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COMPARATIVE STUDY OF GRAFTING OF METHACRYLAMIDE ONTO SAGO STARCH USING CERIC AND PERSULFATE IONS AS FREE RADICAL INITIATORS

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ABSTRACT
Methacrylamide was grafted onto sago starch through a free- radical polymerization process using ceric and persulfate ion initiator systems. The effects of reaction variables, namely initiator concentration and monomer concentration, polymerization temperature and time, material-to-liquor ratio and the presence of alcohol were systematically investigated to determine optimal grafting conditions. The formation of graft copolymers was confirmed using gravimetric estimation method. Structural, thermal and morphological characteristics of the synthesized graft copolymers were examined by FTIR spectroscopy, X-ray diffraction, thermogravimetric analysis, differential scanning calorimetry and scanning electron microscopy. In addition, the biodegradability of graft copolymers was qualitatively assessed using two microorganisms viz. (a) Escherichia coli (a bacterium) and (b) Aspergillus niger (a fungus). The optimal conditions for graft copolymerization were established.

Keywords: *Methacrylamide, Biodegradation, Ceric ion, Graft copolymerization, Persulfate ion, Sago starch*

INTRODUCTION
Starch is a naturally occurring, renewable, inexpensive and fully biodegradable biopolymer. Despite these advantages, starch exhibits poor mechanical strength and dimensional stability, limiting its direct commercial use. Chemical modification, such as graft copolymerization, improves functional properties including elasticity, sorbancy, thermal resistance, microbial stability and ion exchange capacity while retaining biodegradability [1]. Starch based graft copolymers are widely applied as superabsorbents, drug carriers, soil stabilizers and flocculants [2].

Graft copolymerization of starch can be initiated using various free radical initiators such as azo-bis-isobutyronitrile (AIBN) [3], potassium persulfate (PPS) [4-5], ammonium persulfate (APS) [6], potassium permanganate [7], ceric ammonium nitrate (CAN) [5] etc.

Sago starch (SS), a low-cost carbohydrate polymer, has received comparatively less attention for grafting than starches such as maize, potato, cassava etc [8]. Methacrylamide (MAM) is a water soluble, relatively less toxic, polar and economical, but its grafting onto starch is underexplored [9]. The first report on the synthesis of starch-g-methacrylamide was probably the one published by Gruber et al. using Scott xanthate method [10]. Meltem has successfully grafted methacrylamide onto starch in an aqueous medium using benzoyl peroxide as the radical initiator [11]. As a matter of fact, no literature is available on grafting of methacrylamide (MAM) onto sago starch and is quite neglected, which prompted the present investigation.

In view of growing environmental concerns, it is essential to evaluate the biodegradability of newly developed starch- based graft copolymers to assess their environmental compatibility.

This work presents a comparative study on the grafting of methacrylamide onto sago starch using ceric and persulfate ion initiators, including the effect of various. The effect of grafting on thermal and morphological properties was examined, and biodegradability was qualitatively evaluated using *Escherichia coli* (ATCC 95922) and *Aspergillus niger* (ATCC 16414).

EXPERIMENTAL:

Materials:
Prior to use, sago starch was dried at 110°C for 10 h. Methacrylamide of analytical grade was used as received without additional purification. Ceric ammonium nitrate (CAN) being hygroscopic in nature was dried in air oven at 110°C for approximately 6 h, while potassium persulfate (PPS) was used as supplied. Stock solutions of CAN (0.1mol/L) were prepared in 1M nitric acid and PPS solutions of the same concentration were prepared in distilled water.

Preparation of Graft Copolymer:
Polymerization reactions were carried out in a beaker kept in a thermostat. A uniform slurry was prepared by dispersing dried sago starch (2 g) in 70 mL distilled water. A predetermined amount of initiator (ceric ammonium nitrate or potassium persulfate) was added to the slurry and allowed to interact with starch for 10 minutes to generate active free radical sites on starch backbone. Subsequently, a known quantity of methacrylamide was added and total volume of reaction was adjusted to 100 mL using distilled water. The reaction mixture was continuously stirred at 400 rpm throughout the process. After a desired reaction time period, the graft copolymer was filtered through preweighed Whatman filter paper no. 41 and repeatedly washed with warm water to remove homopolymethacrylamide. Finally, the resulting sago starch-g-poly(methacrylamide) (S-g-PMAM) was dried in a vacuum oven at 80°C for 24 h to remove excess of water and was weighed. Control reactions were performed under identical conditions using sago starch alone, without the addition of monomer.

Grafting Parameters:
The percentage grafting efficiency (%GE), percentage grafting (%G) and % Add-on were calculated using following formulae [1, 9]:

$\%GE = 100\ (W_2 - W_1)/W_3$

$\%\ G = 100\ (W_2 - W_1)/W_1$

$\%\ Add\text{-}on = 100\ (W_2 - W_1)/W_2$

Where, W_1, W_2 and W_3 are the weights of starch, graft copolymer and monomer respectively. Percentage Grafting (% PG) indicates the graft copolymer yield relative to the weight of the starch backbone, whereas grafting efficiency (%GE) represents the yield relative to the amount of monomer charged.

