

# UV-Radiation Induced Graft Copolymerization of Methyl Methacrylate onto Sodium Salt of Partially Carboxymethylated Psyllium

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**ABSTRACT:** UV-radiation induced grafting of methyl methacrylate onto sodium salt of partially carboxymethylated psyllium has been carried out using ceric ammonium nitrate as a photoinitiator in an aqueous medium. The reaction variables including concentrations of initiator, nitric acid, monomer, and amount of the backbone as well as time and temperature have been varied for establishing the optimized reaction conditions for grafting. The influence of these reaction conditions on the grafting yields has been discussed. The overall activation energy of grafting

has been calculated. The infrared spectroscopic, thermogravimetric analysis, and scanning electron microscopic techniques have been used for the characterization of graft copolymers. © 2011 Wiley Periodicals, Inc. *J Appl Polym Sci* 124: 4945–4952, 2012

**Key words:** graft copolymers; polysaccharides; sodium salt of partially carboxymethylated psyllium (Na-PCMPsy); UV-radiation; optimum reaction condition; activation energy ( $E_a$ )

## INTRODUCTION

Psyllium (Psy) is a natural plant polysaccharide obtained from *Plantago ovata* and its mucilage is composed of neutral arabinoxylan (arabinose 22.6% and xylose 74.6%). Psy forms mucilage in water, which is composed of arabinoxylan having straight xylose chain with arabinose branches at 2 or 3 position. Psy mucilage is an anionic natural polysaccharide consisting of pentosan and uronic acid obtained from the seeds plantago Psy.<sup>1–6</sup> Psy is a natural biodegradable polymer that decomposes before it melts and hence can not be easily fabricated. To overcome this difficulty, the carboxymethylated derivative of it, that is, sodium salt of partially carboxymethylated psyllium (Na-PCMPsy) can be modified by grafting of different vinyl monomers onto it using radiation methods. The introduction of carboxymethyl groups in the Psy molecules will lead to the combined influence of the following factors:

1. carboxymethyl groups increase the swellability of Psy, thereby facilitating the diffusion of monomer and initiator and
2. the ionization of carboxymethyl groups along the Psy chains introduces negative charges,

which attract ceric ions to the backbone, leading to the backbone that will be available for the monomer and thereby increasing the reactivity of Psy.<sup>7–10</sup>

Graft copolymerization is a well known technique to impart a new property or enhance the existing properties in the parent polymer with minimum degradation of the original properties. The nature of the changes in the properties depends on the type of monomer being grafted, the percentage of grafting, the method of grafting, and the distribution of the grafted chain throughout the parent polymer. The UV-radiation technique is well known for initiation of graft copolymerization and is among the most promising and practical method because of its simplicity. The graft copolymerization by low energy UV-radiation possesses certain advantages like less degradation of the backbone polymer and control over the reaction.<sup>7,11,12</sup> Moreover, the UV-radiation equipment is more cost effective than the high energy radiation equipment. Therefore, a photoinitiator is generally used to promote the graft copolymer reaction. Upon absorption of energy, the photoinitiator is decomposed into reactive radicals or transfer its energy to other molecules in the system thereby facilitating the graft copolymer reaction.<sup>13–20</sup>

This article presents the results of the evaluation of the optimized reaction conditions of grafting of methyl methacrylate (MMA) onto Na-PCMPsy by using the UV-radiation method and characterization

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of the graft copolymer has been ascertain by using different techniques.

## EXPERIMENTAL

### Materials

Psy was supplied by Gujarat Sat-Isabgol Factory (Unjha, Gujarat, India). Na-PCMPsy was prepared from Psy and the purification and the measurement of degree of substitution were followed as discussed earlier.<sup>21,22</sup> The  $\overline{DS}$  was found to be 0.13. MMA (Fluka) was washed with 2% NaOH aqueous solution, followed by washing with distilled water, then dried over anhydrous calcium chloride, and finally purified with usual procedure. Ceric ammonium nitrate (CAN; Analar grade, Qualigens, Glaxo India) was used as photoinitiator. All other reagents and solvents used were of reagent grade. Nitrogen gas was purified by passing through freshly prepared alkaline pyrogallol solution. The double distilled water was used for the preparation of solutions and in polymerization reactions.

