

Comparative Isoconversional Thermal Analysis and Degradation Kinetics of Acetylated *Mimosa Pudica* (Touch-Me-Not) Hydrogel

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Abstract: Comparative thermal degradation and kinetics of hydrogel of *Mimosa pudica* (MPH) and acetylated *Mimosa pudica* hydrogel (AMPH) were investigated. Thermogravimetric (TG) analyses of MPH and AMPH was carried out and analyzed using isoconversional method. Kinetic parameters such as energy of activation (E_a) were calculated by fitting the thermal data to Flynn–Wall–Ozawa (FWO) and Kissinger methods. The order of thermal degradation reactions (n) was evaluated using Kissinger method. TG curves of both MPH and AMPH exhibited two step exothermic degradation. The E_a values calculated by Kissinger and FWO models for first step of degradation of AMPH were found in the range of 105.41-108.49 and 153.67-163.77 kJ mol⁻¹ for the second step which were comparable with the E_a values of the unmodified MPH. The mean values of integral procedural decomposition temperature (IPDT) for MPH and AMPH were found to be 338 °C and 346 °C, respectively. The mean comprehensive index of thermal stability (ITS) was found to be 0.55 for MPH and AMPH showing both materials much stable than many commercially available ingredients used in drug designing.

Keywords: *Mimosa pudica*, Acetylation, Hydrogel, Thermal degradation, Decomposition kinetics.

Introduction

Hydrogels are cross-linked hydrophilic polymers that can absorb large quantities of water without dissolving. They have potential applications in drug delivery systems and development of engineered tissues due to their structural similarity to extracellular matrix of tissues, biocompatibility, availability and environment friendly nature [1-3]. Hydrogels have gained attraction in adsorption of proteins [4] and metal ions [5], contact lenses, wound dressing [6], waste water treatment and agriculture [7], catalyst carriers [8] and hygienic products [9].

Polysaccharides have attracted researchers due to their uses in daily life and pharmaceutical, medicinal and various other industries. The polysaccharides are modified, e.g., acetylated, to get desirable physical and chemical properties [10, 11]. These materials whether acetylated or not, are generally subjected to testing prior to use, particularly in pharmaceutical industry. In this connection, one of the important parameter is to get information about the thermal stability and degradation kinetics of polysaccharidal materials which may give information about the use, storage and handling [12, 13]. Thermally more stable polysaccharidal materials may have higher shelf-life [14].

Mimosa pudica L. is a sensitive plant that has been recognized for its wide spectrum of medicinal applications [15-18]. Besides medicinal applications, *Mimosa pudica* (MP) hydrogel has been evaluated for sustained release of drugs and bucoadhesive studies [19, 20].

Scientists are keen to evaluate physical and chemical properties of polysaccharides and polysaccharide based hydrogels and their derivatives for potential applications. For this purpose, thermal studies of such materials are important. Recent reports revealed that thermal stability of materials (polysaccharides) used in pharmaceuticals (tablets) is important to enhance the shelf life of pharmaceuticals [14, 21, 22]. A valuable tool to enhance the thermal stability of polysaccharides [23, 24] is their esterification [25]. In this regard, TG analysis of MPH and AMPH can be used to determine thermal stability, degradation kinetics and thermodynamic properties because, to the best of knowledge, thermal studies of said materials have not been reported yet.

Herein, we report on comparative isoconversional thermal analyses (FWO and Kissinger methods) to determine the thermal stability (ITS, IPDT), degradation kinetics (E_a , n , Z) and thermodynamic parameters (ΔH , ΔS , ΔG) of

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hydrogelable polysaccharide isolated from MPH and its acetylated derivative. Therefore we are reporting is the topic of interest in order to find its applications in pharmaceuticals.

Experimental

Reagents and Chemicals

Seeds of MPH were purchased from local market. Acetic anhydride, 4-dimethylaminopyridine (DMAP), ethanol and *n*-hexane used were obtained from Fluka. All these chemicals were used without further purification. Deionized water was used throughout the study.

Measurements

FTIR spectroscopy of MPH and AMPH was carried out using FTIR Instrument IR Prestige-21 (Shimadzu, Japan). KBr pellet technique was employed to obtain the FTIR spectra of MPH and AMPH. Moisture content of the sample pellets was removed at 50 °C under vacuum for 4 h before analysis. ¹H-NMR (400 MHz) spectra were recorded on Bruker instrument using DMSO-*d*₆ as a solvent and 64 number of scans were accumulated. TG analyses of MPH and AMPH were performed on a simultaneous thermal analyzer SDT, Q-600 (TA Instruments, USA), under N₂ flowing at rate of 100 mL min⁻¹. Each sample was heated at four different heating rates, i.e., 5, 10, 15 and 20 °C min⁻¹ from ambient to 800 °C.

Extraction of Hydrogel from MPH seeds

The MPH seeds (20 g) were washed with 100 mL distilled water to remove dust particle on the surface and then soaked in 1000 mL distilled water for 10 h. Mucilage was separated from seeds by using cotton cloth after blending seeds with kitchen blender. Mucilage was washed with *n*-hexane twice to remove lipophilic substances. Air dried gel was powdered by grinder. Yield of the hydrogel obtained was 9%.