Infrared Spectral Analysis:
The IR spectra of sago starch and graft copolymers were recorded using KBr pellets on a Shimadzu FTIR- 4200 spectrophotometer over the frequency range 4000 – 600 cm^{-1}.

Thermogravimetric Analysis:
STA7300 Hitachi Thermal Analysis model was used to perform thermogravimetric analysis sago starch and graft copolymers. Samples were heated at a rate of 10°C/min from ambient temperature up to 500°C under a nitrogen atmosphere.

Differential Scanning Calorimetry:
13-15 mg of sample of sago starch and graft copolymer samples were subjected to DSC using a STA7300 Hitachi model up to 500°C with a heating rate of 10°C/min. under nitrogen purge.

Differential Thermal Analysis:
DTA measurements were performed on STA7300 Hitachi Thermal Analysis system under a nitrogen atmosphere by heating up to 500°C at a rate of 10°C/min.

X- Ray Diffraction Analysis:
Crystallinity of sago starch and graft copolymers was examined using Shimadzu- XR 6100 X- ray diffractometer over a 2θ range of 5° to 80°.

Scanning Electron Microscopy:
Surface morphology of graft copolymers and pure starch were studied using Zeiss Gemini 300 field emission scanning electron microscope. Film samples were mounted on specimen stabs and coated with thin film of gold by ion sputtering method. The micrographs were taken at magnification of 1000 and 500 using 15kV accelerating voltage.

Biodegradation Studies:
Biodegradability was qualitatively evaluated using a carbon-free minimal salt medium inoculated with *Escherichia coli* and *Aspergillus niger*. Graft copolymers were used as the sole carbon source, and microbial growth was assessed after 14 days using nutrient agar and Sabouraud's dextrose agar plates. Table 1 illustrates different types of controls and distribution of test samples. Media and sample controls served as negative

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controls, whereas culture controls acted as positive controls to confirm microbial viability and to assess degradation of the graft copolymers as the sole carbon source.

Table 1: Description of control and test setups used for biodegradation studies

Experimental Setup	Media	Culture	Sample
Media control	Present	Absent	Absent
Culture control	Present	Present	Absent
Sample control	Present	Absent	Present
Test	Present	Present	Present

RESULTS AND DISCUSSION:

Evidence for grafting:

Gravimetric estimations: Grafting of methacrylamide onto sago starch was confirmed by an increase in weight of residue relative to the control sample. This gain in weight of starch forms the basis to calculate %GE, % G and % Add-on.

Infrared spectral analysis:
Sago starch exhibited a broad absorption band at 3301.60 cm^{-1} corresponding to -OH stretching vibrations of anhydroglucose units (AGU) (Fig. 1 A). Similar bands were observed in graft copolymers (S-g-PMAM) prepared using PPS (3301.99 cm^{-1} (Fig. 1 B)) and CAN (3292.43 cm^{-1} (Fig. 1 C)), indicating the participation of -OH groups during grafting, with band broadening arising from overlapping O-H and N-H vibrations. Furthermore, the appearance of new absorption bands at 1634 cm^{-1} for S-g-PMAM (PPS) and 1632 cm^{-1} for S-g-PMAM (CAN), assigned to amide C=O stretching, provided clear evidence of methacrylamide grafting. Characteristic absorptions corresponding to aliphatic C-H stretching (~2900 cm^{-1}), -CH_2 scissoring (~1300 cm^{-1}), and C-O stretching (~ 1000 cm^{-1}) were present in both pure starch and grafted starch. This confirmed the grafting of MAM onto starch.