### UV-graft copolymerization

The UV-radiation grafting reactions were carried out in 500 mL glass vessel. A weighed amount of Na-PCMPsy (0.2–1.0 g) was dissolved in 146.32 mL of conductivity water in the photochemical reactor and the solution was stirred with continuous bubbling of a slow stream of nitrogen gas for an hour at 55°C and thereafter 20 min at room temperature. Freshly prepared CAN solution (2.5 mL;  $0.5 \times 10^{-3}$  to  $10.0 \times 10^{-3}$  mol L<sup>-1</sup>) in nitric acid (Nil-0.5 mol L<sup>-1</sup>) was added to the photochemical reactor and contents were also then flushed with purified nitrogen gas and freshly distilled MMA (0.037–0.370 mol L<sup>-1</sup>) was added and put the reaction vessel in the UV-reactor cabinet. Thus, the solution was irradiated with continuous stirring for different time intervals (0.5–10 h) in the temperature range 20–45°C. After completion of the grafting reaction, the irradiated sample solution was removed carefully and the crude graft product was isolated by centrifugation. It was then purified by washing with dilute nitric acid and 90% methanol and finally washed with pure methanol. Thus, the crude copolymer sample of Na-PCMPsy-g-Polymethylmethacrylate (PMMA) obtained was dried under vacuum at 40°C.

The grafting yields, namely, percentage of grafting (%G) and percentage of grafting efficiency (%GE) and rate of polymerization ( $R_p$ ) was evaluated by using expressions as mentioned earlier.<sup>7,23</sup>

### UV-radiation source

The UV-radiation source was purchased from Scientific Aids and Instruments Corporation (SAIC, Ma-

dras, India). The UV-reactor was then assembled with immersion well containing 125 W medium pressure mercury lamps. The medium pressure lamps have much more intense arcs and radiate predominantly 254, 365, or 425 nm radiation.

### IR spectra

Infrared (IR) spectra of Psy, Na-PCMPsy ( $\overline{DS} = 0.13$ ), Na-PCMPsy-g-PMMA, and PMMA were taken in KBr pellet using Nicolet Impact 400 D Fourier transform infrared spectrophotometer.

### Thermogravimetric analysis

The thermal behavior of Psy, Na-PCMPsy ( $\overline{DS} = 0.13$ ), Na-PCMPsy-g-PMMA, and PMMA has been examined in an inert atmosphere at a heating rate of 10°C min<sup>-1</sup>. with the help of the Mettler Toledo Star SW 7.01 thermogravimetric analyzer.

### Scanning electron microscopy

Model ESEM TMP + EDAX, Philips make has been used to obtain the micrographs of Psy, Na-PCMPsy ( $\overline{DS} = 0.13$ ), and Na-PCMPsy-g-PMMA graft copolymer samples.

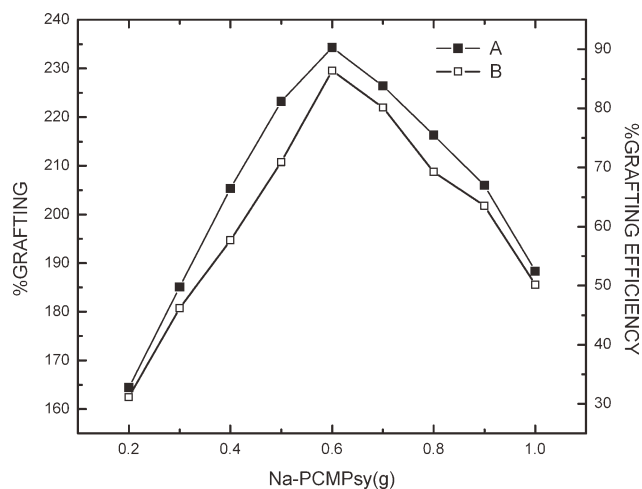
## RESULTS AND DISCUSSION

### Determination of optimum reaction conditions

The reaction variables including concentrations of initiator (CAN), nitric acid, monomer (MMA) as well as time and temperature have been varied for establishing the optimized reaction conditions for grafting.

#### Effect of substrate concentration

The influence of varying amount of Na-PCMPsy on %G and %GE is shown in Figure 1. It can be observed from this figure that, the value of %G and %GE is found to be increased with increase in Na-PCMPsy concentration and the maximum value is reached at 0.6 g of Na-PCMPsy concentration in the case of grafting of MMA. Beyond this optimum concentration of Na-PCMPsy, %G and %GE values are found to be decreased with further increase in Na-PCMPsy concentration. The results can be explained on the basis of the fact that the greater the amount of Na-PCMPsy for a given amount of monomer, the greater is the amount of complex formed during the course of irradiation and consequently the higher is the number of active sites generated on the polymeric substrate during the subsequent decomposition of the complex. As a result of this, the grafting yields are found to be increased with the increase in

