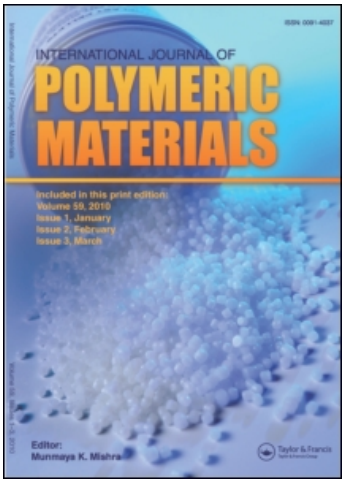

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Synthesis, Thermal Properties and Antibacterial Activity of Polyketones

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Polyketones were prepared by the Friedel-Craft reaction from 1,3-dimethoxy benzene, chloroacetic acid and dichloroalkanes, *i.e.*, dichloromethane and 1,2-dicholoroethane. The polyketones were characterized by IR spectra and vapour pressure osmometry. The thermal properties were studied by the thermogravimetry and differential scanning calorimetry. The kinetic parameters for the thermal decomposition reaction were evaluated by the methods of Broido and Doyle. The synthesized polylaketones shows antimicrobial properties against bacteria, fungi and yeast.

Keywords: Polyketones; Synthesis; Thermal properties; Antibacterial activity

INTRODUCTION

Microorganisms plays a vital role in the structural degradation of various useful chemical compounds. The materials susceptible towards such a microbial degradation includes textile fibers, paints, marine, coatings, electrical insulation, food packaging material, storage tank lining, pharmaceutical material and hydrocarbon fuel system [1–3]. During last decade extensive work has been carried out to prevent

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such degradation using certain biocides especially based on polymeric system. With this view polyketones were prepared and tested for their biocidal properties using *Bacillus substilis*, *Pseudomonas fluorescens Aspergilline niger*, *Trichloderma longibranchiatum*, *Rhodotorules minuta* and *Saccharomyas cerevisiar* as test microorganism.

EXPERIMENTAL

All the chemicals used for the synthesis were of analytical grade purity.

Friedel-Crafts polyketones were prepared according to published procedures (4 and 5), particular conditions of the synthesis are reported in Table I and reaction scheme shown in Figure 1.

The experimental details for the characterization of the resins are the same as reported earlier [4, 5].

For the antimicrobial study of polyketones, bacteria strains (*B-subtilis P. fluorescent* and *E-coli*), fungal strains (*A-niger*, *T. longibrachiatum* and *P. Chrysogenum*) and yeast strain (*R. minuta*, *S. cerevisia* and *P. stipitis*) were selected.

RESULT AND DISCUSSION

Polymerization condition and characteristic of polyketones are presented in Table I. All the polyketones are highly coloured ranging from brown to black in colour. Polymers are soluble in acetone, dimethylformamide, dioxane *etc.*, and soften in the range of 130–175 degree celsius. The chlorine content of soluble polyketones varied from 6.0 to 7.0% [6]. The number average molecular weight of the resin varied from 2350 to 2890.

The structural assignment of IR peak are as follows.

Aromatic substitution was confirmed by the presence of CH in plane and out of plane bending around 1020–1080 and 800–860 cm^{-1} respectively. The prominent sharp band at around 1700 cm^{-1} is due to carbonyl group and a band around 670 cm^{-1} is attributed to C—Cl [7] the IR spectra of these resins resemble each other in all aspects which suggest that all the resins are linear polyketones differing in their molecular weights.

TABLE I Condition for the preparation of polyketones. (Reaction temperature: 130°C; Reaction time: 4 hrs; Solvent; Nitrobenzene (25 ml))