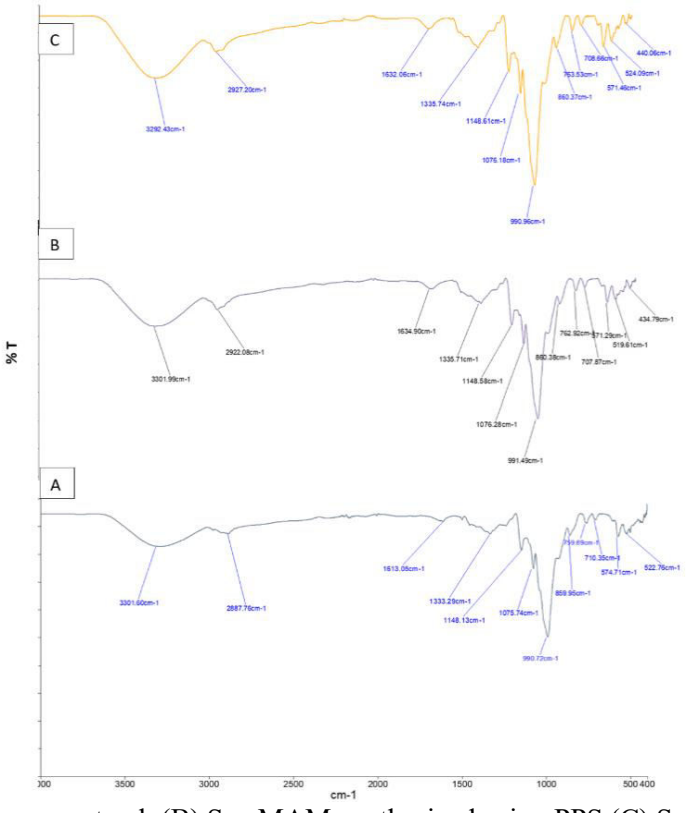

Fig. 1 IR spectra of (A) Pure sago starch (B) S-g-MAM synthesized using PPS (C) S-g-MAM synthesized using CAN at optimum conditions

Effect of initiator concentration:

Table 2 show that the percentage grafting (%G), percentage grafting efficiency (%GE) and percentage add-on (% Add-on) increase with increasing concentrations of potassium persulfate (PPS) and ceric ammonium nitrate (CAN) till it reached a maximum value. Beyond this concentration, a decline in these parameters is observed.

Table 2: Effect of initiator concentration on grafting of Methacrylamide (MAM) onto sago starch

PPS initiated				CAN initiated			
[PPS] (mol/L)	%G	%GE	%Add On	[CAN] (mol/L)	%G	%GE	%Add On
0.0005	0.90	1.80	0.89	0.0005	5.84	11.67	5.51
0.001	0.95	1.90	0.94	0.001	5.88	11.76	5.55
0.002	1.99	3.99	1.95	0.002	10.00	20.00	9.09
0.004	2.85	5.71	2.77	0.003	7.98	15.97	7.39
0.006	1.76	3.52	1.73	0.004	7.91	15.83	7.33
0.008	1.78	3.56	1.75	0.005	7.86	15.72	7.28
0.010	1.36	2.72	1.34	0.006	2.69	5.39	2.62

Experimental conditions: sago starch = 2 g; [MAM] = 0.1175 mol/L; reaction time = 180 min; temperature = 30 °C; total reaction volume made to 100 mL using distilled water.

The observed increase in grafting parameters with initiator concentration can be attributed to the enhanced formation of free radical sites on the starch backbone, which facilitates grafting of methacrylamide chains. However, at higher concentrations of PPS and CAN, excess initiator promotes side reaction such as premature termination of growing graft chains, initiation of homopolymerization and primary radical termination leading to partial oxidation of starch. In addition of non- availability of grafting sites on starch backbone limits further grafting. The maximum %GE achieved for MAM using PPS and CAN were 5.71% and 20% at 0.04 mol/L and 0.02 mol/L concentrations respectively. Comparative analysis clearly demonstrates that CAN is a more efficient initiator for grafting MAM onto sago starch, as it yields significantly higher grafting efficiency at a lower initiator concentration.

Effect of Monomer Concentration:

Table 3 exhibit the effect of monomer concentration on the grafting of methacrylamide (MAM) onto sago starch using PPS and CAN. Grafting efficiency (%GE) decreases with increasing monomer concentration from 0.117 mol/L to 0.705 mol/L for both initiators. For the PPS- initiated system, percentage grafting (%G) and percentage add- on (% add- on) initially increase and then decrease at higher monomer concentrations. In contrast, the CAN- initiated system exhibits a continuous decline in %G and % add- on with increasing monomer concentration. The optimum monomer concentration for maximum %G was found to be 0.470 mol/L for PPS and 0.117 mol/L for CAN.

Table 3: Effect of [MAM] on the grafting of sago starch in the presence of PPS and CAN as initiators

	PPS initiated			CAN initiated		
Monomer Conc. (mol/L)	%G	%GE	% Add On	%G	%GE	% Add On
0.117	2.85	5.71	2.77	10.00	20.00	9.09
0.235	2.90	2.90	2.82	7.18	7.18	6.69
0.352	2.92	1.95	2.84	7.11	4.74	6.64
0.470	3.94	1.97	3.79	6.98	3.49	6.52
0.587	2.40	0.96	2.34	6.92	2.77	6.48
0.705	1.30	0.43	1.28	5.69	1.89	5.38

Experimental conditions: sago starch = 2 g; reaction time = 180 min; temperature = 30 °C; total reaction volume made to 100 mL using distilled water. For PPS initiated reaction: [PPS] = 0.004 mol/L. For CAN initiated reaction: [CAN] = 0.002 mol/L

The decrease in %GE with increasing monomer concentration is attributed to the preferential involvement of MAM in homopolymerization rather than grafting onto starch macroradicals. Increased reaction medium

viscosity at higher monomer concentrations supports enhanced homopolymer formation. The initial rise in %G and % add-on, particularly in the PPS system, arises from greater monomer availability near starch backbone. At higher concentrations, extensive homopolymerization and formation of lumpy polymer aggregates limits monomer diffusion toward relatively immobile starch macroradicals, resulting in reduced grafting. These diffusion and competition effects explain the existence of an optimum monomer concentration for effective grafting.

