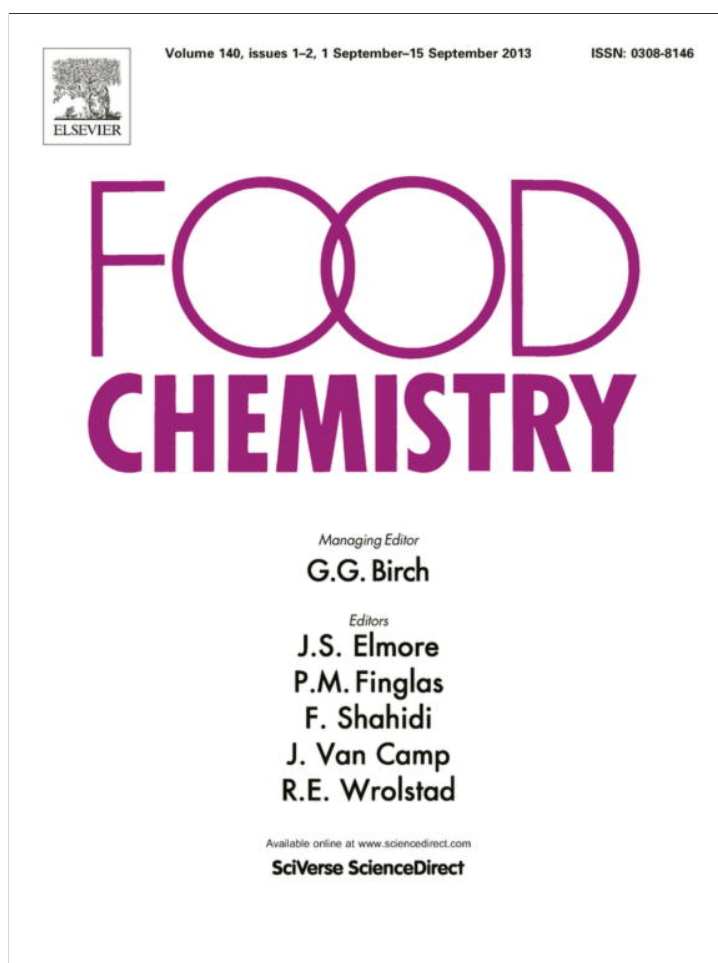


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Analytical Methods

Thermal analysis of some natural polysaccharide materials by isoconversional method

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ABSTRACT

Isoconversional thermal analysis of some important polysaccharides from functional foods is reported. Various thermal parameters including apparent activation energy (E_a), pre-exponential factor (A) were worked out, and the fitness of data to different models describing the degradation kinetics of polysaccharides was studied. The polysaccharides from *Mimosa pudica* (MP), *Plantago ovata* (PO), *Argyrea speciosa* (AS), *Acacia nilotica* (AN), *P. ovata* husk (HK) and *Acacia modesta* (AM) exhibited multistep degradation while those from *Astragalus gummifer* (AG), *Salvia aegyptiaca* (SA) and *Ocimum basicilicum* (OB) degraded mainly in single step. Generally, the degradation was exothermic. The average E_a values as determined by Flynn–Wall–Ozawa method were found to be in the range 132–187 kJ mol⁻¹. The mean comprehensive index of thermal stability (ITS) fell in the range 0.33–0.43. All the materials under investigation except those from SA and AS appear to be as stable as some of the important commercial materials used as pharmaceutical ingredients. Model-fitting analysis revealed that the major degradation step follows first-order kinetics.

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1. Introduction

A large number of gums and mucilages of edible plant items are natural hydrogels with excellent water holding capacity. They are being used as functional foods, food additives and herbal medicine since long (Duke, 2002; Morton, 1990). Some of them have been used as materials for tissue engineering, food technology, pharmaceutical excipients and drug carriers (Hoffman, 2002; Iqbal, Akbar, Hussain, Saghir, & Sher, 2011; Lee & Mooney, 2001; Lin & Metters, 2006; Munarin, Petrini, Farè, & Tanzi, 2010; Niño-Medina et al., 2010; Peppas, Bures, Leobandung, & Ichikawa, 2000). These materials are becoming more and more popular because of being safe, biodegradable and biocompatible. The increasing importance of these materials has motivated the scientists to study their physical and chemical properties in order to find their property-based applications. For most of the applications it is important to know thermal stability of a material to be used. Thermal stability, moisture content, glass transition temperature and other thermodynamic parameters can conveniently be determined by use of thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC) (Iqbal et al., 2011; Zohuriaan & Shokrolahi, 2004).