Resin number	1,3-*di-methoxy benzene (mol)*	*Chloro acetyl chloride (CAC) (mol)*	1,2-*Di-chloro ethane (DCE) (mol)*	*Di-chloro methane (DCM) (mol)*	*Aluminium chloride (mol)*	*Yield (%)*	*Physical state and softening range* (°C)*	*Chlorine (%)*	*Remarks***
1	0.02	0.02	–	–	0.04	59.6	Yellowish brown powder 148 – 160	6.5	CAC and $AlCl_3$ were mixed and 1,3-DMB + $PhNO_2$ was added within 10 min.
2	0.04	0.04	–	–	0.06	68.4	Blackish brown powder 161 – 175	7.0	As above
3	0.02	0.02	–	–	0.04	58.7	Dark brown powder 152 – 164	6.2	To $AlCl_3$, 1,3-DMB + CAC + $PhNO_2$ were added within 30 min.
4	0.02	0.01	0.01	–	0.04	60.0	Blackish brown powder 147 – 160	6.0	To $AlCl_3$ + CAC + $PhNO_2$ 1,3-DMB was added and contents were kept at 60°C for 1 hr. and to this DCE was added.
5	0.02	0.01	0.01	–	0.04	58.4	Blackish brown powder 145 – 147	6.3	To $AlCl_3$ + DCE + $PhNO_2$ 1,3-DMB was added and contents were kept at 60°C for 1 hr. and to this CAC was added.
6	0.02	0.01	–	0.01	0.04	58.7	Blackish brown powder 140 – 152	6.4	To $AlCl_3$ + CAC + $PhNO_2$ 1,3-DMB was added and contents were kept at 60°C for 1 hr. and to this DCM was added.
7	0.02	0.01	–	0.01	0.04	58.0	Blackish brown powder 130 – 151	6.1	To $AlCl_3$ + DCM + $PhNO_2$ 1,3-DMB was added and contents were kept at 60°C for 1 hr. and to this CAC was added.

*From DSC thermograms.
**The general method for preparation is already given in text. Here specific changes for each preparations are indicated.

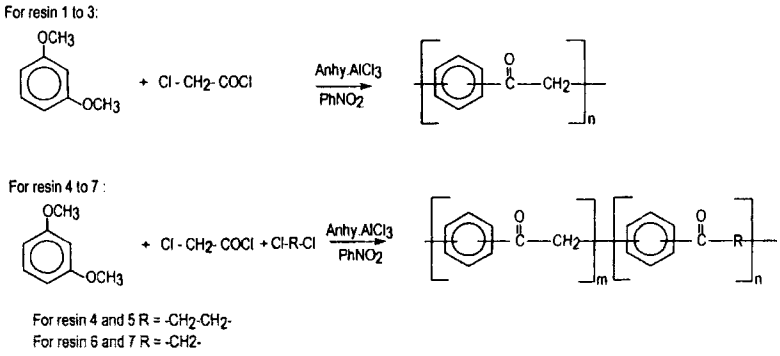

FIGURE 1 Reaction scheme.

Thermal characterization of all resins were carried out by TG and DSC. The thermal data and various kinetic parameters of thermal degradation are presented in Table II. Data indicated that the degradation starts around 200–280°C indication that these polyketones exhibit moderate thermal stability. The Broido method [8] was applied to the analysis of the thermogravimetric data to calculate energy of activation (E_A) of the degradation reaction this degradation of resin range from 83–116 KJ mol^{-1}. The temperature characteristic for the degradation has been calculated for the polyketones from Doyle's method [9] and are listed in Table II. The decomposition range and initial decomposition are different for all the resins. This clearly indicates that the mode and range of decomposition are different for all the polyketones.

Result listed in Tables III to V shows the effect of polyketones on the growth of bacteria, fungi and yeast – respectively. From the result it is revealed that the polyketones shows significant inhibition of growth with in 48 hours. During this period control culture exhibited maximum growth. Resin 1 and 2 are prepared from 1,3-dimethoxy benzene and CAC only shows significant growth inhibition of all the microorganism. Resin 3 to 5 gives 2–48% growth and resin 6 and 7 gives 4 to 55% growth of microorganism. Result listed in Table IV shows the effect of polyketones on the growth, all the three fungi at or early stage. Which is evident by negligible utilization of sugar from the medium and in significant changes in the pH as compared to control.

TABLE II Kinetic parameters of the polyketones using thermogravimetry

Resin	*Weight loss (%) at temperature upto*					*Decomposition temperature range*	*IPDT*[a]	*Energy of activation*[b] *Ea*	*Heat of fusion*[c] ΔH_f
number	300°C	400°C	500°C	600°C	700°C	(°C)	(°C)	$KJ \cdot mole^{-1}$	$(J \cdot gm^{-1})$
1	13	22	39	82	85	225–590	465	90.0	24.0
2	15	19	40	90	–	280–620	470	106.0	31.3
3	14	23	55	89	–	255–585	450	103.0	26.7
4	16	25	62	87	90	210–635	445	83.0	22.9
5	10	21	53	79	80	240–625	450	99.0	33.0
6	07	15	38	86	–	220–570	500	116.0	28.0
7	06	12	39	75	78	205–575	498	109.0	25.0

Rate of heating:10°C/min.
[a] Doyle's method.
[b] Broido method.
[c] From DSC thermogram.