Effect of Polymerization Temperature:

In the present work, graft copolymerization was carried out on granular sago starch; therefore, the polymerization temperature was maintained below the onset of starch gelatinization temperature (60°C). The effect of temperature on grafting using potassium persulfate (PPS) and ceric ammonium nitrate (CAN) in the range of 25°C to 50°C is summarized in Table 4. An increase in %G, %GE and % add-on was observed with increasing temperature up to an optimum value of 30°C, beyond which a decline in these parameters occurred. The initial enhancement in grafting may be attributed to increased monomer mobility, improved diffusion toward starch macroradicals, swelling of starch granules leading to lager surface area and accelerated chain propagation at elevated temperatures.

Table 4: Effect of polymerization temperature on the grafting of MAM onto sago starch in presence of PPS and CAN as initiators

	PPS initiated			**CAN initiated**		
Temperature (°C)	%G	%GE	% Add On	%G	%GE	% Add On
25	0.36	0.18	0.36	6.54	13.09	5.67
30	3.94	1.97	3.79	10.00	20.00	9.09
35	2.31	1.16	2.26	5.39	10.79	5.12
40	1.96	0.98	1.93	5.18	10.37	4.93
45	1.93	0.97	1.89	4.32	8.65	4.15
50	1.81	0.90	1.77	3.78	7.57	3.65

Experimental conditions: sago starch = 2 g; reaction time = 180 min; total reaction volume made to 100 mL using distilled water.

For PPS initiated reaction: [PPS] = 0.004 mol/L; [MAM] = 0.470 mol/L.

For CAN initiated reaction: [CAN] = 0.002 mol/L; [MAM] = 0.117 mol/L.

The decrease in grafting parameters beyond 30°C temperature is possibly the result of partial dissolution of starch causing change in amylose to amylopectin ratio, increased starch oxidation, and enhanced chain transfer reactions unfavourable to grafting. Thus, 30°C was established as the optimum temperature for grafting MAM using both initiators.

Effect of Polymerization Time:

The effect of polymerization time on grafting of MAM onto sago starch using PPS and CAN is depicted in Table 5. Low graft yields at the initial stage indicate an induction period of 60 min. Following this period, %G, %GE and % add-on increase, reaching a maximum at 90 min for PPS- initiated grafting and 120 min CAN initiated grafting, after which the values level off. This behaviour can be attributed to progressive depletion of monomer and initiator during the reaction. From a practical standpoint, a reaction time of 60 min was found to be sufficient for the synthesis of graft copolymers using both initiators.

Table 5: Effect of polymerization time on the grafting of MAM onto sago starch in presence of PPS and CAN as initiators

	PPS initiated			**CAN initiated**		
Time (minutes)	%G	%GE	% Add On	%G	%GE	% Add On
30	5.51	2.76	5.23	4.03	8.06	3.87
60	5.73	2.86	5.42	7.42	14.83	6.90
90	4.39	2.19	4.20	7.89	15.77	7.31
120	4.09	2.05	3.93	10.47	20.94	9.48

150	3.93	1.96	3.78	10.56	21.12	9.55
180	3.94	1.97	3.79	10.00	20.00	9.09

Experimental conditions: sago starch = 2 g; temperature = 30 °C; total reaction volume made to100 mL using distilled water.

For PPS initiated reaction: [PPS] = 0.004 mol/L; [MAM] = 0.470 mol/L.

For CAN initiated reaction: [CAN] = 0.002 mol/L; [MAM] = 0.117 mol/L.

Effect of material-to-liquor ratio:
Table 6 shows the effect of material-to-liquor ratio on the grafting of MAM onto sago starch using PPS and CAN. The material-to-liquor was varied by decreasing the solvent volume in the reaction mixture. A decrease in volume of liquor resulted in enhanced grafting, which may be due to an increased frequency of effective collisions between starch macroradicals and monomer molecules. Maximum % GE was obtained at material-to-liquor ratios of 1:3.33 for PPS and 1:20 for CAN. A further decrease in solvent led to a decline in grafting parameters, likely due to restricted mobility of monomer molecules in the reaction medium.

Table 6. Effect of material – to – liquor ratio on the grafting of MAM onto sago starch in presence of PPS and CAN as initiators

PPS initiated					CAN initiated				
Material to liquor ratio	Volume of liquor (mL)	%G	%GE	%Add On	Material to liquor ratio	Volume of liquor (mL)	%G	%GE	%Add On
1:16.67	100	3.94	1.97	3.79	1:33.33	100	10.47	20.94	9.48
1:13.33	80	3.99	1.99	3.84	1:26.66	80	11.22	22.44	10.09
1:10	60	4.01	2.01	3.85	1:20	60	12.26	24.53	10.92
1:6.67	40	4.08	2.04	3.92	1:16.66	50	8.21	16.43	7.59
1:3.33	20	4.43	2.22	4.24	1:13.33	40	5.27	10.54	5.01
-	-	-	-	-	1:6.66	20	4.90	9.81	4.68

Experimental conditions: sago starch = 2 g; temperature = 30 °C.