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In the present work we are reporting isoconversional thermal analysis of polysaccharide materials isolated from *Plantago ovata* (PO), *P. ovata* husk (HK), *Salvia aegyptiaca* (SA), *Ocimum basicilicum* (OB), *Mimosa pudica* (MP), *Acacia modesta* (AM), *Argyrea speciosa* (AS), *Acacia nilotica* (AN) and *Astragalus gummifer* (AG). These materials are widely being used in food and pharmaceutical preparations as thickener, stabilizer, emulsifier etc. The gels from PO, AG and AN are also approved (US-FDA) pharmaceutical additives. Seeds of OB and SA are used in folk medicines and in some parts of Asia these are added in ice desserts (regionally known as 'Falooh-dah') and beverages. They are source of dietary fibre (Al-Yousuf, Bashir, Ali, Tanira, & Blunden, 2002; Gorai, Gasmi, & Neffati, 2011; Hosseini-Parvar, Matia-Merino, Goh, Razavi, & Mortazavi, 2010). The gum extracted from OB has sheer-thinning properties and has been suggested for use as a functional additive in food products (Hosseini-Parvar et al., 2010). The gum from AM is used for various ailments and its suspension in water, locally called as 'Zhuble sharbat', is used as a health tonic (Ahmad et al., 2011). The AS gel has also been suggested for use in pharmaceutical preparations (Singh et al., 2010). The gels from PO and MP have recently been evaluated as drug carriers for sustained drug delivery (Ahuja, Kumar, & Yadav, 2010; Akbar, Iqbal, Chaudhary, Yasin, & Massey, in press; Iqbal et al., 2011; Singh, Kumar, Langyan, & Ahuja, 2009). Chemical composition of most of these materials has been reported by several workers (Azuma & Sakamoto, 2003; Chalk, Stoddart, Szarek, & Jones, 1968; Chatterjee & Mukherjee, 1958; Farooqi,