Modification of MPH

Hydrogel was acetylated to study the effect of acetylation on thermal properties of hydrogel. For this purpose, hydrogel (1.0 g) was suspended in DMSO and acetic anhydride (10 mL) was added. Reaction was preceded in the presence of DMAP (40 mg) as a catalyst. The mixture was stirred at 80 °C for 6 h and acetylated product was precipitated and

washed with ethanol. Product (sample 4) was dried under vacuum at 50 °C overnight.

Yield: (1.20 g); degree of substitution (DS, calcd. by acid base titration): 1.86; FTIR: 1747 (CO_{Ester}), 2926 (CH and CH₂), 1431 (CH₂), 3414 cm⁻¹ (OH); ¹H-NMR (DMSO-*d*₆; 400 MHz; NS 64): 1.97 (CH₃), 3.01-4.70 (Repeating unit- Hs). Comparable FTIR and ¹H-NMR spectra were obtained for the rest of samples.

Determination of Degree of Substitution (DS)

The DS of acetylation on MPH was calculated by acid-base titration method. For this purpose, 100 mg of AMPH was added in 50 mL of 0.1M NaOH aqueous solution and stirred continuously overnight for complete saponification. The pH of the solution was maintained at 7 by adding 0.01M HCl solution. Then a known quantity of 1M NaOH solution was added to the neutral solution and excess base was neutralized with 0.1M HCl solution. DS of acetylation was calculated using following equation (eq. 1);

$$DS = \frac{[n.NaOH \times M(RU)]}{[Ms - Mr(RCO) \times n.NaOH]} \quad (\text{eq. 1})$$

where, *n*.NaOH is the number of moles of NaOH added after saponification, M (RU) is molar mass of repeating unit of the polymer, Ms is the mass of sample taken and Mr (RCO-) is molar mass of ester functionality.

Thermal Analysis and Degradation Kinetics

Thermal decomposition temperatures, initial (T_{di}), maximum (T_{dm}) and final (T_{df}) of each thermal degradation step were calculated from TG curves of MPH and AMPH. Different isoconversional methods were employed for the kinetic studies of thermal data. The FWO method [26-30] is used for the calculation of kinetic parameters. It was the first isoconversional linear integral method (eq. 2).

$$\ln \beta = \ln \frac{AE_a}{Rg(\alpha)} - 5.331 - 1.052 \frac{E_a}{RT} \quad (\text{eq. 2})$$

where, β the heating rate; *A* the pre-exponential factor; *R* the general gas constant and *T* is the temperature at the conversion rate (α). The α is calculated by using below given relation (eq. 3);

$$\alpha = \frac{m_o - m_t}{m_o - m_f} \quad (\text{eq. 3})$$

where, m_o and m_f are the initial and final mass, respectively; m_t is the sample mass at any temperature T .

FWO method may be termed as a model-free approach, because it is based on the assumption that rate of thermal degradation reaction depends upon temperature only for a fixed extent of conversion. For a fixed value of α at different heating rates, a graph is plotted between $\log\beta$ and $1000 T^{-1}$ which gives a straight line graph and the value of E_a is calculated by the slope of this straight line graph.

In Kissinger's method, it was assumed that the temperature of maximum deflection in differential thermal analysis is also the temperature at which the reaction rate is maximum. Kissinger's method [31] uses following equation (eq. 4);

$$\ln\left(\frac{\beta}{T_m^2}\right) = -\frac{E_a}{RT_m} + C \quad (\text{eq. 4})$$

A plot of $\log(\beta/T_m^2)$ versus $1000 T_m^{-2}$ for a constant conversion gives the E_a at that conversion.

Equation for the determination of shape index by Kissinger method is given below which can be used to find out the order of reaction n ;

$$S = \frac{\left[\left(\frac{d^2\alpha}{dt^2}\right)_L\right]}{\left[\left(\frac{d^2\alpha}{dt^2}\right)_R\right]} \quad (\text{eq. 5})$$

The values of $(d^2\alpha/dt^2)_L$ and $(d^2\alpha/dt^2)_R$ are obtained from the 2DTG curve where subscripts "L" and "R" indicate the data on left and right sides of the 2DTG peak. The value of "S" helps to calculate the n using equations as under (eq. 6 and eq. 7);

$$n = 1.88S \quad (S \geq 0.45) \quad (\text{eq. 6})$$

$$n = 1.26S^{0.5} \quad (S \leq 0.45) \quad (\text{eq. 7})$$

Integral procedural decomposition temperature (IPDT) and comprehensive index of intrinsic thermal stability (ITS) were calculated to

evaluate the thermal stabilities of the samples using method proposed by [32]. The IPDT and ITS values were determined from TG curves of all four heating rates and mean values are reported for MPH and AMPH. The thermodynamic parameters such as change in enthalpy (ΔH), change in entropy (ΔS) and change in Gibbs free energy (ΔG) were calculated by The Eyring-Polanyi equation [33] which is written as under (eq. 8):

$$k = \frac{k_B T}{h} e^{\frac{-\Delta G}{RT}} \quad (\text{eq. 8})$$

where h is Planck's constant, ΔG is the Gibbs energy of activation, and k_B is Boltzmann's constant.