For PPS initiated reaction: [PPS] = 0.004 mol/L; [MAM] = 0.470 mol/L; reaction time = 60 min. For CAN initiated reaction: [CAN] = 0.002 mol/L; [MAM] = 0.117 mol/L; reaction time = 120 min.

Note: "-" indicates no corresponding experimental point

Effect of Solvent:
Since methacrylamide (MAM) exhibits limited solubility in alcohols, the effect of methanol, ethanol and isopropanol on grafting parameters (Table 7) was examined. Alcohol- water mixture was prepared in 1:3 ratio, maintaining a total reaction volume of 100 mL. The presence of alcohol resulted in decline in % G, %GE and % add-on. This reduction is attributed to changes in the polarity and dielectric constant of the reaction medium. With increasing alkyl chain length from methanol to isopropanol, the dielectric constant decreases due to reduced miscibility with water, despite the constant size of the polar hydroxyl group. These observations indicate that graft copolymerization of MAM onto sago starch is governed primarily by the polarity and dielectric constant of the medium rather than the intrinsic polarity of the monomer. Consequently, water was found to be the most suitable solvent for effective grafting.

Table 7: Effect of solvent on the grafting of MAM onto sago starch in presence of PPS and CAN as initiators

Solvent	Dielectric constant of medium	PPS initiated			CAN initiated		
		%G	%GE	% Add On	%G	%GE	% Add On
Water	81	5.74	2.87	5.43	10.47	20.94	9.48
Water: Methanol	68.9	1.88	0.94	1.85	8.29	16.58	7.65
Water: Ethanol	66.8	1.63	0.81	1.59	7.33	14.67	6.83
Water: Isopropanol	65.2	1.31	0.66	1.29	5.74	11.49	5.43

Experimental conditions: sago starch = 2 g; temperature = 30 °C; total volume = 100 mL. Water: Alcohol mixtures = 3:1 (v/v).

For PPS system: [PPS] = 0.004 mol/L; [MAM] = 0.470 mol/L.

For CAN system: [CAN] = 0.002 mol/L; [MAM] = 0.117 mol/L.

Characterization of Grafted Starch:

Thermogravimetric Analysis:

Figures 1-3 shows thermal behaviour of pure sago starch, S-g-PMAM using PPS and CAN as initiators. Both pure starch as well as graft copolymers exhibited two step TG/DTG profiles. The initial weight loss up to 256°C is attributed to the removal of physically bound moisture. Pure sago starch showed a major weight loss of 75.38% at a T_{max} (maximum decomposition temperature) of 309°C, with a total loss of 85% at 583°C. In comparison, S-g-PMAM synthesized using PPS displayed a lower total weight loss (82%) at the same T_{max}, indicating enhanced thermal stability. S-g-PMAM synthesized using CAN exhibited a major weight loss of 76% at a slightly higher T_{max} of 315°C, with an overall degradation pattern comparable to starch. These results confirm that grafting with methacrylamide improves the thermal stability of sago starch. Detailed decomposition stages for pure starch and graft copolymers are summarized in Table 8.

Table 8: Thermogravimetric data of starch and its graft copolymers

Polymer	No. of Stages	Temperature Range	T max	% Weight Loss	% Residue at 583°C
Starch	1	37- 256	91	9.63	
	2	256- 583	309	75.36	15.01
S-g-MAM (CAN)	1	36- 252	76	9.33	
	2	252- 484	315	76.03	14.64
S-g-MAM (PPS)	1	37- 258	95	9.85	
	2	258- 482	309	72.68	17.47