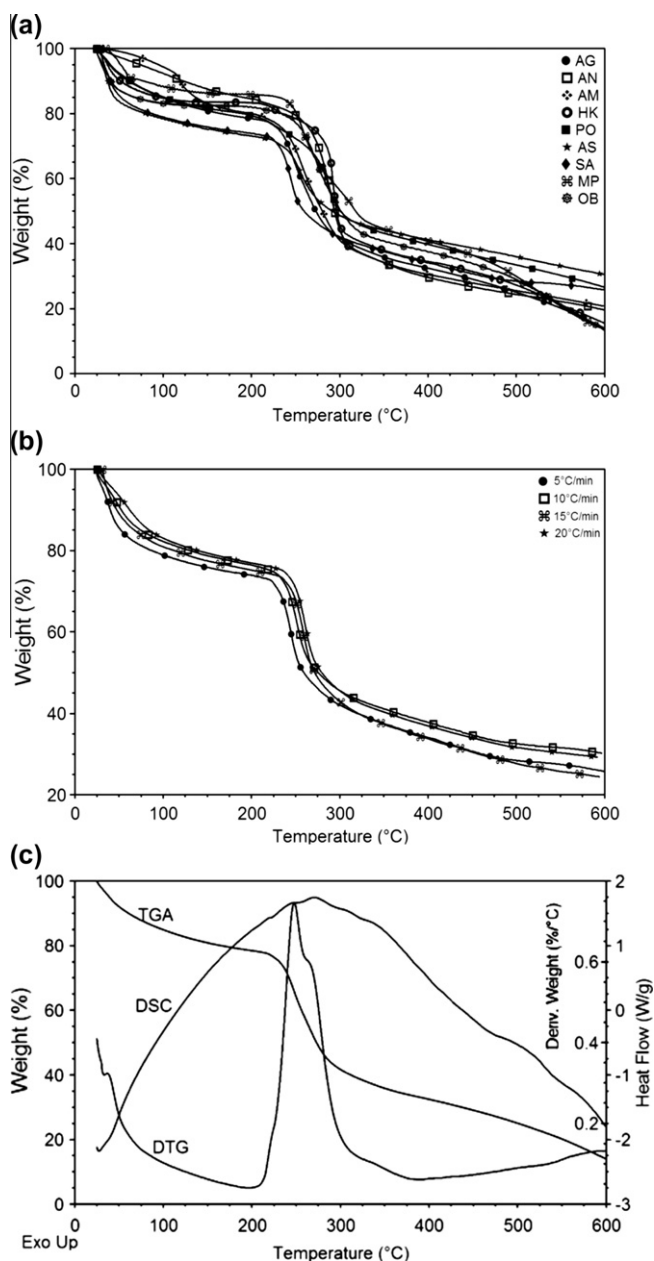


Fig. 1. (a) TGA curves of polysaccharides under study at heating rate of 5 °C min⁻¹. MP: *Mimosa pudica*; PO: *Plantago ovate*; AS: *Argyrea speciosa*; AN: *Acacia nilotica*; HK: *Plantago ovata* husk; AM: *Acacia modesta*; AG: *Astragalus gummifer*; SA: *Salvia aegyptiaca*; OB: *Ocimum basicilicum*. (b) TGA curves of SA at different heating rates (c) Representative TGA, DTG and DSC curves for SA at heating rate of 5 °C min⁻¹.

Table 1
Thermal and compensation effect parameters for polysaccharides^a.

Sample code	Compensation equation parameters			E_a (kJ mol ⁻¹)	lnA	IPDT (°C)	ITS	ΔH^*	ΔS^*	ΔG^*
	a	b	$ r $							
AG	-2.410	0.222	0.999	187.0	43.8	241	0.38	182.7	114.6	122.9
AN	-2.203	0.214	0.999	157.5	35.8	254	0.40	152.8	47.4	126.1
AS	-2.395	0.219	0.999	157.6	36.9	226	0.35	153.2	57.1	123.0
AM	-2.414	0.218	0.999	132.6	31.3	269	0.42	128.1	10.4	122.5
MP	-2.199	0.208	0.998	165.0	36.6	270	0.43	160.6	54.6	131.6
OB	-2.444	0.213	0.999	164.7	37.6	261	0.41	160.2	62.7	126.0
PO	-2.414	0.212	0.999	154.9	35.2	247	0.39	150.2	42.5	126.4
SA	-2.343	0.223	0.999	169.6	40.2	213	0.33	165.3	84.7	121.4
HK	-1.437	0.212	0.999	175.4	38.7	262	0.41	170.7	71.5	130.2

^a Mean E_a , IPDT and ITS values are reported from different heating rates up to 600 °C. lnA is calculated from mean E_a .

Kapoor, & Khan, 1977; Fischer et al., 2004; Saghir, Iqbal, Hussain, Koschella, & Heinze, 2008; Seigler, 2003; Tharanathan & Anjaneyalu, 1972; Tharanathan & Anjaneyalu, 1975; Tischer, Iacomini, & Gorin, 2002). As far as their thermal studies at multiple heating rates are concerned there appears to be no such reports available in literature, however, a few of them have been studied by use of single-heating rate methods. It is now well established that the single-heating rate methods do not provide accurate information regarding kinetic parameters (Vyazovkin, 2006; Vyazovkin et al., 2011). In this work we have used model-free and model-fitting approaches in order to determine the so-called 'kinetic triplet' consisting of E_a , A and the functional form ($g(\alpha)$) of the model that describes the thermal degradation pattern of gels.

2. Materials and methods

2.1. Materials

The materials used in this study were: PO husk and seeds; seeds of SA, OB and MP; gums of AG, AM, AN and AS; acetone; acetic acid; methanol. The plant materials were obtained from local market and the chemicals were from E. Merck, Germany. The chemicals were used without further purification. Double-distilled water was used throughout this study.

2.2. Isolation of polysaccharides

The polysaccharides from PO seeds (Iqbal et al., 2011) and husk (Saghir et al., 2008) were isolated by use of reported methods. For isolation of gels from seeds of OB, SA and MP the reported methods (Iqbal et al., 2011) were used with slight modifications as follows. The seeds (15 g) were washed with distilled water (50 cm³) and transferred to a 2-L beaker containing distilled water (500 cm³). They were allowed to swell overnight and then blended with blunt blades using kitchen blender. The polysaccharide was separated out by vacuum filtration through muslin cloth and air-dried at room temperature (30 ± 3 °C) to a constant weight. The gel from AS was better isolated as above by omitting the blending step. The isolated AM and AN gels were obtained from local market and were purified by dissolving them (20 g) in distilled water (150 cm³), centrifugation and filtering out the insoluble material followed by air-drying the resulting polysaccharide to a constant weight. The AG gel, obtained from the local market, was freed from extraneous matter by suspending it in distilled water for about 2 h followed by removal of clear gel with the help of a stainless steel spatula and drying in an oven at 35 ± 1 °C overnight.