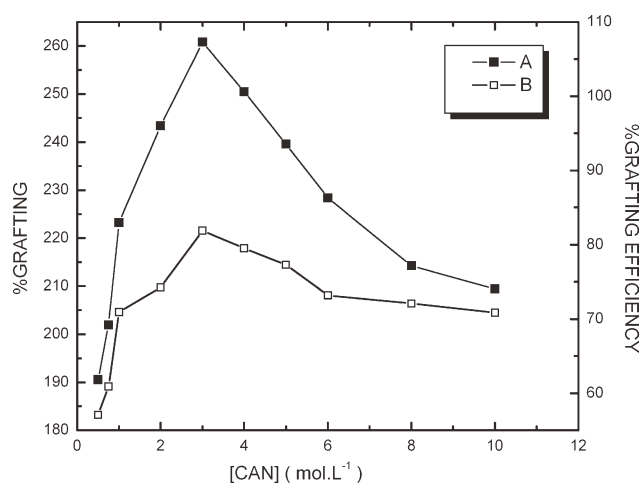


**Figure 1** Effect of amount of Na-PCMPsy on (A) %G or (B) %GE.

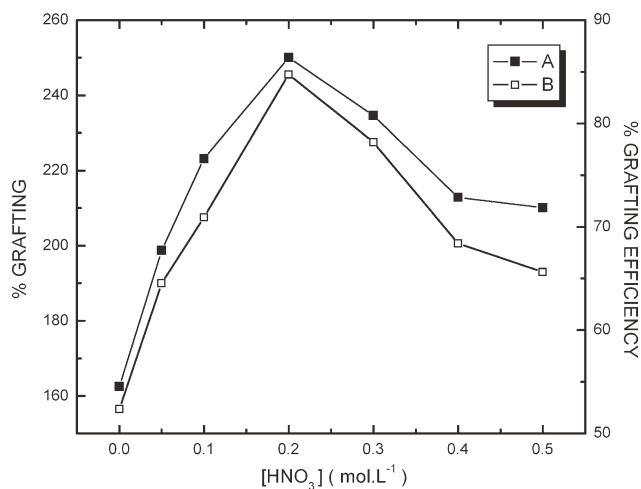
the backbone concentration. The observed decrease in %G and %GE with further increase in the amount of Na-PCMPsy indicates the formation of homopolymer (PMMA).<sup>7,11,12,24</sup>

#### Effect of photoinitiator concentration

The effect of the photoinitiator concentration of  $Ce^{+4}$  ions on the grafting yields is shown in Figure 2. It can be seen from this figure that with increasing ceric-ion concentration, %G and %GE are found to increase and reaches maximum value of 260.77% at  $[Ce^{+4}] = 3.0 \times 10^{-3} \text{ mol L}^{-1}$ ; however, with further increase in  $[Ce^{+4}]$ , the values of %G and %GE are decreases. Thus, the observed increase in %G within the photoinitiator concentration (CAN) range  $0.5 \times 10^{-3}$  to  $3.0 \times 10^{-3} \text{ mol L}^{-1}$  may be due to the fact that within these concentration range, the complex formation between the  $-OH$  groups and carboxylate anion of Na-PCMPsy and  $Ce^{+4}$  is facilitated and the



**Figure 2** Effect of of CAN concentration on (A) %G or (B) %GE.

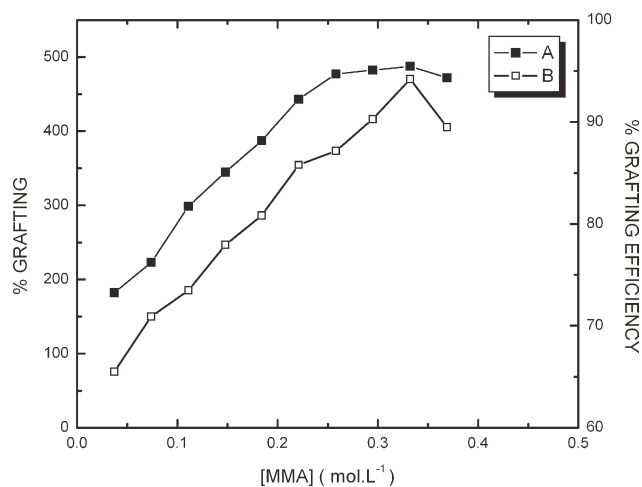


**Figure 3** Effect of nitric acid concentration on (A) %G or (B) %GE.

photodecomposition of the complex leads to the formation of more active sites. Thus, this activation along the backbone that has taken place is immediately followed by the graft copolymerization of MMA onto the Na-PCMPsy. The observed decrease in the grafting yields (%G and %GE) at higher photoinitiator concentration, that is, beyond  $[Ce^{+4}] = 3.0 \times 10^{-3} \text{ mol L}^{-1}$ , may be due to the efficiency of  $Ce^{+4}$  to take part in the termination of growing grafted chains. Furthermore, homopolymer (PMMA) formation at higher initiator concentration, which competes with the grafting reaction for the available monomer (MMA) could also lead to a decrease in %G and %GE. Similar results are also reported in the literature.<sup>25-27</sup>