The data were analyzed by the use of Universal Analysis 2000 software, version 4.2E (TA Instruments, USA), and MS Excel® 2010.

Results and Discussion

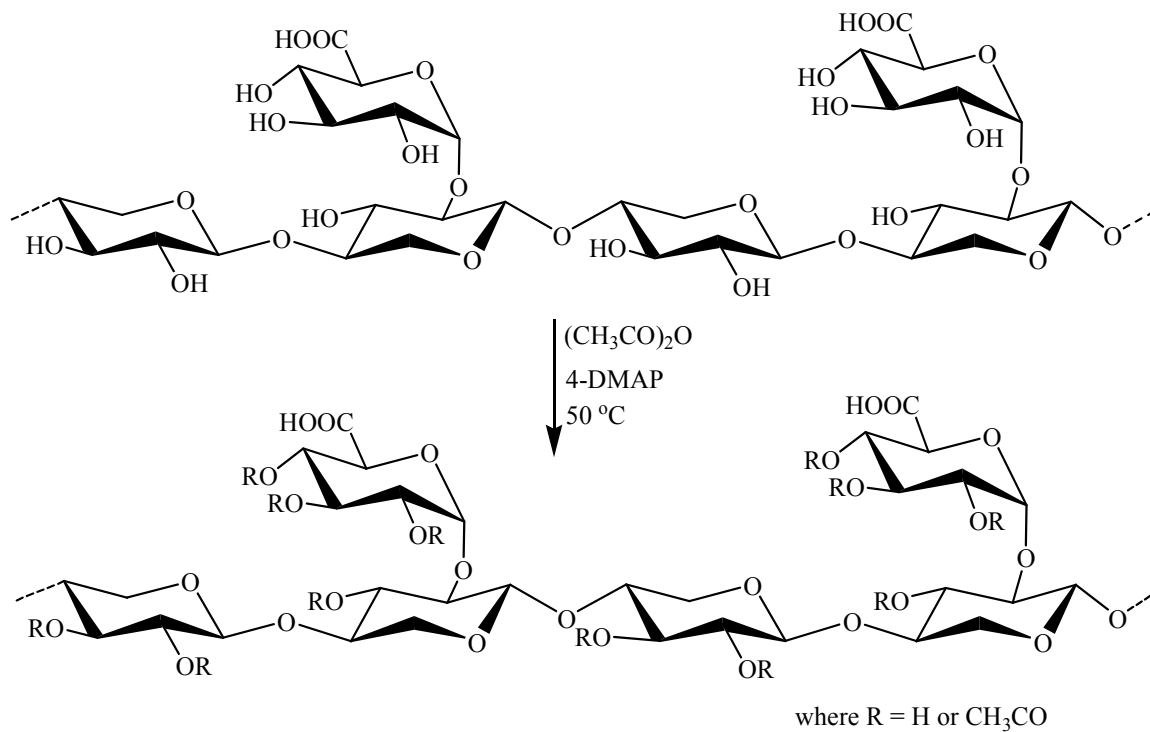
Synthesis and Characterization

Acetylation of MP hydrogel (MPH) was carried out to obtain acetylated MPH derivative, i.e., AMPH. MPH was reacted with acetic anhydride in the presence of DMAP (40 mg in each reaction) as a catalyst (Scheme-1) which is a widely accepted method for acetylation of polysaccharides [34]. Degree of substitution (DS) of acetylation 1.86 was obtained while using large molar ratio of acetic anhydride to anhydroglucose (1:12) in the presence of 4-dimethylaminopyridine (DMAP) as a catalyst. The AMPH derivatives were therefore fabricated with DS 0.35-1.86. The reaction conditions and results are given in Table-1. The degree of substitution (DS) was calculated in triplicate from acid-base titration after saponification and mean values are being shown. The products obtained were soluble in DMSO and DMAc.

Table-1: Reaction conditions and results of acetylation of MPH (1.0 g) using acetic anhydride in the presence of DMAP (40 mg) catalyst.

Sample	Molar Ratio ^a	Yield/g	DS ^b	Solubility
1	1:3	0.89	0.35	DMSO
2	1:6	1.02	0.81	DMSO, DMAc/LiCl+Δ
3	1:9	1.18	1.74	DMSO, DMAc/LiCl+Δ
4	1:12	1.20	1.86	DMSO, DMAc/LiCl+Δ

^aAnhydroglucose unit: acetic anhydride; ^bDegree of substitution calcd. by acid base titration after saponification.



Scheme-1: Synthesis of acetylated MPH.