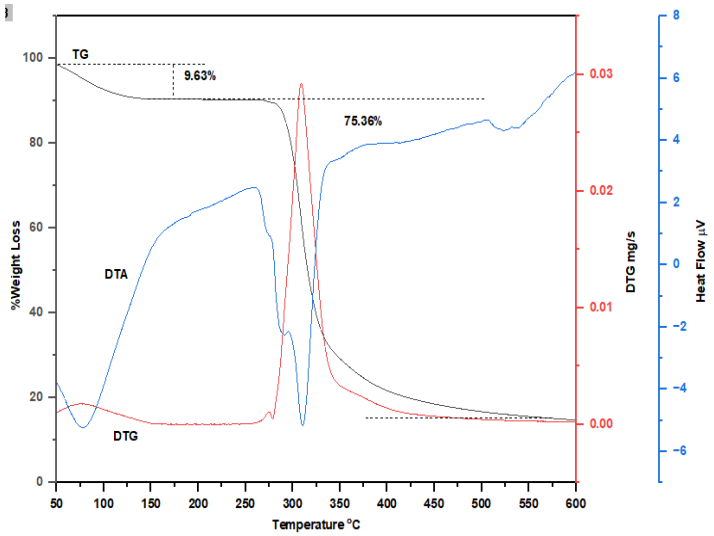

Fig. 1 TGA, DTG and DTA for pure sago starch

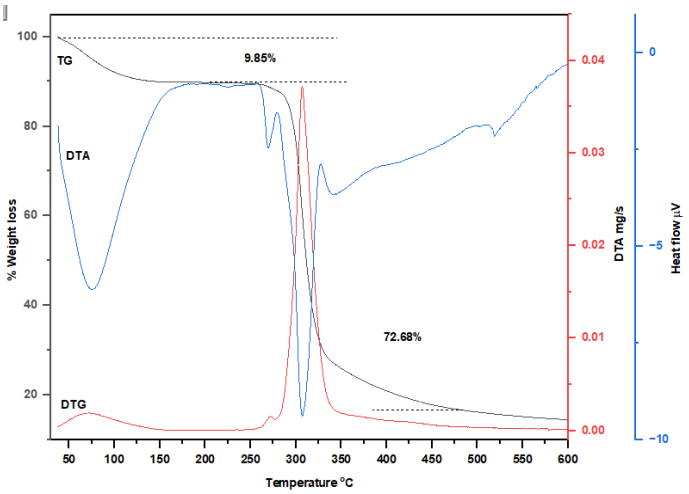

Fig. 2 TGA, DTG and DTA for S-g-MAM (PPS)

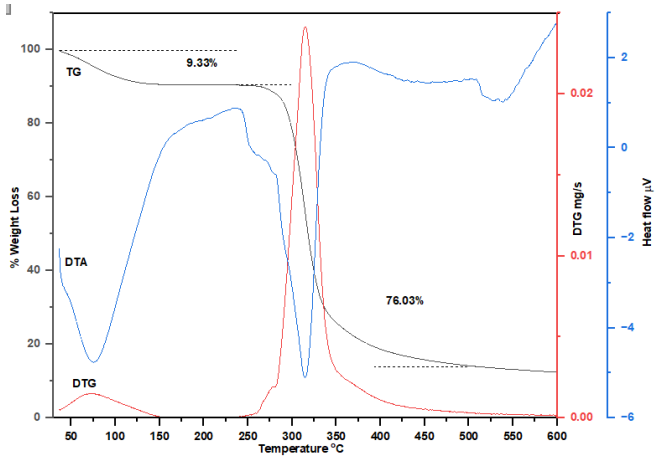

Fig. 3 TGA, DTG and DTA for S-g-MAM (CAN)

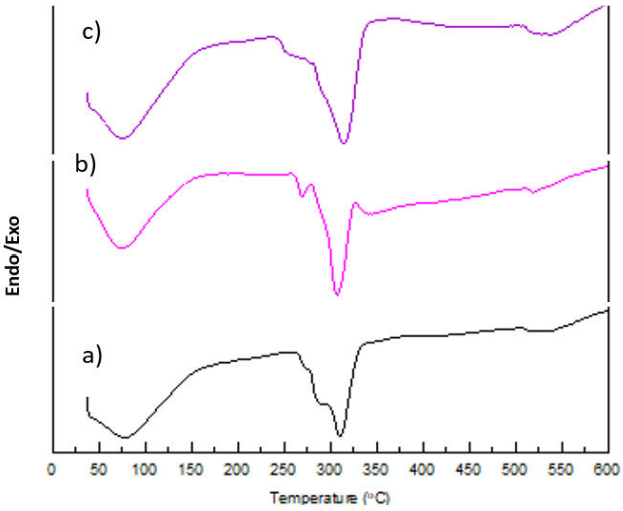

Fig. 4 DSC curve of (a) Pure Sago Starch (b) S- g-MAM (PPS) (c) S-g-MAM (CAN)

Differential Scanning Calorimetry:

The DSC curve for pure and grafted sago starch is illustrated in Fig. 4., with thermal parameters tabulated in Table 9. The first endothermic transition in each DSC curve corresponds to the glass transition temperature (Tg). Pure sago starch containing 11-15% moisture exhibited a Tg at 80°C, consistent with the plasticizing effect of water reported in the literature. Grafted starch samples showed lower Tg values, indicating that grafted

methacrylamide chains act as internal plasticizers [9]. The enthalpy change (ΔH) for pure starch was 46 J.g-1, while lower ΔH values for graft copolymers suggest reduced crystallinity upon grafting.

Table 9: DSC data for insoluble sago starch and its graft copolymers

Polymer	Endotherm/°C				ΔH/J. g^{-1}
	Onset	Midpoint	Peak 1	Peak 2	
Starch	43	80	286	310	46
S-g-MAM (CAN)	41	76	254	315	36
S-g-MAM (PPS)	40	75	271	307	34

Differential Thermal Analysis:

Figures 1-3 exhibit the DTA thermograms of pure sago starch and its graft copolymers, with corresponding data summarized in Table 10. The first endothermic peak in each DTA curve corresponds to the glass transition temperature (T_g) and aligns closely with the T_g values obtained from DSC (Table 9). Additional endothermic peaks observed in the DTA curves may be associated with oxidative degradation of the polymers [12]. These results confirm the consistency of thermal transitions measured by DSC and DTA for both pure sago starch and graft copolymers.