2.3. Thermal analysis

TGA and DSC analysis were carried out with a simultaneous thermal analyzer SDT, Q-600 (TA instruments, USA), under

nitrogen atmosphere at a flow rate of $100 \text{ cm}^3 \text{ min}^{-1}$ using 3.0–10.0 mg of the sample. The data were obtained at four different heating rates, i.e., 5, 10, 15 and $20 \text{ }^\circ\text{C min}^{-1}$ from ambient to $600 \text{ }^\circ\text{C}$. The data were analysed by the isoconversational Flynn–Wall–Ozawa (FWO) method (Eq. (1)).

$$\ln \beta = \ln \frac{AE_a}{Rg(\alpha)} - 5.331 - 1.052 \frac{E_a}{RT} \quad (1)$$

where $g(\alpha) = \int_0^\alpha \frac{d\alpha}{f(\alpha)}$; β the heating rate; A the pre-exponential factor; R the general gas constant and T is the temperature at the conversion α . The FWO method was the first isoconversational linear integral method developed by Flynn and Wall (1966) and Ozawa (1965). This method is based on the assumption that for a fixed extent of conversion, the reaction rate depends upon temperature only. Thus it eliminates the dependence of reaction kinetics on any model, that may be represented by an integral form $g(\alpha)$. Therefore, this may be termed as a model-free approach. This method uses data obtained at different heating rates, thus at fixed α , the plot of $\log \beta$ vs $1/T$ will be a straight line, the slope of which permits the calculation of E_a . The α is defined as $(w_o - w_t)/(w_o - w_f)$, where w_t is the weight of the sample at any temperature T , w_o the initial weight and w_f is the final weight at the temperature at which the mass loss is approximately negligible.

Thermal stabilities of the polysaccharides were determined by integral procedural decomposition temperature (IPDT) and comprehensive index of intrinsic thermal stability (ITS) by Doyle (1961). This method is considered to be most appropriate and reliable to determine these parameters because it takes into account the whole TGA curve by measuring area under it. The ITS and IPDT values were determined from TGA of all four heating rates and mean values are reported for each polysaccharide. The life-times of polysaccharides were also predicted by model-free approach based on Eq. (2). The model-free approach eliminates the limitations of other methods such as ASTM E1641 and E698 methods which assume that the E_a remains constant throughout the degradation step. Therefore, in cases where E_a is not constant in a step the model-free approach is more appropriate (Vyazovkin et al., 2011).

$$t_\alpha = \frac{\int_0^{T_\alpha} \exp\left(-\frac{E_a}{RT}\right) dT}{\beta \exp\left(-\frac{E_a}{RT_0}\right)} \quad (2)$$

This relationship exploits the variation of activation energy with α . The integral in the numerator has no analytical solution, however, it can be evaluated by different approximations. In this study we used the Senum–Yang fourth degree approximation.

The data were analysed by the use of Universal Analysis 2000 software, version 4.2E (TA Instruments, USA), and MS Excel® 2010. Hierarchical cluster analysis (HCA) was performed to classify the materials with similar thermal properties by use of Statistica 8 and dendrogram were drawn using weighted pair-group average and Euclidean distance.

3. Results and discussion

The isolated polysaccharides were white, off-white or light brown in colour. They showed good swelling in water. The protein content as determined by use of BCA Protein Assay Kit (Pierce, Thermo Scientific) was found to be in the range 0.01–0.5% which may be considered to have negligible effect on thermal properties. Thermal behaviour of the isolated polysaccharides was studied by TGA and DSC from ambient to $600 \text{ }^\circ\text{C}$. In TGA they exhibited an endothermic weight loss of 8–20% in the $80\text{--}120 \text{ }^\circ\text{C}$ range, which was due to the loss of trapped water (Iqbal et al., 2011; Peng & Wu, 2010; Popescu, Popescu, Lisa, & Sakata, 2011; Yang, Yan, Chen, Lee, & Zheng, 2007). The major weight loss (18–36%) occurred in