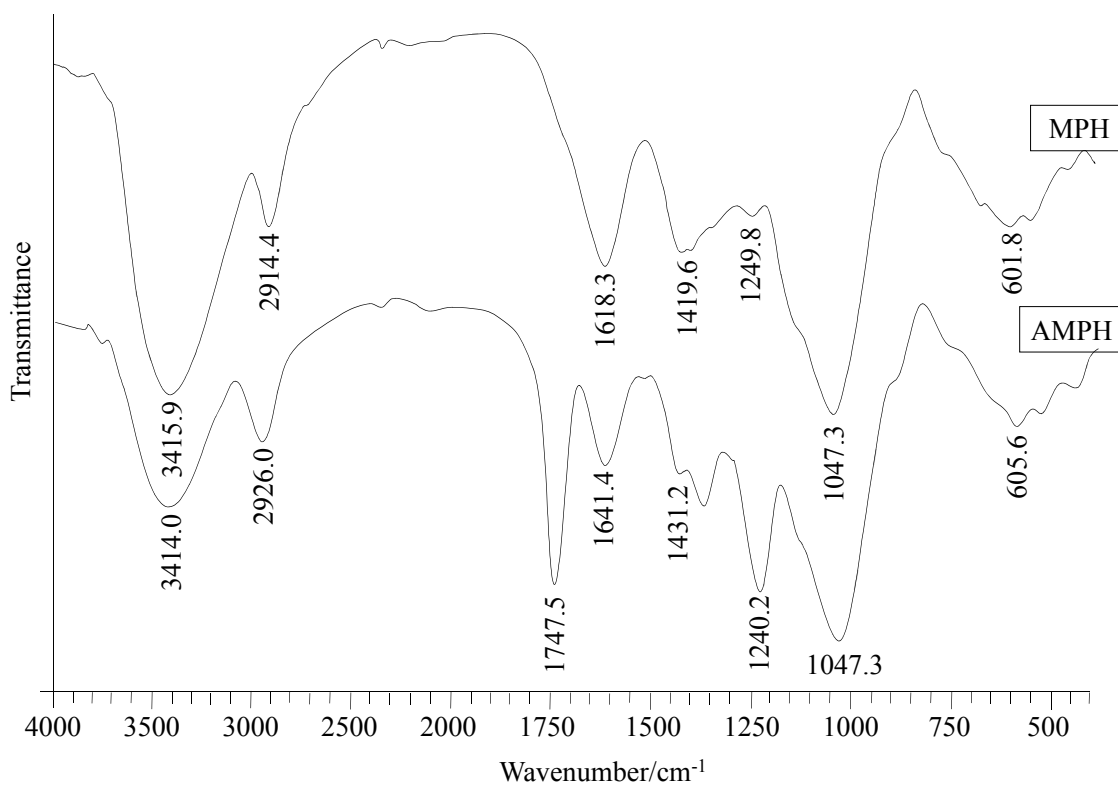


Fig. 1: FTIR (KBr) spectra of MPH and AMPH 4 showing distinct ester carbonyl signal.

The polysaccharide acetates are characterized using FTIR spectroscopic analyses for determination of success of acetylation as ester carbonyl peaks are easily recognizable. Therefore, MPH and AMPH were characterized using FTIR (KBr) spectroscopic analysis (Fig. 1) by making pellets of samples. The spectrum of AMPH **4** showed a distinct ester peak at 1747 cm^{-1} indicating successful acetylation of MPH. Other characteristic broad peak of still present OH groups was found at 3414 cm^{-1} . Characteristic signals of COC of polymer backbone centered at 1047 cm^{-1} whereas, CH_2 stretching appeared at 1431 cm^{-1} . Distinct ester peaks along with all characteristic signals of MPH indicated successful formation of AMPH.

Typical $^1\text{H-NMR}$ ($\text{DMSO-}d_6$; 400 MHz) spectrum of sample **4** (Fig. 2) showed the success of acetylation. Methyl Hs of acetyl groups attached on to MPH polysaccharides appeared at δ 1.97 ppm. The MPH polysaccharide repeating unit Hs were detectable at δ 3.01-4.70 ppm.

Thermal analysis

Thermal degradation behavior of both MPH and AMPH (sample **4**) was studied in the temperature range of ambient to $800\text{ }^\circ\text{C}$ using multiple heating rates. The TG curves of MPH and AMPH showed 9% and 6% mass loss, respectively between $50\text{-}130\text{ }^\circ\text{C}$ temperature ranges, indicating the loss of trapped water [23, 35]. TGA of both MPH and AMPH showed two step degradations. Overlay TG and DTG curves of MPH and its acetylated derivative (Sample **4**) recorded at multiple heating rates are shown in Fig. 3 and results are shown in Table-2. The initial (T_{di}) and final (T_{df}) thermal degradation temperatures of MPH was found in the range $248\text{-}266\text{ }^\circ\text{C}$ and $372\text{-}384\text{ }^\circ\text{C}$, respectively for first thermal degradation step. The T_{di} and T_{df} observed for second step were $412\text{-}427\text{ }^\circ\text{C}$ and $499\text{-}523\text{ }^\circ\text{C}$, respectively. The mass loss was approx. 56-58% in the first step while 26% in the second step at various heating rates. Whereas, the T_{dm} values were found in the range of $293\text{-}307\text{ }^\circ\text{C}$ and $461\text{-}493\text{ }^\circ\text{C}$ for degradation step 1 and 2, respectively.

Table-2: Thermal decomposition temperatures of MPH and AMPH **4** at various heating rates.