Table 10: DTA data for insoluble sago starch and its graft copolymers

Polymer	Peak Temperatures/°C			
Starch	78	292	310	530
S-g-MAM (CAN)	74	-	314	534
S-g-MAM (PPS)	76	307	341	518

X- Ray Diffraction Analysis:

Starch granules are semi- crystalline, with clustered amylopectin branches contributing to crystallinity, which accounts for approximately 20- 25% of the starch granule volume [13]. The XRD patter of pure sago starch (Fig.5 a) displays three diffraction peaks between 2θ = 15- 25°, confirming its crystalline nature. Upon grafting with methacrylamide, these peaks become less intense and slightly broadened (Fig.5 b & c), indicating a reduction in crystallinity. This suggests that grafting involves not only the amorphous regions but also affects the crystalline domains of the starch granules.

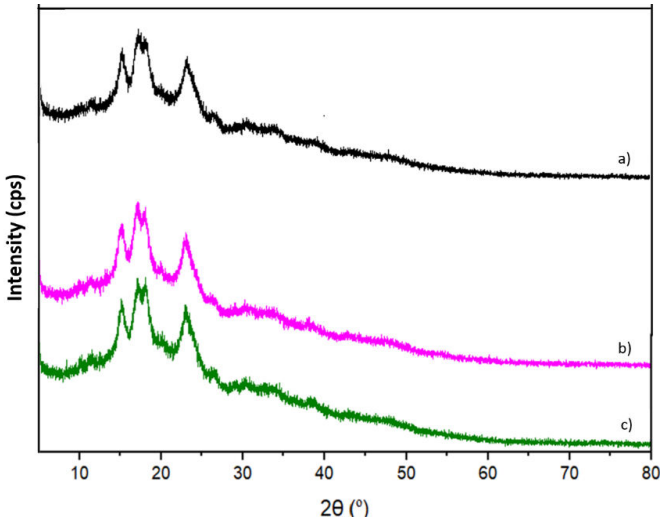

Fig. 5 Wide angle X- ray diffraction pattern of (a) Pure Sago Starch (b) S-g-MAM (PPS) (c) S-g-MAM (CAN)

Scanning Electron Microscopy:

The effect of grafting on the surface morphology of starch was investigated by SEM. Micrographs of pure sago starch ×1000 magnification (Fig. 6 a) reveal irregular, oval- shaped granules with smooth surfaces. In grafted starch (Fig. 6 b & c), the granules exhibit increased surface roughness and irregularity, with many appearing connected by a thin coating of grafted PMAM chains. These observations indicate that the water- soluble methacrylamide forms a surface layer on the starch granules while preserving their overall structural integrity.

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Biodegradability of sago starch.

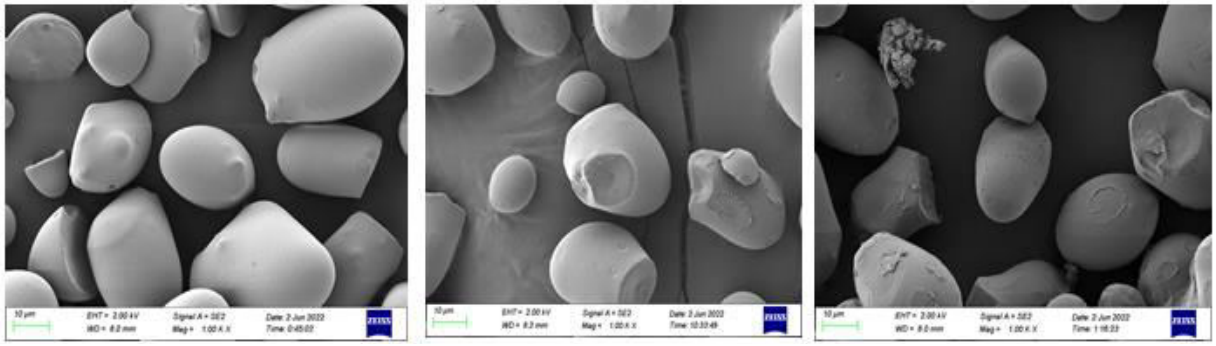

Fig. 6 SEM of (a) Pure Sago Starch (b) S-g-MAM (PPS) (c) S-g-MAM (CAN)

Qualitative screening of biodegradation of graft copolymers:

The biodegradability of the graft copolymers was evaluated by measuring their ability to serve as the sole carbon source for microbial growth. The increase in microbial growth was observed in the terms of colonies. Significant growth of both *E. coli* and *A. niger* was observed on grafted starch (Fig. 7 a & b), confirming that grafting with methacrylamide does not hinder the biodegradability of sago starch.