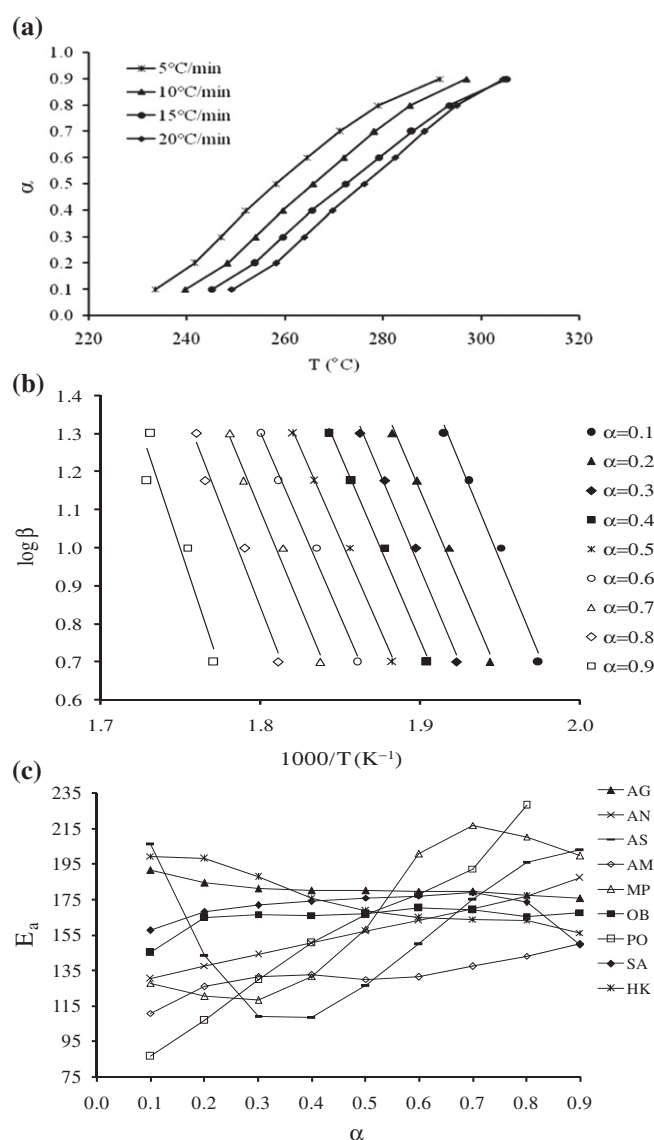


Fig. 2. (a) Representative α - T curve for AG (b) Representative FWO plot for AG to calculate E_a . (c) Dependence of E_a on α for polysaccharides.

the range $225\text{--}325 \text{ }^\circ\text{C}$ (Fig. 1a), which was due to major degradation of the polysaccharide structure. This step was associated with a wide exothermic enthalpy change as shown in the DSC scan (Fig. 1c). The mean ITS and IPDT values were found to be in the range 0.33–0.43 and $213\text{--}270 \text{ }^\circ\text{C}$, respectively (Table 1), which are indicative of good thermal stability of the materials. The isolated polysaccharides could be classified on the basis of their thermal behaviour (HCA) as shown in Fig. 3. It can be seen that SA and AS polysaccharides with ITS values 0.35 and 0.33, respectively, are on the lower side and they appear to form one major group while others form the second major group containing small groups at various similarity levels. As the IPDT and ITS are calculated from the area under the TGA curve, the SA and AS depicted lower values due to higher moisture contents (SA = 20%, AS = 21%) in them (Fig. 1a).

3.1. Flynn–Wall–Ozawa analysis

The apparent E_a values for major stage of decomposition were calculated by FWO method at different conversions ($\alpha = 0.1\text{--}0.90$ with 0.1 increment). Typical α - T and FWO plots for AG are shown in Fig. 2a and b respectively. The E_a - α curves

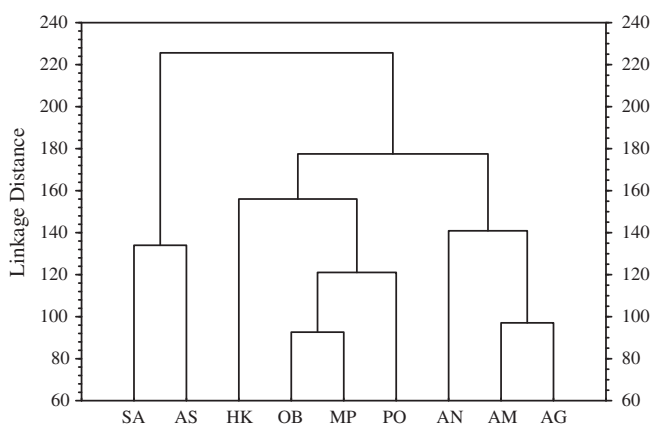


Fig. 3. Thermal classification of polysaccharides: Dendrogram showing similarity levels of thermograms for polysaccharides.