Sample	Step	$T_{d_i}/^\circ\text{C}$	$T_{d_m}/^\circ\text{C}$	$T_{d_f}/^\circ\text{C}$	Mass loss/ % at T_{d_f}	Char yield/ Mass/%
MPH	I	248-266	293-307	372-384	56-58	13-14 at $600\text{ }^\circ\text{C}$
	II	412-427	461-493	499-523	83-84	
AMPH	I	231-241	315-333	381-404	68-71	9-11 at $600\text{ }^\circ\text{C}$
	II	430-448	469-485	501-517	88-90	

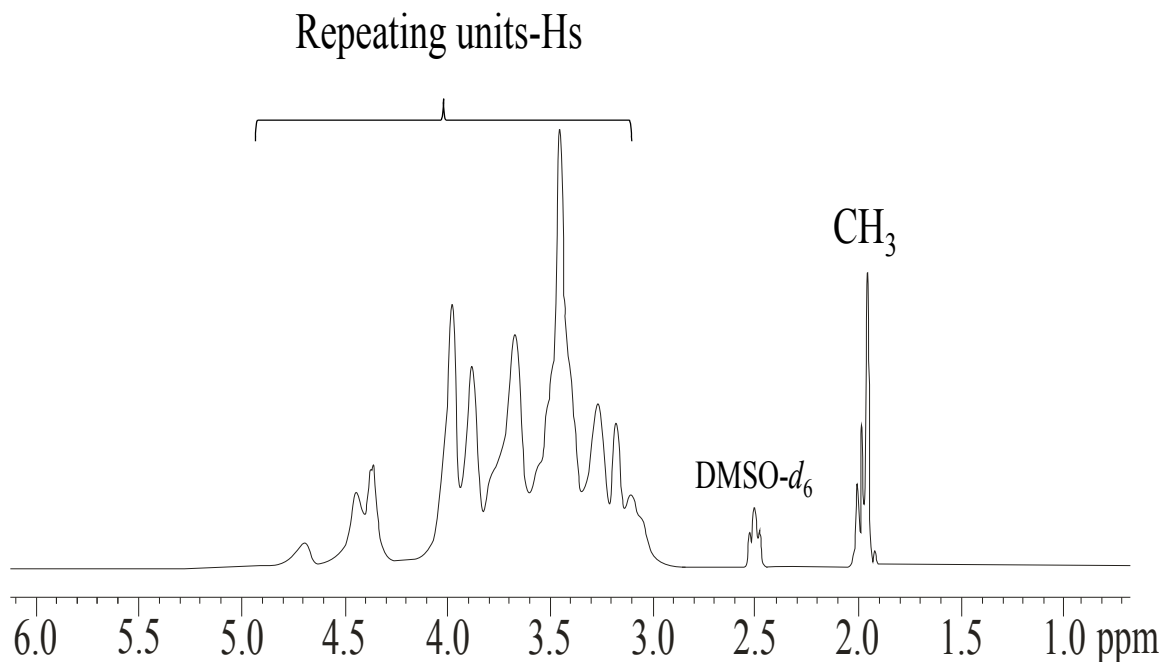


Fig. 2: $^1\text{H-NMR}$ ($\text{DMSO-}d_6$; 400 MHz; NS 64) spectrum of AMPH **4**.