#### Effect of acid concentration

The values of the grafting yields (%G and %GE) for the photo-grafting of MMA onto Na-PCMPsy at different nitric acid concentrations are depicted in Figure 3. It can be observed from this figure that there exists an optimum concentration of nitric acid at  $0.20 \text{ mol L}^{-1}$ , which affords maximum percentage of grafting and grafting efficiency. The values of %G and %GE are found to be decreased beyond the respective optimum concentration of nitric acid. It is interesting to note that even at zero concentration of nitric acid, an appreciable value of percentage of grafting is observed this may be due to the possibility that, even in absence of acid, in an aqueous medium Na-PCMPsy ionizes fully to a greater extent, which facilitates the diffusion of monomer and photoinitiator leading to appreciable values of grafting. The values of %G and %GE increase in the beginning with increase in nitric acid concentration may be due to decrease termination rate of the growing polymer chain, or an increase in the initiation rate.



**Figure 4** Effect of monomer concentration (MMA) on (A) %G or (B) %GE.

However, beyond the optimum concentration of acid, the decrease in %G and %GE could be attributed to a corresponding reduction in ceric-Na-PCMPsy complex formation and an increase in termination rates.<sup>28,29</sup>

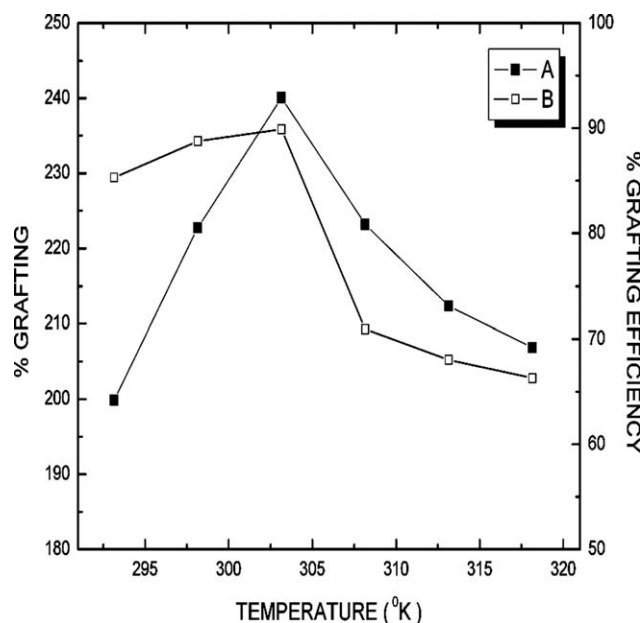
#### Effect of monomer concentration

It is evident from Figure 4 that %G increases steadily up to  $0.332 \text{ mol L}^{-1}$  and upon grafting of MMA and beyond this concentration, it decrease slowly. The continues increase in %G, upon increasing the monomer concentration (MMA), as observed in Figure 4, could be attributed to variety of reasons. First, complexation of Na-PCMPsy increase with monomer, which is required for monomer reactivity, that would be favored with increase in monomer concentration. The second reason might be due to the gel effect,<sup>30</sup> that is, increase in viscosity of the medium owing to the solubility of the homopolymer (PMMA) in its own monomer (MMA), which could be more pronounced with increase of monomer concentration. This causes hindrance in termination, particularly by coupling of growing polymer chains. However, the other steps in the photo-graft copolymerization process, namely initiation, propagation, and radical chain processes are not going to be affected to the same extent by increasing viscosity because the mobility of the polymer chains is restricted by the backbone structure. Besides this, the gel effect also causes swelling of Na-PCMPsy, thus facilitating the diffusion of the monomer (MMA) to the growing grafted chains and at the active sites on the backbone, there by enhancing grafting. Beyond optimum concentration, the values of %G decreases; this may be due to after optimum concentration lesser availability of monomer (MMA) for photo-grafting and most of it may be used for homopoly-

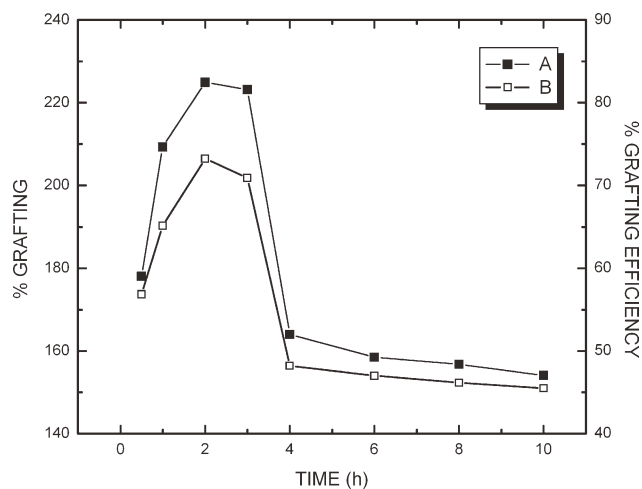
merization. The increase in the value of %GE by increasing the monomer concentration could be attributed to the greater availability of grafting sites to monomer. However, the decreasing trend of %GE as shown in Figure 4 is probably due to the competition between the homopolymerization and grafting, where the former prevails over the latter at higher monomer concentration.