(Fig. 2c) indicated the dependence of E_a on the degree of conversion α . The polysaccharides from MP, AS and PO showed strong dependence of E_a on α suggesting a multistep degradation pattern for these materials. The E_a value of OB, SA and AG remains almost constant in the region $\alpha \geq 0.2$ indicating that these polysaccharides may be decomposed in one step. The E_a for HK, AN and AM varies with α , which suggests a multistep degradation of these materials (Vyazovkin, 2006; Vyazovkin et al., 2011). The multistep decomposition may be attributed to the diversity of sugar content of the material. The average activation energies are given in Table 1.

Since FWO method does not provide a direct estimate of the pre-exponential factor A , this factor was calculated by use of the compensation effect relationship (Vyazovkin, 2006; Vyazovkin et al., 2011) defined by Eq. (3).

$$\ln A = a + bE_a \quad (3)$$

where a and b are the compensation parameters. This relation suggests that any change in $\ln A$ shall be accompanied by a corresponding change, in a linear fashion, in E_a as calculated by use of Eq. (4). The a and b were determined by model-fitting approach using Coats–Redfern Eq.

$$\ln \frac{g(\alpha)}{T^2} = \ln \left[\left(\frac{AR}{\beta E_a} \right) \left(1 - \frac{2RT}{E_a} \right) \right] - \frac{E_a}{RT} \quad (4)$$

where \bar{T} is the average experimental temperature. The compensation parameters (a and b) and E_a (from FWO method) were put in the Eq. (3) to give A . The $g(\alpha)$ models used for solving the Eq. (4) in the present work are listed in Table 2. By use of this equation plots of $\ln(g(\alpha)/T^2)$ vs $1/T$ yield a set of kinetic parameters, A and E_a ; thus, according to Eq. (3) the values of a and b were obtained. The $\ln A$ values calculated from this equation using average FWO activation energies are listed in Table 1. The best model was selected on the basis of (i) the correlation coefficient (ii) the closeness of the activation energy with that determined by FWO method. Thus the first order model (F1) was found to be the best of the ten for most of the polysaccharides. The AN, AS, AM, MP, PO and HK polysaccharides exhibited a multistep decomposition with first order kinetics for the rate-determining step, whereas OB, SA and AG showed single step decompositions. This finding supports the general practice of using Broido method for determination of kinetic parameters of polysaccharides where the reaction mechanism is assumed to be of first order. In case of HK the data also fits well in A2 model and for others the diffusion models, D1 and D3, also showed good fits (Table 2). All the polysaccharides under investigation exhibited very high stability (life time > 20 years) at 40 °C, except PO and AM (life time about 1 month), as predicted by the model-free analysis

Table 2 Kinetic parameters for polysaccharides determined by model-fitting approach using Coats–Redfern equation^a.