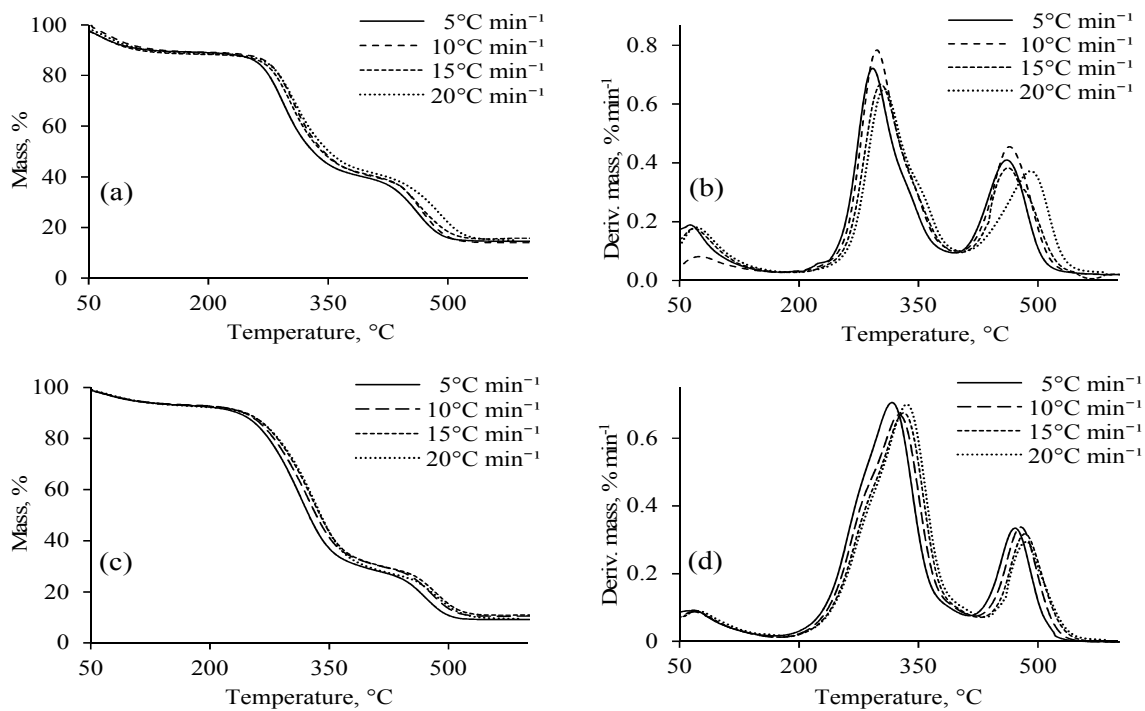


Fig. 3: Overlay TG (a, c) and DTG (b, d) curves of MPH and AMPH 4, respectively at multiple heating rates.

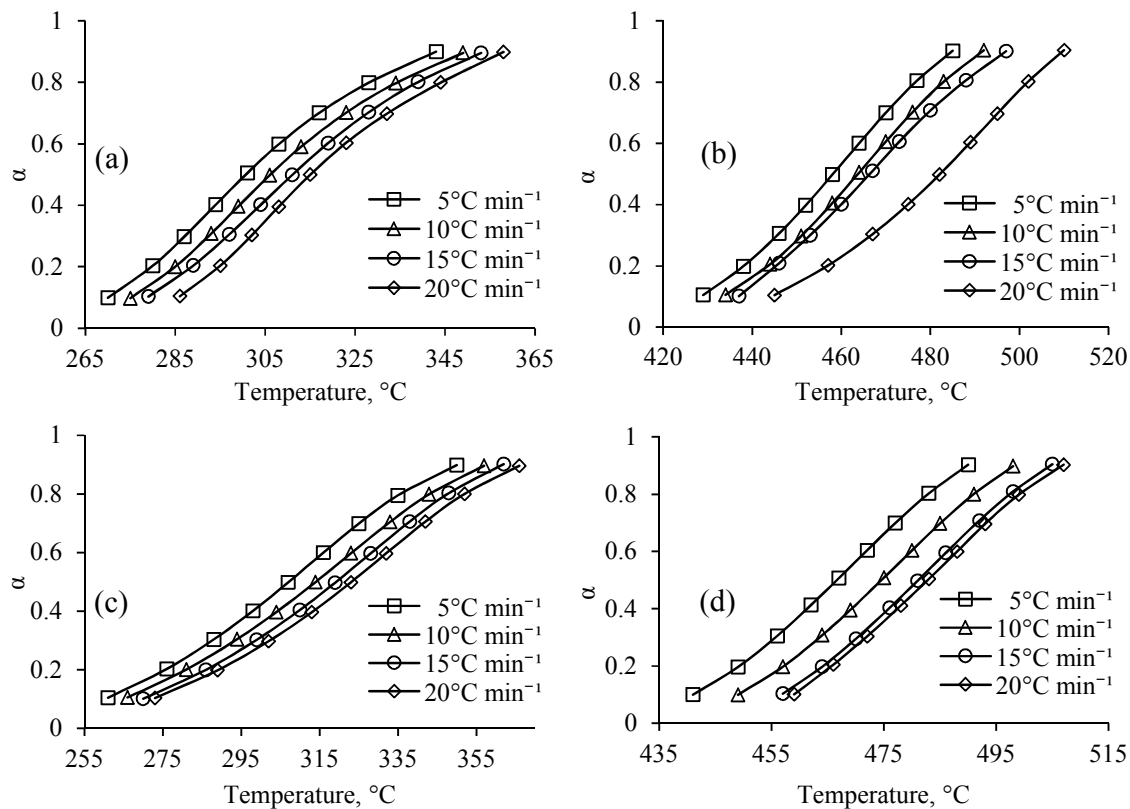


Fig. 4: Plots of α vs. T curves of first (a, c) and second (b, d) thermal degradation steps for MPH and AMPH 4 respectively, at multiple heating rates.

Thermal degradation at different heating rates of AMPH (sample 4) showed T_{d1} for first degradation step was 231-241 °C whereas the T_{d2} values were between 381-404 °C. The first step was major degradation step with 68-71% mass loss. T_{d1} and T_{d2} were observed 430-448 °C and 501-517 °C with 19% mass loss for second degradation step of AMPH. The T_{d2} values of both degradation steps in AMPH found higher than that of MPH were in the range of 315-333°C and 469-485 °C for step 1 and 2, respectively. It is concluded from comparison of decomposition temperature maxima (T_{d2}) of first step of MPH (293-307 °C) and AMPH (315-333 °C), significant thermal stability was imparted to MPH after acetylation (Table-2). Similarly, higher T_{d2} values of second step degradation were found for AMPH as compared to MP. Plots of α vs. T curves of first and second thermal degradation steps for MPH and AMPH at each heating rate are shown in Fig. 4 showing comparable degradation pattern at different heating rates.