#### Effect of temperature

Figure 5 shows the influence of temperature on %G and %GE. It can be observed from the figure that the values of the grafting yields (%G and %GE) are found to be increased from 20 to 30°C but decreases with further increases temperature in the grafting of MMA onto Na-PCMPsy. Thus, the observed increase in %G and %GE with temperature could be interpreted in terms of favorable influence of temperature on: (i) the faster photolysis of the Na-PCMPsy chains, (ii) the swellability of Na-PCMPsy, (iii) solubility of monomer molecules, (iv) diffusion rate of monomer from the aqueous phase to the backbone, and (v) the rates of initiation and propagation of photografting. The observed decrease in grafting efficiency with rising temperature may also be attributed to the solubility of monomer in the aqueous phase at higher temperature, thus increasing the possibility of the monomer to contact Ce (IV), hence more Ce(IV) will be available to initiate the homopolymerization. In addition, at higher temperatures, various hydrogen abstraction and chain transfer reactions might be accelerated, which may lead to decrease in %G and %GE.



**Figure 5** Effect of temperature on (A) %G or (B) %GE.



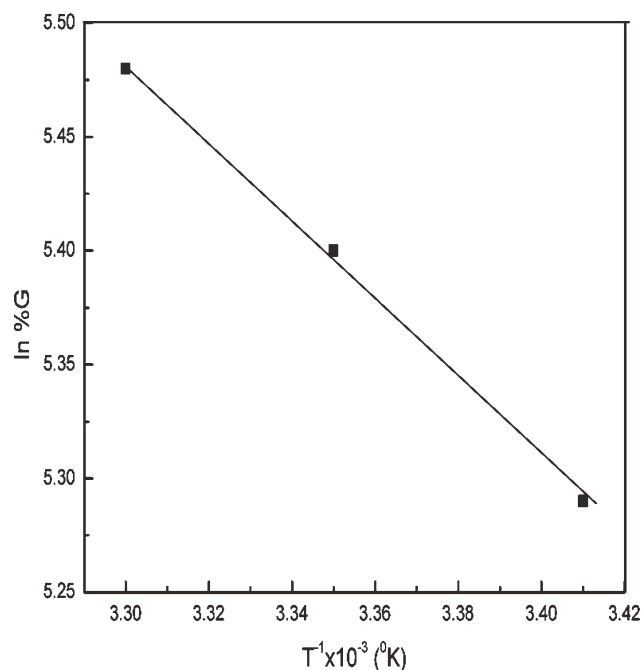
**Figure 6** Effect of reaction time on (A) %G or (B) %GE.

#### Effect of reaction time

The influence of reaction time on grafting yields is shown in Figure 6. It can be observed from the figure that the values of %G and %GE are found to be increased in the beginning and achieved the maximum value of the grafting 224.91% at 2 h in the grafting of MMA. However, beyond these optimum value, %G and %GE are found to be decreased with further increase in time. The result of this figure may be explained on the basis of the fact that with increase in reaction time the number of grafting sites on the backbone increases as a result of which the extent of initiation and propagation of photo-graft copolymerization also increases with time leading to the increase in the grafting yields.<sup>7</sup> But beyond the optimum value of reaction time 2 h, as the available sites for photo-grafting of the monomer (MMA) on the Na-PCMPsy backbone get reduced, the decreases in the value of %G and %GE are observed. The observed decrease in the values of grafting yields is also may be presumably due to the detrimental effect of UV-radiation onto the grafted side chains of PMMA at longer irradiation times in the presence of the photoinitiator. In addition to this, the decrease in the values of %G and %GE beyond optimum reaction time may also be attributed to the depletion in monomer and initiator concentrations and shortage of the available grafting sites.<sup>31</sup> Thus, from the above

**TABLE I**  
Optimized Reaction Conditions

Na-PCMPsy ( $\overline{DS} = 0.13$ )	0.6 g (dry basis)
[HNO <sub>3</sub> ]	0.2 mol L <sup>-1</sup>
[CAN]	$3.0 \times 10^{-3}$ mol L <sup>-1</sup>
[MMA]	0.326 mol L <sup>-1</sup>
Temperature	30°C
Time	2 h
Volume of water	142.29 mL
Total volume	150 mL



**Figure 7** plot of natural log of percentage grafting versus  $1/T$ .

discussion, the optimized reaction conditions obtained in the UV-radiation graft copolymerization of MMA are shown in Table I.