Reaction model	Code	$g(\alpha)$	AG		AN		AS		AM		MP		OB		PO		SA		HK										
			E_a	$\ln A$	$ r $	E_a	$\ln A$	$ r $	E_a	$\ln A$	$ r $	E_a	$\ln A$	$ r $	E_a	$\ln A$	$ r $	E_a	$\ln A$	$ r $									
Power law	P1	$\alpha^{1/4}$	13	0.23	0.849	26	2.83	0.977	11	0.13	0.782	12	0.12	0.898	8	0.60	0.718	14	0.34	0.873	17	0.79	0.888	11	0.19	0.669	72	13.31	0.984
Power law	P2	$\alpha^{1/3}$	20	1.63	0.885	38	5.50	0.981	18	1.15	0.834	18	1.22	0.926	14	0.22	0.800	22	1.91	0.902	26	2.67	0.911	17	1.01	0.738	99	19.30	0.985
Power law	P3	$\alpha^{1/2}$	34	4.99	0.910	61	10.78	0.984	32	4.24	0.871	32	4.35	0.944	25	2.56	0.853	38	5.44	0.923	43	6.59	0.928	30	4.01	0.790	154	31.14	0.986
Power law	P4	$\alpha^{3/2}$	121	24.65	0.932	202	41.35	0.987	113	22.56	0.905	114	22.84	0.960	95	17.76	0.898	132	26.03	0.942	148	29.34	0.944	107	21.88	0.840	480	100.85	0.987
One-dimensional diffusion	D1	α^2	164	34.25	0.935	273	56.38	0.987	153	31.49	0.908	155	31.85	0.962	129	25.16	0.903	179	36.08	0.944	201	40.48	0.946	146	30.59	0.845	643	135.45	0.987
First order	F1	$-\ln(1-\alpha)$	114	24.00	0.977	186	38.64	0.999	108	22.28	0.959	107	21.92	0.991	90	17.65	0.953	124	25.19	0.982	139	28.23	0.980	105	22.19	0.911	440	93.08	0.993
Avrami–Erofeev	A1	$[-\ln(1-\alpha)]^{1/4}$	22	2.28	0.960	40	6.10	0.999	20	1.83	0.928	20	1.75	0.984	16	0.71	0.907	24	2.56	0.970	28	3.34	0.968	20	1.81	0.853	103	20.27	0.992
Avrami–Erofeev	A2	$[-\ln(1-\alpha)]^{1/3}$	32	4.75	0.967	56	9.83	0.999	30	4.13	0.941	30	4.02	0.987	24	2.51	0.927	35	5.14	0.975	40	6.19	0.973	29	4.11	0.877	140	28.49	0.992
Avrami–Erofeev	A3	$[-\ln(1-\alpha)]^{1/2}$	53	9.66	0.973	88	17.15	0.999	49	8.76	0.951	49	8.58	0.990	41	6.36	0.942	58	10.25	0.979	65	11.81	0.977	48	8.72	0.896	215	44.77	0.993
Three-dimensional diffusion	D3	$[1-(1-\alpha)^{1/3}]^2$	210	43.11	0.966	340	69.49	0.998	197	39.95	0.946	197	39.68	0.985	167	31.95	0.942	228	45.28	0.973	255	50.65	0.971	191	39.46	0.895	797	166.62	0.993
Contracting sphere	CS	$1-(1-\alpha)^{1/3}$	100	19.48	0.963	166	32.86	0.997	94	17.86	0.941	94	17.73	0.983	79	13.76	0.935	109	20.57	0.970	123	23.29	0.969	91	17.61	0.886	394	81.83	0.993
Contracting cylinder	CC	$1-(1-\alpha)^{1/2}$	94	18.33	0.956	156	31.13	0.996	88	16.76	0.931	88	16.73	0.978	74	12.90	0.925	103	19.37	0.963	115	21.96	0.963	85	16.45	0.873	372	77.61	0.992

^a Best fit models are in boldface.

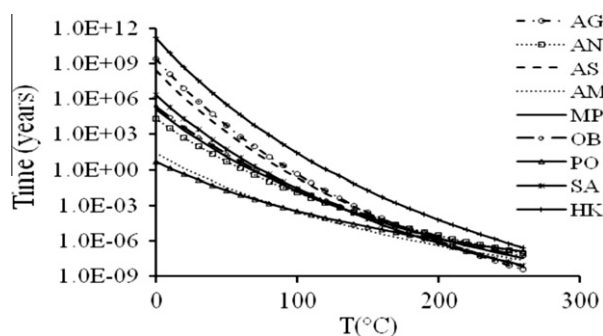


Fig. 4. Life time prediction of polysaccharides at different temperatures and 5% degree of conversions for polysaccharides.

(Fig. 4). The isocoverational method provides more accurate values of E_a and A than the single-heating rate method, therefore, we also report here relatively more reliable values of very useful activation parameters ΔS^* , ΔH^* and ΔG^* (Table 1).

4. Conclusions

Thermal degradation of polysaccharides from MP, PO, AS, AN, PO husk and AM appeared to be a multistep process while those from AG, SA and OB followed single step kinetics. The degradation was exothermic in nature. Mean comprehensive index of thermal stability (ITS) ranged from 0.33–0.43. The SA and AS with ITS values 0.33 and 0.35, respectively, showed relatively lower stability than others due to higher moisture content. The ITS values of MP, PO, PO husk, AN, OB, AG and AM are comparable with some of the commercial polysaccharides like HPMC. Kinetic triplets consisting of apparent activation energy, pre-exponential factor and thermal degradation model, for the major decomposition step, were found to be 132–187 kJ mol⁻¹, 31.3–43.8 and of first order respectively.

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