Degradation Kinetics

Different kinetic parameters; energy of activation (E_a) and frequency factor (A) were evaluated using FWO and Kissinger isoconversional methods. The order of thermal degradation reactions (n) was calculated by Kissinger method. In FWO method, the E_a values for both stages of thermal decomposition were calculated at different conversions ($\alpha = 0.1-0.90$ with 0.1 increment) therefore FWO is most trustable method in this regard. The E_a values of first and second degradation steps for MPH were found to be 110.33 and 148.90 kJ mol⁻¹, respectively (Table-3). Whereas, the E_a values of both degradation steps of AMPH (sample 4) were found to be 108.49 and 153.67 kJ mol⁻¹ respectively. The comparable E_a values calculated by FWO method showed that acetylation does not significantly affect the degradation rates of MP. FWO plots between $\log\beta$ and $1000 T^{-1}/K$ for each

thermal degradation step at several degree of conversion for MPH and AMPH are shown in Fig. 5.

In Kissinger method, the maximum decomposition temperature at each heating rate is taken in to account and values of E_a were calculated by the slope of straight line graph between $\log(\beta/T_m^2)$ and $1000 T_m^{-2}/K$ (see Table-3, Fig. 6). The E_a values calculated were 105.04 and 148.91 kJ mol⁻¹ for both degradation steps of MP. Whereas, E_a values of both degradation steps of AMPH were found to be 105.41 and 163.77 kJ mol⁻¹, respectively. The E_a values calculated by Kissinger method were in close accordance to those by FWO method. Each thermal decomposition step of both MPH and its acetylated derivative (AMPH) exhibited first order kinetics as calculated by Kissinger method.

Thermodynamic Analysis

The thermodynamic parameters such as ΔH , ΔG and ΔS were calculated from TG data for both MPH and AMPH (Table-3). The IPDT and ITS values are important thermal parameters to determine thermal stability calculated from the area under TG curves rather than degradation temperatures. The mean value for ITS was found to be 0.55 for both MPH and AMPH. ITS values for MPH (0.55 ±0.03) and AMPH (0.55 ±0.02) are higher than the similar kind of polysaccharides obtained from *Astragalus gummifer* (0.38), *Acacia nilotica* (0.40), *Argyrea speciosa* (0.35), *Acacia modesta* (0.42), *Ocimum basicilicum* (0.41), *Plantago ovata* (0.39), *Salvia aegyptiaca* (0.33) and *P. ovata husk* (0.41) clearing greater stability of MPH and AMPH [22]. Moreover, the mean IPDT values were found to be 338 and 346 °C for MPH and AMPH, respectively. Hence, relatively higher values of IPDT indicated that AMPH was thermally as stable as MPH.

Table-3: Thermal degradation kinetics and thermodynamic parameters of MPH and AMPH 4.

Sample	Method	Step	r	n	E_a (kJ mol ⁻¹)	lnA	ΔH^*	ΔS^*	ΔG^*	IPDT	ITS
MPH	FWO	I	0.975	-	110.33	24.95	105.57	56.61	138.01	338 ±0.5	0.55 ±0.03
	Kissinger	I	0.988	0.93	105.04	24.17	100.27	63.50	136.68		
	FWO	II	0.996	-	148.90	23.79	142.76	70.08	194.51		
	Kissinger	II	0.996	0.87	148.91	23.03	142.77	76.40	199.18		
AMPH	FWO	I	0.994	-	108.49	24.46	103.51	61.90	140.57	346 ±0.3	0.55 ±0.02
	Kissinger	I	0.999	1.02	105.41	24.64	100.43	60.64	136.73		
	FWO	II	0.986	-	153.67	26.98	147.42	43.73	180.28		
	Kissinger	II	0.999	0.99	163.77	27.82	157.52	36.22	184.73		