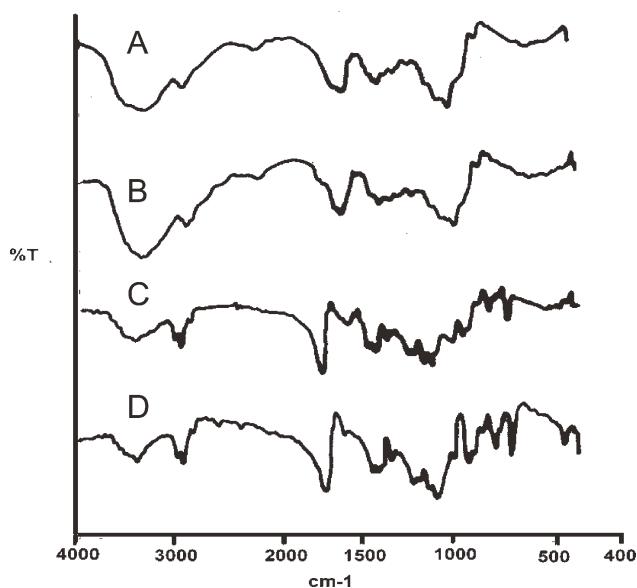
#### Evaluation of energy of activation

The natural log of %grafting ( $\ln\%G$ ) versus  $1/T$  is plotted for the initial portion of the curve, that is, 20–30°C (cf. Fig. 5) as shown in Figure 7 and these values are found to fall on a straight line as is evident from this figure. The overall activation energy of grafting ( $E_a$ ) is calculated from the results of Figure 7 and is found to be 14.38 KJ mol<sup>-1</sup>. similar results have reported in literature and calculated similarly the overall activation energy 8.70, 34.66, and 48.16 KJ mol<sup>-1</sup>.<sup>32,33</sup>

## EVIDENCE OF GRAFTING

### IR-spectra

Figure 8(A) shows the IR spectrum of Psy. The presence of a very strong and broad absorption band at  $\sim 3407$  cm<sup>-1</sup> is assigned to  $-\text{OH}$  stretching. Reasonably sharp absorption band  $\sim 2928$  cm<sup>-1</sup> may be attributed to the  $-\text{CH}$  stretching. The absorption band appeared at  $\sim 1640$  cm<sup>-1</sup> is due to hydration of water. The  $-\text{CH}_2$  bending in Psy is assigned to absorption at  $\sim 1465$  cm<sup>-1</sup> and the frequency at  $\sim 1345$  cm<sup>-1</sup> is attributed to  $-\text{CH}$  bending. The bending of  $-\text{OH}$  is probably distributed at  $\sim 1325$  and  $\sim 1427$  cm<sup>-1</sup> frequencies.



**Figure 8** IR spectra of (A) psyllium, (B) Na-PCMPsy, (C) Na-PCMPsy-g-PMMA, and (D) PMMA.

The IR-spectrum of Na-PCMPsy ( $\overline{DS} = 0.13$ ) [Fig. 8(B)] shows somewhat reduced intensity of the absorption at  $\sim 3337\text{cm}^{-1}$ , due to  $-\text{OH}$  stretching, indication that some of the  $-\text{OH}$  groups present in Psy sample are involved in carboxymethylation. The presence of a band at  $\sim 2929\text{cm}^{-1}$  is due to  $-\text{CH}$  stretching. The band due to water (bending of water) appeared at  $\sim 1640\text{cm}^{-1}$  in Psy sample [cf. Fig. 8(A)] is absent in the Na-PCMPsy sample. The asymmetric and symmetric vibrations due to  $-\text{COO}-$  moiety are assigned to  $\sim 1605\text{cm}^{-1}$  and  $\sim 1423\text{cm}^{-1}$  respectively. This can be attributed to the incorporation of carboxymethyl groups in Psy.

The IR-spectra of Na-PCMPsy-g-PMMA and PMMA are shown in Figure 8(C,D), respectively. In Figure 8(C) showed absorption bands of Na-PCMPsy and additional strong absorption band at about approximately  $1730\text{--}1750\text{cm}^{-1}$  assigned to  $-\text{C}=\text{O}$  stretching of ester group ( $[\text{bond COOCH}_3]$ ) characteristic of methacrylates. Moreover, in Figure

8(D), the isolated grafted chains, that is, (PMMA) it shows the characteristic peak of  $-\text{C}=\text{O}$  stretching at about approximately  $1730\text{--}1750\text{cm}^{-1}$ . This may be attributed to the fact that hydrolysis of the graft copolymer give back the grafted chains (PMMA). Thus, the results of Figures 8(A–D) provide a substantial evidence of grafting MMA onto Na-PCMPsy.