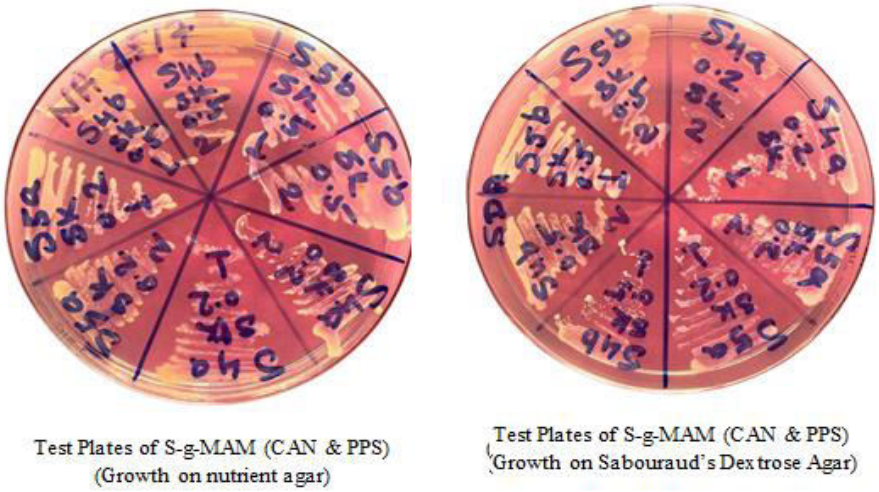

Fig. 7. Biodegradation study of starch graft copolymers using Nutrient Agar and Sabouraud's Dextrose Agar

CONCLUSIONS

Methacrylamide was successfully grafted onto sago starch using both ceric and persulfate ion initiators. Optimum reaction conditions determined in this study are summarized below:

Reaction Parameters	MAM (CAN)	MAM (PPS)
Initiator Concentration (mol/L)	0.002	0.004
Monomer Concentration (mol/L)	0.117	0.470
Polymerization Temperature (°C)	30	30
Polymerization Time (Minutes)	120	60
Material-to-liquor ratio	1:20	1:3.33

Ceric ions proved to be more efficient, yielding higher grafting efficiency under milder conditions. The grafting process fulfils several green chemistry criteria, including the use of renewable raw materials, aqueous reaction medium, ambient temperature, and absence of organic solvents. Grafting improved the thermal and morphological properties of starch while retaining its biodegradable nature, making these materials promising for environmentally benign applications.

REFERENCES

1) Athawale V D, Rathi S C (1999) Graft Polymerization: Starch as a model substrate. JMS - Reviews in Macromol. Chem. and Phy. 39(3): 445 - 480

2) Kavlani N, Sharma V, Singh L (2012) Various techniques for the modification of starch & the applications of its derivatives. Intern. Res. J. Pharma. 3(5): 25 – 31

3) Meltem C, Mehmet S (2002) Synthesis and characterization of starch- poly (methyl methacrylate) graft copolymers. J. App. Polym. Sci. 86 (1): 53 – 57

4) Qudsieh I Y M, Razi A F, Muyibi S A, Yunus W Z et al. (2004) Preparation and characterization of poly (methyl methacrylate) grafted Sago Starch using potassium persulfate as redox initiator. J. Appl. Polym. Sci. 94(5): 1891 – 1897

5) Razi A F, Qudsieh I Y M, Yunus W Z, Ahmad M, Rahman M Z, (2001) Graft copolymerization of methyl methacrylate onto Sago Starch using ceric ammonium nitrate and potassium persulfate as redox initiator systems. J. Appl. Polym. Sci. 82(6): 1375 - 1381

6) Abdulganiyu U, Sanagi M M, Salisu A, Ibrahim W A W, Karim K J A, Keyon S A (2016) Preparation and characterization of starch grafted with methacrylamide usimg ammonium persulphate. Mat. Lett. 185 (15): 173- 176

7) Mostafa K M (1995) Graft polymerization of acrylic acid onto starch using potassium permanganate acid (redox system). J. App. Polym. Sci. 56 (2): 263-269

8) Lele V, Kumari S, Niju H (2018) Syntheses, Characterization and Applications of Graft Copolymers of Sago Starch – A Review. Starch/ Stärke 70(7): 1700133

9) Athawale, V. D., Rathi S. C., Lele, V. (1998) Graft copolymerization on to starch. I. Grafting of Methacrylamide using ceric ammonium nitrate as an initiator. *Eur. Polym. J.*, 34 (2), 159 – 161

10) Gruber E., Alloush S., John K., Schruz J. (1972) Structure and properties of grafted potato starch. I. Morphology and solubility. *Starch – Stärke*, 24, 251 – 258

11) Celik M. (2006) Preparation and characterization of starch-g-polymethacrylamide copolymers. *J. polym. Res*., 13, 427-432

12) Athawale V D, Lele V V (2000) Thermal studies on granular maize starch and its graft copolymers with vinyl monomers. Starch – Stärke 52: 205- 213

13) Swinkles J J M (1985) Sources of starch, its chemistry and physics. Int. G. M. A. van Beynum & J. A. Roels (Eds.), Starch conversion technology, New York: Marcel Dekker