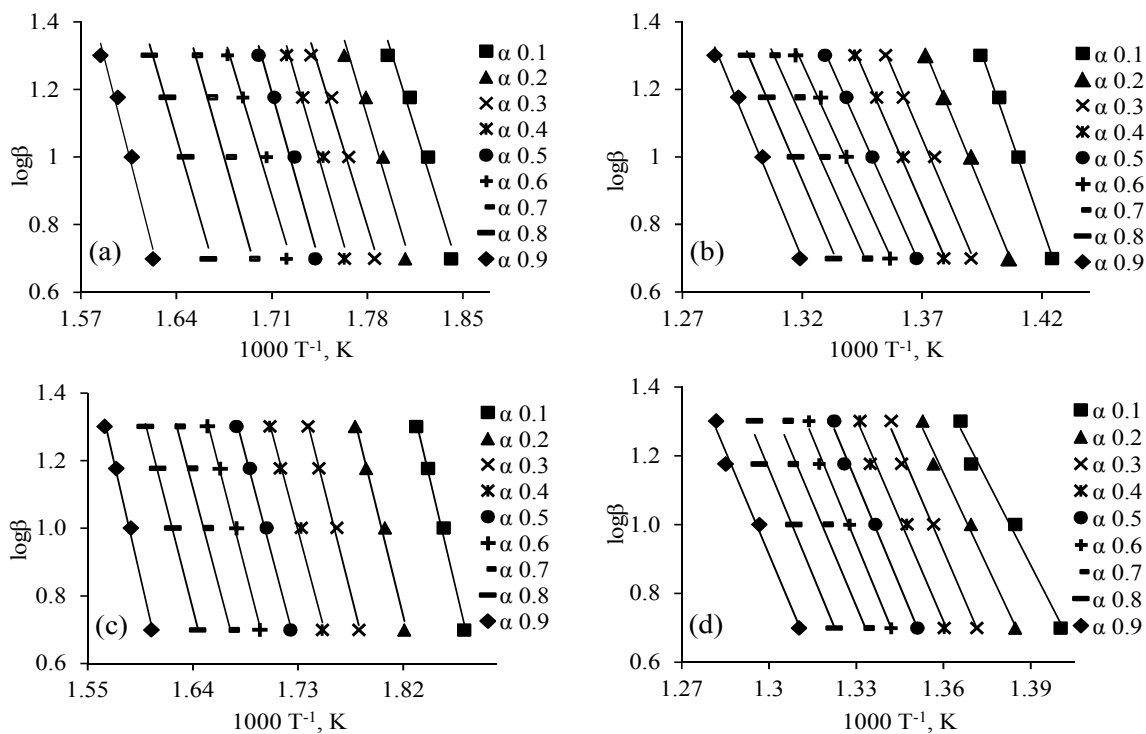


Fig. 5: Flynn-Wall-Ozawa (FWO) plot between $\log\beta$ and $1000 T^{-1}/K$ for first (a, c) and second (b, d) thermal degradation steps at several degree of conversion for MPH and AMPH 4, respectively.

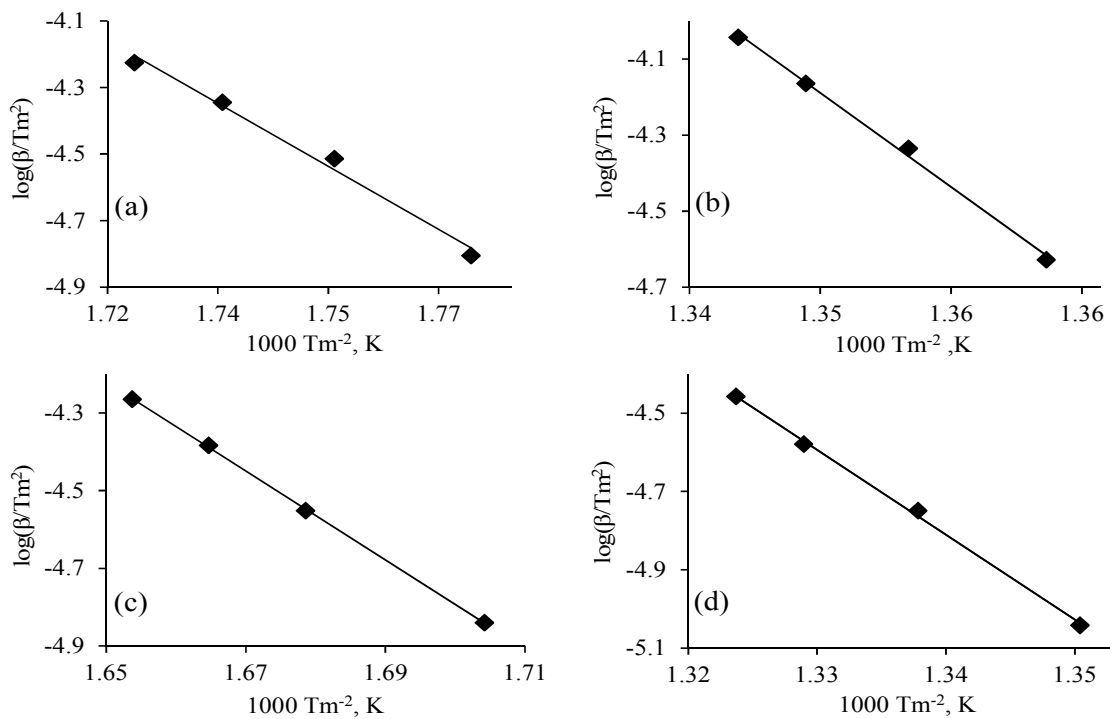


Fig. 6: Plots of Kissinger method between $\log(\beta/T_m^2)$ and $1000 T_m^{-2}/K$ for first (a, c) and second (b, d) thermal degradation step of MPH and AMPH 4, respectively.

Conclusion

Both MPH and AMPH showed two steps thermal degradation. The acetylated derivative of MPH showed less moisture content due to incorporation of hydrophobic acetyl group. The removal of moisture was an endothermic process whereas the degradation steps were exothermic in nature. Both MPH and AMPH have ITS value 0.55 showing higher stability making it a competitive candidate to the commercially available polysaccharides. IPDT value for AMPH was 346 °C, higher than that for MPH; 338 °C. Kinetic and thermodynamic parameters were also calculated and their values were found higher for acetylated derivative of MPH than unmodified gel of MP. All the degradation steps of both MPH and AMPH followed first order kinetics.

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