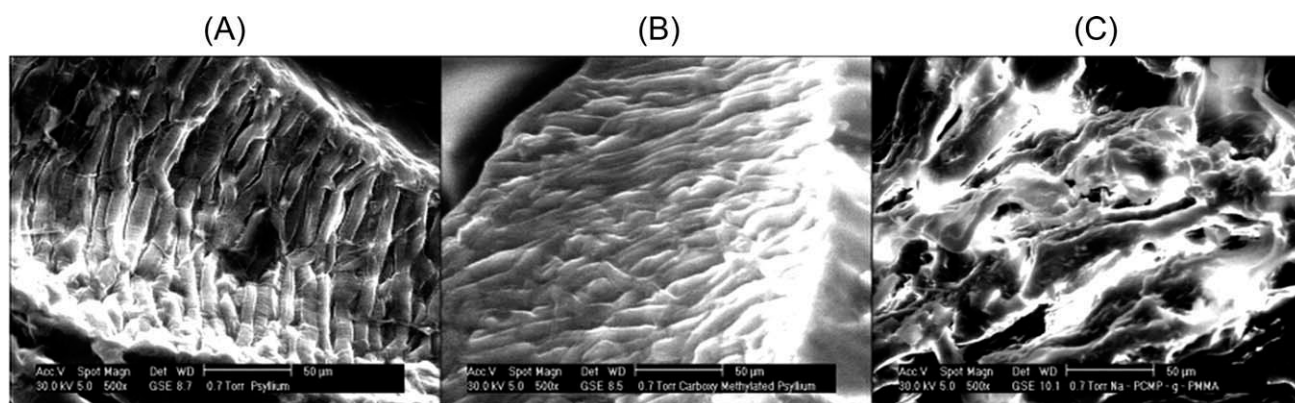
### Scanning electron microscopy

The scanning electron micrograph of Psy [Fig. 9(A)] shows fiber-like appearance structure. Upon carboxymethylation of Psy, the structure of Psy gets improved as shown in [Fig. 9(B)], wherein the morphology of the fiber-like structure is modified in such a way that because of the introduction of the hydrophilic groups onto Psy, the fibers seem to have got swollen and exhibited smooth surface. The surface topology of Na-PCMPsy ( $\overline{DS} = 0.13$ ) is shown in Figure 9(B). Upon comparing the morphology of the grafted sample [Fig. 9(C)] with ungrafted material, it is clearly evident that the grafted chains have drastically changed the topology of the Na-PCMPsy ( $\overline{DS} = 0.13$ ). As shown in Figure 9(C), the lumpy morphology is observed with MMA. The scanning electron micrograph of Na-PCMPsy ( $\overline{DS} = 0.13$ ) [Fig. 9(C)] further reveals the additional surface deposits indicating that grafting has taken place.

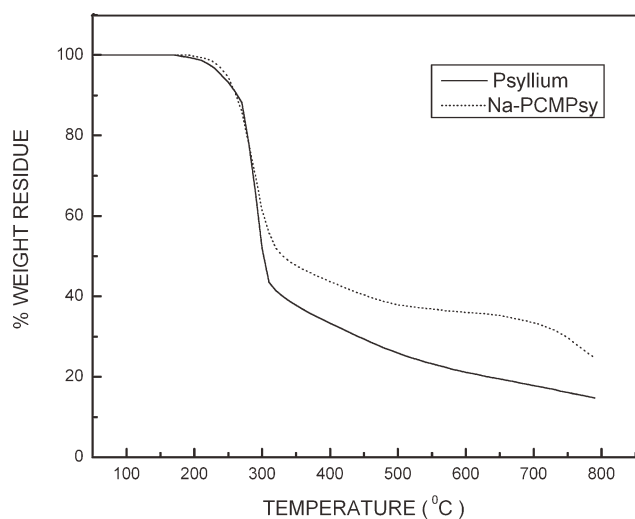
### Thermogravimetric analysis

Psy, Na-PCMPsy, and its graft copolymer and homopolymer

Figure 10 show the primary thermograms of Psy and Na-PCMPsy obtained at a scan rate  $10^\circ\text{C min}^{-1}$ . The overall degradation of Psy (Figure 10) involves only single step. The sample begins to decompose at  $170^\circ\text{C}$ , rapidly loses 57.5 % of its weight up to  $315^\circ\text{C}$ . Beyond  $315^\circ\text{C}$ , the weight loss is slow and gradual up to about  $780^\circ\text{C}$  leaving 14.5 % residual weight. In the temperature range  $315\text{--}780^\circ\text{C}$ , the