TABLE III Effect of polyketones on the growth of *B. subtilis*, *P. fluorescens* and *E. coli*

Incubation time	*Growth of B. subtilis*								*Growth of P. fluorescens*								*Growth of E. coli*							
	Resin number[a]								*Resin number*[a]								*Resin number*[a]							
(*h*)	*Con*[b]	1	2	3	4	5	6	7	*Con*[b]	1	2	3	4	5	6	7	*Con*[b]	1	2	3	4	5	6	7
20	70	3	4	5	3	2	2	2	60	3	3	3	2	4	4	9	58	3	2	2	2	4	3	4
24	78	3	4	5	3	3	2	2	65	3	3	3	2	11	4	9	67	3	2	4	2	7	3	8
28	85	6	4	6	3	6	5	11	80	3	5	4	2	15	7	9	75	4	2	4	3	11	4	15
32	98	6	4	6	4	6	5	22	85	3	5	4	3	34	7	15	88	4	5	5	4	16	5	19
36	100	6	4	6	4	27	6	29	90	3	5	4	3	50	7	45	95	6	5	5	4	18	5	28
40	100	6	5	7	10	29	6	40	94	5	5	5	3	7	11	55	100	6	7	5	7	18	5	36
44	100	6	6	7	12	55	6	45	100	6	5	6	10	65	11	62	100	6	7	5	7	20	5	40
48	100	6	7	7	12	60	6	55	100	8	5	6	20	70	11	74	100	6	7	5	7	20	5	40

[a] Concentration of each resin was 500 ppm.
[b] Control does not contain any of the resin.

TABLE IV Effect of polyether-ketones on *A. niger*, *T. longibrachiatum* and *P. chrysogenum*

	A. niger				*T. longibrachiatum*				*P. chrysogenum*			
Resin number	*pH of the solution*	*Sugar utilized* (%)	*Dry weight* (*mg*)	*Growth** (%)	*pH of the solution*	*Sugar utilized* (%)	*Dry weight* (*mg*)	*Growth** (%)	*pH of the solution*	*Sugar utilized* (%)	*Dry weight* (*mg*)	*Growth** (%)
Control**	3.9	99.8	786	100	4.5	99.0	806	100	4.7	98.0	708	100.0
1	5.6	6.1	25	3.2	5.7	10.9	41	5.1	5.6	13.3	32	4.5
2	5.2	11.9	30	3.8	5.8	5.1	28	3.1	5.3	17.4	29	4.1
3	5.3	13.6	35	4.5	4.8	37.6	190	13.5	4.9	36.2	75	10.6
4	5.2	15.4	40	5.1	5.6	17.3	59	7.3	5.2	22.9	109	15.4
5	5.4	8.5	20	2.5	5.1	23.9	88	10.9	5.0	24.0	27	3.8
6	5.1	19.1	65	8.3	5.3	19.2	67	8.3	5.2	22.4	30	4.2
7	4.6	41.0	300	38.2	5.6	15.6	50	6.2	4.3	40.0	250	35.3

*After 40 h.
**Control does not contain any of the resin.

TABLE V Effect of polyketones on the growth of *Rhedotorula minuta, Sacchromyces cerevisiae* and *Pochia stipitis*

	R. minuta		*S. cerevisiae*		*P. stipitis*	
Resin number	*Incubation time (h)* 24	48	*Incubation time (h)* 24	48	*Incubation time (h)* 24	48
Control*	51	100	68	100	71	100
1	4	6	3	6	6	8
2	5	6	4	7	10	12
3	5	7	6	7	7	14
4	7	9	8	8	27	48
5	9	11	6	10	41	15
6	8	10	8	9	22	40
7	10	25	8	18	18	21

*Control does not contain any of the resin.

Resin showed significant inhibition of growth of microorganism except the resin 5 to 7. From the above results it can be seen that antimicrobial activity decreases upon addition of alkyl chain on the resin backbone. The sequence of addition of various reagent plays important role in deciding the microbial properties of the polyketones.

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