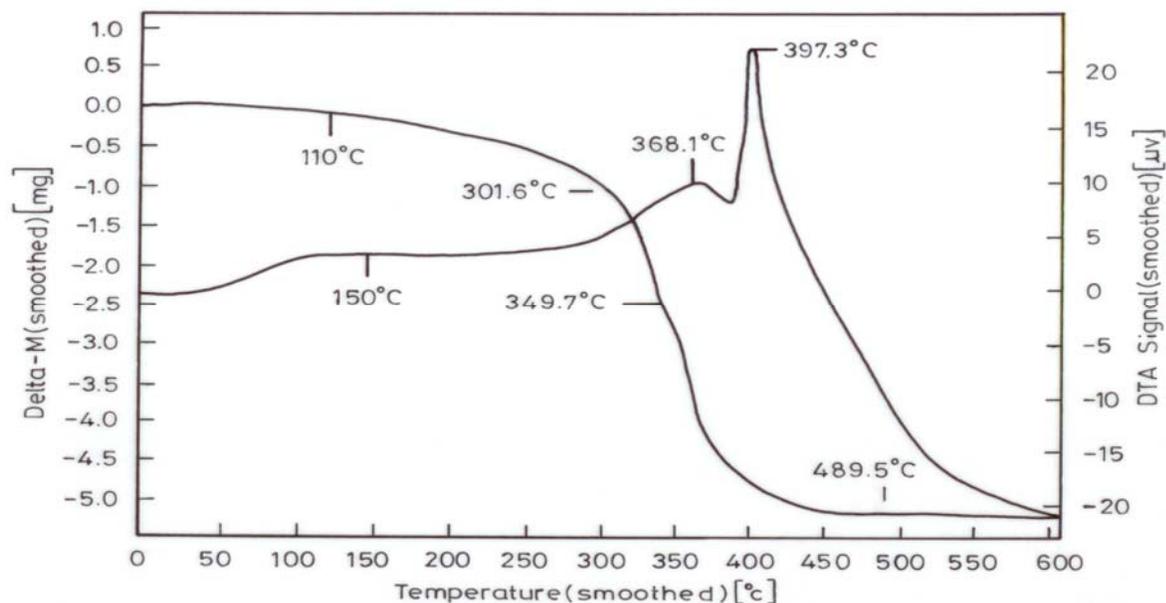


FIGURE 3. The TG-DTA of grafted *Hs*-g-poly(BA).

CONCLUSIONS

Grafting of poly (BA) (Pg: 66.80), poly (BA-co-AM) (Pg: 12.80), poly (BA-co-AA) (Pg: 22.95) and poly (BA-co-4VP) (Pg: 25) onto *H. sabdariffa* fiber in presence of ceric ion initiator yielded the modified and advanced graft copolymers. It is evident from the characterization and chemical evaluation through modern technique that grafting resulted in the morphological transformation that improved the physico-chemico-thermal resistance, hydrophobicity in *H. sabdariffa* fiber. The graft copolymers thus obtained are economic and judicious means to utilize the renewable waste biomass that after use as reinforcement in polymer matrix based bio-composites have improved the mechanical strength for various advanced applications. Use in aerospace, transportation, packaging are some of its application that could serve as pioneer for the advancement of science and technology.

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