**Figure 9** Scanning electron micrograph of (A) psyllium, (B) Na-PCMPsy, and (C) Na-PCMPsy-g-PMMA.

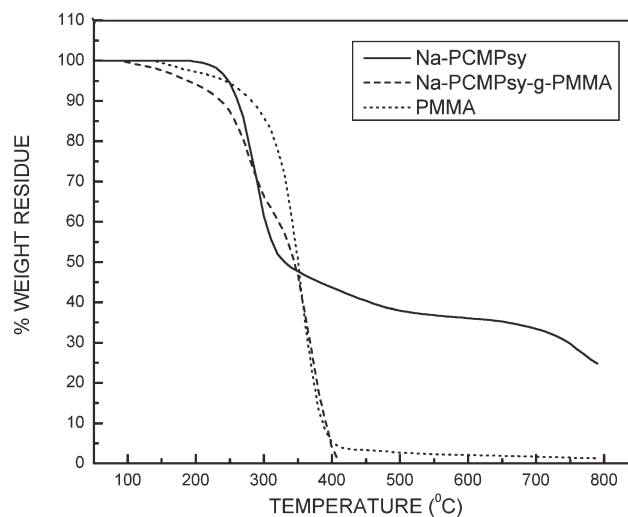


**Figure 10** Thermogram of psyllium and Na-PCMPsy.

sample loses 28 % of its original weight. The maximum rate of weight loss occurs at 295°C.

In the case of Na-PCMPsy (Figure 10) also, a single step of degradation is observed. The decomposition starts at 190°C and proceeds at a faster rate up to 320°C and at this temperature the sample loses 48% of its original weight. However, beyond this temperature, degradation proceeds at a slow rate up to 500°C compared with the degradation proceeded in the earlier temperature range. This temperature range involves about 14% weight loss. With further increase in temperature, the degradation is found to occur at a relatively very slow rate up to 790°C. The temperature at which the maximum rate of weight loss occurs is at 300°C. The overall degradation leaves about 24.5% residue.

The primary thermograms obtained at a scan rate of  $10^{\circ}\text{C min}^{-1}$ , for Na-PCMPsy, Na-PCMPsy-g-PMMA (% G = 387.46), and PMMA in an inert atmosphere are shown in Figure 11. The overall degradation of Na-PCMPsy-g-PMMA comprises of two steps. The first step encompasses a temperature range 90–300°C, involving about 34% weight loss, the rate of weight loss reaching a maximum at 270°C. This step is immediately followed by the second step involving about 65.5 % weight loss over a temperature range 300–415°C with a maximum rate



**Figure 11** Thermogram of Na-PCMPsy, Na-PCMPsy-g-PMMA, and PMMA.

of weight loss at 365°C. The sample leaves about only 0.5 % residue.

In the case of PMMA, the decomposition begins at 135°C and proceeds very slowly up to 320°C involving about 22 % weight loss. Beyond 320°C, the sample degrades very rapidly up to 390°C involving about 70% weight loss. The maximum rate of weight loss occurs at 350°C and literature data also show maximum weight loss of PMMA in between 350–400°C.<sup>34</sup> The degradation is complete at about 705°C leaving about 1% residual weight.

The temperature characteristic values and the value of the integral procedural decomposition temperature (IPDT) of Psy, Na-PCMPsy ( $\overline{DS} = 0.13$ ), Na-PCMPsy-PMMA, and PMMA are tabulated in Table II. The examination of IPDT values indicates that the overall thermal stability of Psy has been increased upon its carboxymethylation, this may be due to introduction of polar groups into it leading to the increased intermolecular and intramolecular interactions, which ultimately imparts higher thermal stability to it but in case of graft copolymer (Na-PCMPsy-PMMA) the observed IPDT value is decreases compared with Na-PCMPsy, this may be due to grafted MMAs are known to depolymerize upon pyrolysis.

**TABLE II**  
Thermal Analysis of Psyllium, Na-PCMPsy, Na-PCMPsy-g-PMMA, and PMMA Samples

Sample	Thermogravimetric analysis data		$T_{\max}$ (°C)		$T_{10}$ (°C)	$T_{50}$ (°C)	IPDT
	$T_i$ , °C (IDT)	$T_f$ , °C (FDT)	Step 1	Step 2			
Psyllium	170	780	295	–	265	300	475
Na-PCMPsy	190	790	300	–	260	330	538
Na-PCMPsy-g-PMMA	90	415	270	365	235	345	213.5
PMMA	135	705	305	–	280	350	368.7

## CONCLUSIONS

UV-grafting of MMA has been successfully carried out onto Na-PCMPsy ( $\overline{DS} = 0.13$ ) using CAN as a photoinitiator and the optimized reaction conditions have been evaluated. Under the optimized reaction conditions, the maximum values of the grafting yields are %G = 387.46 and %GE = 91.26. The spectral (infrared), thermogravimetric analysis, and scanning electron microscopic techniques have been used successfully as the evidence of UV-grafting.

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